Deinking of high-quality offset papers: Influence of consistency, agitation speed, and air flow rate in the flotation stage

FÉLIX CARRASCO, MARIA ANGELS PELACH AND PERE MUTJÉ

ABSTRACT

Flotation deinking of high-quality offset printed papers was studied using a laboratory cell with a nominal capacity of 50 L. Disintegration conditions prior to flotation were held constant to facilitate study of operating conditions during the flotation stage. The analyzed process variables were pulp consistency (0.75%, 1%, 1.25%), air flow rate (500, 750, 1000 L/h), and agitation speed (850, 1150, 1450 rpm). A deinkability factor—based on handsheet brightness after flotation—was used to evaluate process efficiency and establish the optimum flotation conditions. Analysis of the results showed excellent correlation between deinkability factor and flotation operating conditions, and the proposed empirical equation had a regression coefficient r^2 of 0.90. The change in deinkability factor with operating conditions is explained by applying probability theory to predict the frequency of collisions between air bubbles and ink particles.

Application:

A laboratory investigation to determine optimum operating conditions for efficient flotation deinking of high-quality offset printed papers.

Consumption of paper and board continues to grow in both the developed countries and in countries with emerging economies in globalized and open markets, such as Southeast Asia *(1)*. With demand for paper products continually growing, recovery and recycling of secondary fibers has become increasingly important. Indeed, efficient utilization of recycled fiber is a key factor in the industry's efforts toward sustainable forestry. Without recycling, the fiber supply in the world's forests will not be sufficient to keep up with demand *(2)*. Recycling efficiency can be increased further by classifying recovered paper by grade and reusing high-quality fibers to produce high-value papers.

Offset printing has become the dominant printing process. The papers used in offset printing must be strong enough for their intended end use while providing adequate print quality. Several authors *(3-6)* have noted that some offset papers can be printed and deinked to produce a second generation of offset-quality papers. The prospect of recycling offset papers to manufacture a similar product is intriguing, but this will not be possible unless these high-quality printed papers are properly classified for reuse. Otherwise, they will end up as secondary fibers in products with much lower value.

The objective of this study was to examine flotation deinking of high-quality offset-printed papers made from chemical pulps. The efficiency of flotation deinking was analyzed by varying the operating conditions of the flotation stage.

EXPERIMENTAL

Materials

The raw material used in this study was high-quality coated paper with a basis weight of 135 g/m^2. The base paper consisted of chemical pulp (10% long fiber + 90% short fiber) with 10% filler. Total filler content in the coated paper was 39 wt.%. All papers were printed by offset using an ink (black carbon pigment base) that dried at high speed by oxidation in contact with air. Printing density was 1.85, and the percentage of surface printed was 19.3%. Uniform printing text was applied to all of the sheets to avoid variations caused by the raw material.

The following chemicals were used: sodium hydroxide, calcium chloride, sodium silicate, EDTA, and hydrogen peroxide (Panrec Quimica, S.A., Spain); and surfactant (Olinor 1040, Pulcra, S.A., Spain).

Methodology

Disintegration and flotation operations were carried out in a laboratory cell (Pulcell) of 50-L nominal capacity (batch operation) that allows the selection of a wide range of consistency, agitation speed, air flow rate, temperature, and chemical additives. Operating conditions during the disintegration stage were held constant throughout the entire experiment (10% consistency, 50°C, 15 min., 1100-rpm agitation speed, and water hardness of 19°fH). The chemical composition of the suspension also was maintained at a constant level during disintegration [1 wt.% NaOH, 2.5 wt.% Na_2SiO_3, 0.5 wt.% EDTA, 1 wt.% H_2O_2, and 1 wt.% surfactant (based on dry paper)]. After disintegration, the suspension was diluted to 5% consistency with cold water, and samples were taken to make handsheets according to TAPPI T 205.

Consistency (c), %	Agitation speed (N), rpm	DEINKABILITY FACTOR, % Air flow rate (q), L/h 500	750	1000
0.75	850	40.8	35.4	40.4
	1150	56.2	58.9	55.6
	1450	65.4	60.5	60.3
1.0	850	51.7	53.4	52.8
	1150	63.8	71.1	71.3
	1450	70.0	73.4	77.7
1.25	850	50.1	61.8	66.7
	1150	76.2	80.8	80.7
	1450	82.6	83.6	87.9

I. Measured deinkability factor (based on brightness) as a function of pulp consistency, agitation speed, and air flow rate (flotation time t = 12 min)

	Degrees of freedom	Sum of squares	F-ratio	Probability > F
Variable				
Agitation speed (N)	2	2654.12	139.00	0.00
Consistency (c)	2	2166.87	113.48	0.00
Air flow rate (q)	2	75.49	3.95	0.06
Interactions				
$N \cdot c$	4	15.03	0.39	0.81
$N \cdot q$	4	28.55	0.75	0.59
$c \cdot q$	4	103.50	2.71	0.11

II. Statistics describing the effects of variables (consistency, agitation speed, air flow rate) and second-order interactions on deinkability factor

The flotation stage was performed in the 50-L cell with a low-consistency agitator, an air diffuser with regulated feeding, and a foam collector. The operating conditions of the flotation stage were varied to find the optimum operating conditions. Agitation speeds were 850, 1150 and 1450 rpm; pulp consistencies were 0.75%, 1%, and 1.25%; and air flow rates were 500, 750, and 1000 L/h. Water hardness and temperature were maintained at 19°fH and 20°C, respectively, throughout all flotation experiments. Samples were extracted every 3 min up to a total time of 12 min to study the kinetics of deinking during flotation.

The efficiency of the deinking process was evaluated by measuring handsheet brightness using an Elrepho 2000 (Datacolor), as indicated in TAPPI T 452. To quantify this efficiency, handsheet brightness measured in each experiment was compared with a reference standard. The brightness of unprinted paper subjected to the same disintegration conditions could be used as a reference standard. In this case, the global efficiency of the deinking process would be defined by the following deinkability factor:

$$E = \frac{B_F - B_D}{B_B - B_D} \qquad (1)$$

As an alternative reference standard, the brightness of unprinted paper, subjected to the same conditions of both disintegration and flotation, could be used:

$$E^* = \frac{B_F - B_D}{B_{BF} - B_D} \qquad (2)$$

where

E = deinkability factor, the global efficiency of deinking (disintegration + flotation) based on the brightness of disintegrated unprinted paper as a reference

E^* = deinkability factor, the global efficiency of deinking (disintegration + flotation) based on the brightness of disintegrated and flotation-deinked unprinted paper as a reference

B_F = brightness after flotation

B_D = brightness after disintegration

B_B = brightness of unprinted paper subjected to the disintegration stage carried out under the same conditions

B_{BF} = brightness of unprinted paper subjected to the disintegration and flotation stages carried out under the same conditions.

The brightness of the disintegrated unprinted paper and the brightness of the flotation-deinked unprinted paper are nearly the same. Consequently, both equations give the same value for deinkability factor.

RESULTS AND DISCUSSION

Table I shows the deinkability factors obtained at the various operating conditions for the deinking process used in this study. **Figure 1** shows the variation of process efficiency (deinkability factor) with agitation speed during the flotation stage at different levels of flotation consistency and air flow rate. The results in Fig. 1 clearly show that deinkability factor increased with increasing agitation speed. The increase is not linear but polynomial, as discussed later. Another noteworthy phenomenon is the increase in process efficiency with increasing pulp consistency. The lowest consistencies (0.75%) showed the lowest process efficiency, while maximum efficiencies generally occurred at the highest consistencies (1.25%).

No bell-shaped deinking curves were observed during this study, in contrast to results described by Mutjé *et al.* *(7)*. However, their work used a wider range of consistencies, and the paper was laser printed. Nevertheless, Mutjé *et al.* *(7)* found that at consistencies of 1.25% or greater, efficiency decreased with increasing consistency. In the present study, the optimum consistency was the maximum value applied (1.25%). Grossmann *et al.* *(8)* found that a flotation consistency of 1.3% is the maximum value used in traditional flotation cells. Results from these studies corroborate our findings.

The effect of the air flow rate applied during the flotation process was of little significance in the range of operating conditions investigated in this study. This is verified by the results in **Table II**, which shows the variance analysis of effects. The *F*-ratio values clearly indicate that consistency and agitation speeds are very significant variables, with the air flow rate presenting a less significant effect. Secondary interactions between two variables were not significant.

Based on the results obtained from laboratory experiments, an empirical equation relating the deinkability factor (E, %) to the process variables—consistency, agitation speed, and air flow rate—was proposed:

$$E = w\, c^x\, N^y\, q^z \qquad (3)$$

where

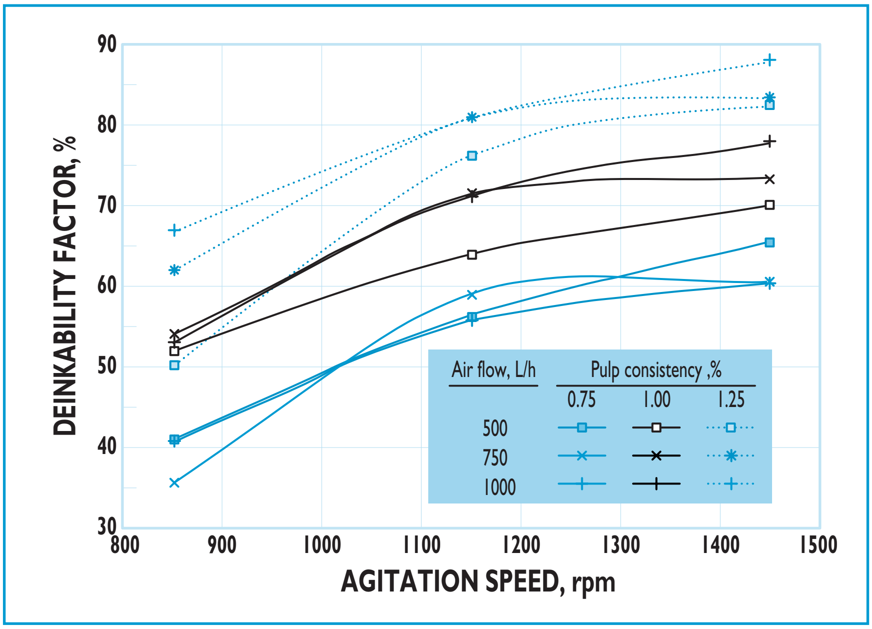
c = pulp consistency, %
N = agitation speed, rpm
q = air flow rate, L/h
w,x,y,z = model coefficients

The model coefficients were obtained through multiple linear regression analysis. Equation 4 shows the fitted equation.

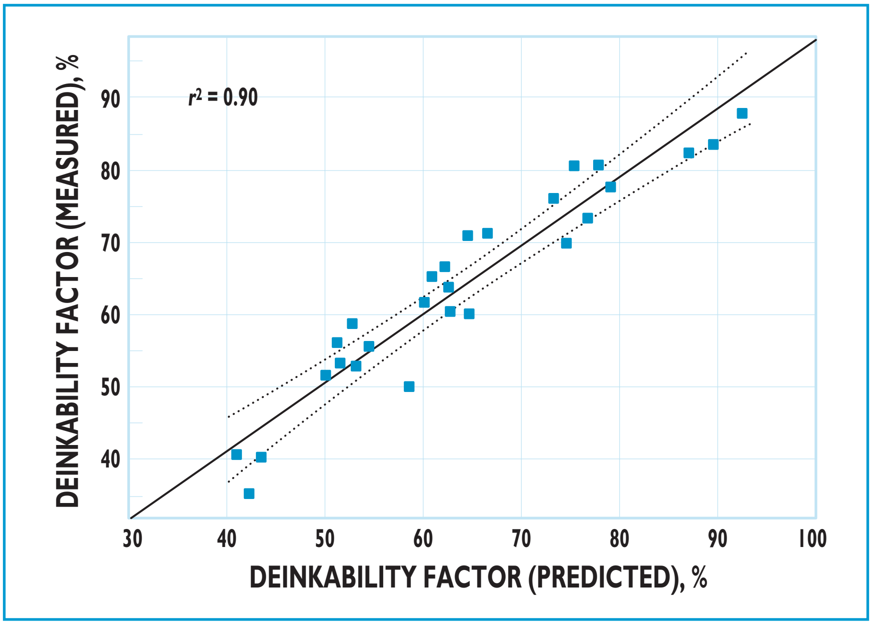
$$E = 0.21\, c^{0.70}\, N^{0.74}\, q^{0.07} \qquad (4)$$

The regression coefficient r^2 for Eq. 4 was 0.90, indicating an excellent fit. These statistical data corroborate the finding that consistency and agitation speeds had a significant effect on deinking efficiency, while the influence of the air flow rate was far less significant. **Figure 2** plots the measured values of deinkability factor vs. the values predicted by Eq. 4. Most of the data points fall within the curves depicting the confidence interval.

The variation of deinkability factor with the operating conditions can be explained using a probability model. This model is based on the probability of collisions between air bubbles and ink particles and has been already described in the literature *(9-12)*. According to this model, the global probability of flotation, P_F, can be expressed as the product of the probabilities of the three individual microprocesses occurring in the flotation macroprocess, as seen in Eq. 5.



1. Deinkability factor as a function of agitation speed at various combinations of consistency and air flow rate (flotation time = 12 min)



2. Comparison of measured and predicted values for deinkability factor

$$P_F = P_c\, P_a\, P_s \qquad (5)$$

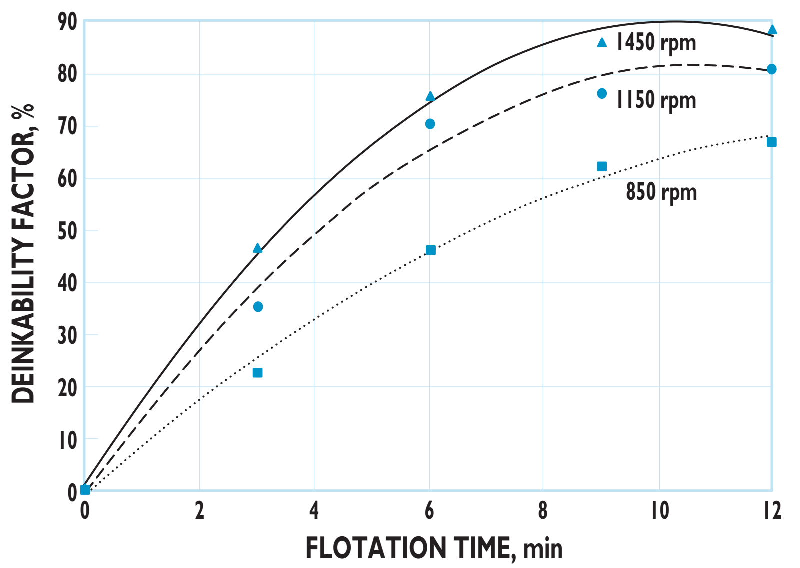
where

P_c = probability of collision between air bubble and ink particle
P_a = probability of particle attachment to bubble particle
P_s = probability of stabilization of the bubble–particle aggregate vs. external forces

The probability model of flotation describes the process in terms of variation in the number of particles within a given volume per unit time *(10)*. Such variation depends on

Flotation time (*t*), min	DEINKABILITY FACTOR, % Agitation speed (*N*), rpm 850	1150	1450
0	0.0	0.0	0.0
3	22.7	35.2	46.5
6	46.0	70.4	75.5
9	61.9	76.0	85.7
12	66.7	80.7	87.9

III. Measured deinkability factor as a function of flotation time and agitation speed (constant conditions of c = 1.25% and q = 1000 L/h)



3. Deinkability factor as a function of flotation time at various levels of agitation speed (consistency = 1.25%, air flow rate = 1000 L/h)

(a) the number of ink particles and number of air bubbles, (b) the number of collisions between ink particles and air bubbles per unit time, and (c) the individual interaction probabilities. The collision probability P_c is higher when the ink particles are larger and the air bubbles are smaller. To maximize the number of collisions between ink particles and air bubbles, the number of small-diameter air bubbles must be increased, a target attained by increasing the agitation speed. Increasing the agitation speed—at constant air flow rate and for a given pulp consistency—increases the disruption of the air bubbles, thus increasing the number of smaller air bubbles. This implies an increase of the specific air surface in contact with the ink particles present in the suspension. In short, in the operating conditions used in the deinking experiments, the process efficiency notably improves upon increasing the agitation level and the pulp consistency and, to a lesser extent, upon increasing the air flow rate. However, if the agitation level or consistency or the air flow rate are too high, a decrease of the stabilization probability P_s could occur, which would lead to a decrease of the deinkability factor. On the other hand, the positive influence exerted by the consistency increase is mainly due to the increase of the number of ink particles per unit volume.

Table III shows the flotation kinetics at different agitation speeds under constant conditions of consistency (1.25%) and air flow rate (1000 L/h). As seen in **Fig. 3**, deinking efficiency at a given flotation time increased with increasing agitation speed. At maximum agitation speed (1450 rpm), deinking efficiency increased significantly during the first 4 min of flotation. From there, the deinkability factor reached an asymptotic value of 88% after 10 min of flotation. This value is considered to be the hyperflotation value under the operating conditions of the experiment, since further increases in flotation time produce minimal variation in efficiency. The asymptotic value is a consequence of the decrease of the number of ink particles per unit volume, which implies a decrease in the probability of collision.

The time constant—defined here as the time required to reach a deinkability factor of 63.2%—is 4.7 min when the agitation speed is 1450 rpm. At 1150 rpm, the growth curve is lower, the time constant is 5.7%, and the asymptote drops to 81%. At 850 rpm, the time constant is 10.2 min and the efficiency asymptote is 67%. Thus it is evident that the time constant decreases (i.e., the deinking process is faster) with increasing agitation speed. At 850 rpm, the air bubbles are neither small enough nor numerous enough to eliminate the ink with the same probability as at 1450 rpm.

CONCLUSIONS

The deinkability efficiency for high-quality offset papers was studied using a laboratory cell (Pulcell) with a nominal capacity of 50 L. To simplify analysis of the flotation stage, the operating conditions during the preceding disintegration stage were held constant. The results demonstrate that within the range of experimental operating conditions, increasing any of the process variables (pulp consistency, agitation speed, air flow rate) increases the deinking efficiency (measured as deinkability factor, based on pulp brightness). Thus the highest efficiency was obtained when the process variables were at their maximum level (pulp consistency 1.25%, agitation speed 1450 rpm, and air flow rate 1000 L/h). A deinkability factor of 88% was obtained under these conditions.

Statistical analysis showed that consistency and agitation speed had a significant effect on the deinkability factor, while the influence of the air flow rate was far less significant.

An empirical equation was fitted to relate the deinkability factor with the operating conditions of the flotation stage:

$$E = 0.21\ c^{0.70}\ N^{0.74}\ q^{0.07}$$

where

E = deinkability factor, %
C = consistency, %
N = agitation speed, rpm
q = air flow rate, L/h

The fit of this correlation was excellent, with a regression coefficient r^2 of 0.90. The F-values of the correlation confirm that consistency and agitation speed were the most significant variables. These results can be explained by applying probability theory to the frequency of collisions between bubbles and ink particles. The probabilities of collision and bubble–ink particle attachment increase with increasing consistency and agitation speed. However, these variables cannot be increased *ad infinitum*. At some point, consistency and agitation speed become excessive, and the probability of destabilizing bubble–ink aggregates increases, thus decreasing deinking efficiency.

Analysis of the observed deinking kinetics demonstrated that the maximum efficiency values (i.e., hyperflotation conditions) depend largely on the agitation speed. Thus, operating at the experimental maximums for consistency (1.25%) and air flow rate (1000 L/h), the maximum attainable deinkability factors at agitation speeds of 850, 1150, and 1450 rpm were 67%, 81%, and 88%, respectively. The time constant of the flotation process also decreases with increasing agitation speed. Thus the duration of flotation deinking can be shortened by increasing the agitation speed. **TJ**

Carrasco is a full professor, Pelach is an assistant professor, and Mutjé is an associate professor in the Laboratory of Paper Engineering and Polymer Materials, Dept. of Chemical Engineering, Universitat de Girona, Av. Lluís Santaló, s/n, 17071 Girona, Spain.

The authors are grateful to the Comisión Interministerial de Ciencia y Tecnologia (CICYT) of the Spanish government for the grant awarded to research projects QUI96-0952 and AMB97-0958.

Received for review June 23, 1997.
Accepted March 24, 1998.

LITERATURE CITED

1. *Fédération Producteurs Pâtes Papiers* 354: 58(1996).
2. Colom, J. F. and Torres, A. L., *Ingeniería Química* 26(301): 57(1994).
3. Pastor, R. B., Torres, A. L., and Colom, J. F., *Investigación y Técnica Papel* 32(126): 803(1996).
4. Matey, M., *Investigación y Técnica Papel* 33(128): 363(1996).
5. Putz, H. J. and Göttsching, L., "Possibilities and limitations of the utilization of deinked pulp (DIP) in high quality paper grades," in *2nd Advanced Training Course on Deinking Technology*, Centre Technique du Papier, Grenoble, France, No. 24, 1995.
6. Bouchard, J. and Douek, M., *J. Pulp Paper Sci.* 20(5): J131(1994).
7. Mutjé, P., Roux, J. C., Delpech, F., and Carrasco, F., *Progr. Paper Recycling* 6(3): 48(1997).
8. Grossmann, H., Schulze, G., and Hanecker, E., *TAPPI 1997 Recycling Symposium Proceedings*, TAPPI PRESS, Atlanta, p. 65.
9. Sutherland, K. L., *J. Phys. Chem.* 52: 394(1948).
10. Schulze, H. J., *TAPPI 1991 Recycling Forum Proceedings*, TAPPI PRESS, Atlanta, p. 161.
11. Pan, R., Bousfield, D. W., and Thompson, E. V., *TAPPI 1992 Pulping Conference Proceedings*, TAPPI PRESS, Atlanta, p. 941.
12. Pan, R., Paulsen, F. G., Johnson, D. A., *et al.*, *TAPPI J.* 79(4): 177(1996).