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Liisa Mäkinen

IMPROVEMENT OF RESOURCE EFFICIENCY IN DEINKED PULP MILL

UNIVERSITY OF OULU GRADUATE SCHOOL;
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IMPROVEMENT OF RESOURCE EFFICIENCY IN DEINKED PULP MILL

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Supervised by
Professor Jouko Niinimäki
Doctor Ari Ämmälä

Reviewed by
Professor Angeles Blanco
Doctor Udo Hamm

Opponent
Professor Samuel Schabel

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University of Oulu Graduate School; University of Oulu, Faculty of Technology, Department of Process and Environmental Engineering, P.O. Box 4300, FI-90014 University of Oulu, Finland
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Abstract

Paper recycling is an ecological strategy for disposing of waste paper, but more importantly, recovered paper is an important source of raw material in the paper and board industry. Deinked pulp production from recovered paper has proved economically viable by comparison with virgin fibre pulp manufacturing, but this viability is now threatened by increasing waste disposal costs, as up to 25% of the raw material available for deinked pulp production can end up in the reject streams, which then have to be disposed of. The most common practice at present is incineration and disposal of the resulting ash in landfills, but tightening legislation has meant that landfills have become very expensive and are likely to be completely banned in the near future. Thus new ways have to be sought for managing the waste problem in deinked pulp production in order to ensure that the recycled paper production remain both economically and environmentally feasible. The aim of this thesis was to study means of improving the material efficiency of a deinked pulp mill without excessively detracting from end product quality or the performance of the combined sludge dewatering stage.

First, an analytical procedure was developed for determining the utilisation potential of reject streams, and a considerable potential for material recovery from these streams was identified. The results presented here show that 80% of the most valuable long fibres from the fine-screening rejects and 15% of the fine material from the flotation froth reject can be recovered. Altogether, with the simultaneous recovery of both categories of reject, it would be possible to improve the material efficiency at the deinked pulp mill by a total of about 5 percentage units, which can be considered significant for process efficiency. Moreover, this would enable the fibre content of the combined sludge to be kept above a certain limit, so that the combined sludge dewatering properties would not be affected. Consequently, the results presented in this thesis provide several widely applicable means for improving the resource efficiency of a deinked pulp mill.

Keywords: deinking, dewatering, fibre fines, fibres, fine material, mineral filler, recovery, recycling, resource efficiency, sludge

Mäkinen, Liisa, Siistausmassan valmistuksen resurssitehokkuuden parantaminen.
Oulun yliopiston tutkijakoulu; Oulun yliopisto, Teknillinen tiedekunta, Prosessi- ja ympäristötekniikan osasto, PL 4300, 90014 Oulun yliopisto
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Tiivistelmä

Paperin kierrätys uusiksi paperituotteiksi on ympäristöystävällinen tapa jätepaperin käsittelemiseksi. Uusiomassan valmistus on myös taloudellisesti kannattavaa verrattuna paperimassan valmistukseen neitseellisistä raaka-aineista. Taloudellinen kannattavuus on kuitenkin vaarassa jätemaksujen alati kasvaessa, sillä siistausmassan valmistuksessa jopa 25 % kierrätyspaperiraaka-aineesta päätyy jätejakeisiin, jotka hävitetään yleisesti polttamalla ja läjittämällä syntynyt tuhka kaatopaikalle. Lainsäädäntö on rajoittanut kaatopaikkasijoittamista ja se on nykyään hyvin kallista. Tulevaisuudessa jätteiden kaatopaikkasijoittaminen voi olla jopa täysin kiellettyä. Jäteongelman ratkaisemiseen tarvitaan siis uusia menetelmiä, jotta kierrätyspaperin valmistus säilyisi sekä taloudellisista että ympäristöllisistä näkökulmista kannattavana toimintana. Tämän väitöskirjatyön tavoitteena oli etsiä keinoja parantaa siistausmassan valmistuksen resurssitehokkuutta palauttamalla käyttökelpoisia materiaaleja jätevirroista takaisin prosessiin siten, ettei heikennetä lopputuotteen laatua eikä lietteidenkäsittelyprosessin toimintaa.

Väitöstyön alussa kehitettiin analyysiproseduuri rejektivirtojen hyötykäyttöpotentiaalin arvioimiseksi ja siistamon rejektijakeissa havaittiin merkittävästi hyödynnettävää materiaalia. Tulokset osoittavat, että 80 % hienolajittelun rejekteissä poistuneista arvokkaista pitkistä kuiduista voidaan palauttaa takaisin prosessiin. Lisäksi flotaation vaahtorejektistä voidaan palauttaa 15 % hienoainetta. Palauttamalla yhtäaikaisesti sekä kuituja hienolajittelun rejekteistä että hienoainetta flotaation vaahtorejektistä, voidaan siistausmassan valmistuksen materiaalitehokkuutta parantaa yhteensä noin 5 prosenttiyksikköä, mikä on merkittävä parannus prosessitehokkuuteen. Kuitujen ja hienoaineen samanaikainen palauttaminen pitää lisäksi lietteiden käsittelyssä seoslietteen kuitupitoisuuden tarpeeksi korkeana, jotta sen vedenerotusominaisuudet säilyvät hyvinä. Tämän tutkimuksen tuloksena löydettiin siis useita laajalti sovellettavissa olevia keinoja siistausmassan valmistuksen resurssitehokkuuden parantamiseksi.

Asiasanat: hienoaine, keräyspaperi, kierrätys, kuitumainen hienoaine, siistaus, talteenotto, täyteaine

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Oulu, April 2013 Liisa Mäkinen

Abbreviations

DIP	Deinked pulp
CEPI	Confederation of European Paper Industries
CMC	Carboxymethyl cellulose
EU	European Union
ERPC	European Recovered Paper Council
INGEDE	International Association of the Deinking Industry (Internationale Forschungsgemeinschaft Deinking-Technik)
IPPC	Integrated Pollution Prevention and Control Directive
LC	Low consistency
LWC	Lightweight coated paper
na	Not analysed
od	Oven dry
ONP	Old newsprint
OMG	Old magazines
RCF	Recycled fibre
SC	Supercalendered paper
TAPPI	Technical Association of the Pulp and Paper Industry

A	Accept
cs	Consistency [%]
dmc	Dry matter content [%]
E_r	Removal efficiency of a component [%]
F	Feed
w_i	Weight of the sample [kg]
R	Reject
RR_m	Mass reject rate [%]

List of original publications

This thesis is based on the following papers, which are referred throughout the text by their Roman numerals:

I Mäkinen L, Ämmälä A, Pirkonen P & Niinimäki J (2012) Sludges - analysis procedure for evaluating the utilisation potential. IPW (1): 36–42.

II Mäkinen L, Körkkö M, Ämmälä A, Haapala A, Suopajärvi T & Niinimäki J (2012) Recovering fibres from fine-prescreening reject at deinking mills. TAPPI J 11(11): 53–62.

III Ämmälä A, Mäkinen L, Liimatainen H & Niinimäki J (2013) Effect of carboxymethyl cellulose and starch depressants on recovery of filler and fines in tertiary flotation. TAPPI J 12(3): 43–50.

IV Mäkinen L, Ämmälä A, Körkkö M & Niinimäki J (2013) The effects of recovering fibre and fine materials on sludge dewatering properties at a deinked pulp mill. Resour Conserv Recy 73(1): 11–16.

The author of this thesis was the primary author of Papers I, II and IV, and a co-author of Paper III. Her main responsibilities in connection with Papers I, II and IV were experimental design, data analysis and reporting of the results. For Paper III she designed and performed the experiments and analysed the results together with the first author. The additional authors participated in the designing and writing of the papers by making valuable comments.

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1 Background

Paper has remained central to our lives despite the predictions associated with the digital revolution. To keep the pulp and paper industry vital, paper recycling is necessity, especially as it is by far the most economical and ecological strategy for disposing of waste paper, being superior to alternatives such as thermal utilisation, incineration or landfilling (Schmidt *et al.* 2007, Villanueva & Wenzel 2007). Recovered paper has been constantly increasing in importance, and it is now the most important source of raw material for the production of paper and board (Ervasti 2010).

One of the most critical issues facing the production of deinked pulp (DIP) from recovered paper is the great number of residues to be disposed of. This means that the resource efficiency of DIP production has declined within the few years due to the deteriorating quality of the raw material (Hudson 2011, Moore 2011). Paper recycling and DIP production has been economically viable compared with virgin fibre pulp manufacturing but now, due to the increasingly stringent waste legislation and increasing costs of waste disposal, the economic viability of DIP production is threatened. New ways of managing the waste problem have to be developed in order to ensure that the recycled paper production remain both economically and environmentally feasible.

Material loss during DIP production can be as high as 25% (Hamm 2010), and because of the limited selectivity of the deinking processes, the reject streams contain some valuable reusable components in addition to contaminants. As the cost of quality recovered paper continues to increase, the loss of valuable material in the form of rejects is becoming both environmentally and economically unacceptable. It would therefore be desirable to be able to recover material from the reject streams in order to improve the material efficiency of a DIP mill, if only suitable means were available for doing so.

2 State of the art

2.1 Deinked pulp production

In general terms, recycled fibre (RCF) processes can be divided into two main categories, based on whether they have a deinking stage or not. Processes with only mechanical cleaning, *i.e.* without deinking, can be used for products such as testliner, corrugating medium, uncoated board and cartonboard, while processes involving both mechanical cleaning and deinking are required for products such as newsprint, tissue, printing and copy paper, magazine papers *e.g.* supercalendered (SC) and lightweight coated (LWC) papers, coated board and cartonboard or market DIP (IPPC 2001).

During the production of deinked pulp, printing inks, stickies and other contaminants that are likely to disturb the processing of the recovered paper or the quality of the final product are removed from the pulp in several processing stages. Generally, large, non-paper elements are removed during pulping, staples and flakes are removed by coarse screening, sand is removed by cleaning, ink and micro stickies (<100–150 µm) are removed by flotation or washing, and macro stickies (>150 µm) and dirt specks are removed by fine screening.

2.1.1 The production line for deinked pulp

Deinking is a process used to produce deinked pulp from recovered paper, which usually consists of a combination of old newspapers (ONP) and old magazines (OMG). The recycled fibre pulp is a mixture of fibres, pigments and fillers, binders, and additives and various other components, including non-paper elements. Consequently, this mixture has to be processed in various ways to meet the quality criteria for the paper to be produced. A deinking line thus consists of sequential unit operations which are connected together in different ways. The arrangement of the unit processes may vary significantly from mill to mill, and some mills have a second or even a third loop in order to achieve a higher quality of pulp. A simplified single-loop deinking process line for producing deinked pulp for newsprint, for example, is presented in Fig. 1.

Fig. 1. A deinking line (with 1 loop) and rejects generated in the course of deinked pulp production. Modified from Paper IV, published by permission of Elsevier.

Deinked pulp production starts with repulping of the recovered paper, in which it is reduced to individual fibres and particles with the aid of warm water and chemicals. The aim is to detach the printing ink from fibres. At the end of the pulper there is a screening section, typically with 6–20 mm holes (Schabel & Holik 2010) to separate out large non-paper elements such as plastic foils, CD's, strings, textiles, wet strength paper, bottles, wires, etc. from the pulp. It is desirable to remove solid contaminants at as early a stage as possible so that they do not disturb the following stages or break down into excessively small particles. Depending on the grade of the recycled paper, about 0.5%–1% of the raw material fed into the process is rejected in pulping (Pöyry 1995).

After pulping, the pulp suspension is run through screens in order to remove debris and solid contaminants from the pulp in processes based on the particle size, shape and deformability of the contaminants. Coarse screening at the beginning of the process line removes large debris such as staples, sand, flakes and grits, while fine screening, which can take place either before or after pre-flotation (or both), is mainly aimed at removing macro stickies and dirt specks. Complete screening of pulp in a single stage is seldom possible without significant fibre losses, and therefore the first-stage screen rejects are rescreened in a second, third or even fourth stage. The last stage (tailing screen) determines the fibre losses of the screening system, being typically about 1%–2% (Hamm 2010, Schabel & Holik 2010). Screens of several kinds are necessary in order to maximise the cleaning efficiency and minimise fibre losses, *i.e.* the screens can be either discs (holes 2.0–3.0 mm) or cylinders (holes 0.8–1.5 mm or slots 0.08–0.4 mm), and either pressurised or at atmospheric pressure (Schabel & Holik 2010).

Larger holes are used at the beginning of the process line, while narrow slots are more typical in the middle and at the end.

Centrifugal cleaning with hydrocyclones is an essential separation process that complements other methods such as screening. Depending on the operation mode, substances that are either lighter or heavier than water are separated out by cleaning. These include mainly plastic foam and other plastic materials, or sand, glass, shives and fragment of metal. The loss in yield attributable to centrifugal cleaning is about 0.5%–1.5% (Pöyry 1995).

Selective flotation is a key process for removing ink and micro stickies, although washing is more frequently used for ink removal in North America, and is in any case common in tissue paper production. In the flotation process air bubbles are fed into the pulp suspension at about 1%–1.2% consistency, so that the hydrophobic particles will become attached to them and rise to the surface, where they form a foam that can be removed. These particles include printing ink, stickies, fillers, coating pigments and binders. Hydrophilic fibres remain in the pulp suspension, of course, although some fibres can end up in the foam on account of mechanical entrainment (Ajersch 1997, Dorris & Page 1997). Several flotation stages may be needed for the efficient removal of contaminants (pre-flotation and post-flotation stages), while in order to minimise losses, one flotation system will usually include two stages (primary and secondary) connected to form a cascade. The flotation froth reject, consisting of the pre-flotation and post-flotation secondary stage rejects, is the largest waste stream produced at a DIP mill, accounting for about 8–16% of the raw material (Hamm 2010).

Dewatering and thickening of the pulp is necessary in order to increase its consistency in the subsequent processes and separate water loops. Dewatering is typically carried out with a combination of a disc filter and a screw press, while dissolved air flotation (DAF) is used for cleaning thickening filtrates. The process water clarification rejects (DAF sludges) consist of colloidal material, fillers, fibres, fines and ink. The loss of yield via DAF sludges is about 1%–5% (Hamm 2010).

To achieve higher quality, pulps may be dispersed and bleached. Dispersion breaks down the residual contaminants to a size at which they usually no longer interfere. A post-bleaching stage may be added at the end of the deinking line if improved optical characteristics are desired.

Effluents from the DIP process are treated in an external waste water treatment plant, generally involving three stages, called the primary, secondary and tertiary treatments. The primary stage is intended to remove solid matter from the effluent, the principal methods being clarification, flotation and filtration (Hynninen 2008), while the secondary treatment uses microbes to break down dissolved and suspended biological matter. The most frequently used secondary processes are activated sludge treatment, aerated lagoons and biofilters (Hynninen 2008). Activated sludge treatment has two main stages: an aeration tank and a clarification to settle out the biological floc (Fig. 2). Most of the activated sludge is pumped back into the aeration tank, while any excess sludge is removed from the process. Sometimes a tertiary treatment, *e.g.* chemical oxidation or activated carbon adsorption, can be used to further improve effluent quality.

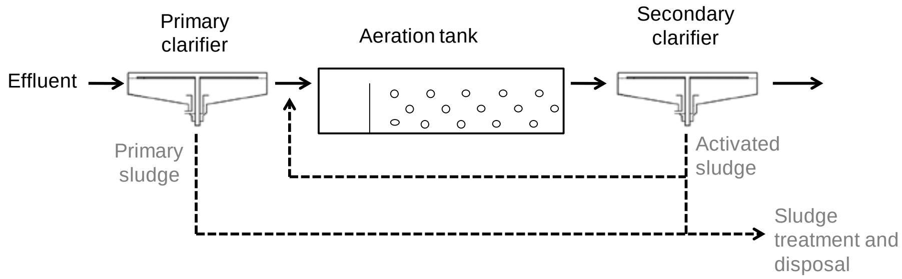

Fig. 2. Principal stages in effluent treatment and the sludges generated in the course of them.

2.1.2 Reject and sludge treatment at a deinked pulp mill

Coarse rejects from recovered paper pulping and coarse screening have no recycling potential and are usually dumped or, if practicable, incinerated (IPPC 2001). Metal wires and staples can be sold as metal waste, but all the other reject and sludge streams produced in the mill area and effluent treatment are typically mixed together into a combined sludge before further treatment (Engel & Moore 1998, Scott & Smith 1995), the first stage in which is dewatering. Dewatering is an essential stage regardless of the method of recycling or disposal, as all the common utilisation and disposal methods benefit from a high dry matter content. The combined sludge is often dewatered in two-stage processes with initial dewatering using gravity tables or drums and disc thickeners, followed by high-consistency dewatering in belt filter presses or screw presses (Hamm 2010). Chemical conditioning of the sludge with polyelectrolytes is a common practice for improving its dewatering properties (Lo & Mahmood 2002, Ray *et al.* 2011, Wenzel 1996).

After dewatering, the combined sludge is usually either burned or used for landfilling (Dahl *et al.* 2008, Monte *et al.* 2009). Incineration and disposal of the ash in landfills is currently one of the most common practices for disposing of rejects and sludges in the pulp and paper industry (Engel & Moore 1998, Monte *et al.* 2009), the objectives of the incineration stage being to reduce the volume of waste, render the organic components inert, immobilise harmful substances in the ash, and make use of the energy generated (Hamm 2010). Due to the high moisture content of sludge even after dewatering, however, the energy value of combined sludge is low and direct incineration of the dewatered sludge can be energy-deficient process, implying that incineration is in reality only a means of reducing the volume of the sludge (Monte *et al.* 2009).

Besides incineration and landfill sites there are some industrial applications and other possible means of utilising reject and sludges, *e.g.* in land applications, including fertilizers, soil amendment and earthworks (Aitken *et al.* 1998, Barriga *et al.* 2010, Engel & Moore 1998, Webb 2000), in thermal processes, including incineration with energy recovery, pyrolysis, steam reforming, wet oxidation and supercritical water oxidation (Engel & Moore 1998, Fitzpatrick & Seiler 1994, Frederick *et al.* 1996, Gavrilescu 2008, Gidner & Stenmark 2002, Kay 2002, Monte *et al.* 2009, Ouadi *et al.* 2012), the production of building materials, including panels, tiles, blocks and briquettes (Blanco *et al.* 2008, Cernec *et al.* 2005, Gerischer *et al.* 1998), applications utilising the mineral part of the sludge through incineration, including filler recovery (Dahlblom *et al.* 2004, Engel & Moore 1998, Kunesh *et al.* 2010), and as an ingredient of asphalt, bricks or cement (Albinas & Zivile 2003, Biermann *et al.* 1999, Cernec *et al.* 2005), in absorbent manufacturing (Engel & Moore 1998, Glenn 1998, Ilyina *et al.* 2000) or in glass aggregate production (Carroll & Reeves 1999, Engel & Moore 1998). The alternative uses for sludge and ash are an excellent option if a user can be found near the mill and if long-term contracts can be concluded (IPPC 2001, Scott & Smith 1995).

2.1.3 Yield and quality aspects

Main goal of the deinking steps is to eliminate contaminants from the pulp in order to meet the optical appearance requirements. Depending on the waste paper grade and requested quality of the product, a total of 15%–25% of the material entering a deinked pulp mill will be rejected (Hamm 2010). These rejects include deleterious materials such as printing ink, stickies and other non-paper contaminants, but also useful fillers, fibres and fines.

Printing ink which remains in the DIP pulp affects the quality of the paper, can adhere to stickies and can cause fouling problems (Pöyry 1995). Ink gives the paper a grey appearance and large ink particles may be visible as specks. The average ink application during the printing process is about 10–20 kg dry ink per ton of paper, which thus means only about 1%–2% of the total mass (Renner 2000), while the yield loss during the separation of ink in flotation can be 16% (Hamm 2010). The selectivity of the ink flotation process is indeed poor.

Flotation losses depend on a large number of parameters (Ajersch 1997, Dorris & Page 1997, Süss *et al.* 1994), namely those affecting the hydrophobicity of fibres, fillers and fines (chemical nature of particles) and those affecting the state of pulp flocculation (Carré *et al.* 2001). Deinking has been found to be best achieved at low consistencies, while a drop in efficiency can be observed at high consistencies near and above 1.2% (Chaiarrekij *et al.* 2000). The removal of large specks is also more difficult at higher consistencies (Julien-Saint-Amand & Perrin 1991), and it has been suggested that at consistencies of 1% and above (Julien-Saint-Amand 1999) fibres tend to form networks and flock, causing ink aggregates to detach from the gas bubbles and thus lowering the flotation efficiency (Julien-Saint-Amand & Perrin 1991, McKinney 1999).

Deinking reject composition analyses have shown that not so many fibres pass into the flotation froth reject (Carré *et al.* 2001, Hanecker 2007, Korpela 1996, Perrin & Julien-Saint-Amand 2006), but that the losses during flotation are mainly due to mineral fillers, the flotation tendency of which is a complex and poorly understood aspect of deinking. The filler content of a flotation froth reject can vary from about 58% to 70% (Carré *et al.* 2001, Hanecker 2007). It is thus clear that solid losses via the flotation froth reject could be most easily reduced if mineral fillers were to be recovered. In most cases this would mean accepting a final pulp with an increased filler content, but this would affect both the processability of the DIP (*e.g.* low retention, slow drainage, web breaks) and the quality of the end product, effects which might be negative (*e.g.* increased dusting tendency and lower bulk, strength and elasticity) or positive (increased light scattering ability, higher gloss) (Pöyry 1995). There have in fact been demands expressed recently for higher filler levels in papers in order to reduce costs (Simonson *et al.* 2012). Recycled filler should of course also meet certain quality requirements set for paper fillers. Primarily, its brightness should be sufficient and its abrasiveness should be low. Brightness is affected by impurities in the fillers, and Carré *et al.* (2001) have stated that recycled fines and fillers from flotation froth reject contain more ink than unfloated fillers and fines. Although it is not known at the current state of the art whether the ink is free or attached to fillers and fines. Ben *et al.* (2004) have proposed that despite the high amount of contaminants in the ultrafine fraction of DAF rejects, there is no significant difference in cleanliness between the fibres and fines in DAF rejects and those in a deinked pulp. The abrasiveness of fillers is affected by particle size and angularity, which can change during incineration of the sludge. This can be a disturbing factor mainly in the case when the filler is recovered from the ash (Moilanen *et al.* 2000).

Stickies remain one of the main problems in the manufacturing of quality paper from recycled fibres (Doshi 2008). They cause problems in the deinking process line, in the paper machine system and for the end user: they form deposits which can lead to web breaks, tears and holes in the paper machine, they can stick to the rolls and clog the wires, they detract from the cleanness of the end product and they can make two sheets of paper stick together (Pöyry 1995). Macro stickies are removed primarily by means of pressure screens, and considerable efforts have been made during the last few years to develop screens in this direction: basket slot sizes have decreased, for example, and new basket, rotor and housing types have been designed (Doshi *et al.* 2010, Heise 2000). Hot dispersion and post-flotation also form a common process module for supporting the removal of macro stickies (Heise 2000), whereas micro stickies can be removed very efficiently by flotation (Sarja 2007).

It is apparent that contaminants which cause problems in the paper machine and affect product quality have to be removed from the pulp, but there are also some fibres that are rejected due to their degradation to fines: it has been demonstrated, for example, that cellulose fibres resist the deinking process 3–7 times before they become too short (Blanco *et al.* 2004, Van Kessel 2002), *i.e.* fibres which have already undergone several recyclings tend to be lost in the reject streams even though they could still be suitable as natural filler for low-grade graphic paper (Grossmann 2007). Also, DIP processes do not selectively remove poor quality fibres but also reject some good quality fibres (Korpela 1996), which must therefore be regarded as losses (Hanecker 2007). It is thus likely that the material efficiency of a deinked pulp mill could be increased by fibre recovery, which would also be a way of minimising the quantity of residues.

Fibres in the screening and cleaning rejects represent more than half of the total rejected fibres (Korpela 1996, Moore 2011), and with this fact in mind, Perrin & Julien-Saint-Amand (2006) and Korpela (1996) have recommended an additional reject stage or optimisation of the existing reject stages for fibre recovery from the screening and cleaning steps. In practice, however, the number of stages is limited to two or three. Material recovery may be easier from some reject streams, such as screening and cleaning rejects, than from others, and therefore the recovery of material from the purest reject streams, near the production should be aimed at, not from the sludge, in which the rejects have already been mixed together. Fibres and other raw materials have also been recovered from primary sludges, however (Moss & Kovacs 1994, Ochoa de Alda 2008), and from waste water (Trutschler 1999) or even dried sludge (Kringstin & Sain 2005, 2006).

Some attempts have been made to recover fibres and other materials from reject streams: *e.g.* the recovery of valuable materials from deinking DAF rejects by column flotation has been studied by Ben *et al.* (2006). Similarly, Matzke *et al.* (1996) examined the recovery of fines and fillers from wash filtrate and froth reject by a joint filtrate flotation treatment method, while Fjallstrom *et al.* (2002) studied material recovery from wash filtrates. Froth washing to prevent material loss during flotation deinking has been demonstrated by DeLozier *et al.* (2005) and Robertson *et al.* (1998), and methods for separating ash and fibres from deinking effluents for reuse have been studied by Dorica & Simandl (1995). In addition, a large research project aimed at enhancing the competitiveness and sustainability of the paper recycling industry was carried out by European research organisations in 2004–2008 (Galland *et al.* 2007). Evidently, the recovery and internal utilisation of material will be the driving force for capital investments in improved deinking process efficiency in the next decade.

2.2 Pressure from legislation and consumers

With the growing environmental awareness, there is now a trend towards increased use of renewable resources and eco-friendly products. Consumers are more environmentally conscious than ever before and people want to recycle and to buy products made from recycled material (Miranda & Blanco 2009). As the population on our planet increases, people are becoming more aware of the pressure on resources and the need for improved efficiency. Increased sensitivity to environmental issues is shifting consumer behaviour towards ecologically conscious preferences, including greater acceptance of recycled products.

Wood is a renewable, biodegradable and sustainable material, which makes wood, pulp and paper suitable raw materials and products for the future (CEPI 2011, Kibat 2012). Papermaking is also one of the oldest recycling industries and one of the leading branches nowadays. Virgin fibre and recycled paper are in fact complementary parts of the same system and neither can manage without the other (CEPI 2011). Recovered paper is an economical source of fibre and recycled paper production avoids the dumping of paper in landfills, but the virgin fibre input into graphic paper products remains essential for the recycled fibre loop.

The European paper and board industry is committed to achieving higher levels of recycling – so that the target paper recycling rate of 66% by 2010 has already been met, and a new target, 70%, has been set for 2015 (ERPC 2011). The promotion of recycling has many environmental benefits, but increased collection rates have also had a major impact on the quality of the recovered paper (Miranda *et al.* 2011). Producing high-quality end products from low-quality raw materials can be costly, due to increased handling, cleaning and waste disposal costs (Hudson 2011, Moore 2011).

The disposal of waste in landfills has become very expensive, and it may even be banned completely in several European countries within the next few years due to tightening of the EU Landfill Directive (99/31/EC). The basis of the European waste legislation is the framework directive on waste (2008/98/EC), one of the main elements in which is the “waste hierarchy”, which has been the guiding principle in European waste management policy. The foremost task of the hierarchy is to prevent or reduce the generation of waste as far as is physically possible, while its secondary task covers problems of reuse, recycling and recovery of material, to be followed in the next step by energy recovery. As a final option, waste should be safely disposed of if none of the previous steps succeed in eliminating it. At the top end of the waste management strategies would thus be zero waste, in which all by-products are either reused or returned cleanly to the soil (CEPI 2011).

The European pulp and paper industry produces about 11 million tons of waste (rejects, sludge and ash) each year, of which 70% are generated from recycled paper production (Monte *et al.* 2009). The utilisation or disposal of wastes and residues has become a crucial issue in the recycled paper industry, residual management being a significant part of the costs of producing recycled paper products (Engel & Moore 1998). The production of DIP may even be economically uninteresting due to the rather low yield and high disposal costs, since Moore (2011) has calculated that a contaminant level of only 2%–3% can lead to yearly losses of more than $700 000 (equal to about 550 000 €) when more than 500 tons of recovered paper are used per day. The loss of valuable material in the rejects evidently becomes economically unacceptable when a yield increase of only a couple of percentage units could lead to savings of millions. Mills are therefore attempting to recover as much usable material from their raw furnish as possible in order to minimise the amount of waste to be disposed of. Focusing on the minimisation of waste at source is invariably the most cost-effective approach, so that Abou-Elela *et al.* (2008) state, for example, that the implementation of pollution prevention measures such as fibre recovery proved to be highly cost-effective compared with end-of-pipe treatments in a board paper mill.

The costs and environmental impacts of solid waste disposal for deinking mills is of growing concern, and mills have sought options for turning residuals into resources by finding alternative ways of utilising or disposing of them (see Chapter 2.1.2). These methods are mainly external uses, however, which entails a risk of over-dependence on external users who can determine prices among themselves. In addition, whether the reuse of sludge or ash produced by incineration is feasible depends on the market demand for these materials (IPPC 2001). For these reasons, waste minimisation through internal use, for instance, will become very important, and thus the future emphasis will be more on improved efficiency.

3 The problem and the aims of the research

3.1 The problem

The loss of valuable material in the form of rejects threatens the economic viability of DIP production, since disposal of the great number of residues in landfills is economically undesirable and environmentally unacceptable. Thus new ways of managing the waste problem through improved efficiency should be developed in order to ensure that paper production from recycled sources remains both economically and environmentally feasible.

3.2 Aims of this research

The aim of this thesis was to study means of improving the material efficiency of a deinked pulp mill without excessively detracting from end product quality or the performance of the combined sludge dewatering stage. The purposes of the experiments were to assess the utilisation potential of deinked pulp mill reject streams, to develop practices for their utilisation and to consider the overall picture of resource efficiency improvement in the deinked pulp mill. The objectives were thus to acquire an understanding of:

1. the utilisation potential of reject streams,
2. the recovery of long fibres from fine screening rejects,
3. the recovery of filler and fine materials from the flotation froth rejects, and
4. the limitations affecting the relationship between sludge dewatering properties and material efficiency improvement in a deinked pulp mill.

One further aim of this thesis was to encourage mill personnel, researchers and decision makers to use of the new understanding of these matters when designing rational and environmentally sustainable strategies for improving the resource efficiency and sustainability of deinked pulp mills.

3.3 Structure of the research

The first part of this thesis discusses the utilisation potential of waste streams. Rejects from a deinked pulp mill have been studied earlier *e.g.* by Carré *et al.* (2001), Fjallström *et al.* (2002), Hanecker (2007), Korpela (1996), Kyllönen *et al.* (2010) and Matzke *et al.* (1996), so that the potential of certain reject streams is already known. Nevertheless, a totally new, systematic and illustrative analytical procedure for determining the utilization potential of rejects and sludges was developed here in Paper I.

The loss of valuable long fibres in fine screening rejects is a well known, and for that reason the second part of the research involved observation of the recovery of these fibres. There have been no earlier studies of this topic, so that Paper II provides proof of concept for recovering long fibres from fine screening rejects.

The use of depressants in DIP production is quite a new area of research although some existing studies can be found (Heimonen & Stenius 1997, Zeno *et al.* 2010). A new way of using depressants in a DIP mill was considered in Paper III when examining their use in the tertiary flotation stage. This study can also be regarded as demonstrating the concept of filler recovery from the flotation froth reject.

The last part of the research discusses the correlation between fibre content and the dewatering properties of the sludge. It is known in the industry that a higher fibre content in the sludge to be dewatered improves the dewatering process, but there have only been a few studies devoted to this issue (Kyllönen *et al.* 2010, Lo & Mahmood 2002), and they do not include experiments with sludges that differ in fibre contents. Thus Paper IV, which includes experiments with sludges of decreasing fibre contents, provides valuable information on the effect of fibre content on sludge dewatering.

4 Materials and experimental environments

4.1 Samples

The research deals with deinked pulp made from mixtures of old newsprint (ONP) and magazines (OMG) (EN 643 paper grade 1.11) and used for the production of newspaper, SC or other printing and writing papers. The sludge and reject samples were obtained from a European deinking mill using a 60/40 ratio of ONP to OMG as the raw material, which represents a common situation in DIP mills. The samples included the fine pre-screening reject, the fine pre-screening accept, the fine screening reject, and the flotation froth reject from the deinking mill and the primary sludge and activated sludge from a waste water treatment plant. The experiments were carried out without delay and the samples were stored at 4 °C in the meantime. The characteristics of the samples are shown in Table 2.

Three types of pressure screening process were used at the mill: coarse screening, fine pre-screening and fine screening. Coarse screening was performed at the beginning of the deinking process with 2 mm holes to remove mainly the staples, while the fine pre-screening, which was used as a sampling point in the work reported in Paper II, was performed with 0.20 mm slots prior to pre-flotation. The sample collected was from the final reject after third-stage screening. Fine screening was performed with 0.15 mm slots after the pre-flotation. The reject sample obtained from the fine screening was used in experiments reported in Papers I and IV. The flotation froth reject sample was a mixture of the pre-flotation and post-flotation secondary-stage rejects.

Table 2. Characteristics of the samples.

Sample	pH[1]	Dry matter content[2] [%]	Ash content[3] [%]	Fibre content[4] [%]
Fine pre-screening reject	8.3	1.0	12.9	78.6[5]
Fine pre-screening accept	7.3	2.3	23.6	53.6[5]
Fine screening reject	8.1	1.1	27.5	59.5
Flotation froth reject	7.9	3.6	70.2	1.6
Primary sludge	8.1	3.1	28.8	26.4
Activated sludge	7.7	1.5	40.3	na

[1]SFS 3021
[2]SFS-EN 20638
[3]ISO 1762
[4]Determined by the method described in Paper I.
[5]Tappi T 275 sp-02 (Somerville-type equipment), except that a 150-mesh wire screen was used.

4.2 Laboratory analyses

Measurement of the oven-dry (od) sample mass sets the basis for the experiment, as it is needed in several of the analyses and calculations. This oven-dry mass was calculated from the consistency (*cs*) or dry matter content (*dmc*). Consistency (SFS-EN ISO 4119) is based on filtering and drying the retained sample, and dry matter content (SFS-EN 20638) on the evaporation of water from the suspension. Consistency was measured in Paper II and dry matter content in all the other instances. The samples for the ash content analyses were incinerated at 525 °C according to ISO 1762.

Fibre content was determined by the method developed in Paper I, as described in more detail in section 4.4. For Paper II the fibre content was analysed in terms of solids retained on a 150-mesh wire screen having a nominal aperture of 105 µm in a Somerville screening apparatus (TAPPI T 275).

Low-grammage sheets (Papers I–II) or opaque pads (Paper III) were prepared for the optical measurements. The low-grammage sheets (35–40 g/m^2) were prepared by filtering the suspension onto a filter paper with a porosity of 1–2 µm according to method described by Körkkö (2012), and the opaque pads by filtering a sufficient amount of sample to obtain a basic weight of 225 g/m^2 onto a membrane filter (Sartorius Ø 50 mm, pores Ø 0.45 µm) to form an opaque pad (modified INGEDE Method 1). $ERIC_{700}$ values were assessed from the reflectivity and light scattering coefficients (ISO 22754 and TAPPI T 567), except that the reflectance was recorded at 700 nm, the use of which is not included in the aforementioned standards but is common practice (Körkkö 2012). Brightness was measured from a stack of test media in accordance with SFS-ISO 2470.

Dirt specks were analysed from hyperwashed pulp. Five sheets with a grammage of 70 g/m^2 were prepared and the area of black particles was measured against the total area using a DOMAS image analysis system available commercially from PTS with an Epson 1680 Pro scanner. A constant threshold setting (182) that was 15% lower than the mean grey level of the darkest sample was employed throughout the analysis. The dirt speck results are presented as mean values determined from five sheets. Confidence intervals for a 95% confidence level are presented, assuming normally distributed data.

For the macro stickies analysis, 10 g of pulp (od) was screened with a Somerville screen using a 100 µm slotted aperture. The retained sample was filtered on filter paper, dyed with water-based ink and the hydrophobic particles were highlighted with aluminium oxide powder. The area of white particles on the prepared sheet was then analyzed and converted to the area of macro stickies with respect to the amount of pulp. Three parallel sheets were analysed for all the cases reported as means and with confidence intervals. (INGEDE Method 4)

4.3 Calculations

The term mass reject rate is used to describe the amount of matter removed from the sample by flotation, expressed as a proportion of the flotation feed. This was calculated as follows:

$$RR_{\mathrm{m}} = \frac{w_{\mathrm{R}} \cdot dmc_{\mathrm{R}}}{w_{\mathrm{F}} \cdot dmc_{\mathrm{F}}}, \qquad (1)$$

where RR_{m} is the mass reject rate [%], w_i is the weight of the sample [kg], dmc_i is the dry matter content of the sample [%], and the subscripts R and F stand for the reject and feed, respectively. Consistency was used in the mass reject rate calculations in Paper II instead of dry matter content. The component removal efficiency calculations for macro stickies and dirt specks were based on the component content of the feed and accept samples:

$$E_{\mathrm{r}} = \frac{x_{\mathrm{R}}}{x_{\mathrm{F}}} \cdot 100 = \left[1 - \left(1 - \frac{RR_{\mathrm{m}}}{100}\right) \cdot \frac{x_{\mathrm{A}}}{x_{\mathrm{F}}}\right] \cdot 100\,, \qquad (2)$$

where E_r is the removal efficiency of the component [%], x_i is its content in the sample, and the subscript A stands for accept (Hautala *et al.* 1999, Hautala *et al.* 2009).

4.4 Fractional analysis procedure (Paper I)

In order to increase the utilisation of the rejects generated at a DIP mill it is extremely important to identify their unknown composition. In an effort to evaluate the utilisation potential of sludges and rejects, a simple but nevertheless informative and illustrative analysis procedure was developed.

The procedure can be viewed as a simple flow diagram, as presented in Fig. 3, which also includes labelling of the different fractions. The first stage in the procedure is to remove any disturbing material (such as sand or other coarse material) from the sample which may interfere with the following stages in the analysis. In this procedure the heaviest fraction (coarse) was removed first with an elutriation underflow followed by filtration to remove the finest fraction (ultrafines). The remaining fibrous fraction was fractionated further with chromatographic separation into four sub-fractions: flakes, long fibres, short fibres and fines. The flakes fraction comprised large but light particles such as flakes and shivers, whereas long and short fibre fractions comprised mainly fibres. The fines fraction consisted of filler and other small particles. Lastly, the ultrafines fractions were composed of the fines possessing the smallest dimensions of all. The experimental environments for the fractional analysis procedure are described in the following sections.

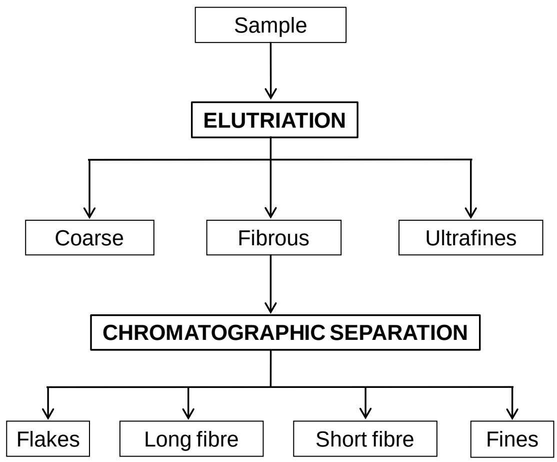

Fig. 3. Fractional analysis procedure for evaluating the utilisation potential of sludges. Modified from Paper I, published by permission of Keppler-Junius GmbH & Co. KG.

4.4.1 Elutriation

The setup used for elutriation is presented in Fig. 4. A total of 20 g of oven-dry sample was diluted to a consistency of 0.3% and fed constantly at 21.6 mL/s from the bottom of the elutriation cell. The velocity of the liquid decelerates when it reaches the upper parts of the cell resulting in the larger and heavier particles (coarse fraction), which cannot exit the cell, settling along the bottom. The remaining liquid was then passed through a 50 µm wire mesh which covered the overflow from the cell to filter the fibrous fraction from the overflow fraction. Finally, when the whole sample had passed through the cell, clean water was fed through until elutriation no longer occurred. The elutriation process took a total of 16 minutes to complete.

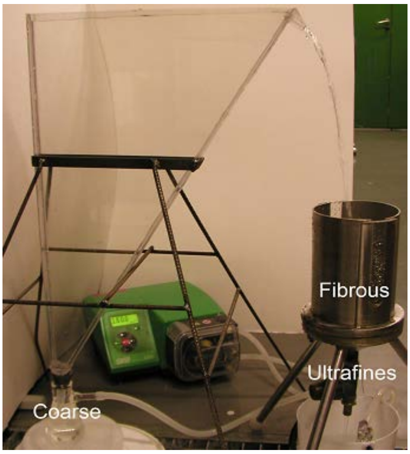

Fig. 4. The elutriation setup. Modified from Paper I, published by permission of Keppler-Junius GmbH & Co. KG.

4.4.2 Chromatographic separation

Chromatographic separation was performed using a tube flow fractionation method (Metso Fractionator) in order to divide the fibrous fraction obtained in the elutriation stage into four sub-fractions according to size (Laitinen 2011), the boundary values for which are quite similar to those achieved in the Bauer-McNett classification (R14, R28, R48 and R200 together with P200). For mass fraction determination, the fractions were filtered onto a membrane filter (Sartorius Ø 50 mm, pores Ø 0.45 µm), dried and weighed.

4.5 Long fibre recovery (Paper II)

The recovery of long fibres was studied by means of laboratory flotation experiments for the untreated fine pre-screening reject (fine pre-screening reject without dispersion) and dispersed (fine pre-screening reject after LC dispersion) fine pre-screening reject. In addition, the possibility of recycling dispersed fine pre-screening rejects directly back into the pre-flotation feed was considered. The experimental environments and the arrangement for the long fibre recovery experiment are described in the following sections.

4.5.1 LC dispersion

Dispersion was performed using a laboratory-scale high-frequency dispergator (ZRI-homogenizer, Haarla Oy, Tampere, Finland), which does not damage fibres (Suopajärvi *et al.* 2009). The device consists of a stator, in the form of a series of concentric rings that contain slots. Embedded between the stator rings is a rotor with a frequency of 300 Hz (18 000 rpm). The pulp was heated to 45 °C prior to dispersion, and its consistency was adjusted to 0.9%. The pulp was dispersed in 225 g (od) batches, which were fed into the centre of the stator and forced (at a pressure of 10 bars) to flow outwards through the concentric rings. The gap between the stator and the rotor during the operation of the dispergator was of the order of 1 mm. A manual valve was used to control the through-flow and the pressure, which was set to 1 bar.

4.5.2 Flotation

A Voith Delta25 batch laboratory flotation cell was used for these flotation experiments. The batch size was 21 litres, and the airflow was kept constant (7.4 L/min) for all the experiments. The samples were preheated to 45 °C. No additional chemicals were used because the samples already contained the flotation chemical residue, but the hardness was measured and adjusted to 18°dH with $CaCl_2$ (German hardness, equivalent to 128 mg Ca^{2+}/L). Each batch, and also the froth that had been removed, was weighed, and samples were taken before and after flotation for analysis.

4.5.3 Experimental arrangement

The long fibre recovery experiment was conducted by carrying out two series of flotations (Fig. 5): a separate flotation of the untreated or dispersed fine pre-screening reject (test series 1) and a mixed fine pre-screening reject and accept flotation (test series 2). The contaminant levels of the separate untreated and dispersed reject flotation experiments were compared with those of the fine pre-screening accept sample from the deinking mill which were used to indicate the reference level of the contaminants in the pulp. The fine pre-screening accept was floated to obtain reference values for the removal efficiencies of the contaminants during the deinking line flotation for comparison with the mixed flotation experiment. Since the composition of the fine pre-screening reject was markedly different from that of the accept (Table 2), the same fibre consistency was chosen for each flotation experiment rather than the same total consistency.

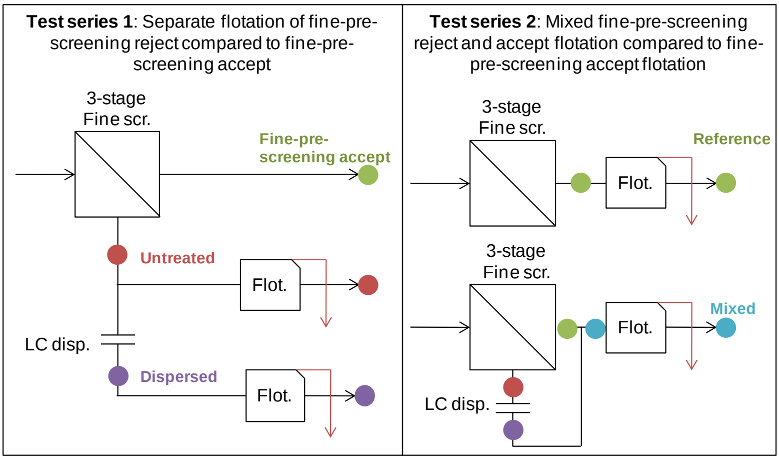

Fig. 5. Experimental arrangements for the long fibre recovery experiment. Paper II, published by permission of TAPPI.

The fine pre-screening reject sample from the deinking mill was used in the untreated flotation experiment. The flotation was performed at 45 °C and 0.8% total consistency, which corresponds to a fibre consistency of 0.63%. Flotation was performed to achieve hyperflotation (*i.e.*, all of the contaminants that floated were removed), which resulted in a reject rate of 18% by weight.

In the dispersed flotation experiment the fine pre-screening reject sample from the deinking mill was preheated to 45 °C and dispersed with a laboratory-scale high-frequency dispergator at a consistency of 0.9%. The total consistency was then adjusted to 0.8%, which corresponds to a 0.63% fibre consistency, with warm tap water to maintain the temperature level (45 °C). Since the goal of this section was to achieve the same mass reject rate as in the untreated flotation experiment; flotation was performed to achieve hyperflotation, which resulted in a reject rate of 20% by weight.

Reference values for the removal efficiencies of contaminants in the deinking line flotation were obtained by floating the fine pre-screening accept sample from the deinking mill. A total consistency of 1.2% was selected for the fine pre-screening accept flotation experiments, because this value is widely used as the flotation consistency for deinking processes (Schabel &Holik 2010, Schwarz & Kappen 2010, Spielbauer 1999). The total consistency of 1.2% resulted in a 0.64% fibre consistency for the fine pre-screening accept. The temperature during flotation was 45 °C, and the reject rate was limited to 14% by weight.

A mixture of the dispersed fine pre-screening reject (10%) and the untreated fine pre-screening accept (90%) was preheated to 45 °C and floated to determine whether the dispersed fine pre-screening reject could be fed back into the pre-flotation feed of the deinking process. To emphasize a possible difference in the removal of contaminants between the flotation arrangements, a ratio of 10/90 for the dispersed reject and fine pre-screening accept was chosen instead of a 1/99 ratio. Mixed flotation was performed at a consistency of 1.2%, which resulted in a 0.67% fibre consistency with a 14% reject rate by weight.

4.6 Fine materials recovery (Paper III)

A fine materials recovery experiment was conducted to determine whether recovery of fillers and fibre fines from the flotation froth reject is possible by refloating froth with or without the use of depressants. The experimental environments and arrangement for this fine materials recovery experiment are described in the following sections.

4.6.1 Flotation

A Voith Delta25 batch laboratory flotation cell was used for the flotation experiments. The batch size was 16 L, and the airflow was kept constant at 3.6 L/min. Each batch was preheated to 45 °C, and the consistency was the same as that of the original flotation froth reject (dry matter content 3.9%). The froth generated was continuously removed, weighed and analysed.

4.6.2 Experimental arrangement

Since the flotation froth reject contained residues of the flotation chemicals (fatty acids), 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) commonly used in the pulp and paper industry and had a medium chain length and charge density (-5.0 meq/g at pH 7), and the starch was a non-ionic starch (charge density -1.0 µeq/g at pH 7). The dosages used were 90 g/t, 122 g/t and 244 g/t (od) for the CMC and 244 g/t and 488 g/t for the starch. Both depressants were diluted to a 1% solution before addition to the flotation cell.

4.7 Sludge dewatering properties after material recovery (Paper IV)

The study of sludge dewatering properties after material recovery was aimed at determining the limitations on the relationship between the sludge dewatering properties and the improvements in material efficiency achieved at a deinked pulp mill by investigating the dewatering properties of sludge samples that contained variable amounts of fine screening and flotation froth rejects. The experimental environments and the arrangement for the sludge dewatering study are described in the following sections.

4.7.1 Sedimentation in a centrifuge

Analytical centrifugation (LUMiFuge, L. U. M. GmbH, Germany) was used to measure the compressibility of the sludges. Two samples from each experiment, each with a volume of 1.8 mL, were centrifuged at a speed of 3000 rpm (corresponding to a centrifugal force of 1300 g) for 10 minutes. The minimum value for the sample position (sediment height) was used as an indicator of compressibility.

4.7.2 Gravity filtration

After chemical conditioning (2 kg/t Fennopol K5060 polymer, Kemira Oyj, Finland), the sludge samples were dewatered in 3 litre batches for 10 minutes with a wire fabric (Metso Fabrics Inc., Finland) that had an air permeability of 150 m^3/m^2 min (p = 200 Pa). The mass of the accumulating filtrate was weighed during filtration, and the dry matter contents of the cake and the filtrate were determined after filtration. The cakes were collected and stored at 4 °C for dewatering in a filter press the following day.

4.7.3 Press filtration

The cakes obtained from the gravity filtration process were divided into batches and pressed with a laboratory press filter (Labox 30, Outotec Larox, Finland). The batch size was 150 g and three parallel pressings were conducted for each sludge sample. The pressing was conducted in the form of a one-sided filtration, using the same wire fabric as in the gravity filtration. The pressing experiments were conducted by first densifying the samples at a pressure of 4 bar for 90 seconds, followed by pressing them under a pressure of 12 bar for 30 seconds. The pressing performance was evaluated by determining the dry matter content of the cakes and the filtrates.

4.7.4 Experimental arrangement

Altogether 12 sludge samples were prepared by mixing (as oven dry basis) the samples of the fine screening reject, flotation froth reject, primary sludge and activated sludge from the mill in different combinations as shown in Table 3. The composition of the combined sludge from the mill was taken as a reference case (Experiment 1), the sample being prepared by mixing the fine screening reject (6.4 g), flotation froth reject (53.2 g), activated sludge (3.2 g) and primary sludge (37.2 g). The other samples were prepared by changing the percentages of the fine screening reject and/or flotation froth reject as follows: (1) in Experiments 2–6 fibre recovery was increased relative to the actual fine screening reject stream, *i.e.* the amount of fine screening reject in the prepared sludge samples was reduced by 0%, 20%, 40%, 60%, 80% and 100%, (2) in Experiments 7–9 the recovery of fine materials (such as fibre fines and mineral fillers) was increased relative to that in the flotation froth reject stream, *i.e.* the amount of flotation froth reject in the prepared sludge samples was reduced by 10%, 20% and 50%, and (3) in Experiments 10–12 the simultaneous recovery of fibres and fine materials were studied *i.e.* the amount of fine screening reject in the prepared sludge samples was reduced by 80% and the amount of flotation froth reject by 10%, 20% and 50%. The amount of activated and primary sludge taken remained the same in all the experiments, but the amounts of other two components were varied, thus the activated and primary sludge percentages altered as well. In Experiment 2, for example, only the amount of fine screening reject was reduced (by 20% compared to Experiment 1, *i.e.* from 6.4 g to 5.12 g), while the amounts of the other components remained constant. This meant that, the total amount of sample decreased from 100 g to 98.72 g, which affected the percentages of the all other components as well

No specific preservation method was used for the sludge samples since the experiments were conducted immediately *i.e.* within two days of sampling.

Table 3. Compositions of the sludges used in the sludge dewatering experiments. Paper IV, published by permission of Elsevier.

Experiment number	Material recovery from reject fractions [%]		Composition of the reject and sludge samples [%]				Contents of the combined sludge [%]	
	Fine screening reject	Flotation froth reject	Fine screening reject	Flotation froth reject	Activated sludge	Primary sludge	Fibre content*	Ash content**
1	0	0	6.4	53.2	3.2	37.2	14.5	49.0
2	20	0	5.2	53.9	3.2	37.7	13.9	49.2
3	40	0	3.9	54.6	3.3	38.2	13.3	50.7
4	60	0	2.7	55.3	3.3	38.7	12.7	49.8
5	80	0	1.3	56.1	3.4	39.2	12.1	51.6
6	100	0	0.0	56.8	3.4	39.8	11.4	52.1
7	0	10	6.7	50.6	3.4	39.3	15.2	48.5
8	0	20	7.1	47.6	3.6	41.7	16.0	47.8
9	0	50	8.7	36.2	4.3	50.7	19.1	42.4
10	80	10	1.4	53.4	3.6	41.6	12.7	50.2
11	80	20	1.5	50.5	3.8	44.2	13.4	48.5
12	80	50	1.9	38.9	4.7	54.5	16.1	43.6

*These values were calculated according to the fibre content of each reject fraction as presented in Table 2 and the percentage compositions of the reject fractions in the sludge sample.

**Measured according to standard ISO 1762.

5 Results

5.1 Utilisation potential of DIP mill reject streams (Paper I)

The fine screening reject consisted mainly of good quality fibres (60%) with high brightness (Fig. 6). Only the amount of macro stickies was such that it would hinder reuse. Thus stickies require some additional treatment such as separation, dispersion or inactivation before the fine screening reject can be recycled back into the process.

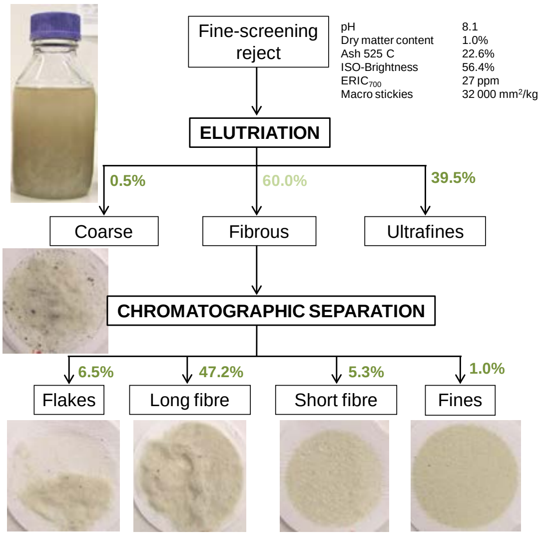

Fig. 6. Proportions of the various fractions in the fine screening reject.

The flotation froth reject comprised mainly fine-grained filler with some ink and fibre fines (Fig. 7), but its measured ash content was very high relative to that of the fine screening reject. The flotation froth reject is the largest stream of waste produced during the processing of recovered paper: amounting to about 15% of the dry weight of the paper produced. Thus there is a considerable potential for filler recovery.

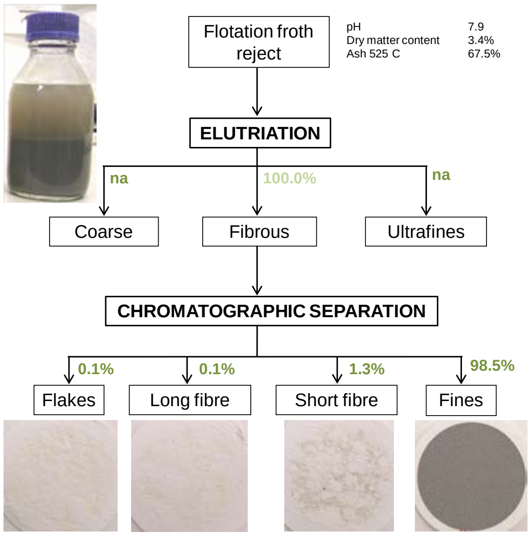

Fig. 7. Proportions of the various fractions in the flotation froth reject.

5.2 Recovery of material from DIP mill reject streams

5.2.1 Long fibre recovery (Paper II)

The amount of macro stickies and dirt specks is sufficient to hinder reuse of the good quality fibres from fine screening rejects. If successful fibre recovery from the fine screening rejects is required, these contaminants have to be processed.

The quantities of different-sized macro stickies in the fine pre-screening reject before and after LC dispersion and after flotation of the dispersed fine pre-screening reject are presented in Fig. 8. The quantities in the fine pre-screening accept are also presented to indicate the acceptable level of macro stickies in the pulp. Dispersion reduced the quantity of macro stickies with particle sizes of 2000 µm or larger, and slightly increased the quantity of 400–1000 µm stickies although the difference remained within the error bars. The total quantity of macro stickies was clearly less after flotation, than before, but was still 3 times higher than in the fine pre-screening accept.

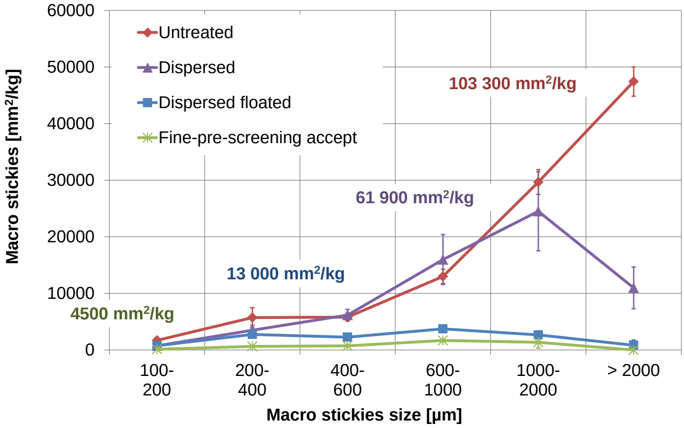

Fig. 8. Macro stickies in the fine pre-screening reject before and after dispersion and after flotation of the dispersed fine pre-screening reject. The numerical values refer to the total quantities of macro stickies in the samples. Paper II, published by permission of TAPPI.

The quantities of different-sized dirt specks in the fine pre-screening reject before and after LC dispersion and after flotation of the dispersed fine pre-screening reject are presented in Fig. 9. The quantities in the fine pre-screening accept are also presented to indicate the acceptable level of dirt specks in the pulp. The dirt specks, especially the large ones (>250 µm), were fragmented into smaller-sized particles by the dispersion process, allowing them to be removed so efficiently by flotation that the total quantity was even less than in the fine pre-screening accept.

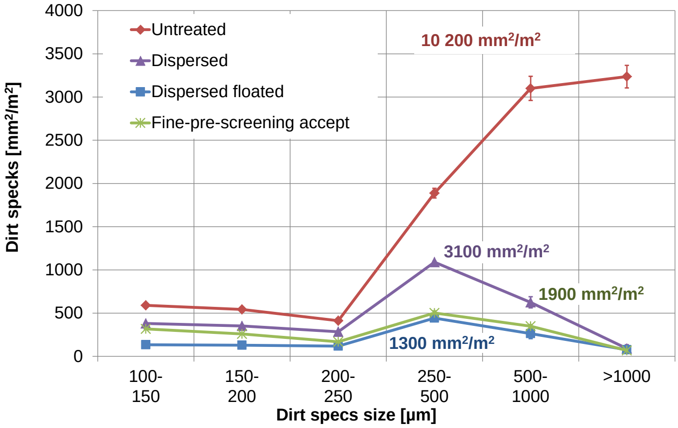

Fig. 9. Dirt specks in the fine pre-screening reject before and after dispersion and after flotation of the dispersed fine pre-screening reject. The numerical values refer to the total quantities of dirt specks in the samples. Paper II, published by permission of TAPPI.

The removal efficiencies for the different-sized macro stickies in the flotation experiments are presented in Fig. 10. The total removal efficiency (LC dispersion + flotation) and separate removal efficiencies for the LC dispersion and flotation stages (dashed lines) are presented for the LC-dispersed fine pre-screening reject. Negative removal efficiency was observed for the macro stickies ranging from 400–1000 µm over LC dispersion stage, indicating that the amount of stickies in this size range increased slightly as a result of the dispersion. The removal efficiency for the macro stickies during the flotation stage for the LC-dispersed fine pre-screening reject was greatest for the size class >200 µm, and the combined removal efficiency of the dispersion and flotation stages was high for all sizes of macro stickies. A higher removal efficiency was achieved for large stickies (>400 µm) than for smaller-sized stickies (<400 µm). The same trend can be observed for the reference and mixed samples, the removal efficiency of the large macro stickies being greatest, and the smaller stickies having lower removal efficiencies. No removal efficiency for macro stickies of size >2000 µm could be calculated for the reference flotation experiment due to their absence from these samples. The removal efficiency of the smallest-sized macro stickies (100–200 µm) in flotation was greatest in the case of the untreated fine pre-screening reject (nearly 40%), whereas the removal efficiency for the smallest-sized macro stickies in flotation was below 20% in the other samples. The removal efficiency for the macro stickies in flotation in the case of the untreated fine pre-screening reject varied between 20% and 50%.

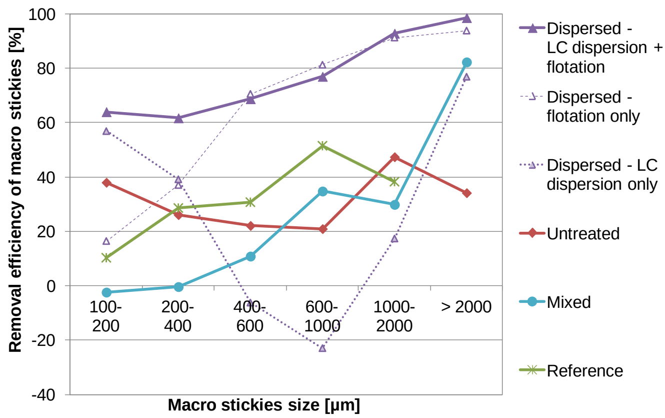

Fig. 10. Removal efficiencies of macro stickies in the flotation experiments (Dispersed, Untreated, Mixed and Reference). Both the total removal efficiency (LC dispersion + flotation) and separate removal efficiencies for the LC dispersion and flotation stages (dashed lines) are presented for the Dispersed experiment. Paper II, published by permission of TAPPI.

The removal efficiencies for the different-sized dirt specks in the flotation experiments are presented in Fig. 11. The total removal efficiency (LC dispersion + flotation) and separate removal efficiencies for the dispersion and flotation stages (dashed lines) are presented for the LC-dispersed fine pre-screening reject. The removal efficiency for the largest dirt specks (>1000 µm) was lowest in the flotation of the LC-dispersed fine pre-screening reject, but the large dirt specks had already been removed in the dispersion stage, which effectively dispersed dirt specks of size >500 µm, so that the removal efficiency over the LC dispersion and flotation stages together was high. Thus the removal efficiency for dirt specks was greatest for the LC-dispersed fine pre-screening reject, followed by the reference, then the mixed sample and finally the untreated fine pre-screening reject, which had the lowest removal efficiency. The removal efficiency for dirt specks remained relatively constant in each flotation experiment. Only a slight variation in the removal efficiencies for the largest dirt specks could be observed between the flotation experiments.

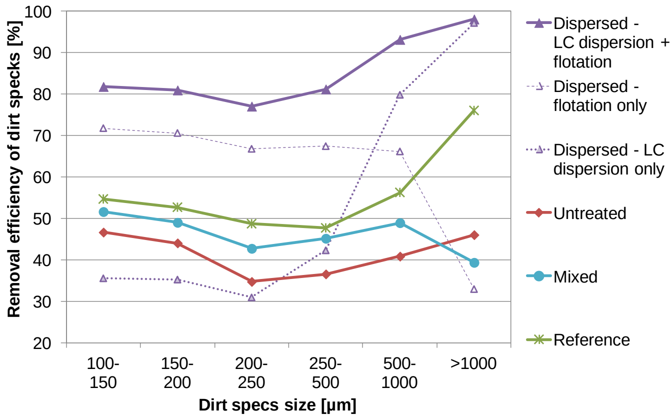

Fig. 11. Removal efficiencies for dirt specks in the flotation experiments (Dispersed, Untreated, Mixed and Reference). Both the total removal efficiency (LC dispersion + flotation) and separate removal efficiencies for the LC dispersion and flotation stages (dashed lines) are presented for the Dispersed experiment. Paper II, published by permission of TAPPI.

5.2.2 Fine materials recovery (Paper III)

The flotation froth reject, a mixture of pre-flotation and post-flotation secondary-stage rejects in a ratio of approximately 2:1, had a dry matter content of 3.9%, a pH of 8, an ISO brightness of 33%, and an ash content of 69% at 525 °C.

The brightness gains during the reflotation of this flotation froth reject using either CMC or starch as the depressant are presented in Fig. 12 and Fig. 13, respectively. Notable brightness gains were achieved only at the higher reject rates, *i.e.* the reject rate needed to be 60%–80% in order to attain an increase in brightness of 20 percentage points. A higher brightness gain for a given reject rate was achieved using CMC as the depressant than in the reference case, where flotation was performed without additional chemicals, but the difference became apparent only with reject rates greater than 50%. Also, too large a CMC dose negated the positive effect, so that with a CMC dose of 244 g/t 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 dose was high enough, 488 g/t in this case. The effect nevertheless remained lower than for CMC doses of 90 and 122 g/t. A starch dose of 244 g/t yielded no difference relative to the reference.

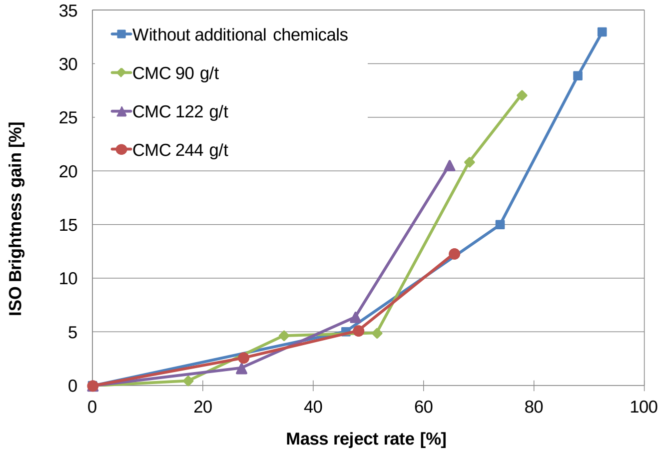

Fig. 12. Effect of CMC on brightness gain during reflotation of the flotation froth reject. Paper III, published by permission of TAPPI.

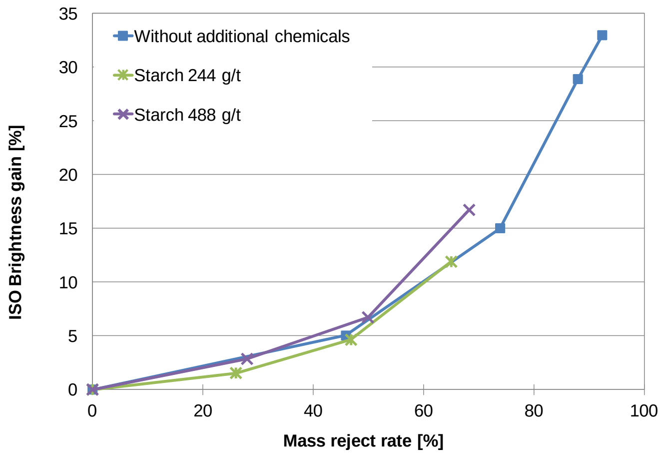

Fig. 13. Effect of starch on brightness gain during reflotation of the flotation froth reject. Paper III, published by permission of TAPPI.

5.3 Sludge dewatering properties after material recovery (Paper IV)

Fibre recovery from reject streams may have crucial effects on sludge dewatering properties. These experiments investigated the effects of material recovery on the combined sludge dewatering process.

The heights of the sediments after 10 minutes of centrifugation are presented as a function of the sludge fibre content in Fig. 14. As the fibre content decreased, the sediment height was also clearly observed to decrease, indicating an increase in the compressibility of the sludge. Compressibility of the sludges describes the pressing properties of the sludges. Highly compressible sludges such as activated sludge are known to be difficult to dewater (Qi *et al.* 2011). The sludge samples in Experiments 2, 7, 8 and 11 had compressibilities that were similar to those in the reference Experiment 1. The compressibilities of the sludge samples that had fibre contents of less than 13% were notably larger than that of the reference sample, indicating that these samples were more difficult to dewater. The limiting point of the increased sludge compressibility is indicated by a circle in Fig. 14 and the same limiting point was noted later in the pressing experiments, as shown in Fig. 15.

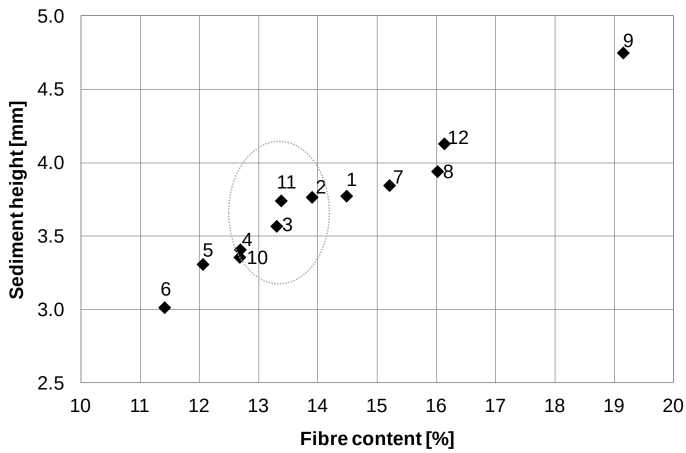

Fig. 14. Heights of the sediments after 10 minutes of rotation of the combined sludge samples at 1300g as a function of the sludge fibre content. Paper IV, published by permission of Elsevier.

The dry matter content of the cakes after pressing as a function of the sludge fibre content is presented in Fig. 15. The dry matter content of the cake clearly decreased with the fibre content. Experiments 2, 3, 7, 8, 11 and 12 had approximately the same dry matter content of the cake as did the reference Experiment 1. The dry matter content of the cakes with a fibre content of less than 13% was notably less than that of the reference cake sample, indicating that the dewatering of those samples was less effective than that of the reference sample. The limiting point of the dry matter content of the cake is indicated by a circle in Fig. 15. The same limiting point was noted earlier in the centrifugation results (Fig. 14).

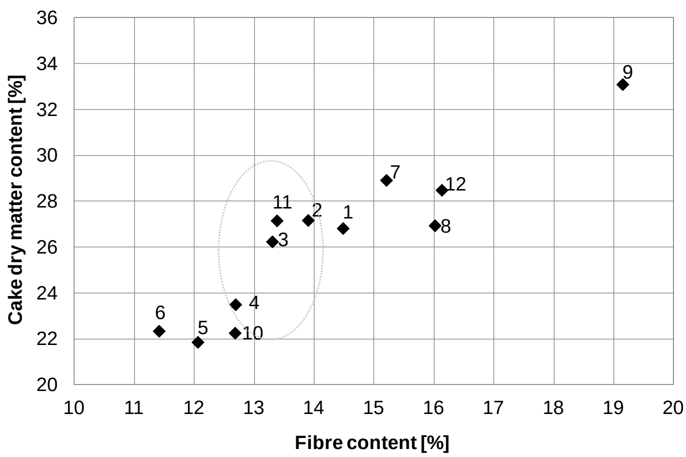

Fig. 15. Dry matter content of the cakes obtained after pressing the combined sludge samples as a function of the fibre content of the sludge. Paper IV, published by permission of Elsevier.

6 Discussion

Sustainability operations in DIP production start with maximising the yield. The unit operations are designed to separate the unwanted fraction (*i.e.* contaminants) from the fibre fraction. Unfortunately, current processes for removing the unwanted fraction also remove a portion of the valuable raw material. It is necessary to analyse the content of each reject stream in order to determine the potential for further material recovery, after which the second phase is to find methods for returning this material to the main process in such a way that its quality is acceptable. Besides quality aspects, it is also important to bear the whole picture in mind, for what seem to be small changes at one point in the process (*e.g.* fibre recovery) can have a significant impact elsewhere (*e.g.* in sludge dewatering).

6.1 Improving the material efficiency of DIP production

According to earlier studies (Carré *et al.* 2001, Korpela 1996) and experimental data (Paper I), two potential reject streams for further material recovery can be identified, namely, the fine screening reject and the flotation froth reject. Thus, further emphasis was placed here on recovering material from these reject streams.

6.1.1 Potential for long fibre recovery

The fine screening rejects are perhaps the most feasible streams for fibre recovery, as the fibres contained in them have proved to have high brightness and a low residual ink content together with great length, indicating that they possess an elevated reuse value (Paper I). The fine screening reject consisted mainly of good quality fibres, so that only the amount of macro stickies and dirt specks hindered its reuse. Thus these contaminants require some additional treatment before the fine screening reject can be recycled back into the process.

The results presented in Paper II indicate that it is possible to recover fibres from fine screening rejects by removing macro stickies and dirt specks with flotation. Since it has been suggested that flotation may remove small particles (50–250 μm) more efficiently than larger ones (Sarja 2007, Schabel & Holik 2010), a certain level of size reduction was considered for macro stickies and dirt specks. It was noticed, however, that it is at least equally important to increase the hydrophobicity of the contaminants by expanding their clean surface area by means of dispersion. The results obtained in Paper II indicate the important role of clean hydrophobic particle surfaces for selective separation during flotation, which is a less frequently reported effect of dispersion. The positive effect of dispersion on the efficiency of contaminant removal by flotation has previously been attributed to the size reduction (Gao *et al.* 2012) or shape change (Perrin & Julien-Saint-Amand 2006) imposed on the contaminants in the course of dispersion. It was observed in Paper II, however, that the removal efficiency for all size classes of stickies and dirt specks was better after dispersion, and that for large macro stickies it was even better than for smaller stickies. It was assumed that the surfaces of the large stickies were modified by dispersion more than were those of the smaller ones, and that the surface of the smaller stickies remained coated with debris, leaving them less active and unable to attach to the air bubbles.

In the experiments described in Paper II, dirt specks and macro stickies were removed from the fine screening reject very efficiently during flotation, and the removal efficiency might be even further improved by optimisation of the flotation. Heise *et al.* (2000) also used flotation for removing residual stickies, although they did so by treating the fine screening tailing accepts. Good contaminant removal efficiencies after dispersion were observed with a separate flotation unit operating at a low consistency (<1%) rather than returning the dispersed reject stream to pre-flotation (Paper II). With the method presented in Paper II, it is possible to recover 80% of the most valuable long fibres from the fine screening rejects, implying that the yield in a deinking mill could be increased by at least 1–1.5 percentage units.

6.1.2 Potential for filler and fine materials recovery

The flotation froth reject stream is the largest waste stream produced in a DIP mill, accounting for more than half of the total amount of reject (Hamm 2010), but it still contains plenty of reusable fillers and fine material. Thus the recovery of material from it in order to reduce the amount of waste is of great interest. The flotation froth reject was composed mainly of fine-grained filler with some ink and fibre fines (Paper I & III). The fibre content of flotation froth has also been found by Carré *et al.* (2001) to be generally low, so that fillers and fines in the flotation froth reject contained more ink than unfloated fillers and fines, leading to the conclusion that recovering fillers and fines from the flotation froth reject may detract from the optical properties of the pulp (Carré *et al.* 2001). In Paper III, however, a considerable brightness gain was reported in a tertiary flotation stage that enabled material to be recovered from the froth without damaging the optical properties of the pulp.

In the experiments reported in Paper III the flotation froth reject from a DIP mill was refloated. It might be possible to achieve a small increase in material efficiency in the flotation stages by adjusting the flotation performance in the primary or secondary stage, although existing flotation processes are already quite well optimised. Hence, our approach was to extend flotation by means of an additional, *i.e.*, tertiary, flotation stage.

An acceptable brightness gain was achieved by virtue of this tertiary flotation stage, and the use of depressants, especially CMC, further increased its selectivity, so that a greater amount of usable material could be recovered. To achieve a brightness gain of 25 percentage points, approximately 15% of the remaining material can be recovered by tertiary flotation, and this can be increased to approximately 25% by using CMC. A brightness gain of 25% is sufficient for newsprint production, although a higher brightness gain may be required for higher grades of graphic paper, so that the recovery rate would fall short by 5–10 percentage points. Recovery of 15–25% of the material in the flotation froth reject could result in an increase of about 2–4 percentage units in the yield.

6.2 Total resource efficiency of DIP production

According to the results presented in Papers II and III, the material efficiency of a DIP mill can be improved by recovering material from the reject streams, but this recovery affects the composition and dewatering properties of the combined sludge, so that these dewatering properties have to be considered when material is to be recovered from the reject streams.

Crucial influences can be brought to bear on the sludge dewatering process if valuable fibres are recovered solely from the fine screening rejects. In addition, if filler and fines material is recovered solely from the flotation froth reject, this will increase the filler and fines content of the pulp in the paper machine, and in some cases, runnability may not tolerate such an increase. If material is recovered from the fine screening reject and the flotation froth reject simultaneously, however, the filler content of the pulp will increase less, and the proportion of fibres in the combined sludge will remain high enough (Paper IV).

The results presented in Paper IV, clearly indicate that the dewatering properties of the combined sludge declined when more fibres were recovered from it, confirming earlier reports of an obvious effect of fibre content on the dewatering properties of sludge (Kyllönen *et al.* 2010, Lo and Mahmood 2002). The reason for the better dewatering properties exhibited by the sludge samples with a higher fibre content was assumed to be that the fillers in the sludge may become attached to the fibres by agglomeration (Gess 1998), thus preventing the small filler particles from migrating into the pores of the cake and causing cake blinding, which is one of the main reasons for difficulties in sludge dewatering (Qi *et al.* 2011). If the quantity of fibres is reduced too much, a saturation point is reached at which the fibres are unable to bind any more filler and the cake starts clogging. Another reason that has been suggested is the high cake compressibility brought about by low fibre content, which has a negative effect on sludge dewatering properties (Qi *et al.* 2011). The fibres improve the mechanical strength of the cake when under pressure and thus detract from its compressibility, as seen in an improved sediment height when the fibre content is higher. The same limiting point at a fibre content of less than 13% was noted in Paper IV in both the compressibility results and dry matter content of the cake when under pressure. Thus two separate analyses indicated that there is a limiting point for the fibre content beyond which the dewatering properties of the sludge deteriorate. This point probably varies from mill to mill. The limiting fibre content in the present set of experiments was 13%, at which it was observed that the sludge dewatering properties were notably poorer than those of the reference sludge sample in Experiment 1.

With regard to the recovery of materials in a DIP mill, the limiting fibre content was already reached in this case after recovering 40% of the fibres from the fine screening reject stream (Experiment 3), whereas it would be possible to recover at least 80% of those fibres (Paper II). To maximise the resource efficiency of such a mill, the deterioration in the sludge dewatering process should be taken into account by compensating for the recovery of fibres with a simultaneous recovery of fillers and fine materials from the flotation froth reject stream, which would allow the fibre content in the combined sludge to be controlled properly.

As a consequence, the optimal recovery proportions would be an 80% recovery of fibres from the fine screening reject stream and a 20% recovery of fillers and fine materials from the flotation froth reject stream (Experiment 11). Given these rates, it would be possible to increase the yield of the DIP mill by approximately 5 percentage points without causing any deterioration in the dewatering properties of the sludge or in the quality of the end product.

6.3 Process concepts

The above results are promising and show that material recovery from DIP reject streams is feasible, but since recovery could be performed in several ways, more thorough investigations would be needed in order to optimise the recovery stages. Some of the options for the recovery of the material from reject streams will be discussed in this section.

A simple example of a recovery process is presented in Fig. 16, which describes the recovery of fine materials from the flotation froth reject stream by tertiary flotation, and that of fibres from the fine screening reject stream, similarly by a separate flotation after the dispersion of hydrophobic contaminants. Although the reject streams are treated here in isolation, optimisation of the reject treatment parameters would minimise other parameters such as the process investment costs, energy consumption and land area requirements.

One option could be combined treatment of the reject streams for material recovery, with combined flotation and even combined dispersion prior to flotation. Dispersion could also be beneficial for the flotation froth reject, to detach ink from the fillers and fines. Carré *et al.* (1997) suggest the use of a kneader-type disperser for ink detachment from fillers and fines instead of the high-speed disperser, used here to disperse the contaminants in fine screening rejects. Thus optimisation of the dispersion devices would be needed to find the optimal devices one for each reject stream. Separate dispersion and flotation stages for both rejects are of course one alternative, although in this case additional devices would be needed, which would increase the investment and operation costs. However, since the fine screening reject stream only represents 2% of the total stock flow, the capacity of the dispersion and flotation devices required for that stream would be much smaller than in the main line. In the case of separate reject treatments, the use of column flotation for treating the flotation froth reject could be considered in order to minimise the land area needed and maximise the fibre yield and ash selectivity (Ben *et al.* 2006, Chaiarrekij *et al.* 2000, Dessureault *et al.* 1995, Dessureault *et al.* 1998, Vashisth *et al.* 2011).

Recirculation of the dispersed fine screening rejects back into the pre-flotation feed would be appealing from an industrial viewpoint, but this was found to be unworkable due to poor contaminant removal in mixed flotation

(Paper II), evidently brought about by the high consistency during mixed flotation, whereas low fibre consistency was seen preferable for fine screening reject flotation. Lower fibre consistency for reject flotation could be also achieved by combining the fine screening reject with the flotation froth reject, which is a further argument to support combined reject treatment.

Low fibre consistency for fine screening reject flotation could be also achieved if there were a fractionation stage in the main line of the deinking mill and the fine screening reject is circulated back into the short fibre flotation after dispersion. Fractionation of the deinked pulp and the separate treatment of the fractions have been discussed lately as the ultimate process for upgrading the quality of deinked pulp (Aregger 2008, Eul *et al.* 1990, Mäkinen 2008, Mäkinen *et al.* 2010). In this case the fibre consistency in short fibre flotation may be low enough to remove macro stickies and dirt specks. Also, the use of CMC in the short fibre flotation might be reasonable way of improving the flotation efficiency, so that the tertiary flotation stage for the flotation froth would no longer be necessary.

Altogether, there are many interesting options for recovering material from reject streams, including separate or combined flotation and dispersion stages, fractionation or the use of additives such as CMC in the flotation stages. And of course, the effects of material recovery on sludge dewatering have to be kept in mind. Thus, the whole economic feasibility of the system will have to be carefully considered.

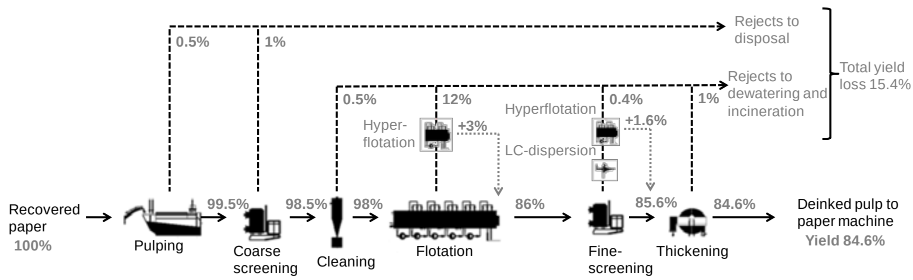

Fig. 16. A deinked pulp line after the insertion of material recovery from the reject streams. Modified from Paper IV, published by permission of Elsevier.

7 Conclusions

The results presented in this thesis provide a means for improving the material efficiency of a deinked pulp mill by recovering filler, fine materials and fibres from the reject streams. The method preserves the performance of the combined sludge dewatering stage while at the same time avoiding any excessive deterioration in end product quality. The simultaneous recovery of fibres from the fine screening rejects and fine materials from the flotation froth reject would make it possible to improve the material efficiency of a deinked pulp mill by a total of around 5 percentage units.

Valuable long fibres are fairly easy to recover from fine screening rejects as it is possible with low consistency dispersion and separate flotation to purify the fine screening rejects to a level that enables reuse of the long fibre fraction without detracting from the quality of the end product. 80% of the most valuable long fibres can be recovered from the fine-screening rejects, implying at least a 1–1.5 percentage units increase in the material efficiency of the deinking mill.

Approximately 15% recovery of fine materials and filler possessing adequate brightness for use in newsprint production is possible by refloating the flotation froth reject in a tertiary stage, and increased recovery is even possible using CMC as a depressant in a reflotation stage. If the sludge dewatering properties are also taken into account, the optimal proportion would be an 80% recovery of fibres from the fine screening reject stream and a 20% recovery of fillers and fine materials from the flotation froth reject stream.

The present findings indicate that substantial improvements in deinked pulp mill resource efficiency can be achieved by recovering material from reject streams. The valuable new information provided in this thesis should encourage the design of totally new, environmentally sustainable and cost-efficient deinking strategies.

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