Effect of carboxymethylcellulose and starch depressants on recovery of filler and fines in tertiary flotation

ARI ÄMMÄLÄ, LIISA MÄKINEN, HENRIKKI LIIMATAINEN, AND JOUKO NIINIMÄKI

ABSTRACT: Yield loss in state-of-art deinking processes for newsprint is usually 15%–20%. Economic and environmental concerns associated with reject handling point to a need to improve yield. This paper describes a study conducted to determine whether it is possible to recover fillers and fiber fines from flotation froth reject of an old newsprint/old magazine processing mill by refloating froth with and without the use of depressants. The recovery of approximately 10%–15% of material was observed without the use of additional flotation chemicals in the reflotation stage. Carboxymethylcellulose (CMC) was observed to be a functional depressant that enhanced fiber fines recovery in particular and, to some extent, china clay recovery but lowered calcium carbonate recovery. The recovery increased to 20%–30% with a dosage of approximately 1 kg CMC/ton of pulp. In addition, starch had a depressing effect, but this effect was weaker than that of CMC, even with much higher dosages.

Application: Mills seeking environmentally sound and economical deinking strategies may be encouraged to test CMC as a filler and fines depressant in their secondary flotation stages.

Recycled pulp has become the major raw material source for paper and board manufacturing. Recycling is economically and environmentally advisable, but a drawback is the large loss of material requiring disposal. The yield loss of the deinked pulp (DIP) manufacturing for newsprint is usually around 15%–20%; deinking flotation is responsible for approximately half [1]. Today, landfilling of the DIP mill rejects and sludges, usually after incineration, is expensive; within the next few years, it may even be prohibited in Europe. Thus, there is immense pressure to improve the material efficiency at DIP mills.

Flotation deinking is performed either in a one- or two-loop process. The two-loop process consists of pre- and postflotation, both of which often consist of two stages: primary and secondary. Preflotation removes most of the free ink detached in pulping. The pulp is then dewatered and dispersed at high consistency, which frees the remaining attached ink on the fibers and disintegrates large contaminants that will be removed during postflotation. In a simplified one-loop process, dispersion and the second loop are omitted. Other process stages in the deinking line also remove unwanted components that would cause runnability problems and quality deterioration in the end product. In general, large, nonpaper elements are removed during pulping; staples and flakes are removed during coarse screening; sand is removed during cleaning; ink and microstickies are removed during flotation; and macrostickies and dirt specks are removed during fine screening. Because the separation processes are not ideal in terms of selectivity, usable material such as fibers, fillers, and fines is lost during all the stages.

The main loss of valuable long fibers occurs during fine screening and the main loss of fillers and fines occurs during flotation; therefore, these reject streams have the highest potential for material recovery. In our previous study, we demonstrated that fiber recovery of approximately 80% from the fine-screening reject of a DIP mill was possible by dispersing macrostickies at a low consistency and then removing them during a separate flotation stage [2]. If fibers are recovered, then recovering some fillers and fines without changing their content in the paper machine is also possible. In addition, there is still room in many cases for an even higher filler and fines content recovery without sacrificing runnability of paper machines. The recycled fillers and fines must fulfill the optical property requirements to maintain the quality of the paper to be produced.

Methods to increase the yield by enhancing the selectivity of flotation in DIP processes have been studied intensively in recent years, although studies involving the use of depressants to attain increased selectivity appear to be limited. Water glass (sodium silicate) is generally used as a pH buffer and peroxide stabilizer and is also known to depress the flotation of filler [3]. Depressants are chemical agents that selectively modify the surfaces of particles to inhibit the adsorption of particles and thus reduce the tendency of those particles to become attached to the rising air bubbles. Polysaccharides such as carboxymethylcellulose (CMC) and starch typically are used as depressants in mineral flotation; they are known to depress materials such as calcite minerals [4]. Also, CMC has been known as a readily floatable silicate mineral depressant [5]. These additives, therefore, might act as functional depressants in recovery from flotation froth rejects where china clay and calcium carbonate content is 60%–80% by weight. Starch is known to readily adsorb on fiber surfaces, while CMC adsorbs on celluloses only in solutions containing high background electrolyte concentrations [6], such as DIP process waters. Because fiber fines tend to enrich in flotation reject [7] due to true flotation [8] or entrainment [9], there is great potential to increase their recovery using depressants in the secondary or tertiary reflotation stage.

Rather than increase yield in the primary flotation stage, the aim of this study was to examine the possibility of recovering fillers and fines from flotation froth reject. The use of CMC and starch depressants for intensifying flotation selectivity by refloating the froth reject was investigated. The research was focused on the reflotation stage because the nonexistent fiber network is beneficial for the retention of contaminants on bubbles [10]; thus, the possible depressing effect would be more apparent. The selectivity of flotation froth reject components—ink, china clay, calcium carbonate, and fines and fibers—was analyzed during the flotation process. The results indicated that the depressants used have the potential to improve filler and fines recovery.

MATERIALS AND METHODS

Flotation froth reject

Flotation froth reject for the study was collected from a European deinking mill that produces newsprint using a mixture of old newsprint and old magazines as the raw material (60/40 share) and has a yield of 82%. Conventional alkaline soap chemistry is used at the mill; peroxide, sodium hydroxide, water glass, and soap are fed into the pulper. The flotation froth reject was a mixture of pre- and postflotation secondary-stage rejects in a ratio of approximately 2:1. The reject had 3.9% dry matter content, pH of 8, ISO brightness of 33%, and ash content of 69.0% and 50.0% at 525°C and 900°C, respectively. Metal analyses showed that aluminum and calcium represented more than 96% of all metals, an indication that almost all ash originated from china clay and calcium carbonate.

Chemicals

The flotation froth reject contained residues of the flotation chemicals (fatty acids); therefore, no additional flotation chemicals were used in the flotation experiments. Instead, CMC and starch were tested as depressants to improve the selectivity of the flotation. The CMC was a standard grade polymer (Finnfix 30 from CPKelco; Atlanta, GA, USA) commonly used in the pulp and paper industry; it had a medium chain length and charge density (-5.0 mEq/g at pH 7). The starch was nonionic starch (charge density -1.0 µEq/g at pH 7). The dosages used (o.d. basis) were 90 g/ton, 122 g/ton, and 244 g/ton for the CMC and 244 g/ton and 488 g/ton for the starch. Both depressants were diluted to a 1.0% solution before addition to the flotation cell.

Flotation

A Voith Delta 25 batch laboratory flotation cell (Voith GmbH; Ravensburg, Germany) was used for the flotation experiments. The batch size was 16 L, and the airflow was kept constant (3.6 L/min). Each batch was preheated to 45°C, and the consistency was the same as the original flotation froth reject consistency (dry matter content of 3.9%). The generated froth was continuously removed, and the removed froth was weighed and analyzed.

Analyses

The dry matter content at 105°C (SFS-EN 20638, "Determination of dry matter content") and the ash content at 525°C and 900°C were determined according to the ISO 1762 ("Determination of residue [ash] on ignition at 525 degrees C") and ISO 2144 ("Determination of residue [ash] on ignition at 900 degrees C) standards, respectively. For the optical measurements, a sufficient amount of sample to obtain a basis weight of 225 g/m² was filtrated into a membrane filter (Sartorius Ø 50 mm, pores Ø 0.45 µm) to form an opaque pad (INGEDE Method 1 "Test sheet preparation of pulps and filtrates from deinking processes"). Brightness was analyzed from two parallel pads using an L&W Elrepho spectrometer (Lorentzen & Wettre; Kiska, Sweden) with an illumination of C/2. The SFS-ISO 2470 method ("Paper, board and pulps: measurement of diffuse blue reflectance factor [ISO brightness]") was used in the residual ink determination. Metals were determined from digested (U.S. EPA 3052) samples with inductively coupled plasma optical emission spectrometry (ICP-OES, Perkin Elmer Optima 5300DV, Perkin Elmer; Waltham, MA, USA).

Calculations

Reject rate

The term mass reject rate (RR_m) is used to describe the amount of matter removed from a sample by flotation. The mass reject rate was calculated using Eq. (1) and represents the proportion of rejected material to the flotation feed. Feed and reject are abbreviated as F and R, respectively.

$$RR_{\mathrm{m}} = \frac{w_{\mathrm{R}} \cdot dmc_{\mathrm{R}}}{w_{\mathrm{F}} \cdot dmc_{\mathrm{F}}} \cdot 100\% \tag{1}$$

where RR_m is the mass reject rate [%], w is the weight of the sample [kg], and dmc is the dry matter content of the sample [%].

Ink content

The ink content of the samples was measured as $ERIC_{700}$, which is based on the reflectivity measurement at 700 nm. The measurement correlates to the ink content but does not represent the true ink content. The true ink content is suggested to be dependent on $ERIC_{950}$ as follows in Eq. (2) [11]:

$$x_{ink} = 0.00561 \times ERIC_{950} \tag{2}$$

According to Körkkö et al. [12], $ERIC_{700}$ yields 1.57 times higher measurement values than $ERIC_{950}$; consequently, the true ink content can be calculated using Eq. (3):

$$x_{ink} = \frac{0.00561}{1.57} \times ERIC_{700} \tag{3}$$

where $ERIC_{950}$ is the effective residual ink content measured at 950 nm [ppm], $ERIC_{700}$ is the effective residual ink content measured at 700 nm [ppm], and x_{ink} is the true ink content [%].

Calcium carbonate and china clay content

The weight difference between samples incinerated at 525°C and 900°C can be used to evaluate the calcium carbonate and clay contents. If the weight difference is assumed to be due only to carbonate calcination to calcium oxide and carbon dioxide, then the contents of calcium carbonate and clay in the original sample can be calculated as follows in Eq. (4) [13]:

$$x_{CaCO3} = \left(x_{ash525} - x_{ash900}\right)\frac{M_{CaCO3}}{M_{CO2}} \tag{4}$$

The content of clay is then (Eq. [5]):

$$x_{Clay} = \left(x_{ash525} - x_{CaCO3}\right) \tag{5}$$

where x_{ash525} is the ash content incinerated at 525°C [%], x_{ash900} is the ash content incinerated at 900 °C [%], M_{CaCO^3} is the molar mass of calcium carbonate (100 g/mol), and M_{CO^2} is the molar mass of carbon dioxide (44 g/mol).

In this method, all inorganic substances are assumed to be clay and calcium carbonate. In mill samples, however, there are also other inorganic materials: metal salts and possible other minerals such as talc and titanium dioxide. If their amounts are low, the method will give reasonable results.

Fiber and fines content

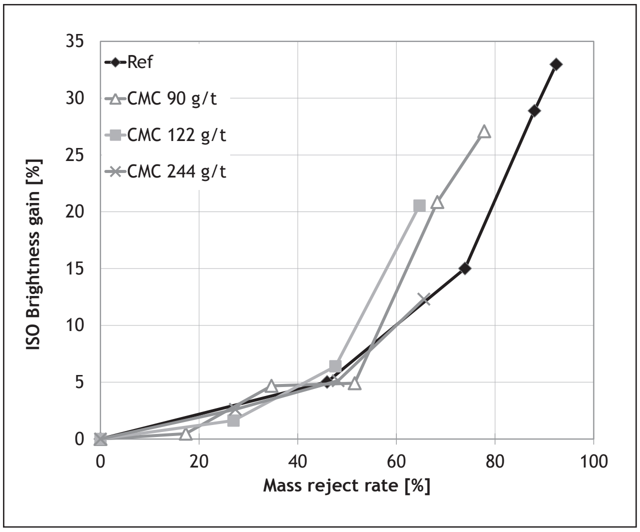
The fiber and fines content can be calculated as the balance (Eq. [6]):

$$x_{fiber} = 100 - x_{Clay} - x_{CaCO3} - x_{i\,nk} \tag{6}$$

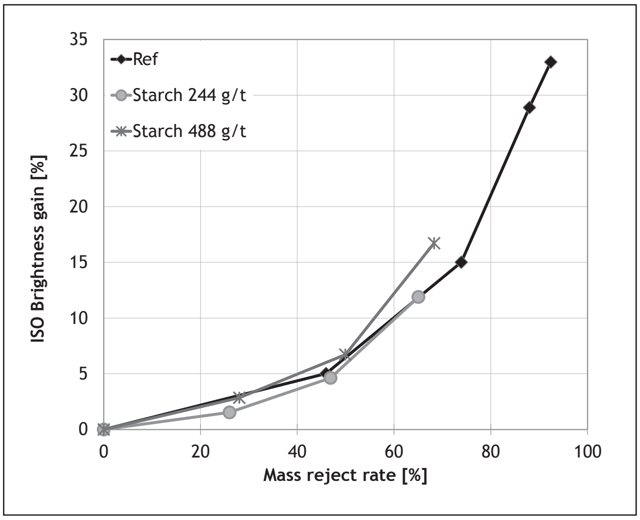
RESULTS

Brightness gain in flotation with depressants

Considerable brightness gains were achieved only at the higher reject rates by reflotation of flotation froth reject. To attain an increase of 20 percentage points, the reject rate needed to be 60%–80%. A higher brightness gain for a given reject rate was achieved using CMC as the depressant (**Fig. 1**) compared with the reference where flotation was performed without additional chemicals. However, the difference became apparent only after the reject rates were greater than 50%. In addition, too large of a CMC dosage



1. Effect of carboxymethylcellulose on brightness gain.



2. Effect of starch on brightness gain.

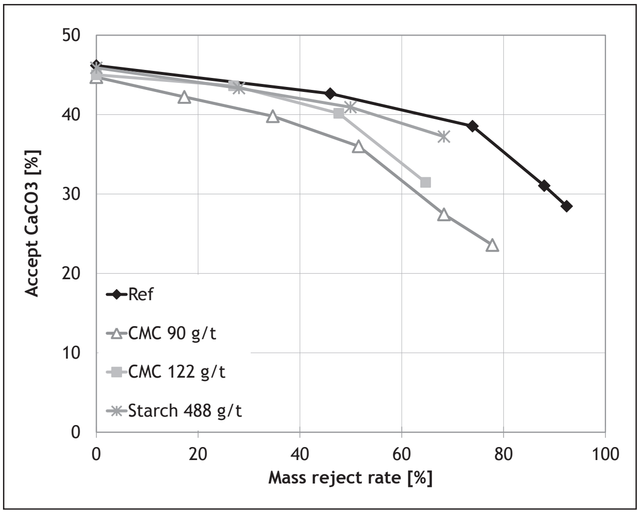
negated the positive effect; with a CMC dosage of 244 g/ton, the brightness gain did not differ from the reference curve.

Compared with the reference, a better brightness gain was also attained using starch at higher reject rates if the dosage was high enough, that is, 488 g/ton in this case (**Fig. 2**). However, the effect remained lower than that for CMC dosages of 90 g/ton and 122 g/ton. With the starch dosage of 244 g/ton, no difference was observed as compared to the reference.

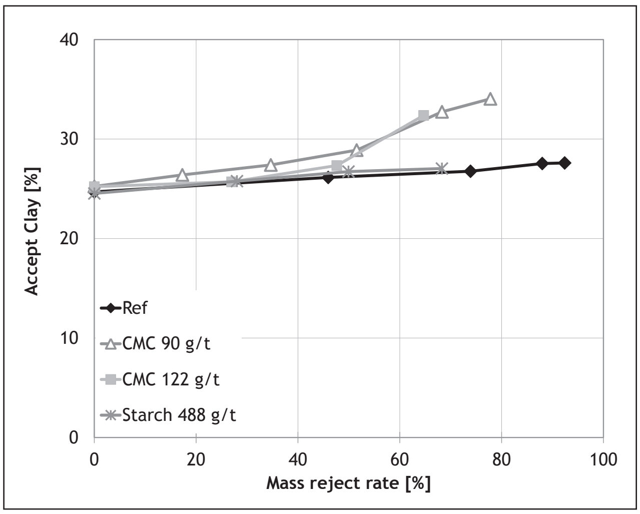
Selectivity of depressants for various components

Figures 3–7 illustrate the changes in the content of the components (calcium carbonate, clay, total ash, fiber and fines, and ink) in the reflotation accepted pulp as flotation proceeded. In these figures, the highest CMC dosage and the lowest starch dosage (both being 244 g/ton) have been removed because they did not differ from the reference.

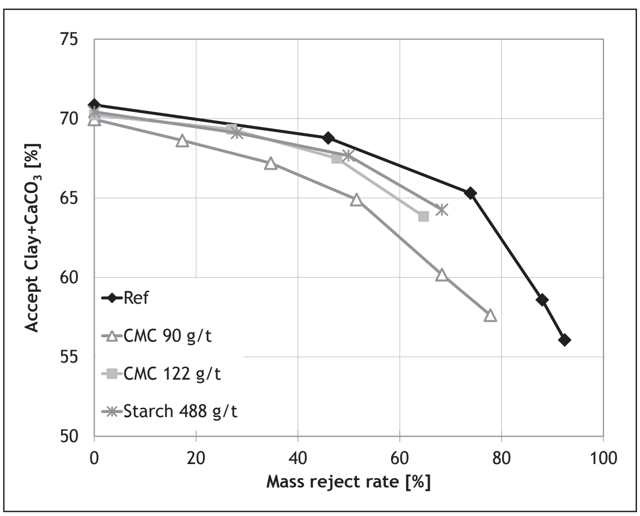
The depressants caused an increase in removal of calcium



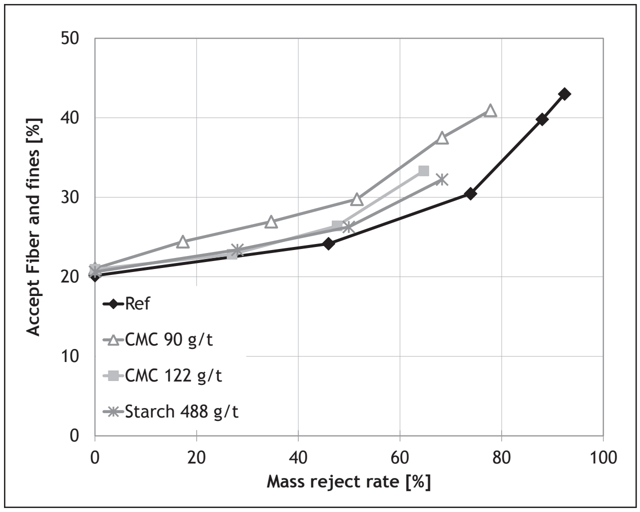
3. Percentage of calcium carbonate in the reflotation accept as a function of the mass reject rate.



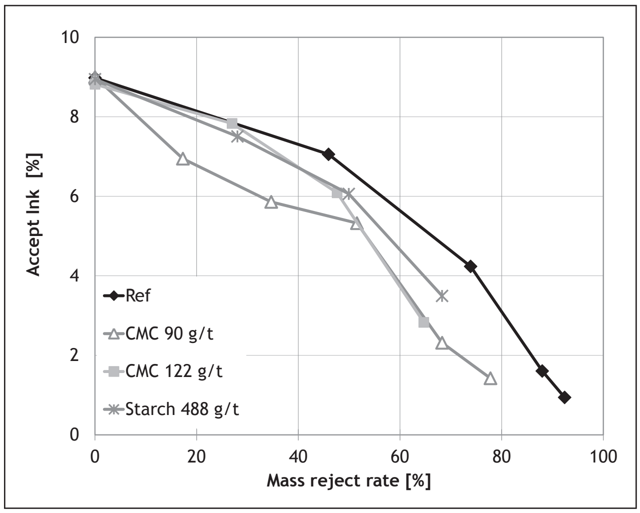
4. Percentage of clay in the reflotation accept as a function of the mass reject rate.



5. Percentage of total ash (i.e., clay and calcium carbonate) in the reflotation accept as a function of the mass reject rate.



6. Percentage of fiber and fines in the reflotation accept as a function of the mass reject rate.



7. Percentage of ink in the reflotation accept as a function of the mass reject rate.

carbonate compared with the reference flotation, which was particularly apparent for reject rates over 50% (Fig. 3). The effect was pronounced for CMC; the lowest CMC dosage of 90 g/ton had a slightly larger effect than the 122 g/ton dosage. The effect of starch was weaker.

The clay content in the reflotation accept (Fig. 4) increased slightly when flotation proceeded. A pronounced effect of CMC was observed at higher reject rates. The effect was quite similar for both dosages, while starch did not cause a depressing effect at all for clay.

The total ash content in the reflotation accept (Fig. 5) decreased slightly at the beginning of flotation and then started to decline more steeply. A CMC dosage of 90 g/ton removed the most filler in these tests, whereas the effect of starch was weaker.

The fiber and fines content in the reflotation accept (Fig. 6) noticeably increased as the flotation proceeded, and the use of depressants improved flotation performance. The best results

	Deinking Losses		Recovery From Rejects		Pulp to Paper Machine			
	Flotation Loss	Fine Screening Loss	Flotation Froth Reject	Screen Reject	Yield	Fiber Content	Fines Content	Filler Content
ref1	**20.0%**	**1.0 %**	**0.0%**	**0.0%**	**79.0%**	**69.0 %**	**16.0 %**	**15.0 %**
rec1.1	20.0%	1.0 %	2.0%	80.0%	80.2%	69.0 %	16.0 %	15.0 %
rec1.2	20.0%	1.0 %	10.0%	80.0%	81.8%	67.6 %	16.4 %	16.0 %
rec1.3	20.0%	1.0 %	20.0%	80.0%	83.8%	66.0 %	17.0 %	17.0 %
rec1.4	20.0%	1.0 %	30.0%	80.0%	85.8%	64.5 %	17.5 %	18.0 %
rec1.5	20.0%	1.0 %	20.0%	0.0%	83.0%	65.7 %	17.2 %	17.2 %
ref2	**15.0%**	**1.0 %**	**0.0%**	**0.0%**	**84.0%**	**69.0 %**	**16.0 %**	**15.0 %**
rec2.1	15.0%	1.0 %	20.0%	80.0%	88.8%	66.2 %	16.9 %	16.9 %
ref3	**25.0%**	**1.0 %**	**0.0%**	**0.0%**	**74.0%**	**69.0 %**	**16.0 %**	**15.0 %**
rec3.1	25.0%	1.0 %	20.0%	80.0%	78.8%	65.8 %	17.1 %	17.1 %
ref4	**15.0%**	**2.0 %**	**0.0%**	**0.0%**	**83.0%**	**69.0 %**	**16.0 %**	**15.0 %**
rec4.1	15.0 %	2.0 %	20.0%	80.0%	88.6%	66.4 %	16.8 %	16.8 %

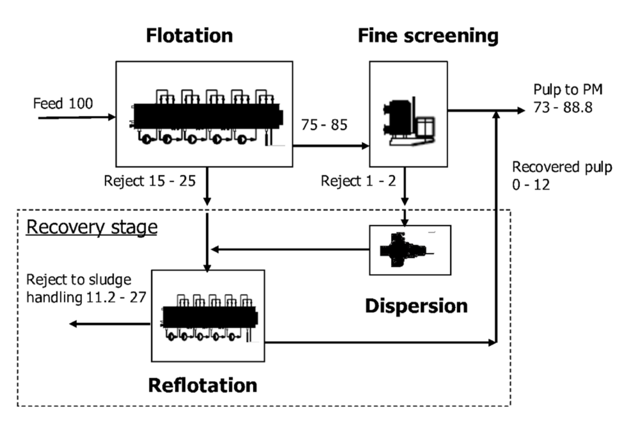
I. Effect of recovery on the yield and pulp components based on the mass balance calculations for a simplified deinking process.

were attained using CMC, and the lowest dosage increased the fiber recovery the most. The effect was quite similar for both CMC dosages. Again, the starch effect was weaker.

The ink content in the reflotation accept (Fig. 7) was significantly decreased as the flotation proceeded. The depressants improved the ink removal selectivity, particularly at higher reject rates. CMC had a greater depressing effect than starch for the dosages investigated.

Effects of reject recovery on pulp for papermaking

Table I presents the yield and percentage of pulp components that were calculated based on the mass balances for various cases. **Figure 8** shows the simplified base process that was considered (flotation and subsequent fine screening).



8. Simplified deinking process with reject recovery stage.

In the reference cases (ref), no recovery occurred; however, the flotation loss and fine screening loss was modified. In the recovery cases (rec), the degree of recovery from the flotation froth and fine screen reject was varied. The following assumptions were made in the calculations:

- The content of fibers, fines, filler, and ink in the feed pulp were 51.0%, 15.0%, 25.0%, and 9.0%, respectively.
- The ink content in the pulp to the paper machine was 0.0%.
- The ratio of fines to filler after the reflotation stage in the recovered pulp was 2:3.
- The flotation froth reject composition did not change with changes in the flotation reject rate.
- The screen reject consisted of only long fibers and macrostickies.
- Macrostickies were dispersed at low consistency before the reflotation stage.

DISCUSSION

The use of depressants, in particular CMC, appeared to be advantageous in reflotation. Selectivity could be increased, and thus, a greater amount of usable material could be recovered. To achieve a brightness gain of 25 percentage points, approximately 15.0% of material could be recoverable without the use of depressants. Using CMC, the amount could be increased to approximately 25%. The ash content in the recovered pulps was approximately the same (i.e., 60.0%). The brightness gain was sufficient for newsprint production; however, for improved newsprint, a brightness gain of 30% may be required, and recovery would then be 5–10 percentage points lower. A limiting factor for recovery is the contamination of fines and filler surfaces. Fines and fillers in the flotation froth reject have been observed to have more attached ink than ones in the flotation accept [14].

Because of the complex chemistry during deinking, it was not clear whether CMC and starch worked as depressants from the mechanism point of view; however, they worked as functional depressants. It can be suggested that the polysaccharides, which tended to attach readily to fiber and fines surfaces in the chemical conditions used [6], resulted in increased surface hydrophilicity. This resulted in more than a doubling of the fines content in flotation accept at reject rates of approximately 70.0%. However, the fibers and fines originating from mechanical pulp can have a slightly hydrophobic nature and consequently have a tendency to be rejected in preflotation, as demonstrated by Körkkö et al. [7]. The lower depressant effect of starch was probably due to its lower charge density and hydrophilicity. Moreover, CMC is known to reduce friction between fibers [6, 15] and lower flocculation. In our experiments, there was no continuous fiber network due to the low amount of fibers; however, microflocs or agglomerates of fines might have existed. It is possible that CMC dispersed those microflocs and thus decreased their tendency to be floated.

Of the minerals, clay was likely able to adsorb CMC and starch via hydrogen bonding between the hydroxyl groups of polysaccharides and filler [16]. This effect increased the hydrophilicity of clay and its content in the reflotation accept as flotation proceeded. Calcium carbonate, however, was enriched in the reject, and the use of starch and especially CMC strengthened its removal. An explanation for this phenomenon may be low flotation pH; Süss et al. [3] have reported low recovery of calcium carbonate at pH near 8. Furthermore, the surface of calcium carbonate could be contaminated by the hydrophobic impurities due to competitive adsorption of trash substances, and thus a depression effect was not seen. Calcium carbonate, originating from paper coatings, can be covered by the hydrophopic coating additives in particular, while clay is used more preferably as a filler without such hydrophopic substances. The observed difference in flotation behavior between pigments was consistent with the observations made by Heimonen and Stenius [17]; however, the effect of CMC and starch was opposite to that observed in their experiments with model substances. Flotation tests using unprinted pigments have demonstrated that flotation behavior may depend on the origin of the pigment; that is, whether it has been used as a filler or a coating [18].

The use of polysaccharides in deinking flotation in the primary stage was studied earlier on a pilot scale by Zeno et al. [19]; however, their approach differed from that of our study. They studied CMC and guar gum as dispersion agents to decrease fiber flocculation under conditions corresponding to the preflotation of a DIP mill. Both agents were observed to enhance flotation selectivity, and the authors concluded that this result was due to decreased fiber flocculation. The depressing effect was also discussed by them. Despite the positive effects of their study, the use of expensive special chemicals in the preflotation trough where all the pulp suspension flowed may not be reasonable. In addition, the dosages used by Zeno et al. [19] were tenfold compared with those used in this study; consequently, the chemical costs of CMC can be estimated to be on the order of EUR 20–40 per ton of deinked pulp. Therefore, the use of polysaccharides as a depressant instead of a dispersant appears to be more feasible because much lower dosages are required. If polysaccharides are used in the secondary or tertiary stage of flotation, where the amount of pulp to be chemicalized is much lower, the additional CMC costs would be approximately EUR 0.5–2 per ton of deinked pulp.

Avoidance of overdosing with CMC is reasonable, not only because of the chemical costs but also because the depressing effect appeared to be diminished at high dosages. This result may be explained by the saturation of CMC on the fines surfaces; after the saturation point, extra CMC may increase their presence on all surfaces or serve as a dispersion agent, causing ink and other particles to be dispersed. The dispersive nature of well-concentrated CMC and consequent decrement of ink removal in flotation has been reported by Johansson et al. [20, 21]. In our study, starch was observed to have a weaker depressing effect than CMC, perhaps because of its lower anionic charge. Even using more than a quadruple dosage of starch compared with CMC did not yield a similar effect to CMC, indicating that starch may not be a good candidate for use as a depressant even though it is much less expensive. Both for starch and CMC optimization, a study would be required to determine the optimal polymer length and charge density.

In this study, the flotation froth reject originating from the secondary stage of flotation was refloated. A small increase in recovery of filler and fines could be achieved by adjusting the flotation performance in the primary or secondary stage; however, in this study, the flotation was extended by an additional tertiary flotation stage. The additional flotation stage favors the selective removal of particles; floatable particles (i.e., ink, fillers, and fines) are mainly removed in the primary stage; ink and fillers are removed in the secondary stage; and ink is removed in the tertiary stage [22]. Although additional separation stages always increase the total selectivity, the number of stages will in practice be limited by operation and investment costs together with raw material and disposal costs. In particular, disposal costs are increasing, and a tertiary stage in mill flotation eventually may become attractive. To intensify existing DIP processes, a tertiary flotation stage is not the only possibility; the use of CMC in the secondary stage might be reasonable. A positive effect is attainable with small dosages, and the amount of pulp to be treated chemically is quite low, depending directly on the reject rate of the primary stage (usually lower than 30%).

If a two-loop DIP line is used, recovery from the secondary stage of postflotation might be appropriate because the reject would be cleaner than the preflotation reject. In addition, several surface-active substances diluted into the pulp from the recovered paper together with the dosed surfactants tend to concentrate into the reflotation stages, decreasing yield and contaminant removal selectivity [23]. During postflotation, the surfactant amount is lower than during preflotation because surfactants are not usually dosed in the second loop; thus, increased efficiency of depressants is expected during this stage.

Considerable material recovery is possible from the flotation froth reject of a DIP mill to be used in newsprint production. Material recovery from flotation froth reject will increase the content of filler and fines by a few percentage points in a paper machine (see Table I and Fig. 8). In some cases, runnability may not tolerate such an increase. However, if the fine screening reject containing mainly long fibers is also recovered, there is less of an increase in filler and fines content. The recovery of up to 80% of fibers from the fine screening reject is possible by dispersing macrostickies at a low consistency and removing them using a separate flotation stage [2].

CONCLUSION

Approximately 10%–15% recovery of fines and filler with adequate brightness to be used in newsprint production is possible without additional chemicals by reflotating froth reject during the tertiary stage. Carboxymethylcellulose is a functional depressant that enhances fiber fines recovery in particular and, to some extent, china clay recovery while lowering calcium carbonate recovery. Due to enhanced selectivity, recovery can be at least doubled, to approximately 20%–30%, using CMC. In addition, while starch also has a depressing effect, the effect is weaker than that of CMC even when using multifold dosages. **TJ**

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We chose this topic because the yield loss at deinked pulp mills is so great that improvements are needed for sustainable deinked pulp production. Flotation froth reject is the largest waste stream produced in a DIP mill, thus the recovery of material from it to reduce the amount of waste is of great interest. This work complements our earlier studies related to improved material efficiency by introducing means to recover filler and fines of sufficient quality from flotation froth rejects.

The most difficult aspect of this study was to monitor the selectivity of the flotation. We solved this by weight content calculations. Although adequate brightness gain was also achieved without additional chemicals, it was interesting to observe that depressants, especially CMC with low dosages, increased further the selectivity of the flotation and thus yield.

Mills can benefit from this study by using this new information in the design of environmentally sound and economical deinking strategies. The results may encourage DIP mills to test CMC as a filler and fines depressant in their present secondary flotation stages.

In next phase we will study how improved material efficiency by recovering fibers, fillers, and fines from reject streams of a DIP mill can be solved without deteriorating dewatering properties of sludge for disposal.

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