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Mika Körkkö

ON THE ANALYSIS OF INK CONTENT IN RECYCLED PULPS

UNIVERSITY OF OULU GRADUATE SCHOOL;
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DEPARTMENT OF PROCESS AND ENVIRONMENTAL ENGINEERING

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Abstract

The amount of printing ink in a pulp suspension produced from recovered paper and its impact on overall brightness is commonly estimated from the reflectance-based ink content measured at a wavelength of 700 nm or 950 nm. The method uses a light scattering coefficient that can be measured from a slightly translucent test medium, i.e. of an opacity less than 97%. This is the case with machine-made papers in most instances. Alternatively, suitable opacity can be achieved by preparing a standard low-grammage sheet on a wire screen, but this results in poor retention of fibre fines, mineral fillers and printing inks, which is especially detrimental to ink measurement when the pulp suspension contains substantial amounts of printing inks. Hence opaque pads are often prepared on filter paper to achieve high retention. Unfortunately their high opacity prevents measurement of the light scattering coefficient, and thus a constant coefficient must be used for the determination of ink content. The aim of this thesis was to clarify the effects of retention and fine material changes on the light scattering coefficient in ink content measurement.

The results showed that the light scattering properties of pulp in the wavelength region used for ink content analysis do not remain constant when the fine material content varies. The grade of the recovered paper, hyperwashing and flotation alter the fine material content and thus affect the light scattering. Printing ink also affects light scattering, but its practical impact is smaller than that of fibre fines and mineral fillers. The light scattering coefficient used for each ink content measurement needs to be representative, otherwise a systematic bias in ink content measurements may result from changes in the nature of the fine material and in its content. It is recommended that the light scattering coefficient should be measured in order to avoid this. The measurement should preferably be performed from a low-grammage sheet prepared on filter paper, as this ensures high retention and a measured value that represents better the initial state of the pulp suspension.

Keywords: deinking, effective residual ink concentration, ERIC, fibre fines, fine material, light scattering, mineral filler, recovered paper, recycling, residual ink

Körkkö, Mika, Jäännösmusteen mittaus kierrätysmassoista.
Oulun yliopiston tutkijakoulu; Oulun yliopisto, Teknillinen tiedekunta, Prosessi- ja ympäristötekniikan osasto, PL 4300, 90014 Oulun yliopisto
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Tiivistelmä

Mustepitoisuusmittaus perustuu hajaheijastukseen joko 700 nm tai 950 nm aallonpituudella ja sitä käytetään usein arvioitaessa keräyspaperista valmistetun massasuspension soveltuvuutta painopaperien valmistukseen ja painomusteen vaikutusta massan vaaleuteen. Mustepitoisuuden mittauksessa käytetään valonsirontakerrointa, joka voidaan mitata hieman läpikuultavasta näytteestä eli näytteen opasiteetin on oltava pienempi kuin 97 %. Tämä opasiteettiraja toteutuu useimmiten paperikoneella tehdyille painotuotteille. Riittävän alhainen opasiteetti saavutetaan myös valmistamalla standardin mukainen matalaneliömassainen arkki viiralle, mutta tämä johtaa kuitumaisten hienoaineiden, mineraalisten täyteaineiden ja painomusteiden alhaiseen retentioon. Matala retentio on erityisen haitallinen piirre mustemittauksen kannalta massoilla, jotka sisältävät huomattavia määriä painomusteita. Siten usein valmistetaan läpikuultamattomia arkkeja suodatinpaperin päälle, joiden retentio on korkea. Korkeasta opasiteetista johtuen näistä arkeista ei voida määrittää valonsirontakerrointa, jolloin mustepitoisuuden määritys perustuu vakiokertoimeen. Tämän väitöskirjan tavoitteena oli selvittää retention ja hienoaineiden muutoksien vaikutuksia valonsirontakertoimeen ja mustemittaustulokseen.

Tutkimuksen tulokset osoittivat että valonsirontakerroin, joka mitataan mustepitoisuuden yhteydessä, ei pysy vakiona hienoainepitoisuuden muuttuessa. Hienoainepitoisuuteen ja siten valonsirontakertoimeen vaikuttavat keräyspaperin laji, hyperpesu ja vaahdotus. Myös painomuste vaikuttaa valonsirontakertoimeen, mutta käytännössä vaikutuksen suurusluokka on pienempi kuin hieno- ja täyteaineilla. Mustepitoisuuden määrityksessä käytetyn valonsirontakertoimen on oltava edustava, muutoin arvot voivat olla systemaattisesti virheellisiä hienoainemäärän tai laadun muuttuessa. Virheen välttämiseksi olisi suositeltavaa määrittää valonsirontakerroin mustepitoisuusanalyysin yhteydessä. Tämä olisi mahdollista tehdä suodatinpaperin päälle valmistetusta matalaneliömassaisesta arkista, jolloin saavutetaan korkea retentio ja näin mitattu arvo edustaa paremmin massasuspension alkuperäistä tilaa.

Asiasanat: ERIC, hienoaine, keräyspaperi, kierrätys, kuitumainen hienoaine, siistaus, täyteaine, valonsironta

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Oulu, June 2012 Mika Körkkö

Abbreviations

APPITA	Australasian Pulp and Paper Industry Technical Association
CIE	International Commission on Illumination (Commission Internationale de l'Eclairage)
CIE LAB	CIE 1976 colour space system: L*, a* and b* coordinates
CPPA	Canadian Pulp & Paper Association, nowadays known as PAPTAC
CTP	Centre Technique du Papier
EN	European Norm
GCC	Ground calcium carbonate
HW	Hyperwashed
HWK	Hardwood kraft pulp
INGEDE	International Association of the Deinking Industry (Internationale Forschungsgemeinschaft Deinking-Technik)
ISO	International Organization for Standardization
L&W	Lorentzen & Wettre, a spectrophotometer supplier
LWC	Lightweight coated paper
od	Oven dry
ONP	Old newsprint
OMG	Old magazines
PAPTAC	Pulp and Paper Technical Association of Canada
PCC	Precipitated calcium carbonate
PTS	Paper Technology Specialists
SC	Supercalendered paper
SFS	Finnish Standards Association
SWK	Softwood kraft pulp
TAPPI	Technical Association of the Pulp and Paper Industry
TMP	Thermomechanical pulp

cs	Consistency [%]
DEM_f	Deinkability factor based on brightness or luminosity [%]
DEM_{LAB}	Deinkability factor based on CIELAB colour system [%]
dmc	Dry matter content [%]
E_r	Removal efficiency [%]
ERIC	Effective residual ink concentration, ink content [ppm]
$ERIC_{700}$	Effective residual ink concentration at 700 nm [ppm]
$ERIC_{950}$	Effective residual ink concentration at 950 nm [ppm]

IE	Ink elimination [%]
k	Light absorption coefficient [m^2/kg]
$\dot{m}$	Mass flow rate [kg/min]
p	Student's t-test distribution
Q_K	Karnis's selectivity index
Q_N	Nelson's selectivity index
$\dot{Q}_V$	Volumetric flow rate [L/min]
r	TAPPI repeatability
R_0	Single-sheet reflectance factor
R_∞	Intrinsic reflectance factor, reflectivity
RI	Residual ink, measurement of ink content [ppm]
$RI_{L\&W}$	Residual ink, a special procedure issued by Lorentzen & Wettre [ppm]
RR_m	Mass reject ratio [%]
s	Light scattering coefficient [m^2/kg]
std	Standard deviation
w	Grammage of the paper sheet [g/m^2]
x	Content of component [%]
$\dot{x}$	Mass flow rate of component [kg/min]
X	Tristimulus factor red
Y	Tristimulus factor green, intrinsic luminance factor, luminosity
Z	Tristimulus factor blue
$\%r$	TAPPI repeatability ratio [%]

List of original publications

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

I Körkkö M, Laitinen O, Vahlroos S, Ämmälä A & Niinimäki J (2008) Components removal in flotation deinking. Prog Pap Recycl 17(4): 15–22.

II Körkkö M, Laitinen O, Haapala A, Ämmälä A & Niinimäki J (2011) Scattering properties of recycled pulp at the near infrared region and its effect on the determination of residual ink. TAPPI J 10(6): 17–22.

III Körkkö M, Haapala A, Liimatainen H, Ämmälä A & Niinimäki J (2011) Challenges in residual ink measurement – Effect of fibre fines and fillers. Appita 64(1): 71–75.

IV Körkkö M, Haapala A, Mäkinen L, Ämmälä A & Niinimäki J (2011) Comparison of test medium preparation methods for residual ink analysis. TAPPI J 10(10): 7–14.

All the listed publications were written by the author of this thesis, whose main responsibilities were the experimental design, data analysis and reporting of the results.

Contents

1 Introduction

1.1 Background

Paper is one of the everyday commodities that it would be very difficult imagine the world without. In the past, graphic paper manufacturing was based on virgin fibres and minerals, but the importance of recovered papers has been constantly increasing during the last four decades. In fact, the volume of recovered paper surpassed the use of virgin raw materials a few years ago (Ervasti 2010).

In the production of graphic papers from recovered paper, the impurities that detract from the quality of the product need to be removed in order to achieve a clean pulp with sufficiently high optical properties (Schabel 2010). The recovered paper stream contains all the materials that became attached to the paper during its production and printing, *e.g.* fibres, fibre fines, mineral fillers, printing ink and adhesives. In addition, a certain amount of extraneous matter, *e.g.* sand, glass, metal and plastic originating from recovered paper collection, handling and storing are also present in the recovered paper stream. These impurities are removed in the deinking process, in which the first unit operation is pulping. This causes the fibres to disintegrate, detaches impurities from the recovered paper and removes large-sized foreign bodies. Smaller-sized impurities remaining in the pulp suspension are removed in the subsequent screening, cleaning, flotation and washing operations. (Schabel 2010). Flotation causes the highest material losses in the deinking line producing printing and writing papers, while in the case of tissue production the highest losses are the result of pulp washing (Hamm 2010).

Printing ink has the highest impact on the optical properties of pulp produced from recovered papers (Carré *et al.* 2010, McKinney 1998). This is removed during flotation (and washing), but only free ink particles can be removed without sacrificing the yield. The optical properties of pulp can also be improved by bleaching the fibre material a yellowish colour and this is normally done in the production of high grades. The fact that the bleaching response is higher for deinked than non-deinked pulps (Rangamannar 2003) highlights the importance of printing ink removal.

Determination of the ink content of a pulp suspension is based on reflectance measurements in a wavelength region higher than 650 nm, where the yellowish colour of the fibre material has a negligible impact on light absorption (Jordan & Popson 1994). By this means the reflectance-based ink content is unaffected by

the changes in the colour of the fibre material induced by alkali darkening or bleaching. The ISO 22754 and TAPPI T 567 standards specify the use of a wavelength of 950 nm, yielding a result that is referred as the Effective Residual Ink Concentration, $ERIC_{950}$ or simply *ERIC*. Since an extended-range spectrophotometer is needed for measurements in this near-infrared region (Villforth *et al.* 2010), a modified version of *ERIC* which can be measured using spectrophotometers of a kind generally to be found in paper industry laboratories has become widely accepted. This modified version uses reflectance measurements at 700 nm and is known as $ERIC_{700}$, Residual Ink and *RI*. Ackermann & Göttsching (2002a) have shown that the light absorption coefficient measured at 700 nm responds linearly to black printing ink equally as well as the coefficient measured at 950 nm. The reading can be different, however, depending on the wavelength chosen, because cyan-coloured printing inks are also accounted for when reflectivity is measured at 700 nm (Ackermann & Göttsching 2002b, Jordan & O'Neill 1994). But even though there is a difference between the wavelengths, both methods are applicable for the analysis of ink content in practical instances.

Reflectance-based ink content can be determined by means of the Kubelka-Munk theory, by a method similar to the optical measurements applied in paper production that use blue or luminous light reflectance. The reflectivity measured indicates the sum effect of light scattering and absorption, whereas the light absorption coefficient indicates the magnitude of light absorption alone. In blue and luminous light reflectance the fine materials are known to alter the light scattering properties of the pulp, and therefore the absorption properties of the pulp can be estimated more accurately using the light absorption coefficient rather than reflectivity. (Pauler 2008, Vaarasalo 1999, Walmsley & Silveri 1999). The ISO 22754 and TAPPI T 567 standards lay down that the *ERIC* has to be determined in accordance with Kubelka-Munk theory, *i.e.* the light scattering coefficient has to be measured during the determination of ink content. For this purpose, the single-sheet opacity must remain below 97% (ISO 22754 and TAPPI T 567).

Determination of the ink content of machine-made papers requires no test medium preparation. The light scattering coefficient can be measured in most instances, as the opacity of machine-made papers remains below 97%. Test medium preparation is needed when the ink content of the pulp suspension is to be determined. The sheets are prepared on a wire screen (nominal aperture 105–125 µm) in accordance with ISO 22754 and TAPPI T 567, even though this

results in washing out the printing ink and other fine materials such as fibre fines and mineral fillers (Dorris 1999, Haynes 2000, Lévesque *et al.* 1995, Lévesque *et al.* 1998, McKinney 1988, Scott 1993). The loss of printing ink during sheet preparation is considered acceptable where deinked pulps are concerned, because then the ink content can be determined by means of the measured light scattering coefficient (ISO 22754 and TAPPI T 567). The loss of printing ink remains only slight, because the deinked pulps contain low amounts of printing ink.

The evaluation of ink content in deinking is also widely used for non-deinked and partially deinked pulps. In these instances the pulp suspensions contain substantial amounts of printing ink, which would be largely rinsed out during sheet preparation on a wire screen. For this reason, INGEDE Method 2 recommends the preparation of opaque pads on filter paper for improving the retention of printing ink (pad preparation is similar to that in ISO 3688 and TAPPI T 218). The preparing of opaque pads resolves the retention issue, but measurement of the light scattering coefficient is prevented due to the high opacity of the pads and the determination of ink content has to be based on a constant value for the light scattering coefficient. This procedure is widely used even when referring to ISO 22754 and TAPPI T 567, despite the fact that use of the measured light scattering coefficient is recommended in these standards. The change in the light scattering properties of pulp at 700 nm and 950 nm remains unclear. This thesis is therefore devoted to studying the effect of fine materials on determination of the light scattering coefficient, ink content and changes in fine material content in deinking applications.

1.2 The research problem

The following problems in reflectance-based ink content measurement were identified:

1. The majority of the losses in deinking arise from the washing and flotation stages. The particle size is known to affect removal in washing, but the removal tendencies of the various recycled pulp components during flotation are unclear. On the other hand, even the selective removal of some components would alter the pulp composition and thus affect the light scattering properties.
2. Changes in fine material content are known to affect the light scattering properties of pulp with blue and luminous light reflectance, but it has not

been shown whether such changes also have an effect at 700 nm, the wavelength at which ink content is determined.

3. Use of a constant light scattering coefficient enables the determination of ink content from opaque pads prepared on filter paper, for which a high retention of printing ink is achieved. It remains unclear, however, whether the use of a constant light scattering coefficient may bias the determination of ink content.
4. The high opacity of pads prevents measurement of the light scattering coefficient. This can still be measured from low-grammage sheets prepared on wire screen, but then the retention of printing ink is poor. Low-grammage sheets prepared on filter paper could take advantage of the aforementioned procedures by achieving a high retention of printing ink and having opacity, below 97%, thus enabling measurement of the light scattering coefficient during the determination of ink content. The applicability of such a method has not been discussed or tested in the literature, however.

1.3 Aim and hypotheses

The aim of this thesis was to obtain new information and understanding on the issues affecting reflectance-based ink content measurement. The following hypotheses were formulated and tested in order to solve the above-mentioned research problems:

1. The selectivity of fibre fines during flotation is higher than that of mineral fillers. The removal of fibre fines and mineral fillers changes the composition and light scattering properties of the pulp.
2. Fibre fines and mineral fillers also improve the light scattering properties of pulp at a wavelength of 700 nm, that used for the measurement of ink content.
3. Determinations of ink content are biased when a constant light scattering coefficient is used, due to the change in fine material content. When the measured light scattering coefficient is used to determine ink content, the values obtained account for the changes in fine material content.
4. The light scattering coefficient can be measured and high retention achieved when a low-grammage sheet is prepared on filter paper.

1.4 Outline of the thesis

This thesis is organized in five chapters. Chapter 1 briefly traces the background and presents the research problem, the aim of the thesis and the hypotheses advanced. Our present understanding of the optical properties of recovered paper in relation to ink content measurements is presented in Chapter 2. Materials and methods for testing the hypotheses are presented in Chapter 3, and the results are summarised and discussed in Chapter 4. Finally, the conclusions to be reached are given in Chapter 5.

2 Present understanding of optical properties in relation to ink content measurements

We experience a wide range of colours in our everyday lives, and know that the colours of daylight, lamps and objects vary. The prerequisite for a sensation of colour is light. We do not perceive colours in the dark, for instead the rod cells in the retina respond to differences in the amount of light, producing a black-and-white sensation. Colour is perceived under brighter conditions, employing cone cells of three types, sensitive to red, green and blue light. The sensation of shades of colour is produced in the human brain on the basis of the relative intensity of these three types of cone cell. (Hunt 1987, Rihlama 1999).

In the science of light and colour we speak of *photometry*, referring to visible light measurement, or more specifically *colorimetry*, referring to colour measurement, whereas the measurement of invisible radiation (including ultraviolet and infrared light) is known as *radiometry* (Johnston 2001). Visible light consists of electromagnetic radiation of wavelengths between 380 nm and 780 nm, although the lower end (380–400 nm) and upper end (700–780 nm) depend on lighting conditions for their visibility (Rihlama 1999).

Part of the illumination light is scattered from the paper and it is from this diffusely reflected light that the optical appearance of the object is recognised. In a similar manner, the scattered light forms the basis for the reflectance-based optical analysis. Reflectivity (the intrinsic reflectance factor, R_∞) is the reflectance measured from an object of thickness great enough that doubling of it would not change the reading. (Pauler 2008). A perfect diffuser scatters all the illumination light, so that it seen as white and has a reflectivity of 100%. In the opposite case, where all the light is absorbed, the object is seen as black and the reflectivity reading is 0%. In between these two extremes are grey samples in which partial absorption of the illumination light appears over the entire wavelength region in equal strength, so that the reflectivity lies somewhere between 0% and 100%. (Vaarasalo 1999).

The colour of objects results from the absorption of light in a specific wavelength region. In general, shades of violet-blue scatter in the wavelength region 380–500 nm, shades of green at 500–580 nm and shades of orange-red at 580–780 nm, with light absorption taking place in the remainder of the visible light spectrum (Pierce & Marcus 1994). All the shades of colour are the result of varying absorbing behaviour of the illumination light as a function of its wavelength (Pauler 2002).

2.1 History of light and colour measurement

The oldest detector of light and colour is undoubtedly the human eye, but the sensation of colour is highly subjective, being affected by the mental and physical conditions of individuals and the surrounding light (Hunt 1987, Johnston 2001). For these reasons there was already considerable interest in the late 19th century in constructing a detector that could measure light and colours objectively (Johnston 2001). The first crude reflectometer was constructed in 1912 (Nutting 1912, Taylor 1920), and after several development phases, the measurement of scattered light came into regular use from 1927 onwards (Lewis 1933).

At that time several light sources, filters and measurement geometries were used for measurement purposes, resulting in a definite need to standardize measurement conditions in order to obtain comparable results. The International Commission on Illumination (CIE, Commission Internationale de l'Eclairage) dedicated itself to this task and announced its recommendations in 1931. The measurement of colour came to be based on reflectivity in the red (X), green (Y) and blue (Z) light regions. (Hunt 1987, Pauler 2008, Pierce & Marcus 1994, Vaarasalo 1999). Reflectivity measurements based on green light, known as the Y-value, or luminosity (INGEDE Method 2), are still commonly used today, partly for measuring the opacity, light absorption and light scattering coefficients. On the other hand, reflectivity the blue and red regions is of less importance as such, although they are used for the calculation of CIELAB colour shades, for example (Pauler 2008).

Brightness measurement was developed by the Institute of Paper Chemistry in the early 1930's for evaluating the performance of pulp bleaching, *i.e.* removal of the yellowish colour from pulp after production (Crawford 1999, Hatch 1940, Lewis 1933). Brightness measurement could have been based on the reflectivity of blue (Z) light, but instead a Wratten filter was used. This has a central wavelength of 457 nm but the measurement includes all wavelengths in the range 410–520 nm. (Jordan 1996). Brightness measurement has become best-known and most commonly used in its pulp and paper application (Crawford 1999, Vaarasalo 1999).

Brightness measurement has found another area of applicability in deinking. The removal of printing ink was commonly quantified in terms of brightness before the invention of a more selective measure of ink content, and as late as 1987 a deinkability factor (DEM_f) was introduced for estimating ink removal efficiency based on the brightness gain over the flotation process (Putz 1999,

Villforth *et al.* 2010). The high sensitivity of brightness to yellow colour was well known, and deliberations were focused on how to obtain information which is not influenced by the yellowness of the fibre material. Calculation of DEM_f from luminosity instead of brightness (Putz 1999) did reduce the impact of the yellowish colour, but it did not completely solve the problem as luminosity also responds to yellowish colours to some extent. Another approach was established in 1992, when a new deinkability factor (DEM_{LAB}) was introduced that was based on CIELAB colour shades (Putz 1999, Villforth *et al.* 2010). The CIELAB colour system had been established earlier, in 1976, and had become a very popular measure of colour (Pauler 2008, Pierce & Marcus 1994, Vaarasalo 1999).

Reflectance-based ink content measurement was presented for the first time at the 2nd Research Forum on Recycling (Jordan & Popson 1993). The method was a specific case of optical measurements used otherwise in pulp and paper applications. The effect of the yellowish colour of the fibre material was found to be negligible at wavelengths higher than 650 nm, and ink removal or fragmentation could be measured without any interference from fibre colour (Jordan & O'Neill 1994, Jordan & Popson 1994). Furthermore, the reflectivity in this wavelength region could reveal the contribution of printing ink to the overall brightness (Jordan 1996). The printing ink also absorbs invisible, near-infrared radiation, *i.e.* in the wavelength region above 780 nm. Thus a near-infrared wavelength of 950 nm was chosen as appropriate for ink content measurement ($ERIC_{950}$), because then the influence of coloured papers could be neglected during the measurement of black printing ink (Jordan & Popson 1994). This idea reached the status of a TAPPI provisional method in 1997 (TAPPI T 567) and became the prevailing technique in North America.

Extended range instruments were needed for measurements in the near-infrared region, however (Villforth *et al.* 2010). Ackermann & Göttsching (2002a) proposed that the determination of ink content could be based accurately enough on light reflectivity at 700 nm, which could be measured using colorimeters and spectrophotometers of a kind generally to be found in paper industry laboratories. Use of this method was recommended for the first time in INGEDE Method 10, which dates back to December 1999, while nowadays such instructions are included in INGEDE Method 2. The measurement responds to printing ink that absorbs light at 700 nm, including both black printing ink and also cyan, green and blue colours (Ackermann & Göttsching 2002b, Pauler 2002, Pierce & Marcus 1994).

2.2 The Kubelka-Munk theory

Pulp and paper applications are multi-component systems consisting of fibres, fibre fines and mineral fillers in varying proportions. The sum effect of the various components can be estimated using the mathematical theory developed by Kubelka & Munk (1931):

$$R_\infty = 1 + \frac{k}{s} - \sqrt{\left(\frac{k}{s}\right)^2 - 2 \cdot \frac{k}{s}}\,, \tag{1}$$

where R_∞ is the reflectivity, k is the light absorption coefficient [m^2/kg] and s is the light scattering coefficient [m^2/kg]. The light absorption and light scattering coefficients define the ability of an infinitively thin layer to absorb and scatter light, respectively. They obey the mass balance calculation, *i.e.* they are additive when weighted by the mass proportion of the component:

$$k = x_1 \cdot k_1 + x_2 \cdot k_2 + x_3 \cdot k_3 \ldots\,, \tag{2}$$

$$s = x_1 \cdot s_1 + x_2 \cdot s_2 + x_3 \cdot s_3 \ldots\,, \tag{3}$$

where x_i is the mass proportion of the component (Pauler 2008, Vaarasalo 1999, Walmsley & Silveri 1999). The light scattering coefficient is measured in accordance with:

$$s = \frac{R_\infty}{w \cdot \left(1 - R_\infty^2\right)} \cdot \ln \frac{R_\infty \cdot \left(1 - R_0 \cdot R_\infty\right)}{\left(R_\infty - R_0\right)}\,, \tag{4}$$

where w is the grammage of the sheet [kg/m^2] and R_0 is the single-sheet reflectance factor against a black cavity (ISO 9416). The reflectivity (R_∞) is measured from an opaque stack of sheets (ISO 2469) and the measurement of grammage is based on SFS-EN ISO 536 and TAPPI T 410. The light absorption coefficient is calculated from the rearranged equation representing the Kubelka-Munk theory as shown in Eq. 1 (ISO 9416). The calculation uses the light scattering coefficient and reflectivity:

$$k = \frac{s \cdot \left(1 - R_\infty\right)^2}{2 \cdot R_\infty}\,. \tag{5}$$

Use of the Kubelka-Munk theory requires the light-absorbing and light-scattering components to be uniformly distributed in the paper sheet. Furthermore, the optical interactions need to be independent of each other. In general, paper sheets

do not completely fulfil these conditions. (Pauler 2008). The additive rules (Eqs. 2 and 3) are valid only in a certain range (Foote 1946, Pauler 2002). It has been explained that the failure of the additive rules is a result of what is known as the crowding factor (Bruehlman *et al.* 1961, Middleton *et al.* 1994). When the filler content is low, for example, all the particles are able to interact with the illumination light, but as the filler content is increased, the particles form aggregates, so that the specific surface area is lower. In addition, light scattering is affected by light absorption. The use of dyes, for example, absorbs light and reduces scattering in the region where absorption of light appears (Foote 1939, Neuman 2005, Pauler 2008), whereas the light scattering coefficient remains intact in the region where light absorption is not changed (Rundlöf & Bristow 1997). The deviation in the light scattering coefficient becomes significant (more than 10%) when the light absorption coefficient is higher than 7 m^2/kg (Neuman 2005, Rundlöf & Bristow 1997). The Kubelka-Munk theory is applicable with some limitations, however, and is widely used to explain the interactions between the absorption and scattering of light in pulp and paper applications.

2.3 Measurement of ink content

Several variations on the concept of reflectance-based ink content exist. Reflectivity at wavelengths of 700 nm and 950 nm could be used as an indication of ink content, but as it is affected by both light absorption and light scattering (Pauler 2002), the measurement of ink content is more often based on the light absorption coefficient as obtained using the Kubelka-Munk theory. The following sections will summarize the various procedures used to measure ink content, where they differ from each other in the methods by which the test media are prepared and the wavelength at which the reflectance is recorded. The general features of test media preparation will be introduced, but a more detailed comparison can be found in the works of Haynes (2000) and Lévesque *et al.* (1995).

2.3.1 Machine-made paper

Machine-made papers produced from deinked pulp in most instances satisfy the opacity limitation, *i.e.* their opacity is less than 97% (ISO 22754 and TAPPI T 567), thus the light scattering coefficient can be measured. According to the above-mentioned standards, the single-sheet reflectance factor and reflectivity

should be recorded at a wavelength of 950 nm and the light scattering and light absorption coefficients determined using Eqs. 4 and 5. The ink content, known as the Effective Residual Ink Concentration, $ERIC_{950}$, or simply *ERIC*, is a ratio of the light absorption coefficient of the pulp to a constant light absorption coefficient of ink itself:

$$ERIC = \frac{k}{k_{\text{ink}}} \cdot 10^{6}, \tag{6}$$

where *ERIC* is the measured ink content [ppm] and k_{ink} is the constant light absorption coefficient of ink, *i.e.* 10000 m^2/kg.

A modified version, known as $ERIC_{700}$, is determined in accordance with Eq. 6, but with the single-sheet reflectance factor and reflectivity measured at a wavelength of 700 nm. Strictly speaking this is not included in the ISO 22754 and TAPPI T 567 standards, although measurement of the light absorption coefficient at 700 nm is introduced in the INGEDE Method 2.

2.3.2 Low-grammage sheets formed on a wire screen

Sheet preparation on a wire screen is used for deinked pulp samples in accordance with ISO 22754 and TAPPI T 567. A 150-mesh wire screen (nominal aperture 105 µm) is required by TAPPI T 272, whereas a wire screen having a nominal aperture size of 125 µm is appropriate for SFS-EN ISO 5269-1. The preparation of sheets having a grammage of 60 g/m^2 is demanded, but if the opacity exceeds 97%, two approaches are introduced for measuring ink content. According to ISO 22754, a new sheet having a lower grammage should be prepared to meet the opacity limitation on measurement of the light scattering coefficient, or alternatively, TAPPI T 567 lays down that *relative ERIC* values with a constant light scattering coefficient can be measured when the light scattering coefficient does not vary substantially. Most commonly, *actual ERIC* values are measured only for deinked pulp where the sheet opacity can be maintained below 97% and the light scattering coefficient measured. The ink content (Eq. 6) is determined from the light absorption coefficient, which is determined from the reflectivity and light scattering coefficient in accordance with Eq. 5.

2.3.3 Opaque pads formed on filter paper

The preparation of opaque pads on filter paper from samples containing substantial amounts of ink, *i.e.* samples from deinking processes, is in accordance with the INGEDE Method 1. Opaque pads (225 g/m^2) formed on filter paper have a high retention of fine materials, including small-sized ink particles, because the filter paper and the higher thickness of the pad prevents fine material losses (Dorris 1999, Haynes 2000, Lévesque *et al.* 1995, Lévesque *et al.* 1998, McKinney 1988, Scott 1993). This enables better representativeness of the original pulp suspension to be achieved.

High opacity of the pad, however, prevents measurement of the light scattering coefficient, and for that reason a constant value has to be used for the determination of ink content (Eq. 6) via the light absorption coefficient (Eq. 5). Use of the following light scattering coefficients has been reported: 42 m^2/kg (INGEDE Method 2 2007), 45 m^2/kg (Walmsley & Silveri 1999), 46.9 m^2/kg (Haynes 2000) and 50 m^2/kg (Vahey *et al.* 2007). The newest version of the INGEDE Method 2 (2011), no longer gives a constant value for the light scattering coefficient.

The ink content can also be measured from opaque pads using a non-standard procedure ($RI_{L\&W}$) as suggested for use with Lorentzen & Wettre's Elrepho 070 spectrophotometer (Anon 2004). In this special procedure the light absorption coefficient (Eq. 5) is calculated from the average reflectivity at 690 nm and 700 nm using a constant light scattering coefficient of 55 m^2/kg, and finally the ink content is taken to be:

$$RI_{L\&W} = 0.8919 \cdot (k - 0.7)^2 + 65.849 \cdot (k - 0.7) + 24.713 , \qquad (7)$$

where $RI_{L\&W}$ is the ink content according to the Lorentzen & Wettre procedure and 0.7 is the light absorption coefficient of unprinted paper.

The third procedure that can be used with opaque pads is ink elimination (*IE*). This is a percentage value based on light absorption coefficients over the flotation stage:

$$IE = \frac{k_{UP} - k_{DP}}{k_{UP} - k_{unpr}} \cdot 100 , \qquad (8)$$

where *IE* is the ink elimination and the sub-indices UP, DP and unpr stand for undeinked pulp, deinked pulp and unprinted pulp, respectively (INGEDE Method 2, Ackermann & Göttsching 2002c). As the light scattering coefficient cannot be

measured from opaque pads, it is set as constant, and thus Eq. 8 can be rearranged to Eq. 9. The term ($[1–R_{\infty,\mathrm{unpr}}]^2/R_{\infty,\mathrm{unpr}}$) in the equation can be neglected if no value for unprinted sample reflectivity is available. (INGEDE Method 2).

$$IE = \frac{\left(\frac{\left(1-R_{\infty,\mathrm{UP}}\right)^2}{R_{\infty,\mathrm{UP}}}\right) - \left(\frac{\left(1-R_{\infty,\mathrm{DP}}\right)^2}{R_{\infty,\mathrm{DP}}}\right)}{\left(\frac{\left(1-R_{\infty,\mathrm{UP}}\right)^2}{R_{\infty,\mathrm{UP}}}\right) - \left(\frac{\left(1-R_{\infty,\mathrm{unpr}}\right)^2}{R_{\infty,\mathrm{unpr}}}\right)} \cdot 100\,. \qquad (9)$$

2.3.4 Low-grammage sheets formed with a closed water loop

Sheet preparation with a closed water loop is included in the INGEDE Method 1. This implies that the drained water is circulated during the preparation process and sheets are discarded until a balance in the white water is achieved. According to the method, a balance should be achieved by discarding five sheets, but it has been observed that a balance in the white water is sometimes not achieved even after nine sheet preparations (Carré 2005). This laborious method was originally employed for determining ink elimination in accordance with Eq. 8 (INGEDE Method 10), but this method is an optional procedure in the current versions of the INGEDE Methods and ink elimination is determined from pads (Eq. 9), as explained in Section 2.3.3.

2.3.5 Summary of ink content measurement

The opacity of machine-made papers is usually below 97%, and the ink content can be determined from the measured light scattering coefficient and reflectivity. When the sample of interest is in the form of a pulp suspension, a suitable test medium needs to be prepared. The preparation of slightly translucent sheets on a wire screen enables measurement of the light scattering coefficient, but results in poor retention of printing ink. High retention of printing ink can be achieved by preparing opaque pads on filter paper, but then the high opacity prevents measurement of the light scattering coefficient.

3 Materials and Methods

3.1 Sample preparation

In Paper I the removal of components in flotation was studied using a batch of recovered papers including old newsprints (ONP) and old magazines (OMG) in the proportion 50/50, while in Paper II the individual print products were pulped separately and the desired mixtures were prepared by mixing the resultant pulps. Pulp mixtures were prepared by mixing pulped ONP1 with hyperwashed pulp produced from unprinted supercalendered paper in the proportions 25/75, 50/50 and 75/25. ONP1 was also used after pulping and after hyperwashing (HW) for precipitated calcium carbonate (PCC) filler addition.

Table 1. Recovered papers and virgin pulps used in the present experiments.

Study	Abbreviation	Paper or pulp type	Printing	Note
Paper I	Mix 1	ONP/OMG mixture 1	Yes	Flotation
Paper II	HW ONP1	Hyperwashed ONP1	Yes	PCC filler addition
	25% ONP1	ONP1 / HW SC1 25/75	ONP Yes, SC No	PCC filler addition
	50% ONP1	ONP1 / HW SC1 50/50	ONP Yes, SC No	PCC filler addition
	75% ONP1	ONP1 / HW SC1 75/25	ONP Yes, SC No	PCC filler addition
	100% ONP1	ONP1	Yes	PCC filler addition
Paper III	HW ONP2	Hyperwashed ONP2	Yes	GCC filler addition
	HW ONP2	Hyperwashed ONP2	Yes	Kaolin filler addition
	TMP	Thermomechanical pulp	No	Washing fines out
	HWK	Hardwood kraft pulp	No	Washing fines out
	SWK	Softwood kraft pulp	No	Washing fines out
	ONP3	Newsprint 3	No	Washing fines out
	SC2	Supercalendered 2	No	Washing fines out
	LWC1	Lightweight coated 1	No	Washing fines out
Unpublished	ONP4	Newsprint 4	Yes	Washing fines out
	SC3	Supercalendered 3	Yes	Washing fines out
	LWC2	Lightweight coated 2	Yes	Washing fines out
Paper IV	Mix 2	ONP/OMG mixture 2	Yes	Comparing test media

HW stands for hyperwashing, ONP, SC and LWC for old newsprint, supercalendered and lightweight coated papers, respectively. PCC is precipitated calcium carbonate and GCC ground calcium carbonate.

In Paper III, ONP2 was pulped, hyperwashed, and used for ground calcium carbonate (GCC) and kaolin filler addition. Thermomechanical (TMP), hardwood kraft (HWK) and softwood kraft (SWK) virgin pulps were obtained as a pulp

suspension directly from mills, while unprinted ONP3, SC2 and LWC1 papers were pulped and used for washing out fine materials. In addition, printed ONP4, SC3 and LWC2 papers were pulped and used for washing out fine materials in accordance with unprinted papers (Unpublished study). The printed papers were taken from the same batch as unprinted papers, so they were as similar as possible to each other, except printed papers were run through the printing process. In Paper IV a 50/50 mixture of ONP/OMG was used for comparing methods for preparing test media.

3.1.1 Pulping

The pulps for the experiments were prepared using a pilot-scale pulper in Paper I and a laboratory-scale pulper in Papers II–IV. The pilot-scale pulper was a Metso OptiSlush™ drum pulper designed for research purposes, having a diameter of 2 metres (Kankaanpää 2002, Suhonen 2002), while the laboratory-scale pulper was a planetary-type mixer (Kenwood™ KM020), which is similar to the Hobart™ N50 suggested for the INGEDE Method 11p. Warm tap water (45ºC) was used to adjust pulping consistency to 16% in all the pulping experiments, and alkaline chemical conditions were used. The dosages of pulping chemicals were as follows: sodium hydroxide 10 kg/t, sodium silicate 18.6 kg/t (as a product, SiO_2/Na_2O mole ratio 2.5), hydrogen peroxide 10 kg/t and Serfax MT90 soap 6 kg/t.

3.1.2 Flotation

Flotation was performed only in Paper I, where the removal of the various pulp components was studied. A pilot-scale Metso OptiBright MC™ flotation cell (Fig. 1) having a volumetric capacity of 1 m^3 was used (Aho & Heimonen 2002, Heimonen 2001). The cell comprises four equal-sized sectors aligned in sequence so that the feed rate of the pulp suspension determines the through-flow of each sector. The level of the pulp suspension in the flotation cell was controlled by adjusting the level of the accept purging pipe. Each sector had a rotor-assisted aeration zone and a free-flowing separation zone connected to each other. Air was mixed into the pulp suspension in the aeration zone, resulting in an upward flow of the pulp suspension due to its lower density and a downward flow in the separation zone, *i.e.* a spiral flow inside the sector. Air bubbles collected the hydrophobic particles during flotation and formed a layer of froth on the top of the suspension that was purged from the cell in the form of an overflow. The froth

from each sector was collected separately, and thus four reject ratios were achieved in a single run.

The pulp was coarse-screened at a 2% consistency prior to flotation, using a 1.5 mm perforated screen. A side-stream of the coarse-screened accept was fed to flotation via dilution, and the rest of the coarse-screened accept was directed back to the dump chest, *i.e.* coarse screening was operated in loop mode. The pulp side-stream was diluted to 1% consistency with process water having a hardness of 20ºdH (German hardness, equal to 142 mg Ca^{2+}/L). The volumetric feed rate of the diluted pulp was 1 L/s, resulting in a residence time of around 15 minutes. An air ratio of 133% was obtained by using an air flow of 80 L/min, defined by dividing the air flow by the volumetric feed rate of the pulp suspension. The aeration rotor was run at a relative speed of 70% (2009 rpm).

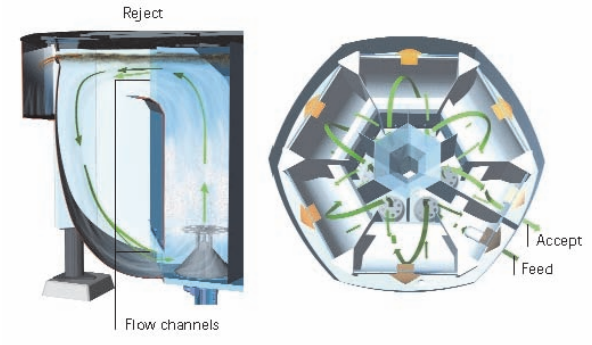

Fig. 1. Side view of the Metso OptiBright MC™ flotation cell sector (left) and top view of the whole cell (right) showing the flows of pulp suspension and froth. The air dispersion rotors are also shown in the picture. The cell shown has 6 sectors, but otherwise it is similar to the pilot-plant cell that has four sectors (Heimonen 2001). Photo courtesy of Metso Corporation.

3.1.3 Hyperwashing and washing

The term *hyperwashing* refers to a procedure where all the fine materials are washed out from the pulp suspension with an excess amount of water (Carré *et al.* 1994). Hyperwashing was performed in the present experiments using a Somerville-type screening machine (TAPPI T 275) equipped with a 150-mesh wire screen (nominal aperture 105 µm) to prepare the *HW ONP1*, *HW SC1* and *HW ONP2* pulps, as shown above in Table 1. Complete fine material removal was achieved using an excessive washing time and a constant water flow of 8 L/min.

The same device and wire screen were used for partial fine material removal in addition to hyperwashing. In the case of the *TMP*, *HWK*, *SWK*, *ONP3*, *SC2*, *LWC1*, *ONP4*, *SC3* and *LWC2* pulps (Table 1) the washing time was varied from 30 seconds to 10 minutes to obtain samples having a variable fine material content.

3.1.4 Addition of fresh fillers

Fresh fillers in proportions ranging from 0% to 40% were added to the pulp suspension prior to sheet preparation. PCC was added to five pulp suspensions in Paper II and GCC and kaolin to the *HW ONP2* pulp suspension in Paper III.

3.2 Preparation of test media for optical analysis

Optical properties were determined from (1) opaque pads formed on filter paper in Papers I and IV, (2) low-grammage sheets formed on a wire screen in Paper IV, and (3) low-grammage sheets formed on filter paper in Papers II–IV. The pH of pulp suspensions was adjusted to 7.3 (±0.3) in all the test media preparations. After preparation, the test media were pressed at 400 kPa for 2 minutes and acclimatized to paper laboratory conditions (SFS-EN 20187, TAPPI T 402) in a circulating air cabinet overnight prior to the optical measurements.

3.2.1 Opaque pads on filter paper

Opaque pads of diameter 160 mm and grammage 225 g/m^2 were prepared from the pulp suspension as outlined in INGEDE Method 2, ISO 3688 and TAPPI T 218. A fast filter paper having a pore size of 5–13 µm (WVR Qualitative filter paper 413) was placed on top of a plastic wire screen in a Büchner funnel. The evenly moistened filter paper and plastic wire were pressed tightly against the bottom of the Büchner funnel during the pouring of a 1% pulp suspension. Immediately after pouring, a vacuum was applied to the suction bottle to seal the filter paper with the plastic wire onto the bottom of the funnel. This procedure was followed to avoid markings on the pads and to ensure that the filter paper could be detached from the pads after filtration.

3.2.2 Low-grammage sheets on a wire screen

Low-grammage sheets were prepared in a sheet mould as outlined in SFS-EN ISO 5269-1 and TAPPI T 272. The mould was equipped with a wire screen having a nominal aperture size of 125 µm, and sheet formation took place in a square area of 272.25 cm^2. An amount of pulp suspension equal to a grammage of 47.5 g/m^2 was weighed and poured into the sheet mould, which was filled above the wire screen. The mould was then filled up to the mark which denotes a volume of 10 litres above the screen and drained after the mixing of the pulp-water suspension. The sheets were transferred to the pressing process with a blotter.

3.2.3 Low-grammage sheets on filter paper

Low-grammage sheet preparation on filter paper is the most novel test medium preparation method and has not yet been standardized. This method was studied in order to overcome problems related to the retention of printing ink and the possibility of measuring the light scattering coefficient from the same sheet, as summarised in Section 2.3.5. Sheet preparation took place in a sheet mould where a high-retention filter paper (pore size of 1–2 µm) had been placed on top of a wire screen. Ashless Munktell 00H filter papers were preferred because their surface was smooth enough to ensure that it was easy to peel the filter paper off the conditioned sheet.

Before sheet formation, the mould was filled up to the surface of the wire screen and the evenly moistened filter paper was placed on top of the screen, after which the upper part was closed. A sufficient amount of pulp suspension was weighed in to reach the target grammage of 30 or 40 g/m^2 over a sheet area of 272.25 cm^2. The weighed suspension was diluted to a volume of 250 ml, poured into the sheet mould and the purge valve was opened. When the excess water had drained off, the resulting sheet was transferred with the filter paper onto a paperboard (that absorbs the water released during pressing) and two blotters and one pressing plate were placed on top of the sheet. After pressing, the plate and the uppermost blotter were removed and the sheet with the filter paper (and one blotter) were acclimatized overnight. After the conditioning period, the filter paper was detached from the sheet with great care so that the surface was not damaged.

3.3 Analyses

Measurement of pulp mass in an oven-dry (od) state forms the basis for the experiment, as it is needed in several of the analyses and calculations. The oven-dry mass of the pulp was calculated from its consistency (*cs*) or dry matter content (*dmc*). Consistency (SFS-EN ISO 4119, TAPPI T 240) is based on filtering and drying the retained pulp, whereas dry matter content (SFS-EN 20638) is related to water evaporation from the pulp suspension. Consistency was measured in Papers II–IV for all the samples with a consistency less than 20%, and dry matter content in all other instances. In Paper I the dry matter content was measured for all the samples studied.

3.3.1 Ash content and measurement of the added filler

The measurement of ash content (ISO 1762, TAPPI T 211) was based on incineration of the pulp at 525ºC. The amount of added filler was evaluated from the ash analyses of the prepared sheets, by subtracting the ash content of pulp without any filler addition from the result.

3.3.2 Mass fractions

The mass proportions of the pulp components were measured in Paper I using a tube flow fractionator as described by Laitinen *et al.* (2006). The fractions comprised flakes, long fibres, short fibres and fines. The fibre fines content was calculated by subtracting the ash content from the fines content.

3.3.3 Fine material content

The fine material content was measured with the same Somerville-type screening machine (TAPPI T 275) that was used for hyperwashing with a 150-mesh wire screen (nominal aperture 105 µm) in Papers II and III. The fine material content was obtained by subtracting the fibre retention percentage from 100. The retained fibre ratio was measured as the ratio of the oven-dry mass of retained fibres to the initial amount of pulp. When partial fine material washing was in question (Paper III), the fine material content was obtained by subtracting the retained pulp ratio from the ratio obtained for hyperwashing. In Paper IV the fine material content

was analysed from the pulp suspension in the same manner, except that the wire screen was the same as in the sheet mould (nominal aperture 125 µm).

3.3.4 Reflectivity, light scattering coefficient and ink content

Sheets acclimatized to the paper laboratory conditions were trimmed to a size of 150 mm by 150 mm and further divided into four pieces having of size 75 mm by 75 mm each. An opaque stack was produced from eight sheet pieces, *i.e.* two sheets, and reflectivity was measured from the four topmost pieces (representing one sheet). The same was repeated for opposite side of the pieces and for the other four pieces that had served as a background for the measurement. As the pads were opaque, four measurements were taken from each side without piling them up. The reflectivity values were recorded at 700 nm (ISO 2469).

The light scattering coefficient at 700 nm and luminous light scattering coefficient were measured only for sheets having an opacity (ISO 2471, TAPPI T 519) less than 97%, according to Eq. 4. The measurements of the light scattering coefficient were based on ISO 9416, ISO 22754 and TAPPI T 567, and the grammage measurements on SFS-EN ISO 536 and TAPPI T 410.

Ink content (Eq. 6) was determined via the light absorption coefficient (Eq. 5). This took place in accordance with ISO 22754 and TAPPI T 567 except that the reflectivity was recorded at 700 nm. Ink content was also measured using a non-standard $RI_{L\&W}$ procedure (Eq. 7) suggested for use with the Lorentzen & Wettre Elrepho 070 spectrophotometer (Anon 2004).

3.4 Calculations

3.4.1 Free ink content

The free ink content ($ERIC_{Free}$) was calculated by subtracting the attached ink content ($ERIC_{HW}$), determined after hyperwashing of the pulp sample (Carré *et al.* 1994), from the total ink content of the pulp:

$$ERIC_{Free} = ERIC - ERIC_{HW} . \quad (10)$$

3.4.2 Mass reject ratio, removal efficiency and selectivity

The mass flow rates of the feed and reject were calculated as follows:

$$\dot{m} = \frac{dmc}{100} \cdot \dot{Q}_V , \tag{11}$$

where $\dot{m}$ is the mass flow rate [kg/min], *dmc* is the dry matter content [%] and $\dot{Q}_V$ is the volumetric flow rate [L/min]. The mass reject ratio was calculated from the mass flow rates:

$$RR_m = \frac{\dot{m}_R}{\dot{m}_F} \cdot 100 , \tag{12}$$

where RR_m is the mass reject ratio [%] and the subscripts R and F stand for reject and feed, respectively. As the reject samples were taken from four consecutive sectors, the cumulative mass flow rates were obtained by summing mass flow rates of the rejects. The cumulative mass reject ratios were obtained from the cumulative mass flow rates of the reject, while the mass flow rate of the feed was the rate of flow into the flotation cell (Hautala *et al.* 2009).

The mass flow rate of a component was calculated as follows:

$$\dot{x} = \dot{m} \cdot \frac{x}{100} , \tag{13}$$

where $\dot{x}$ is the mass flow rate of the component [kg/min] and x is the content of component [%]. The calculation of removal efficiency was based on:

$$E_r = \frac{\dot{x}_R}{\dot{x}_F} \cdot 100 = \left[1 - \left(1 - \frac{RR_m}{100} \right) \cdot \frac{x_A}{x_F} \right] \cdot 100 , \tag{14}$$

where E_r is the removal efficiency of a component [%] and subscript A stands for the accept (Hautala *et al.* 1999, Hautala *et al.* 2009). In the case of the reject, the cumulative mass flow rate of a component was calculated by summing mass flow rates from each sector. The efficiency of printing ink removal was based on the calculated free ink content of the feed and accept flows. The calculated removal efficiencies were plotted against the mass reject ratio and the selectivity curve introduced by Nelson (1981) was fitted to them. The curve fitting was done by adjusting Nelson's selectivity index in the following equation so that the best fit was achieved:

$$E_r = \frac{RR_m}{1 - Q_N + Q_N \cdot RR_m} , \tag{15}$$

where Q_N is Nelson's selectivity index, defined as:

$$Q_N = 1 - \frac{x_A}{x_R}. \quad (16)$$

A positive selectivity index ($0 < Q_N < 1$) points to enrichment of the component concerned in the froth (reject), where the ideal selectivity is described as $Q_N = 1$. In the case of a split flow, $Q_N = 0$, the content values for the accept and reject are equal. A negative selectivity index ($-\infty < Q_N < 0$) is a result of component enrichment in the deinked pulp (accept). Curves illustrative of this could be achieved by means of Nelson's selectivity index, although positive and negative Nelson's selectivity indices cannot be directly compared as their ranges are not the same. For that reason, another selectivity index was also used:

$$Q_K = 1 - \frac{x_I}{x_{II}}, \quad (17)$$

where Q_K is Karnis's selectivity index, x_I is given for the fraction in which the content is lower and x_{II} for the fraction in which the content is higher (Karnis 1997). Thus, Karnis's selectivity index has a finite range ($0 \leq Q_N \leq 1$), but it does not indicate in which fraction (accept or reject) a component has been enriched. When the accept content is lower, Q_N is positive and equal to Q_K, whereas when Q_N is negative, Q_K was calculated as follows:

$$Q_K = 1 - \frac{1}{1 - Q_N}. \quad (18)$$

3.4.3 Mass balance calculation of ink content

The effect of fillers on the light absorption coefficient was estimated in Papers II and III according to its mass-weighted relation (Walmsley & Silveri 1999). In a two-component system, *i.e.* pulp and mineral fillers, Eq. 2 can be written as follows:

$$k_{MB} = \left(1 - \frac{x}{100}\right) \cdot k_{pulp} + \frac{x}{100} \cdot k_{filler}, \quad (19)$$

where k_{MB} is the light absorption coefficient in accordance with the mass balance calculation [m^2/kg], x is the percentage of filler [%], k_{pulp} is the measured light absorption coefficient of the pulp [m^2/kg] and k_{filler} is the light absorption coefficient of the mineral filler [m^2/kg]. Assuming that the light absorption coefficient of fresh mineral filler is negligible, Eq. 19 reduces to:

$$k_{\mathrm{MB}} = \left(1 - \frac{x}{100}\right) \cdot k_{\mathrm{pulp}} . \tag{20}$$

Calculation of the percentage deviation in ink content was based on:

$$ERIC_{\mathrm{Dev}} = \frac{ERIC - ERIC_{\mathrm{MB}}}{ERIC_{\mathrm{MB}}} \cdot 100 , \tag{21}$$

where $ERIC_{\mathrm{Dev}}$ is the percentage deviation [%], $ERIC$ is the measured ink content [ppm] and $ERIC_{\mathrm{MB}}$ is the ink content in accordance with the mass balance calculation [ppm]. The $ERIC_{\mathrm{MB}}$ values were calculated from the light absorption coefficients according to Eq. 6 in Paper II, while Eq. 7 was used in Paper III. The effect of the mass balance calculation can be understood quite simply from the following: the higher amount of inkless filler the sheet contains, the less inked fibres will be present, and thus less ink.

3.4.4 TAPPI repeatability

The calculation of TAPPI repeatability was based on ten individual replicates, according to TAPPI T 1200. The repeatability (r) of the ash content and fine material content data in Paper IV was calculated from:

$$r = 2.77 \cdot std , \tag{22}$$

where *std* stands for standard deviation. TAPPI repeatability ratios (%r) were calculated for light scattering coefficients and ink content in Papers II–IV and for reflectivity in Papers IV:

$$\%r = \frac{2.77 \cdot std}{average} \cdot 100 . \tag{23}$$

3.4.5 t-test statistical analysis

Statistical analysis was used to evaluate the significance of the results in Paper IV. A t-test based analysis was run in equal-variance or unequal-variance mode depending on the results of the F-test for variances. A normal distribution of the data was assumed. The statistically significant differences between two mean values were evaluated using Student's t-test distribution (p-value). Ten replicates were used for testing.

4 Results and Discussion

4.1 Removal of fine materials

The removal of components was studied using Nelson's and Karnis's selectivity indices, which have been used previously in screening, cleaning and fractionation studies (Hautala *et al.* 2009, Karnis 1997, Nelson 1981). These are powerful tools, as they point to component removal independent of the mass reject ratio, whereas cleanliness and removal efficiency, for example, are dependent on the mass reject ratio.

Removal efficiencies for fibre fines, mineral fillers, short fibres and long fibres in flotation as functions of the mass reject ratio are presented in Fig. 2 by means of curves illustrative of Nelson's selectivity index, which were fitted by adjusting Q_N in Eq. 15.

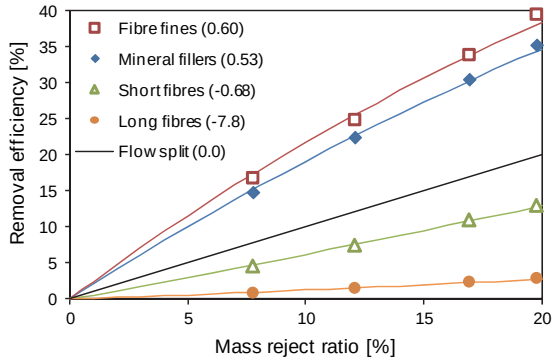

Fig. 2. Removal efficiencies of components during pilot-plant flotation. Nelson's selectivity indices, obtained by fitting a curve (Eq. 15) to the experimentally observed data, are shown in parentheses. Modified from Paper I.

Fibre fines and mineral fillers were enriched in the reject and obtained positive Nelson's selectivity indices, while negative indices were obtained for short and long fibres, as they were retained in the accept. Since positive and negative Nelson's selectivity indices cannot be directly compared, Karnis's selectivity indices (Eq. 18) were also calculated. The values of Q_N and Q_K are presented in Table 2.

Table 2. Selectivity indices for the components studied in Fig. 2.

	Nelson's selectivity index	Karnis's selectivity index
Fibre fines	0.60	0.60
Mineral fillers	0.53	0.53
Short fibres	–0.68	0.40
Long fibres	–7.8	0.89

The fibre fines had the highest tendency to become enriched in the froth, *i.e.* the reject from flotation. The floatability of fibre fines apparently arises from ability of calcium soap to adhere to it, as shown by Drabek *et al.* (1998), who used microscopic imaging of the deposition of fine materials at the air-water interface in an impinging jet cell and found that small-sized fibre fines were readily deposited only when calcium soap was present.

The next most selective enrichment in froth was observed for mineral fillers. This can be explained by the same mechanism as for fibre fines, because the PCC filler has been observed to be deposited at the air-water interface even in the absence of calcium soap. The concentration of calcium soap had a negligible effect on the deposition, however. (Drabek *et al.* 1998). The fact that the selectivity of mineral fillers was lower than that of fibre fines might arise from their smaller size or more hydrophilic surface in recovered paper suspensions. Diminishing particle size (in the range of 1-50 µm) has been shown to have reducing tendency to attach onto the bubble (Heindel & Bloom 1997, Somasundaran & Zhang 1998, Thompson 1997). The dispersion and coating agents present in recovered paper suspensions are able to modify the surfaces of fillers to render them more hydrophilic which can explain as well the observed results (Heimonen & Stenius 1997, Letscher & Sutman 1991, Liphard *et al.* 1991).

Short and long fibres were seen to become enriched in the pulp suspension (the accept from flotation), which means that their content in the pulp suspension increases in the course of flotation. The long fibres had tendency to be retained in the accept to a greater extent than the short fibres, indicating that greater the size of the fibre material, the less is removed in flotation, which is in accordance with the results observed by Drabek *et al.* (1998).

The fibre fines content (14%) was about half of the mineral filler content (29%), and for that reason the mineral fillers brought about the most notable alteration in pulp composition. This observation is in accordance with Carré *et al.* (2001), Hamm (2010), Korpela (1996) and Süss *et al.* (1994), who have shown that the reject contains a high amount of ash. The impact of fibre fines on pulp

composition is higher on occasions when its content is higher in the feed to the flotation process.

In addition to flotation, the washing stage (if present in the deinking line) and the laboratory analysis of attached ink can alter the pulp composition. Washing removes fine materials (<100 µm), the removal of particles having a diameter less than 40 µm being particularly efficient (Gilkey 1998). The analysis of attached ink is commonly performed by hyperwashing (Carré *et al.* 1994) using a wire screen having an aperture of 105 µm. This hyperwashing changes the pulp composition even more than deinking washing, because all the fine materials are deliberately washed out.

4.2 Effect of fine materials on light scattering at 700 nm

The results in relation to fine materials affecting light scattering at 700 nm, were studied using three methods: (1) by adding fresh mineral fillers, (2) by washing out fibre fines from a virgin pulp suspension and (3) by washing out fine materials from unprinted and printed papers. Finally, the effect of fine materials on light scattering at 700 nm was compared with their effect in the luminous region, which is commonly used in paper measurements.

4.2.1 Fresh mineral fillers

The light scattering coefficients at 700 nm as a function of added PCC filler for pulps with a variable ink content are shown in Fig. 3. The ink content and fine material content were higher for the pulp samples containing a higher percentage of unwashed ONP. The light scattering coefficients are shown only for samples having an opacity of less than 97%, *i.e.* 75% ONP and 100% ONP samples, those with the highest ink content, are not shown. The light scattering values for mixed pulps (25% ONP and 50% ONP) were higher than those observed for hyperwashed newsprint due to fine materials originating from unwashed ONP. The addition of PCC filler improved the light scattering coefficient at 700 nm regardless of the ink content.

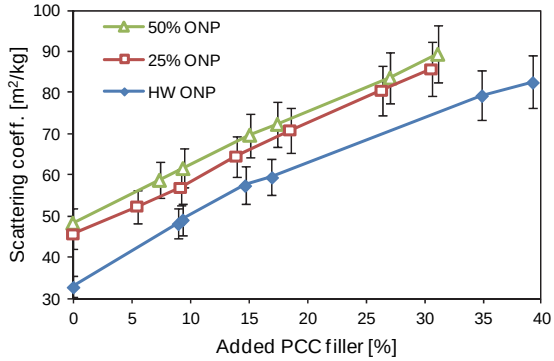

Fig. 3. Light scattering coefficients at 700 nm as a function of added PCC filler. Error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Paper II, published by permission of TAPPI.

The light scattering coefficients at 700 nm for hyperwashed newsprint pulps are shown as a function of added PCC, GCC and kaolin fillers in Fig. 4. The particle size distributions of the aforementioned fillers are shown in Fig. 5.

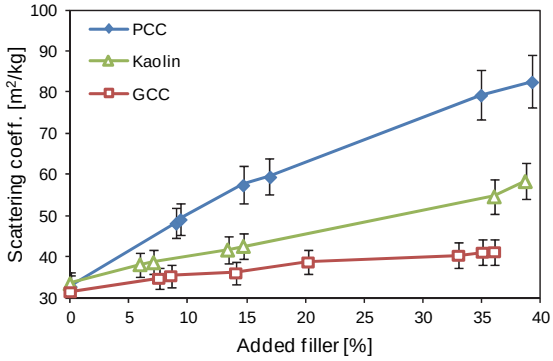

Fig. 4. Light scattering coefficients at 700 nm as a function of added PCC, GCC and kaolin fillers. Error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Modified from Papers II and III.

The addition of fresh fillers improved the light scattering properties of the pulp, where the highest increase was observed for PCC, which had the lowest average particle size (3.1 µm). The lowest improvement in light scattering was observed for GCC, which had the broadest particle size distribution (the average size of 5.0 µm). Kaolin filler had the highest average particle size (7.9 µm) of the studied

fillers and the impact of kaolin on light scattering was in between those of PCC and GCC.

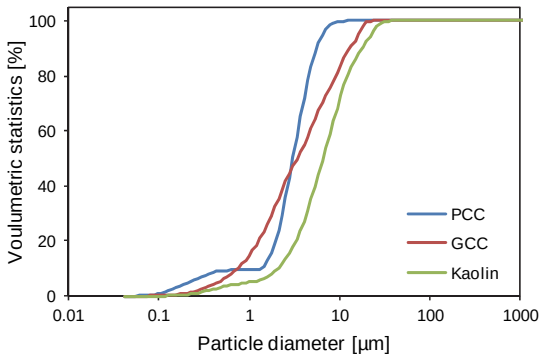

Fig. 5. Volumetric size distribution of fillers used in Papers II and III, as determined with Beckman Coulter LS 13320. The average particle sizes of the PCC, GCC and kaolin fillers were 3.1, 5.0 and 7.9 µm, respectively.

4.2.2 Fresh fibre fines

Light scattering coefficients at 700 nm are shown as a function of fibre fines originating from thermomechanical, hardwood kraft and softwood kraft pulps in Fig. 6. The variation in fibre fines content was achieved by the gradual washing of virgin pulps. Thermomechanical pulp fines improved the light scattering at 700 nm, the increase being about the same as was observed for kaolin filler (Fig. 4). Kraft pulp fines had a negligible effect on light scattering properties at 700 nm, as the coefficients varied within the error bars. Furthermore, the amount of fines was much lower than in thermomechanical pulp.

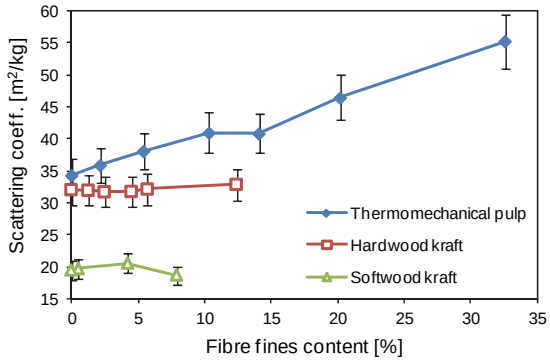

Fig. 6. Light scattering coefficients at 700 nm as a function of fibre fines originating from virgin pulps. Error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Paper III, published by permission of APPITA.

4.2.3 Fine materials from unprinted and printed papers

The mineral fillers and fibre fines found in deinked pulp production originate from recovered paper. Unprinted and printed papers were studied to ascertain that fine materials which have gone through the papermaking process also affect the light scattering properties of pulp. The light scattering coefficient at 700 nm is shown as a function of fine materials originating from unprinted newsprint, supercalendered and lightweight coated papers in Fig. 7. The variation in fine material content was achieved by gradual washing of pulps produced from unprinted papers. All the fine materials studied improved light scattering at 700 nm, the improvement being comparable to obtained with kaolin filler (Fig. 4) and thermomechanical pulp fines (Fig. 6).

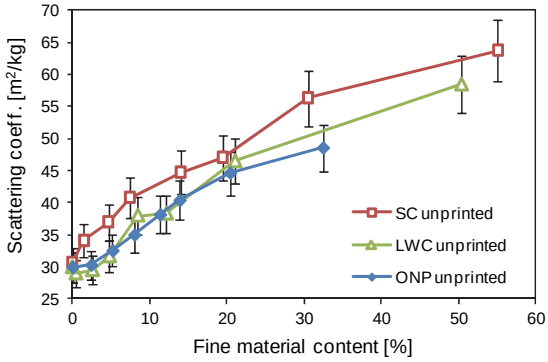

Fig. 7. Light scattering coefficients at 700 nm as a function of fine materials originating from unprinted papers. Error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Paper III, published by permission of APPITA.

The light scattering coefficient at 700 nm is shown as a function of fine materials originating from printed newsprint, supercalendered and lightweight coated papers in Fig. 8. The variation in fine material content was achieved by gradual washing of pulps produced from printed papers like with the unprinted papers. All the fine materials studied improved light scattering at 700 nm.

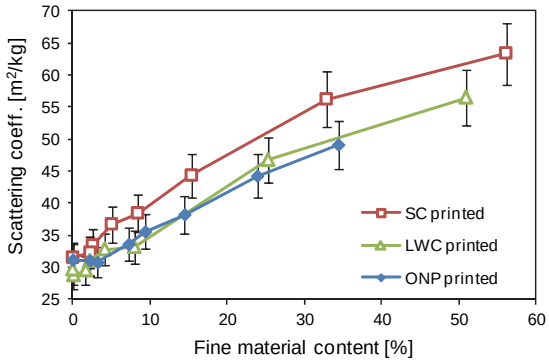

Fig. 8. Light scattering coefficients at 700 nm as a function of fine materials originating from printed papers. Error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6% (Unpublished results).

The fine material content and ash content of the unprinted and printed papers studied in Figs. 7 and 8 are presented in Table 3 with the light scattering and absorption coefficients of entire pulps and hyperwashed pulps. It is expected that

the printing (light absorption) reduces light scattering, and this effect becomes significant (more than 10%) when the light absorption coefficient increases by 7 m^2/kg according to studies done by Neuman (2005) and Rundlöf & Bristow (1997). The studied entire pulps samples of printed papers had high enough absorption coefficient (Table 3) so that their light scattering coefficient should have been lower by 10%–20% than that of unprinted papers. However, this high reduction in light scattering was not observed; for printed newsprint a slightly higher light scattering coefficient was observed while supercalendered paper had slightly lower and the highest decrease (3.4%) was observed for lightweight coated paper. Apparently natural variation in paper quality may contribute to observed results, despite the printed papers were taken from the same batch as unprinted papers. It remains, therefore, unclear if the printing reduces the light scattering as much as supposed by Neuman (2005) and Rundlöf & Bristow (1997) and further research can be recommended to clear the issue. Nevertheless it has to be highlighted that the fine materials alter the light scattering much more efficiently than the light absorption by printing ink. The removal of fine materials from newsprint, supercalendered and lightweight coated papers reduced light scattering coefficient by 18, 32 and 27 m^2/kg, respectively, which in the percentage basis corresponds to 36%, 50% and 48%. Therefore it can be concluded that the changing fine material content affects the light scattering properties of pulp at 700 nm.

Table 3. Characteristics of the papers studied in Figs. 7 and 8.

	Fine material content[1] [%]	Ash content [%]	Light scattering coefficient at 700 nm [m^2/kg]		Light absorption coefficient at 700 nm [m^2/kg]	
	Entire pulp	Entire pulp	Entire pulp	Hyperwashed	Entire pulp	Hyperwashed
ONP unprinted	32.4	10.1	48.5	29.9	2.43	0.77
ONP printed	34.3	10.3	49.1	31.2	13.2	3.28
SC unprinted	55.1	35.0	63.7	30.6	0.77	0.34
SC printed	56.1	35.1	63.3	31.5	13.9	1.74
LWC unprinted	50.4	35.2	58.4	29.9	0.44	0.28
LWC printed	50.9	33.9	56.4	29.6	16.5	0.82

[1] passing through a 150-mesh wire screen

The effect of paper type on the light scattering coefficient can be estimated using the data given in Table 3. The estimation can be justified by the fact that the studied papers represented average recovered paper, as it has been reported that the mineral content of newsprint and uncoated and coated wood-containing

papers varies in the ranges 3%–15%, 5%–35% and 28%–48%, respectively (Haarla 2000). The recovered paper stream entering a deinking mill is a mixture of various paper types, and it is expected that the ONP to OMG ratio will easily vary, but in small steps. To give an idea of this, if an ONP/OMG ratio of 70/30 were to change to 50/50 we would observe a 2.2 m^2/kg improvement in the light scattering coefficient (comparison based on the coefficients of printed papers). This implies that recovered papers do not cause any significant change in light scattering properties during normal mill operations. Changes in the light scattering coefficient can be significant, however, if the recovered paper composition changes substantially. For example, if newsprint was exchanged for supercalendered paper the light scattering coefficient of the pulp suspension would improve by 14 m^2/kg. Such a great change in recovered paper type can be observed in research and development projects, for instance, highlighting the fact that the fine materials affect the light scattering coefficient in practice. Thus a single value for the light scattering coefficient is unrepresentative when used in the determination of ink content if the grade of the paper changes.

In all probability it is the determination of attached ink by hyperwashing that causes the biggest changes in fine material content, as discussed at the end of Section 4.1. The reason is that all the fine materials are deliberately washed out during hyperwashing. By reference to Table 3 it can be estimated that hyperwashing would lower the light scattering coefficient of newsprint, supercalendered and lightweight coated papers by 18, 32 and 27 m^2/kg, respectively. Such marked changes clearly highlight the fact that a single value for the light scattering coefficient is unrepresentative of either an entire pulp or a hyperwashed pulp.

4.2.4 Correlation between light scattering coefficients

The correlation between light scattering coefficients measured using a wavelength of 700 nm and a luminous weighting function is shown in Fig. 9. The correlation is based on all the pulp samples studied in Papers II and III. A linear correlation with luminous light scattering properties was obtained, even though the response was 1.2-fold for the latter.

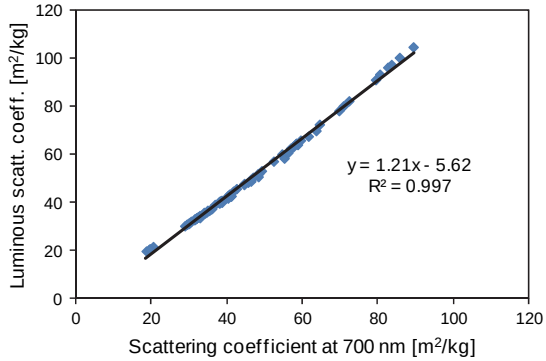

Fig. 9. Correlation between the luminous light scattering coefficient and the light scattering coefficient at 700 nm. Modified from Paper II.

The correlation obtained implies that all the fine materials affecting luminous light scattering also scatter light at 700 nm and that the effect of fine materials on light scattering properties at 700 nm is in accordance with earlier studies. Hubbe *et al.* (2008), Middleton *et al.* (1994), Lopes Velho (2002) and Thomsen (1994) have all shown that mineral fillers improve luminous light scattering, while Allison & Graham (1989), Corson *et al.* (1991), Hubbe *et al.* (2008), Moss & Retulainen (1997) and Retulainen *et al.* (2002) have shown that thermomechanical pulp fines also improve it. Furthermore, it has been shown that kraft pulp fines have a negligible impact or even reduce the luminous light scattering properties of pulp (Hubbe *et al.* 2008, Marton & Alexander 1963, Moss & Retulainen 1997). This highlights the fact that fine materials affect the light scattering properties of pulp at 700 nm, which is used during the determination of ink content. The same also applies to ink content measured at 950 nm, as it has been shown that fine materials also affect near-infrared light scattering (Körkkö *et al.* 2011). The impact of fine materials on near-infrared light scattering has nevertheless been shown to be about 20% less in magnitude.

4.3 Effect of fine materials on measured ink content

Up to now it has been shown that fine materials affect light scattering properties at 700 nm. In this section, the effect of fine materials on the determination of ink content will be explored using a constant light scattering coefficient and measured light scattering coefficients. For the former case two procedures were tested and for the latter only Eq. 6 was tested, as the L&W procedure (Eq. 7) is only recommended for instances when opaque pads have been prepared.

4.3.1 Ink content with constant light scattering

The measured ink content of hyperwashed newsprint as a function of added PCC filler is shown in Fig. 10. The ink content was measured using Eqs. 5 and 6 and a constant light scattering coefficient of 42 m^2/kg. The ink content measurements deviated substantially from the mass balance prediction (Eq. 20) with variations in the PCC filler content.

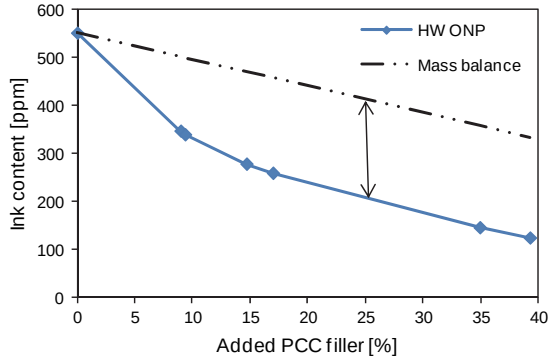

Fig. 10. Ink content (Eq. 6), determined with a constant light scattering coefficient of 42 m^2/kg as a function of added PCC filler. The development of ink content in accordance with the mass balance (Eq. 20) is shown with the straight line. The deviation between these two is highlighted with an arrow. Paper II, published by permission of TAPPI.

The percentage deviations (Eq. 21) between the measured ink content and that calculated from the mass balance, as highlighted with an arrow in Fig. 10, are shown in detail for all the pulps studied in Paper II in Fig. 11. The ink content was higher, the higher was the percentage of unwashed ONP pulp. The measured ink content deviated substantially from the mass balance predictions regardless of the

magnitude of that content, the deviation being highest with the highest amount of PCC filler, with a bias of up to 65% in the measured ink content being observed when the light scattering coefficient improved from 33 m^2/kg to 83 m^2/kg. This is a direct result of using an unrepresentative light scattering coefficient for the determination of ink content (Eqs. 5 and 6), as the PCC filler was able to improve the light scattering properties of the pulp. Thus use of a constant light scattering coefficient for determining ink content is unable to account for the changes in fine material content.

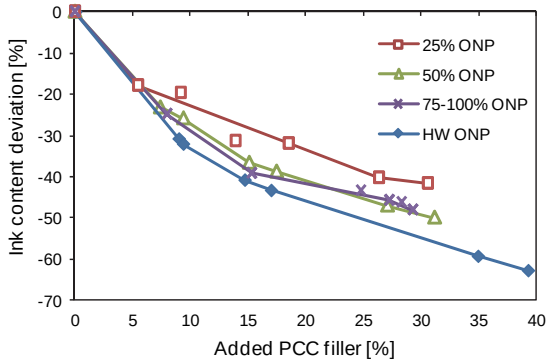

Fig. 11. Percentage deviations in ink content (Eq. 21) as a function of PCC filler. Ink content (Eq. 6) was determined with a constant light scattering coefficient of 42 m^2/kg. Paper II, published by permission of TAPPI.

The determination of ink content can also be based on a special procedure, $RI_{L\&W}$ (Eq. 7). The percentage deviations (Eq. 21) between measured ink content and that calculated from the mass balance for hyperwashed pulps are presented as a function of added PCC, GCC and kaolin fillers in Fig. 12, which shows that the use of this special procedure also resulted in a significant deviation from the mass balance prediction, the highest deviation being observed for PCC and the lowest for GCC. Up to a 70% bias in the measured ink content was observed when the light scattering coefficient improved from 33 m^2/kg to 83 m^2/kg. Likewise, the bias in the ink content as determined in accordance with Eq. 6 is a direct result of using an unrepresentative light scattering coefficient.

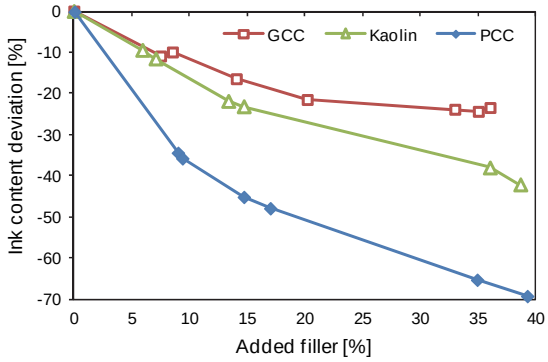

Fig. 12. Percentage deviations in ink content (Eq. 21) as a function of added fillers. Ink content was determined according to the L&W procedure (Eq. 7). Modified from Papers II and III.

The high bias in the ink content results (up to 65%–70%) when a constant light scattering coefficient was used cannot be explained on the basis of the weaknesses of the Kubelka-Munk theory as reviewed in Section 2.2. These weaknesses related to the additive rule governing the light scattering coefficient and the interaction between light scattering and light absorption. Strictly speaking, the determination of ink content with a constant light scattering coefficient uses only the relation between reflectivity and the light absorption coefficient. As the light scattering coefficient is not measured and the additive rule is not used, the reported weaknesses are unable to explain the biases in ink content. Instead, the bias is a result of using an unrepresentative light scattering coefficient.

Although the impact of fine material on the measured ink content was studied separately in this thesis, the discussion can be extended on the basis of the data obtained to cover reflectivity and the light absorption coefficient. The changes in fine material content also affect the reflectivity, as both light scattering and light absorption contribute to reflectivity (Pauler 2002). Even if the measured ink content were reported in the form of light absorption coefficients determined with a constant light scattering coefficient, the same bias would be encountered as with the measured ink content, as has been demonstrated by Körkkö *et al.* (2009). This is easy to understand, as ink content is determined through the light absorption coefficient. It is difficult to predict a representative value for the light scattering coefficient, and therefore this should be measured.

4.3.2 Ink content with measured light scattering

The ink content measured for hyperwashed newsprint as a function of added PCC filler is shown in Fig. 13, where the straight line indicates the mass balance dependence of the ink content (Eq. 20). Ink content (Eqs. 5 and 6) was determined using the measured light scattering coefficient, and the results were in good agreement with the mass balance prediction, as they remained within the boundaries of the error bars, *i.e.* within the TAPPI repeatability ratio of 7.6%.

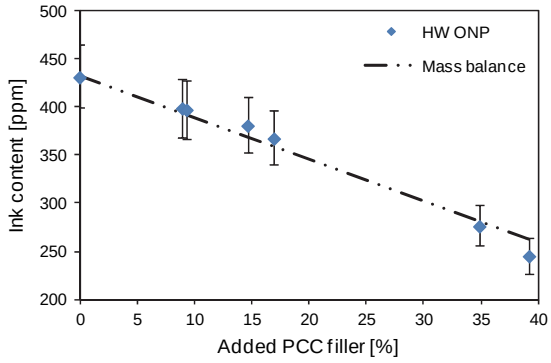

Fig. 13. Ink content (Eq. 6) determined with the measured light scattering coefficient as a function of added PCC filler. The error bars are based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. The development of ink content in accordance with the mass balance (Eq. 20) is shown with the straight line. Paper II, published by permission of TAPPI.

The percentage deviation (Eq. 21) between the measured ink content and that calculated from the mass balance for pulps of variable ink content as a function of added PCC filler is shown in Fig. 14. A positive deviation highlights the fact that the measured value is above the mass balance prediction, while a negative deviation shows the opposite. In most instances (16 out of 21) the percentage deviation was less than the TAPPI repeatability ratio (7.6%). The five samples deviating more than 7.6% from the mass balance had opacities between 95% and 97%. According to ISO 22754 and TAPPI T 567 the opacity needs to be less than 97%, but the high opacity may have still contributed to the error, as it has been shown that the measurement of light scattering and light absorption coefficients becomes increasingly susceptible to error when opacity exceeds 95% (Vahey *et al.* 2007). This is due to fact that when the opacity is high, the single-sheet reflectance comes close to the reflectivity.

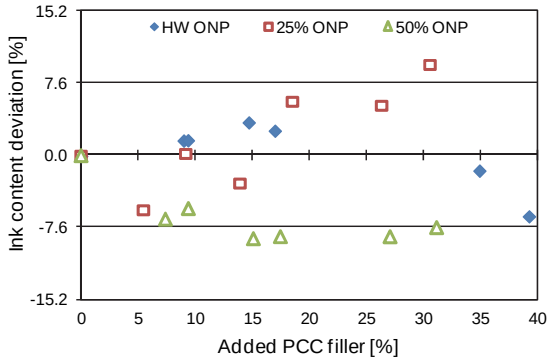

Fig. 14. Percentage deviations in ink content (Eq. 21) as a function of added PCC filler. Ink content (Eq. 6) was determined using the measured light scattering coefficient. The scale of the y axis is based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Paper II, published by permission of TAPPI.

The percentage deviation between measured ink content and that calculated from the mass balance is shown as a function of added PCC, GCC and kaolin fillers in Fig. 15. The measured ink content was in good agreement with the mass balance predictions, as only in two instances were the boundaries of the error bars (7.6%) exceeded. These two samples had the highest load of kaolin filler (over 30%), but their opacity remained well below 95%, so that this could not explain the deviation.

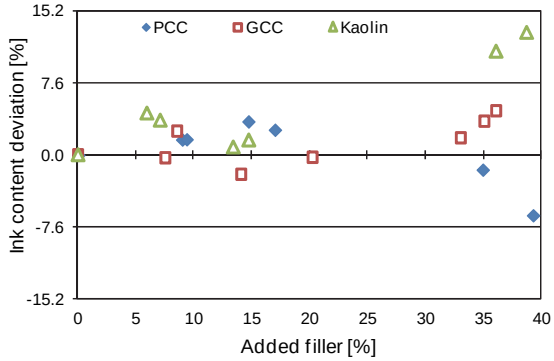

Fig. 15. Percentage deviations in ink content (Eq. 21) as a function of added fillers. Ink content (Eq. 6) was determined using the measured light scattering coefficient. The scale of the y axis is based on a measured TAPPI repeatability ratio (Eq. 23) of 7.6%. Modified from Papers II and III.

When the measured light scattering coefficient is used for determining ink content, the fine materials changing the light scattering properties of the pulp are taken into account and the deviations observed are the result of random error. The deviation is in most instances less than 10%, which is substantially lower than the deviation of up to 65%–70% observed when a constant light scattering coefficient was used. Measurement of the light scattering coefficient can therefore be recommended, as it results in a much more accurate determination of ink content when the fine material content varies.

4.3.3 Calculation of free ink content and removal efficiencies

It is now evident that changes in the fine material content will affect the light scattering coefficient at 700 nm and also the determination of ink content with a constant light scattering coefficient. The free ink content (Eq. 10) is presented in Fig. 16 as a function of the mass reject ratio in the pilot-plant flotation described in Paper I. Three levels of free ink content are presented, which were calculated from Eq. 7 using a constant light scattering coefficient for determining the light absorption coefficient, from Eq. 6 using a constant light scattering coefficient for determining the light absorption coefficient and from Eq. 6 using the measured light scattering coefficient for determining the light absorption coefficient.

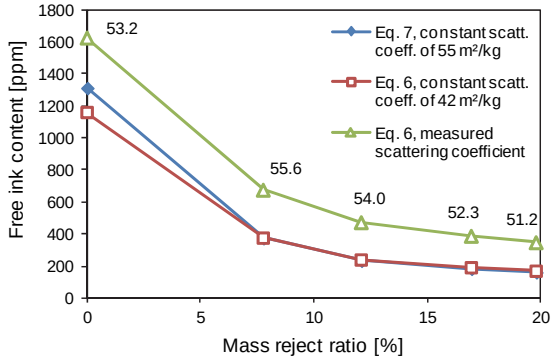

Fig. 16. Calculated free ink content (Eq. 10) during pilot-plant flotation, obtained using three procedures for determining the ink content. Measured light scattering coefficients are also given for the pulp samples. The light scattering coefficient of hyperwashed pulp was 34.6 m^2/kg. Data from Paper I and Körkkö *et al.* (2011).

The free ink content determined with constant light scattering coefficients was very similar for all the accept samples (Fig. 16), but a slight difference was

observed for the feed sample. This was due to the difference between Eq. 6 and Eq. 7, in that the quadratic fit of Eq. 7 results in higher values when the ink content is high. The free ink content determined with the measured light scattering coefficient indicates that that obtained with a constant light scattering coefficient is too low. This is a consequence of the differing light scattering properties of non-deinked and fines-free hyperwashed pulps, as the light scattering coefficient differed by 22 m^2/kg between these samples (Körkkö *et al.* 2011).

The free ink removal efficiencies and Nelson's selectivity indices shown in Fig. 17 were determined from the free ink content values (Fig. 16), and curves illustrating Nelson's selectivity index are also shown, these having been obtained in a similar manner to those for the pulp components studied in Section 4.1. The removal efficiencies based on ink content determined in accordance with Eq. 7 are the same as in Paper I. These removal efficiencies can be compared with those obtained with the other two methods used for measuring ink content in Fig. 16. The removal efficiencies were lower when the measured light scattering coefficient was used for the determination of ink content, which results from the higher free ink content. Thus the bias in ink content due to the use of an unrepresentative light scattering coefficient results in an overestimation of free ink removal during flotation.

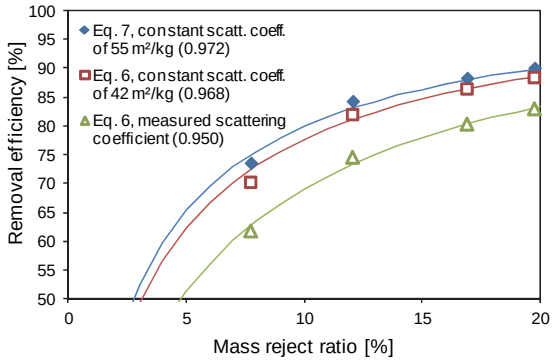

Fig. 17. Free ink removal efficiencies calculated with various ink content measurement procedures during the pilot-plant flotation. Nelson's selectivity indices obtained by fitting a curve (Eq. 15) to the experimentally observed data are shown in parentheses. Modified from Paper I.

The removal efficiencies (Fig. 17) were in good agreement with the selectivity curve whether or not the light scattering coefficient was measured. This indicates

that the light scattering properties of pulp are not changed during flotation as much as one might expect. It was observed separately (Körkkö *et al.* 2011) that the light scattering coefficient improved (by 2 m^2/kg) at low mass reject ratios due to the removal of printing ink. The reason for this is that not only the fine materials but also the printing ink will contribute to light scattering (Neuman 2005, Rundlöf & Bristow 1997). The removal of printing ink reduces the light absorption and therefore improves the light scattering coefficient. At the same time, fine materials are removed, which reduces the light scattering. A reduction in the light scattering coefficient was observed at mass reject ratios higher than 10% and the light scattering coefficient was 2 m^2/kg lower at a mass reject ratio of 20% than in the feed into flotation. As the light scattering coefficient did not decrease linearly during flotation, and as the change was reasonably minor, this would explain why the removal efficiencies were in good agreement with the selectivity curve whether or not the light scattering coefficient was measured.

In hyperwashing all the fine materials are washed out, which causes a much greater change in the light scattering coefficient than does printing ink. The underestimation of free ink content and overestimation of selectivity can be avoided by determining the ink content from the measured light scattering coefficient. We have observed that in some instances the attached ink content can be even higher than the ink content of deinked pulp when a constant light scattering coefficient is used (Körkkö *et al.* 2008). These abnormal ink content levels would result in negative free ink content values and removal efficiencies higher than 100%, which would evidently be incorrect. Furthermore, we have observed that the ink content of deinked pulp can increase during the washing-thickening stage (Körkkö *et al.* 2008). Due to the substantial loss of fine materials at that stage, the ink content could have been expected to decrease or remain the same, but not to increase. Again, this results from using an unrepresentative light scattering coefficient for determining the ink content, and biased ink content results can be avoided by using the measured light scattering coefficient for this purpose.

4.4 Influence of the test medium preparation method

Sheet preparation on a wire screen is known to produce a low retention of fine materials, including printing ink, by comparison with opaque pads formed on filter paper (Dorris 1999, Haynes 2000, Lévesque *et al.* 1995, Lévesque *et al.* 1998, McKinney 1988, Scott 1993). In this thesis, an opaque pad formed on filter

paper and a low-grammage sheet formed on a wire screen were used as a reference for evaluating the applicability of a relatively novel sheet preparation method, *i.e.* low-grammage sheet preparation on filter paper.

4.4.1 Fine materials

Ash and fine material content figures are presented in Table 4 for the pulp suspension, an opaque pad formed on filter paper, a low-grammage sheet formed on a wire screen and a low-grammage sheet formed on filter paper. Preparation of the sheet on a wire screen resulted in a 14%-unit decrease in ash content and a 25%-unit loss of fine materials, while the results for a sheet formed on filter paper were about 10%-units higher for ash content and about 20%-units higher for fine material content. The ash content and fine material content results for the opaque pad and low-grammage sheet formed on filter paper were close to each other. Thus, high retention can be achieved by preparing a low-grammage sheet on filter paper.

Table 4. Ash content and fine material content results obtained for a pulp suspension and various test medium preparation methods. The TAPPI repeatability values are based on Eq. 22.

Analysis	Pulp suspension	Pad formed on paper	Sheet formed on wire screen	Sheet formed on filter paper
Ash content [%]	22.3 ± 1.5	18.1 ± 0.2	8.0 ± 0.3	17.7 ± 1.2
Fine material content[1] [%]	48.4 ± 0.7	41.9 ± 1.8	23.0 ± 0.9	44.8 ± 2.5

[1] passing through a 120-mesh wire screen

4.4.2 Light scattering coefficient at 700 nm

Light scattering coefficients at 700 nm for low-grammage sheets (opacity less than 97%) are presented in Fig. 18. Use of filter paper in the sheet preparation resulted in a 7.2 m^2/kg higher light scattering coefficient, a rise that proved statistically significant, $p < 0.0001$. The higher light scattering coefficient for sheet preparation on filter paper is in accordance with the higher ash content and fine material content (Table 4).

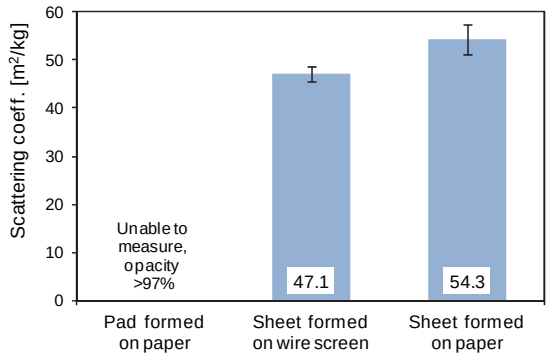

Fig. 18. Light scattering coefficients at 700 nm for various test medium preparation methods. The error bars are based on measured TAPPI repeatability ratios (Eq. 23), which were 3.4% and 5.6% for a sheet formed on a wire screen and a sheet formed on filter paper, respectively. Paper IV, published by permission of TAPPI.

4.4.3 Reflectivity at 700 nm

Reflectivity values at 700 nm are presented in Fig. 19 for a pad formed on filter paper, a low-grammage sheet formed on a wire screen and a low-grammage sheet formed on filter paper. The low-grammage sheet preparation on filter paper resulted in about 5%-units lower reflectivity, which differed statistically significantly from the sheet prepared on a wire screen ($p < 0.0001$). Ink removal during test medium preparation is especially detrimental, as these test media are used for quantifying the ink content. The more printing ink is lost, the less the result represents the original state of the pulp suspension of which the ink content is of interest.

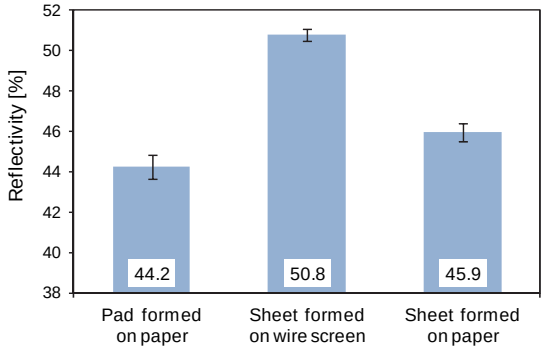

Fig. 19. Reflectivity at 700 nm for various test medium preparation methods. The error bars are based on measured TAPPI repeatability ratios (Eq. 23), which were 1.3%, 0.60% and 0.94%, for a pad formed on filter paper, a low-grammage sheet formed on a wire screen and a low-grammage sheet formed on filter paper, respectively. Paper IV, published by permission of TAPPI.

The lowest reflectivity was found for the pad, this being slightly lower (by 1.7%-unit) than the reflectivity of the sheet formed on filter paper (Fig. 19). The t-test showed that this difference was statistically significant ($p < 0.0001$). High ink retention is therefore achieved when filter paper is used for test medium preparation, although the highest retention of all is achieved when opaque pads are formed on filter paper. Formation of a thicker mat (higher grammage) during test medium preparation has been shown to improve the retention of fine materials, including printing ink (Dorris 1999, Haynes 2000, Lévesque *et al.* 1995, Lévesque *et al.* 1998, McKinney 1988, Scott 1993). Even though high-retention filter paper (1–2 µm) was used for preparing the low-grammage sheet, the relative thinness of the sheet resulted in a slight loss of printing ink.

4.4.4 Ink content at 700 nm

The ink content results obtained with measured light scattering coefficients are presented in Fig. 20. The ink content of the opaque pad formed on filter paper is not presented, as the opacity was higher than 97%. The use of filter paper for sheet preparation resulted in a substantially higher ink content (1.5-fold) that was confirmed to be statistically significant ($p < 0.0001$). Thus the ink content can be

determined reliably based on the measured light scattering coefficient of sheets formed on filter paper, which also achieves a high retention of fine materials.

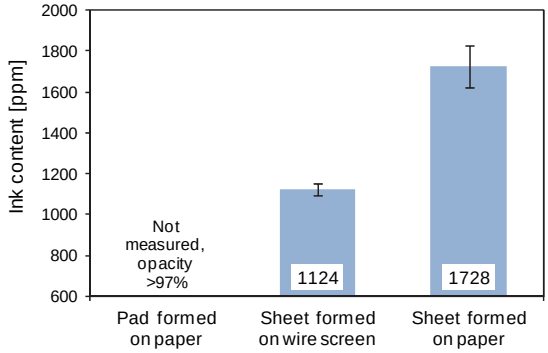

Fig. 20. Ink content (Eq. 6) determined from the measured light scattering coefficient and reflectivity. The error bars are based on measured TAPPI repeatability ratios (Eq. 23), which were 2.8% and 5.9% for a sheet formed on a wire screen and a sheet formed on filter paper, respectively. Paper IV, published by permission of TAPPI.

The sheet formed on filter paper resulted in a good compromise between high retention of printing ink and other fine material, at the same time enabling the ink content to be determined with the measured light scattering coefficient. The advantage of the opaque pad preparation is that the retention of printing ink is slightly higher, but its weakness is that the light scattering coefficient cannot be measured due to the high opacity of the pad. For this reason it seems that the most representative values would be achieved by measuring the reflectivity from an opaque pad and the light scattering coefficient from a sheet prepared on filter paper. This would be especially useful when dealing with highly fragmented printing ink, *e.g.* recovered paper having flexographic printing on it, as superior retention of printing ink can be achieved by preparing opaque pads on membrane filter foils (Körkkö *et al.* 2012).

4.4.5 Repeatability

The random variation during sheet preparation can be estimated from the repeatability ratio results. The repeatability ratios for reflectivity, which is measured from a stack, were very good by comparison with those for the light scattering coefficient, which were measured from a single-sheet. It thus seems that the majority of the inconsistencies in the repeatability ratio for ink content

arise from the measurement of a single sheet (light scattering coefficient). Images of single sheets formed on a wire screen and on filter paper are shown in Fig. 21, where the bright pixels indicate that more light has passed through the sheet, while at the darker points less light has been transmitted through the sheet. In general a sheet formed on filter paper is darker, as a result of the higher retention of printing ink.

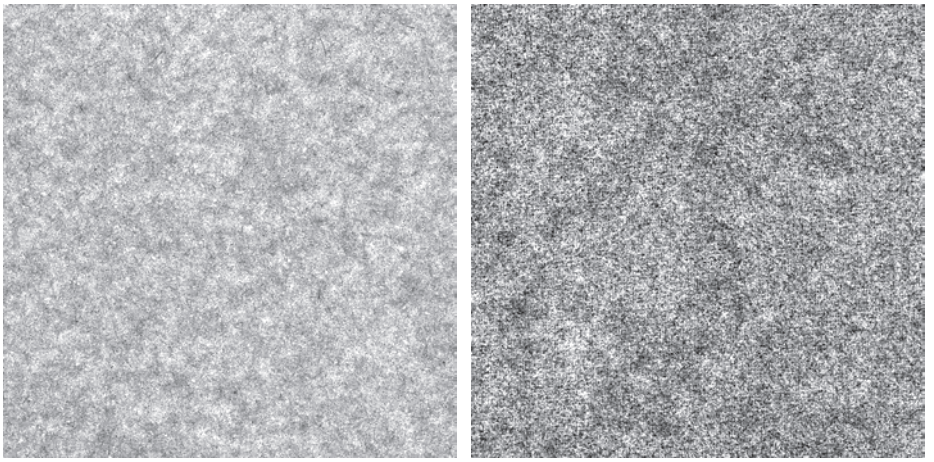

Fig. 21. Grey-scale images of a sheet formed on a wire screen (left) and a sheet formed on filter paper (right). The images were taken with an Epson 1680Pro scanner in light transmission mode and they are at their actual size of 60 mm by 60 mm. Paper IV, published by permission of TAPPI.

Slightly higher variation in grey shades can be observed visually in the sheet formed on filter paper than in the sheet formed on a wire screen, indicating lower uniformity for the first-mentioned. This visual observation is in agreement with the repeatability ratio, which was nearly double for the sheet prepared on filter paper. The low consistency during sheet preparation on a wire screen (see Section 3.2.2) and mixing may contribute to better observed repeatability. This can be compared with the results obtained by Dorris (1999), who observed that standard sheet preparation results in better repeatability than the preparation of opaque test media. Higher repeatability ratios (lower precision) can still be considered acceptable, as ink content values were higher for the sheets formed on filter paper, thus in all probability representing the initial state of the pulp more accurately.

5 Conclusions

The amount of printing ink in a pulp suspension is commonly evaluated in terms of reflectance-based ink content. The ink content is determined from reflectivity and the light scattering coefficient at 950 nm or 700 nm using the Kubelka-Munk theory. In deinking applications the ink content is commonly determined on the basis of a constant light scattering coefficient.

Flotation and washing alters the pulp composition by removing fine materials. Thus the fine material content depends on yield losses in flotation or washing and the composition of the raw material feed, *i.e.* the higher the fine material content, the higher is its impact. Removal efficiency and selectivity in flotation are higher for fibre fines than for mineral fillers, and as printing ink also affects light scattering at 700 nm, flotation performed with reasonable losses (less than 20%) has only a minor effect on the light scattering properties.

Fresh PCC, GCC and kaolin fillers and thermomechanical pulp fines improve the light scattering coefficient at 700 nm, as also do fine materials originating from newsprint, supercalendered and lightweight coated papers which have gone through the papermaking process. Kraft pulp fibre fines have a negligible influence on light scattering at 700 nm, however, and furthermore, their content is low in kraft pulps. Generally, all the fine materials that improve luminous light scattering also improve light scattering at 700 nm, but at about a 20% lower level.

Procedures employing a constant light scattering coefficient are unable to account for changes in fine material content, which results in a biased ink content. This is a result of the ability of fine materials to alter the light scattering coefficient. A bias of up to 65% in the measured ink content is observed with the commonly used procedure (Eq. 6) and up to 70% with the procedure proposed for the L&W Elrepho device (Eq. 7) when the light scattering coefficient changes from 33 m^2/kg to 83 m^2/kg. However, when the coefficient is measured and used for the determination of ink content, the changes in fine material content are already taken into account. In most instances, the ink content values obtained with measured light scattering coefficients are in good agreement with the mass balance predictions, and only in a few instances is the bias higher than 10%, which is considerably less than that observed with a constant light scattering coefficient.

Retention during sheet preparation can be improved substantially by using a filter paper, while at the same time the grammage of the sheet can be kept low enough that the light scattering coefficient can be measured. With the non-

deinked pulp suspension studied here, the ink content was 1.5-fold when a filter paper was used in sheet preparation rather than a wire screen. The use of a filter paper resulted in slightly poorer uniformity in the sheet, as the repeatability ratio of the ink content nearly doubled. A higher repeatability ratio (lower precision) can still be considered acceptable, however, as sheet preparation on filter paper retains more of the printing ink and thus serves better to represent the initial state of the pulp suspension. The retention of printing ink is slightly less for a low-grammage sheet than for an opaque pad, however, despite a filter paper being used in both test medium preparation methods, as higher grammage improves the retention. The most representative measure of ink content can therefore be achieved when the reflectivity is measured from an opaque pad formed on a filter paper and the light scattering coefficient from a low-grammage sheet formed on a filter paper. This is especially advantageous when the pulp suspension contain highly fragmented printing ink. It should be stated, however, that a sheet formed on filter paper results in a good compromise, because reasonably high retention of printing ink is obtained and the light scattering coefficient can be measured from the prepared sheet.

On the basis of the work conducted here it can be recommended that the light scattering coefficient should be measured during the determination of reflectance-based ink content, and that this should be done from a low-grammage sheet formed on filter paper, because it will then represent better the initial state of the pulp suspension. Once measured, the resulting value can be used as a constant provided the pulp composition and recovered paper remains the same. Even when using the same raw material, separate constant values for the light scattering coefficient are needed for whole pulp and hyperwashed pulp. The representativeness of a constant light scattering coefficient should always be checked by measuring it.

The impact of fine materials on light scattering was studied in this thesis using fresh fillers and fibre fines while also fine materials originating from unprinted and printed papers were studied. The impact of printing ink on light scattering could not be fully revealed as the natural variation of paper quality might have contributed to the results. The impact was seen less than that reported in the literature and for that reason further research can be recommended to clear the issue between printing ink and light scattering by examining the effect of printing ink contaminated fine materials on light scattering.

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