# PROYECTO FIN DE CARRERA

INGENIERÍA QUÍMICA

# ESTUDIO DE LA FLOTACIÓN PARA EL DESTINTADO DE LA FRACCIÓN DE FIBRA CORTA EN PULPA PROCEDENTE DE PAPEL RECUPERADO

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# TABLA DE CONTENIDOS

# 1 INTRODUCCIÓN Y OBJETIVOS

El reciclado de papel es un proceso cada vez más importante dentro de la industria papelera, debido tanto a razones económicas como medioambientales. De hecho en los últimos 15-20 años, ha aumentado el uso de papel reciclado para la fabricación de papel comercial: como el usado en pañuelos, periódicos, etc. Este papel reciclado requiere unas condiciones mínimas de algunas de sus propiedades para su uso en papeles de alta calidad, como por ejemplo brillo, contenido de cenizas o tinta residual. Actualmente, alrededor de la mitad del papel y el cartón producido mundialmente se obtiene con papel recuperado.

El principal componente del papel recuperado que ha de retirarse es la tinta, debido a que produce una reducción en el brillo del papel y su blancura. Por ello, los procesos de destintado de papel han sufrido progresivamente mejoras, y las líneas de reciclado cada vez son más complejas. Como parte de dichas mejoras, diversas alternativas de destintado están siendo consideradas con el objetivo de mejorar el proceso.

En este proyecto, se realizó un estudio en detalle de la flotación para la eliminación de la tinta en la fracción de fibra corta de pasta procedente de papel recuperado, dentro de una nueva propuesta de proceso de destintado. Esta propuesta se muestra esquemáticamente en la Figura 1. Básicamente, el papel recuperado (mezcla entre periódicos y revistas) es pulpeado durante un tiempo muy corto y posteriormente separado en fracción de fibra corta (o fracción de finos) y fracción de fibra larga. Con este corto pulpeado se evita que la tinta sufra una excesiva fragmentación y vuelva a depositarse en las fibras. Tras el fraccionamiento, la fracción de fibra larga pasa por un segundo pulpeado, y la fracción de fibra corta se limpia mediante un proceso de flotación. Esta fracción de fibra corta ya flotada se puede combinar con la fracción de fibra larga y ser realimentada a una segunda parte del proceso de producción de papel, para mejorar la limpieza y el brillo de la pasta de papel. (Körkkö *y cols.*, 2010).

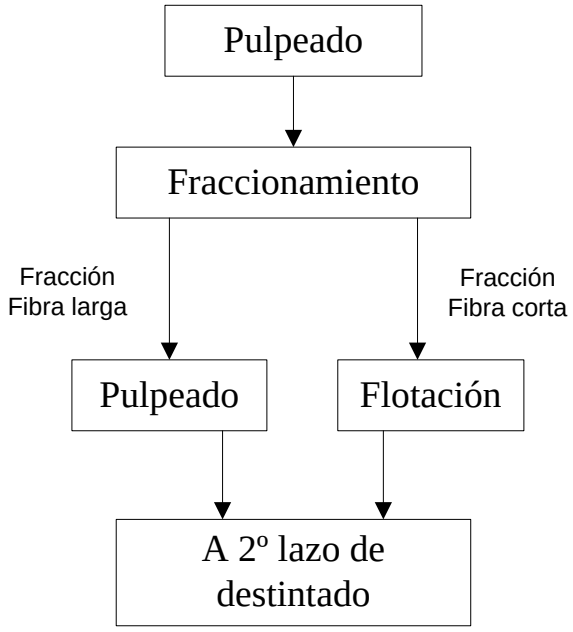


**Figura 1.** Diagrama del destintado alternativo estudiado (Körkkö *y cols.*, 2010).

El objetivo de este proyecto fue obtener más información sobre la flotación de la fracción de fibra corta dentro de la propuesta de destintado de la Figura 1. Para ello, se realizaron diferentes experimentos de flotación variando sus propiedades en búsqueda de los valores óptimos, con los que la muestra consiguiera un mayor brillo con la menor pérdida de fibras posible. Todo el trabajo se llevó a cabo en el Departamento de Ingeniería de Procesos y Medio Ambiente de la Universidad de Oulu (Finlandia), dentro del Laboratorio de Ingeniería de Fibra y Partículas, y su memoria original se puede consultar en el Anexo 1 de este proyecto fin de carrera.

# 2 ANTECEDENTES

## 2.1 LÍNEA DE DESTINTADO CONVENCIONAL

El propósito del destintado es eliminar la tinta y otras substancias contaminantes que pueden afectar el proceso de fabricación de papel o sus propiedades finales. Por este motivo, los procesos en los que se emplea papel recuperado son más complejos que los que usan fibras vírgenes, ya que el papel recuperado es una mezcla de varios tipos de papel con cierta cantidad de contaminantes. Las propiedades finales de la suspensión de pasta de papel dependen directamente de la eliminación de contaminantes, de modo que una baja separación de contaminantes lleva a valores bajos de brillo y calidad del producto final (Lassus 2000 pp. 241, Holik 2000 pp.91). En la página 9 del Anexo 1 se presenta un diagrama con las etapas del proceso de fabricación de papel de periódico.

Las diferentes etapas de una línea convencional de destintado son las siguientes: pulpeado, depuración por tamizado, limpieza por centrifugado, flotación, dispersión, blanqueado y eliminación de agua. Durante el pulpeado se aplican fuerzas mecánicas, térmicas y químicas para despegar las impurezas de las fibras, como la tinta. Otras etapas como la depuración por tamizado, la limpieza por centrifugado y la flotación son etapas de separación en las que se elimina la tinta libre de la suspensión (Eul *y cols.* 1990, Doshi 1997 pp.3). A continuación se explican más detalladamente estas etapas.

### *2.1.1 Pulpeado*

El objetivo del pulpeado es desintegrar el papel en fibras individuales para obtener una suspensión que pueda ser bombeada. Las fuerzas de desintegración aplicadas durante el pulpeado deben de ser mayores que la resistencia del papel o las fuerzas de unión de los contaminantes a las fibras. Pero estas fuerzas de desintegración no deben de ser excesivas para evitar que los contaminantes (como la tinta) se rompan en partículas demasiado pequeñas, lo cual dificulta su posterior eliminación. (Holik 2000 pp. 95-97)

Se necesita que durante el pulpeado se consiga una buena separación entre las partículas contaminantes y las fibras para que posteriormente la suspensión de pasta de papel sea tratada eficientemente. Las fuerzas mecánicas separan la tinta mediante fricción entre fibras y los reactivos químicos consiguen que algunas partículas se vuelvan más hidrófobas. El objetivo es liberar la tinta de las fibras evitando que se vuelvan a adherir a ellas o que se puedan fragmentar. Cuanto más baja es esta fragmentación de la tinta, mayor brillo tiene la suspensión de papel tras la flotación (McKinney 1999 pp. 107, Beneventi *y cols.* 2005, Holik 2000 pp. 151-153). En las páginas 10 y 11 del Anexo 1, se amplía la información sobre el proceso de pulpeado.

### *2.1.2 Depuración por tamizado*

El objetivo de la depuración por tamizado es eliminar los contaminantes sólidos de la pasta de papel recuperado. Se emplean diferentes pantallas o tamices según el tamaño de partícula y la forma de los contaminantes. Habitualmente se tamiza la misma suspensión varias veces, para evitar en la medida de lo posible la pérdida de fibras. En las páginas 11 y 12 del Anexo 1 se amplía la información de esta fase.

### *2.1.3 Limpieza por centrifugado*

La limpieza por centrifugado es otro proceso de separación que complementa a la depuración por tamizado, y su objetivo es eliminar partículas de la suspensión que puedan afectar la calidad del papel. Como partículas contaminantes nos referimos a arena, piezas de metal, grapas o materiales plásticos. Para una eliminación eficiente, es necesario que los contaminantes tengan una densidad mayor a la del agua. Gracias a la limpieza por centrifugado se pueden eliminar partículas más pequeñas que con la depuración por tamizado. (Holik 2000 pp. 134-135)

### *2.1.4 Flotación*

La flotación es el proceso más común para realizar la separación de la tinta de las fibras sobre las que se encuentra depositada. Es un proceso de separación que se basa en la probabilidad de que burbujas de aire se adhieran a las partículas de tinta y las lleven a la superficie de la suspensión formando una capa de espuma. Este proceso es posible gracias al comportamiento hidrófobo de las partículas permitiendo que puedan ser eliminadas, mientras que las fibras son retenidas en la suspensión. (Holik 2000 pp. 151-152, 241)

Una vez que se ha conseguido una buena separación de la tinta y las fibras en el pulpeado, se introduce un flujo de aire a la suspensión y las partículas repelentes al agua se adhieren a estas burbujas de aire formando una capa de espuma en la superficie de la suspensión, partículas contaminantes como tinta, cargas, pigmentos, etc. La espuma formada se retira de la suspensión o mecánicamente, o por rebose o por extracción al vacío (Holik 2000 pp. 151-153, 244). La flotación se presenta más a fondo tanto en el Apartado 2.3 de este proyecto, como en las páginas 13 y 14 del Anexo 1.

### *2.1.5 Dispersión*

El objetivo de la dispersión es romper los contaminantes a un tamaño en el que no interfieran en el proceso y evitar que se vean a simple vista. La dispersión hace que ciertos contaminantes se hagan más pequeños y se favorezca su eliminación por flotación, ya que mezcla reactivos blanqueantes y despega la tinta todavía adherida a las fibras. (Holik 2000 pp. 185-186)

### *2.1.6 Blanqueado*

La etapa de blanqueado mejora las propiedades ópticas de la pasta de papel destintada, como por ejemplo el brillo y la luminosidad. Los reactivos más utilizados son peróxido de hidrógeno ($H_2O_2$), hidrosulfito de sodio ($Na_2S_2O_4$), cloro ($Cl_2$), dióxido de cloro

($ClO_2$), hipoclorito de sodio (NaOCl) y ozono ($O_3$). La elección del reactivo blanqueante depende de la calidad del producto final. (Ackermann 2000 pp. 307)

### *2.1.7 Eliminación de agua*

La eliminación de agua se lleva a cabo mediante un proceso de filtrado por presión y su objetivo es quitar el agua de la suspensión de pasta de papel incrementando su consistencia (porcentaje de masa de papel seco). Esta eliminación de agua produce un ahorro en espacio para un posterior almacenaje de la pasta de papel. Los equipos más comunes para la eliminación de agua en procesos de papel recuperado son los filtros de disco y las prensas de tornillo. (Holik 2000 pp. 168-169, 171-172)

## 2.2 LÍNEA DE DESTINTADO CON FRACCIONAMIENTO

Como se ha comentado en este proyecto, se ha propuesto el estudio de un proceso alternativo para el destintado. La separación de las fibras de papel, y el posterior tratamiento de blanqueado de sus fracciones por separado (fracción de fibra corta y larga) puede ser una manera eficiente de mejorar la pasta de papel destintada y sus propiedades. La separación de las fibras mediante el fraccionamiento reduce el gasto en reactivos químicos (al tratar las fracciones por separado) y también reduce el consumo de energía, al realizar la dispersión sólo en la fracción de fibra larga (Mäkinen *y cols.*, 2010). Alcanzar las exigencias de brillo y blanqueado del producto final es el reto del los procesos de destintado de papel recuperado (Eul *y cols.* 1989). La línea de destinado con fraccionamiento es un desarrollo de la línea de destintado convencional. Con fraccionamiento, la pasta de papel es separada mediante una separación por tamizado en dos fracciones según el tamaño de las fibras: fracción de fibra larga y la fracción de fibra corta (o fracción de finos). De este modo las fracciones pueden ser tratadas por separado y ser empleadas en papeles de diferente grado o calidad. El principio del fraccionamiento se presenta en la Figura 2.

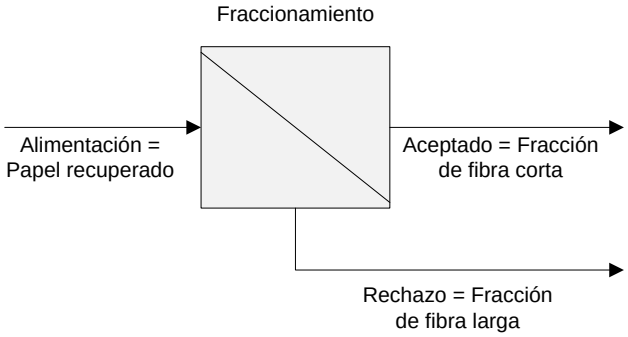


**Figura 2.** Principio de fraccionamiento (Kraschowetz *y cols.*, 2008).

Otro de los motivos de la introducción del fraccionamiento en las líneas de destintado es el incremento del uso de papel recuperado para producir papel y cartón por razones medioambientales. Al aumentar el uso de papel recuperado, se ha aumentado también la cantidad de material no fibroso y contaminantes. Mediante el fraccionamiento, se minimiza el gasto en limpieza y depuración por tamizado debido a que se concentran la mayoría de los contaminantes en una fracción (Kraschowtz *y cols.* 2008, Meltzer 1999 pp. 153). La eficiencia del fraccionamiento depende tres diferentes grupos de factores: propiedades de la fibra (longitud, grosor…), características del equipo de fraccionamiento (tipo de cesta, diseño del rotor) y parámetros de operación (ratio de rechazo, velocidad del rotor). (Meltzer 1999 pp. 156). Esta información se encuentra ampliada en las páginas 16 y 17 del Anexo 1.

El lugar de la etapa de fraccionamiento dentro del proceso de destintado también fue estudiado, como se puede comprobar en las páginas 19, 20 y 21 del Anexo 1.

### *2.2.1 Diferencias entre fraccionamiento y depuración por tamizado*

Si bien físicamente ambos procesos son parecidos, los objetivos son muy diferentes. Así, el objetivo de la depuración por tamizado es la eliminación de partículas y contaminantes no deseados con la menor pérdida de fibras útiles. Por el contrario, el fraccionamiento se centra en la separación de un flujo en dos o más fracciones con diferentes características de fibra. La calidad de una buena depuración por tamizado está determinada por las propiedades del aceptado, con los diversos contaminantes eliminados en el flujo de rechazo. Sin embargo, las propiedades de la fracción de fibra

larga son las que determinan la calidad de un fraccionamiento. En la página 18 del Anexo 1 se muestra una figura con conceptos y diferencias entre depuración por tamizado y fraccionamiento.

### *2.2.2 Propiedades de las fracciones*

Como se ha explicado antes, el fraccionamiento separa las fibras según sus características, como su longitud o flexibilidad. De todos modos, no es posible tener una separación total y estricta de las fibras cortas y largas mediante un fraccionamiento. Lo que se consigue habitualmente es aumentar la concentración de fibras largas en una fracción. (Holik 2000 pp. 127)

Las dos fracciones tienen diferentes características:

- *Fracción de fibra corta:* alta concentración de finos, ceniza y fibras cortas junto con las partículas de tinta libres.
- *Fracción de fibra larga:* alta concentración de fibras largas junto con partículas de tinta adheridas a las fibras.

Gracias al fraccionamiento, se pueden blanquear las fracciones por separado obteniéndose mejores valores de brillo que al añadir estos reactivos químicos a la suspensión de pasta de papel sin fraccionar. La fracción de fibra larga puede ser tratada eficientemente con peróxido de hidrógeno ($H_2O_2$), y la fracción de fibra corta puede ser blanqueada con blanqueantes reductores si fuera necesario. (Eul *y cols.* 1989, Eul *y cols.* 1990, Lapierre *y cols.* 2003, Kraschowetz *y cols.* 2008)

## 2.3 FUNDAMENTOS DE LA FLOTACIÓN

La flotación es un proceso que se emplea para separar la tinta de las fibras del papel recuperado. Esta separación se basa en la probabilidad, y se emplean burbujas de aire que se adhieren a las partículas de tinta y las transportan a la superficie de la suspensión, donde se forma una capa de espuma que se le elimina mecánicamente, mediante succión

o por rebose. La eficiencia de la flotación depende estrictamente de las interacciones hidrodinámicas y químicas entre las partículas de tinta y las burbujas de aire. Para mejorar la selectividad de adhesión entre la tinta y el aire se añaden reactivos químicos como colectores o espumantes. Por lo tanto mediante la flotación se eliminan la tinta y otros contaminantes de la suspensión, gracias a la diferente hidrofobia de estas partículas y las fibras. El principio de la flotación se presenta en la Figura 3.

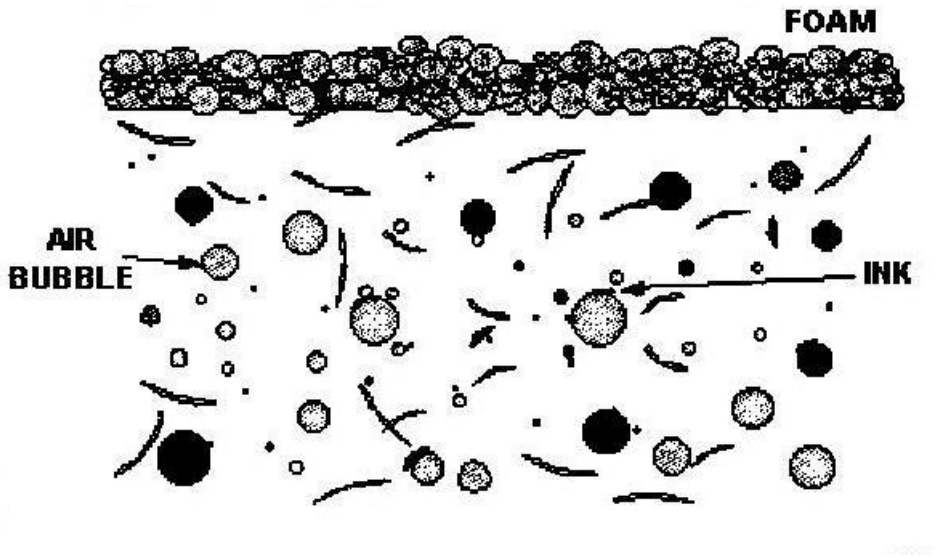


**Figura 3.** Principio de la flotación (Carre & Galland 2007 pp.4)

Durante el destintado por flotación, además de tinta y otros contaminantes, también se elimina material valioso de la suspensión como cenizas, fibras cortas y finos. Para una mejor compresión de la eliminación de componentes durante el destintado por flotación, en la página 23 del Anexo 1 se presentan los cuatro principales mecanismos de transporte que describen la separación de partículas de la suspensión de papel gracias a las burbujas de aire.

### *2.3.1 Fases de la flotación*

Los pasos que conducen a una correcta flotación se ven afectados por múltiples factores como la consistencia, el pH, la temperatura, el tamaño de las burbujas de aire, etc. Según Beneventi (*et al.* 2005), Julien Saint Amand (*et al.* 1999, 2005) y McKinney (1999, pp 107-109), una correcta flotación estaría compuesta por las siguientes fases:

- *Separación de la tinta y la fibra* (se muestra en el apartado 2.1.1).

- *Choque entre la burbuja de aire y la partícula de tinta libre:* Las burbujas de aire y las partículas de tinta tienen que estar lo suficientemente cerca para colisionar y formar aglomerados. La probabilidad de colisión se ve afectada por los tamaños tanto de partícula como de las burbujas de aire, aumentando cuanto menor sea el tamaño de burbuja de aire. En la página 24 del Anexo 1 se presenta una gráfica con la influencia del tamaño de burbuja en el brillo.

- *Formación del aglomerado partícula de tinta-burbuja de aire:* El factor más importante para la formación de este aglomerado es la interacción química superficial entre la tinta y la burbuja. Se añaden reactivos químicos como jabones de ácidos grasos no saturados que reaccionan con la tinta formando un fuerte complejo hidrófobo que se adhiere a la burbuja de aire. Cuanto más grande es el tamaño de la partícula de tinta menos eficiente es la eliminación por flotación.

- *Migración del aglomerado a la superficie:* Pese a que el aglomerado partícula de tinta-burbuja de aire se haya formado, esa unión ha de ser lo suficientemente fuerte para llegar a la capa de espuma. La probabilidad de migración a la superficie es mayor con burbujas de aire de menor tamaño, ya que aumenta el área superficial de contacto.

- *Eliminación de la espuma de la célula de flotación:* Esta eliminación se produce por rebose, ayudado por unas palas rotativas. La estabilidad de la espuma depende de diversos factores, como la gravedad y la turbulencia de la suspensión, que ayudan a que las partículas más grandes se despeguen de las burbujas.

También se estudiaron las pérdidas de fibras y finos, sus mecanismos de pérdida durante la flotación y la influencia del tamaño de burbuja. Toda esta información, así como una pequeña introducción de los estudios previos de flotación de fibra corta (o flotaciones de baja consistencia), se encuentra entre las páginas 26 y 30 del Anexo 1.

# 3 MATERIALES Y MÉTODOS

## 3.1 Materiales

### *3.1.1 Materia prima*

El papel recuperado empleado en los experimentos consistió en una mezcla de periódicos antiguos (ONP) y revistas (OMG). Se considera antiguo al papel recuperado porque tenía más de tres meses. La alimentación para cada producción de pasta en el desintegrador era de un 50% ONP y de un 50% OMG. Las revistas a su vez, estaban compuestas equitativamente por dos tipos de papel revista, SC (Super Calendared) y LWC (Light Weight Coated). El papel LWC se emplea habitualmente para propaganda y el SC es un papel muy satinado, que se emplea en la producción de revistas debido a su calidad superior.

### *3.1.2 Reactivos químicos*

Se emplearon tres reactivos químicos comerciales diferentes – que aparecen denominados en este proyecto como surfactante #1, surfactante #2 y jabón. Los surfactantes #1 y #2 eran de tipo no iónico con diferente composición química, y el reactivo denominado como "jabón" era una mezcla de jabones de sodio y ácidos grasos. No es posible detallar la composición de estos reactivos por motivos de confidencialidad. En cada experimento en el que se empleó jabón, se añadieron 7,2 g de $CaCl_2$ para ajustar la dureza de la suspensión. El lugar en el que se adicionaron los reactivos y su cantidad variaron según cada experimento.

## 3.2 Métodos

### *3.2.1 Descripción del proceso*

*Producción de la pasta de papel (Pulping)*

La pasta fue producida empleando un desintegrador (pulper) Hobart (H600) con un mezclador helicoidal (Figura 4). En cada preparación de pasta se emplearon 2.500 g de papel recuperado, el cual se rasgó en tiras de unos dos centímetros de ancho y se mezcló con agua caliente (45 ºC) para obtener un contenido en sólidos (consistencia) del 15%. El tiempo de pulpeado total fue de 14 minutos.


**Figura 4.** Desintegrador Hobart (H600).

*Fraccionamiento*

El fraccionamiento se llevo a cabo empleando el dispositivo piloto de fraccionamiento PULA del laboratorio, consistente en un tanque de 300 litros, una bomba y una cesta con ranuras de 0,15 mm de anchura. La pasta se diluyó en el tanque de alimentación con agua caliente del grifo (45 ºC) hasta obtener una consistencia aproximada de 0,8%. La pasta diluida se separó empleando una frecuencia de 50 hercios en el rotor de la cesta en dos fracciones: fracción de fibra corta (aceptado) y fracción de fibra larga (rechazo).

*Flotación*

La flotación se llevó a cabo empleando un equipo de flotación Voith Delta 25 equipado con caja de nivel para mantener constante el volumen de líquido mediante el aporte de agua al dispositivo durante la flotación. Tanto el aire como el agua se introducen al equipo de manera continua, mientras que la suspensión de pasta de papel se alimenta de manera discontinua. Las fracciones de fibra corta (o finos) de todos los experimentos de fraccionamiento se emplearon como alimentación para la célula de flotación. Estas muestras de fibra corta se mantuvieron a 45 ºC empleando un calentador eléctrico hasta su introducción en dicho equipo de flotación.


**Figura 5.** Equipo de flotación Voith Delta 25.

### *3.2.2 Análisis*

*Consistencia y cenizas*

La consistencia de las muestras de pasta de papel se realizó según la normativa SFS EN ISO 4119. El contenido en cenizas se determinó mediante ignición de las muestras a 525 ºC según la normativa ISO 1762. Estos datos se emplearon para calcular otras propiedades como la ratio de rechazo de masa ($RR_m$) y la eliminación de cenizas, según las ecuaciones de la página 34 del Anexo 1.

*Hojas de mano*

Éstas son muestras de papel, producidas a partir de la pasta obtenida en cada experimento y realizadas en el laboratorio. A lo largo de este proyecto se ha trabajado con dos clases diferentes de formetas u hojas de mano para la realización de ensayos. Las hojas de mayor tamaño (165 mm x 165 mm) y bajo gramaje (30 $g/m^2$) se prepararon en un molde depositando las fibras de papel sobre un papel de filtro de alta retención (Körkkö *y cols.*, 2010); por otra parte, para las hojas de mano de menor tamaño (ø 39 mm) y mayor gramaje (225 $g/m^2$) se emplearon unos papeles de filtro de membrana de nitratos de celulosa, que evitaban la pérdida de tinta (INGEDE Method 11p, 2009). Una vez preparadas, todas las hojas de mano se aclimataron en el laboratorio antes de las mediciones ópticas, siguiendo la normativa SFS EN 20187.

*Mediciones ópticas*

Se midieron propiedades ópticas como el brillo o la tinta residual de las hojas de mano empleando un espectrofotómetro L&W Elrepho. Los datos se calcularon con la media entre dos mediciones de cada hoja.

- *Brillo:* Definida como la reflectancia de la luz azul. El brillo ISO es un factor intrínseco de reflectancia determinado con un medidor cuya sensibilidad a la luz concuerda con la normativa ISO estándar 2470.

- *Tinta residual:* La presencia de tinta varía el brillo y el color del papel reciclado. La concentración de tinta fue determinada mediante el método ERIC (concentración efectiva de tinta residual) según la normativa ISO 22754. Para calcular ERIC se emplean las fórmulas de las páginas 35 y 36 del Anexo 1.

*Hiperlavado (hyper washing, HW)*

El hiperlavado se llevó a cabo lavando la pasta de papel con un caudal de agua de 8 L/min durante 20 minutos mediante un equipo de depuración por tamizado de tipo Sommerville con un tamiz de 0.15 mm de malla, según la normativa Tappi T 275. Variables como la retención en el hiperlavado y la eliminación de finos se calcularon empleando las ecuaciones de la página 36 del Anexo 1.

## 3.3 Desarrollo de la parte experimental

La parte experimental del proyecto se dividió en cinco diferentes grupos de experimentos de flotación. El primer grupo fueron los experimentos de referencia, tanto para la flotación como para el fraccionamiento. Con el segundo grupo de experimentos se determinaron los valores óptimos de nivel de agua y flujo de aire para la flotación de la fracción corta de papel. En los otros tres grupos de experimentos se utilizaron diferentes reactivos químicos durante la flotación de la fracción corta de papel.

### *3.3.1 Experimentos de referencia*

Este bloque de experimentos se dividió en dos partes: flotaciones y fraccionamiento. De esta manera se obtuvieron muestras de referencia para poder comparar con los resultados obtenidos por muestras de otras flotaciones o fraccionamientos realizados en diferentes condiciones.

*Flotaciones de referencia*

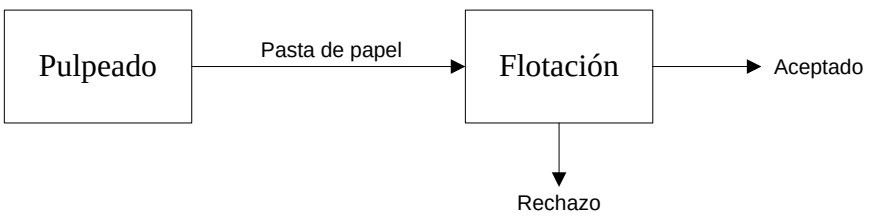


**Figura 6.** Diagrama de las flotaciones de referencia.

Se realizaron 5 flotaciones empleando muestras de pasta de papel sin fraccionar (se denominan en el proyecto como "flotaciones de referencia", pese a que los valores del ensayo nº 41 son los que se emplean como referencia en los resultados). Después del pulpeado, la pasta sin fraccionar se diluyó con agua caliente del grifo (45 ºC) hasta una consistencia de 1,2% y se añadieron 6 mg de jabón por cada gramo de pasta seca en la célula de flotación. En estos 5 experimentos, el nivel de flotación fue de 19 litros y el flujo de aire de 7,4 L/min. Se estudió la influencia del tiempo en la flotación, por lo que se obtuvieron progresivamente una mayor cantidad de rechazo en cada flotación hasta que no se obtuvo más cantidad de rechazo. Las variables de estos experimentos están presentadas en la Tabla 1 y el diagrama del proceso en la Figura 6.

**Tabla 1.** Variables de las flotaciones de referencia.

| ***Nº de test*** | ***Volumen [L]*** | ***Flujo de aire [L/min]*** | ***Jabón [mg/g]*** | ***Rechazo [g]*** |
|---|---|---|---|---|
| 37 | 19 | 7,4 | 6 | 545 |
| 38 | 19 | 7,4 | 6 | 1022 |
| 39 | 19 | 7,4 | 6 | 1533 |
| 40 | 19 | 7,4 | 6 | 2024 |
| 41 | 19 | 7,4 | 6 | 2508 |

*Fraccionamiento*

Después del pulpeado, la pasta sin fraccionar fue diluida con agua caliente del grifo (45 ºC) hasta una consistencia de 1,2%. Para realizar las posteriores flotaciones utilizando únicamente la fracción de fibra corta y empleando reactivos químicos (5.3.3, 5.3.4 y 5.3.5), se llevaron a cabo tres experimentos de fraccionamiento como el presentado en la Figura 7. Se estudió uno de estos fraccionamientos en el cual no se emplearon reactivos durante el pulpeado y se analizaron muestras tanto de la alimentación (feed, F), fibra corta (short fibre, SF) y fibra larga (long fibre, LF). Las muestras de alimentación y fibra corta también fueron hiperlavadas para comparar resultados.

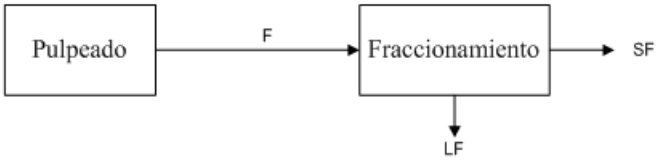


**Figura 7.** Diagrama del fraccionamiento.

### *3.3.2 Estudio del nivel de flotación y flujo de aire durante la flotación*

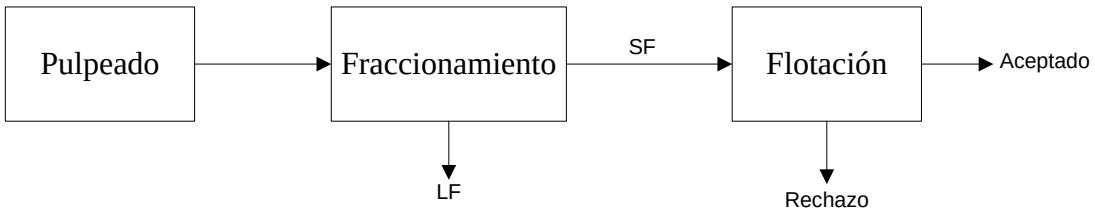


**Figura 8.** Diagrama del proceso para el etudio del nivel de flotación y flujo de aire.

Después del pulpeado y del fraccionamiento, se llevaron a cabo 12 experimentos de flotación separados en 4 grupos de 3 experimentos cada uno. En cada uno de los grupos se modificó el nivel de flotación y el flujo de aire, buscando los valores óptimos de ambas variables (se emplea "nivel de flotación" en el proyecto pese a que se refiere al volumen de carga dentro de la célula de flotación). Una vez fijados tanto el nivel de flotación como el flujo de aire para cada grupo, se realizaron las tres flotaciones. En ellas se estudió la influencia del tiempo en la flotación, por lo que se obtuvo progresivamente una mayor cantidad de rechazo hasta que no se obtuvo más cantidad de rechazo. No se emplearon reactivos químicos en estas flotaciones. Los valores de estas variables se muestran en la Tabla 2 y el diagrama del proceso en la Figura 8.

**Tabla 2.** Valores de nivel de flotación y flujo de aire para las diferentes flotaciones.

| *Nº de test* | *Volumen [L]* | *Flujo de aire [L/min]* | *Rechazo [g]* |
|---|---|---|---|
| 1 | 19 | 7,4 | 610 |
| 2 | 19 | 7,4 | 1174 |
| 3 | 19 | 7,4 | 1705 |
| 4 | 20 | 7,4 | 671 |
| 5 | 20 | 7,4 | 1150 |
| 6 | 20 | 7,4 | 1720 |
| 7 | 19 | 13 | 554 |
| 8 | 19 | 13 | 1008 |
| 9 | 19 | 13 | 1512 |
| 10 | 20 | 13 | 668 |
| 11 | 20 | 13 | 1180 |
| 12 | 20 | 13 | 1737 |

### *3.3.3 Estudio del acondicionamiento previo a la flotación*

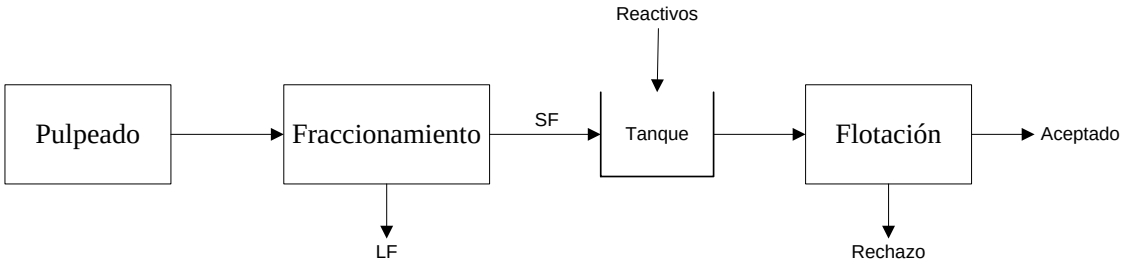


**Figura 9.** Diagrama de las flotaciones con acondicionamiento previo.

A continuación se realizó un estudio de la influencia del acondicionamiento previo a la flotación de la pasta de papel. Como se puede observar en el diagrama de la Figura 9, después del pulpeado y del fraccionamiento se añadió como reactivo una dosis 9,5 mg de jabón por gramo de pasta seca a la fracción de fibra corta. Cinco flotaciones se llevaron a cabo a diferentes tiempos de contacto entre el reactivo y la fracción de fibra corta. El nivel de flotación y el flujo de aire se fijaron en 19 litros y 7,4 L/min (ver apartado 4.2). Los parámetros de estas flotaciones se muestran en la Tabla 3. En cada flotación se recogieron muestras a diferentes tiempos de flotación, obteniendo diferentes cantidades de rechazo que se fueron acumulando hasta que no se obtuvo más cantidad de rechazo. El test 13 se empleó como referencia para los restantes experimentos. El clorato cálcico se añadió en todos los experimentos en la célula de flotación menos para el experimento 18 en el que el $CaCl_2$ y el jabón se añadieron a la vez.

**Tabla 3.** Parámetros para las flotaciones con acondicionamiento previo.

| ***Nº de test*** | ***Reactivo*** | ***Tiempo [min]*** | ***Rechazo [g]*** |
|---|---|---|---|
| 13 | - | - | 771, 1328 |
| 14 | Jabón | 0 | 230, 474, 491 |
| 15 | Jabón | 20 | 202, 438, 553 |
| 16 | Jabón | 40 | 234, 461, 673 |
| 17 | Jabón | 60 | 243, 484, 794 |
| 18 | Jabón + $CaCl_2$ | 60 | 714, 1208, 1655 |

### *3.3.4 Estudio del efecto del pH en la flotación*

Cuatro experimentos de flotación diferentes se llevaron a cabo controlando el pH: en las dos primeras flotaciones se empleó pasta de papel sin fraccionar, y en las dos últimas se empleó la fracción de fibra corta. Durante el pulpeado se añadió como reactivo una dosis de 6 mg de jabón por gramo de pasta seca, como se presenta en la Figura 10.

En cada uno de los 2 primeros experimentos, 1.520 g de pasta de papel se diluyeron en la célula de flotación hasta un nivel de flotación de 19 litros (consistencia del 15%). En la primera flotación se midió el pH de la alimentación (en torno a 7,5), mientras que el pH de la segunda flotación se ajustó a 8,5 en la celda de flotación empleando una solución 0,2M de NaOH.

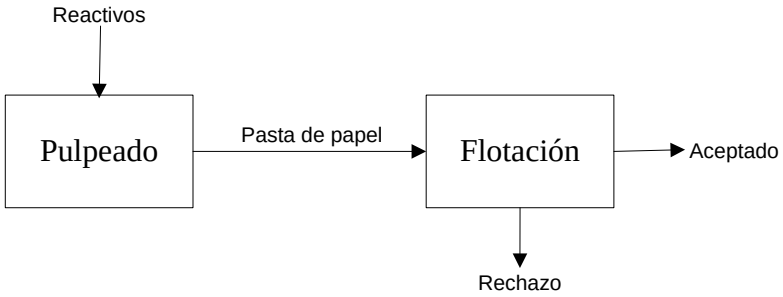


**Figura 10.** Diagrama de las flotaciones de pasta sin fraccionar con control de pH.

Las otras dos flotaciones se llevaron a cabo con la fracción de fibra corta (SF) obtenida tras el fraccionamiento, como se detalla en la Figura 11. En la primera flotación se midió el pH de la alimentación (en torno al 7,9), mientras que el pH de la segunda flotación se ajusto a 8,5 empleando una solución 0,2M de NaOH.

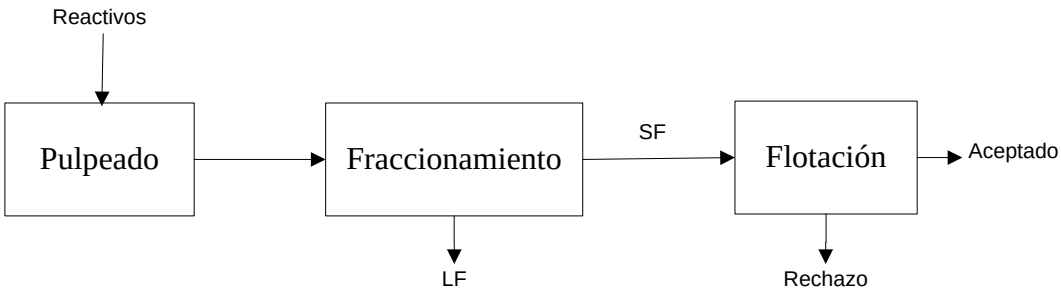


**Figura 11.** Diagrama de las flotaciones de fibra corta con control de pH.

Las variables de todas estas flotaciones se muestran en la Tabla 4. En cada flotación se recogieron muestras a tres diferentes tiempos de flotación, consiguiendo diferentes cantidades de rechazo que se fueron acumulando hasta que no se obtuvo más cantidad de rechazo.

**Tabla 4.** Variables para las flotaciones con control de pH.

| *Nº de test* | *Voumen [L]* | *Flujo de aire [L/min]* | *Nota* | *Rechazos [g]* |
|---|---|---|---|---|
| 19 | 19 | 7.4 | Sin fraccionar | 506, 1091, 2651 |
| 20 | 19 | 7.4 | Sin fraccionar – pH 8.5 | 566, 1141, 2746 |
| 21 | 19 | 7.4 | Fibra corta | 613, 1257, 1756 |
| 22 | 19 | 7.4 | Fibra corta – pH 8.5 | 573, 1219, 1621 |

### *3.3.5 Estudio de la adición de diferentes reactivos químicos de flotación*

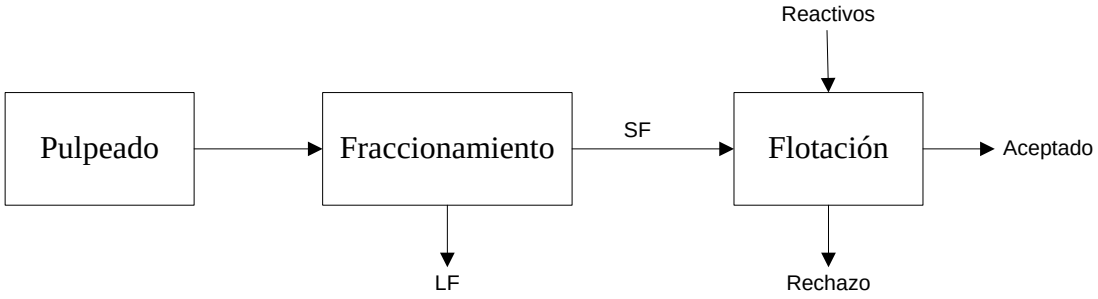


**Figura 12.** Diagrama de las flotaciones con reactivos químicos.

Como se puede ver en la Figura 12, después del pulpeado y el fraccionamiento las flotaciones se llevaron a cabo con la fracción de fibra corta (SF). Se emplearon tres reactivos químicos diferentes en seis experimentos de flotación: jabón, surfactante #1 y surfactante #2. Todos los reactivos químicos se añadieron en la célula de flotación antes de comenzar la misma. Para el jabón, se añadieron dosis de 6 y 9 mg de jabón por gramo de pasta seca respectivamente. Para los surfactantes, las dosis añadidas fueron de 0,6 y 0,9 gramos por gramo de pasta seca. En cada flotación se recogieron muestras tanto de aceptado y como de rechazo a diferentes tiempos de flotación, con diferentes cantidades de rechazo para cada toma que se fueron acumulando hasta que no se obtuvo más cantidad de rechazo. Los parámetros de estas flotaciones se muestran en la Tabla 5.

**Tabla 5.** Parámetros para las flotaciones químicas.

| ***Nº de test*** | ***Reactivo químico*** | ***Dosis [mg/g]*** | ***Rechazo [g]*** |
|---|---|---|---|
| 23 | Jabón | 6 | 238, 382 |
| 24 | Jabón | 9 | 148, 265 |
| 25 | Surfactante #1 | 0,6 | 293, 485, 858 |
| 26 | Surfactante #1 | 0,9 | 337, 591, 832 |
| 27 | Surfactante #2 | 0,6 | 346, 590, 1020 |
| 28 | Surfactante #2 | 0,9 | 388, 616, 1151 |

# 4 RESULTADOS Y DISCUSIÓN

Los resultados están distribuidos en los mismos grupos de experimentos de flotación explicados en el apartado 3.3. Se ha de tener en cuenta que el papel recuperado empleado en estos experimentos es antiguo, por tanto los valores de las muestras serán más bajos en comparación con experimentos de papel fresco. Según Vahlroos *y cols.* (2008), se pueden obtener valores de brillo ISO alrededor de 60% con papel fresco, en vez de los valores alrededor del 50% que se obtienen con revistas y periódicos recuperados.

## 4.1 Experimentos de referencia

*Flotaciones de referencia*

Los valores de brillo, contenido en cenizas y tinta residual obtenidos por medición óptica de hojas de ensayo de cada muestra, se han representado frente al ratio de masa de rechazo ($RR_m$) en las figuras de las páginas 41 y 42 del Anexo 1. Los resultados están presentados en la siguiente tabla:

**Tabla 6.** Resultados de las flotaciones de referencia.

| ***Muestra*** | ***Brillo ISO [%]*** | ***Cenizas [%]*** | ***Tinta residual [ppm]*** | ***Eliminación cenizas [%]*** | ***$RR_m$ [%]*** |
|---|---|---|---|---|---|
| FEED | 45,1 | 24,7 | 1439 | 0,0 | 0,0 |
| A37 | 47,0 | 18,6 | 1216 | 20,2 | 9,0 |
| A38 | 48,3 | 15,5 | 989 | 41,0 | 17,6 |
| A39 | 49,5 | 13,2 | 846 | 50,2 | 21,7 |
| A40 | 50,6 | 11,2 | 738 | 53,8 | 23,8 |
| A41 | 51,8 | 9,8 | 628 | 68,8 | 27,9 |

Según la Tabla 6 y como era de esperar las muestras con altos ratios de masa de rechazo ($RR_m$) son las que tienen menor contenido en tinta residual y cenizas. Se observa que el brillo y la $RR_m$ son proporcionales (aumento de 6,7% unidades de brillo ISO), pese a la pérdida de fibra que se produce por los altos valores de $RR_m$. Debido a que en la flotación se eliminan tanto tinta como cargas del papel, la ganancia de brillo debida a la eliminación de la tinta puede estar contrarrestada por la pérdida de cargas del papel (como colorantes), si el brillo de las cargas es mayor que el brillo de las fibras, como

ocurre para las mezclas de papel recuperado ONP/OMG. Comparado con los datos obtenidos por Körkkö *y cols.* (2008), el valor de brillo obtenido en el Test 41, y que va a ser considerado como blanco de comparación del resto de experimentos, es bastante elevado.

*Fraccionamiento*

Tras fraccionar la alimentación en fibra corta y fibra larga, se analizaron las propiedades ópticas de las hojas de mano de menor tamaño (filtro de membrana) de cada muestra. Los valores obtenidos tanto de la alimentación y como de las fracciones de fibra corta y fibra larga se muestran en la Tabla 7. Tras el fraccionamiento, se observó quelos valores de brillo fueron similares para todas las muestras pese a que la fracción de fibra corta tuviera el mayor contenido en cenizas. Este contenido en cenizas de la fracción de fibra corta fue mayor que el obtenido por Mäkinen *y cols.* (2010), como también fue mayor el valor de la muestra de alimentación. También se obtuvieron valores similares de retención para la alimentación y la fracción de fibra larga. El valor de brillo ISO de las muestras hiperlavadas (HW) fue mayor para la fracción de fibra larga que para la alimentación.

**Tabla 7.** Brillo, contendio en cenizas y retención de la alimentación, fracción de fibra corta y fracción de fibra larga del fraccionamiento.

| | ***Alimentación*** | ***Fibra corta*** | ***Fibra larga*** |
|---|---|---|---|
| Brillo ISO [%] | 44.3 | 44.0 | 42.7 |
| Cenizas [%] | 24.0 | 32.9 | 21.9 |
| Retención [%] | 61.0 | 38.6 | 59.1 |
| Brillo ISO - HW [%] | 44.0 | - | 47.0 |

Se confirma con estos resultados que la fracción de fibra corta acumula una mayor cantidad de cenizas. Observando los datos de brillo se comprueba que el mayor valor lo obtuvo la muestra de fibra larga hiperlavada, lo que sugiere que había gran cantidad de tinta libre en la suspensión (no adherida a las fibras). Comparando el brillo de estas muestras con el obtenido por las flotaciones de referencia, se observa una diferencia de 7,8% unidades entre la muestra A41 y la fracción de fibra corta (SF), según las Tablas 6 y 7. Por lo tanto, gracias a la flotación se pierde una mayor cantidad de cenizas y de tinta, incrementando así el brillo.

## 4.2 Nivel de flotación y flujo de aire durante la flotación

Se midieron las propiedades ópticas de las hojas de mano de menor tamaño (filtro de membrana) de las muestras obtenidas en cada experimento de flotación. Los valores de brillo y contenido en cenizas están representados en las Figuras 13 y 14. Según la Figura 13, sólo se obtuvo una diferencia de 1,0% en el brillo ISO con las condiciones estudiadas. Al comparar estos datos de brillo con los obtenidos por las flotaciones de referencia (A41), se observa que son inferiores. Para el contenido en cenizas, las diferencias entre experimentos fueron muy pequeñas según la Figura 14.

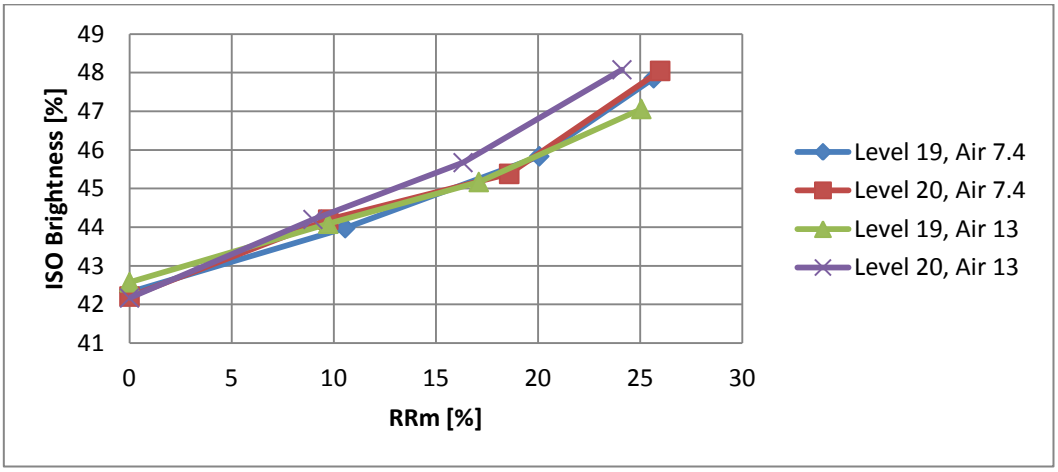


**Figura 13.** Valores de brillo para las flotaciones de nivel de flotación y flujo de aire.

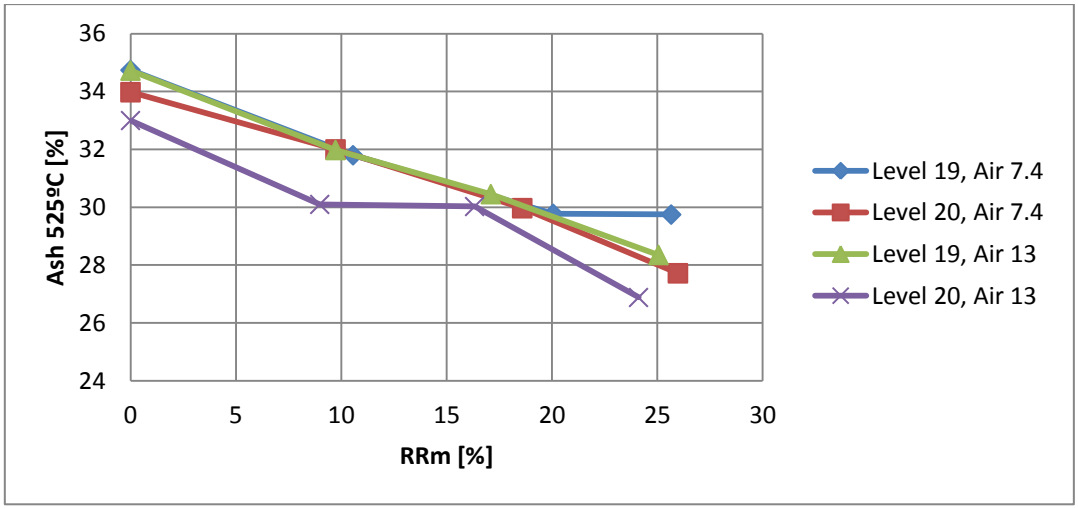


**Figura 14.** Valores de cenizas para las flotaciones de nivel de flotación y flujo de aire.

Variar el nivel de flotación y el flujo de aire no tuvo mucho efecto en el brillo o en el contenido en cenizas, aunque sí que incrementó la velocidad de flotación. Por tanto, se

fijó el nivel de flotación a 19 litros y el flujo de aire a 7,4 litros por minutos para el resto de flotaciones, para gastar la menor cantidad posible de agua y aire.

## 4.3 Acondicionamiento previo a la flotación

Una vez analizadas las formetas de menor tamaño (filtro de membrana), se representaron los valores de brillo y contenido en cenizas de las muestras de alimentación y de fracción de fibra corta post-flotada para cada flotación, y así determinar la influencia del tiempo de contacto de los reactivos químicos previo a la flotación. Se representan estas variables frente a $RR_m$ en las Figuras 15 y 16. Los valores del Test 21, en el que el jabón se añadió en el desintegrador, se muestran también en las gráficas para comparar resultados.

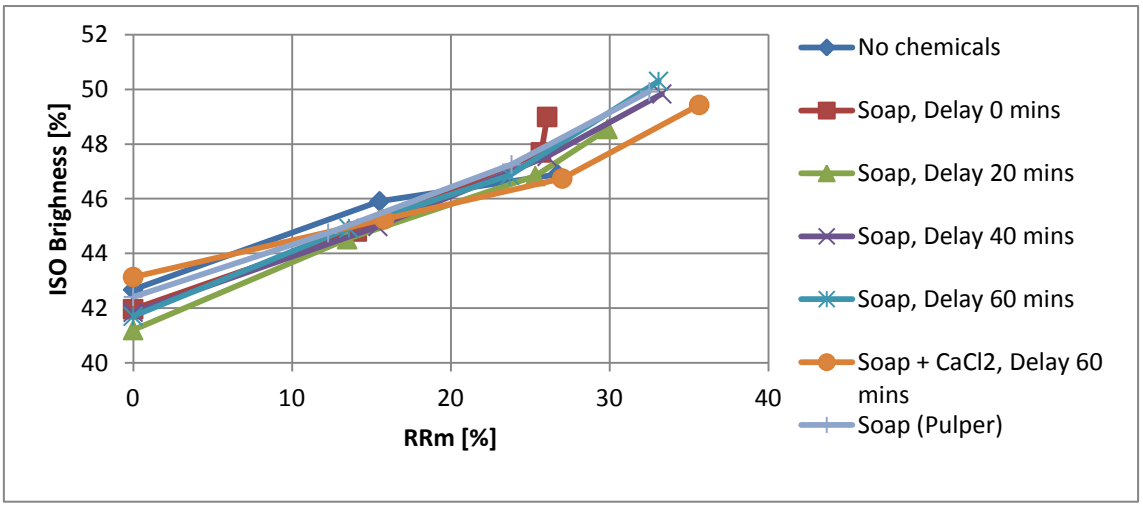


**Figura 15.** Valores de brillo para las flotaciones con reactivo a diferentes tiempos.

Los valores más altos de brillo se obtuvieron con las muestras en las que más tiempo actuaron los reactivos químicos, aunque también con unos altos ratios de masa de rechazo ($RR_m$) con la consiguiente pérdida de material fibroso de la muestra. El uso de reactivos químicos mejoró el brillo, obteniéndose unos valores de brillo ISO levemente más altos (2% unidades) que los de las flotaciones de nivel de flotación y flujo de aire.

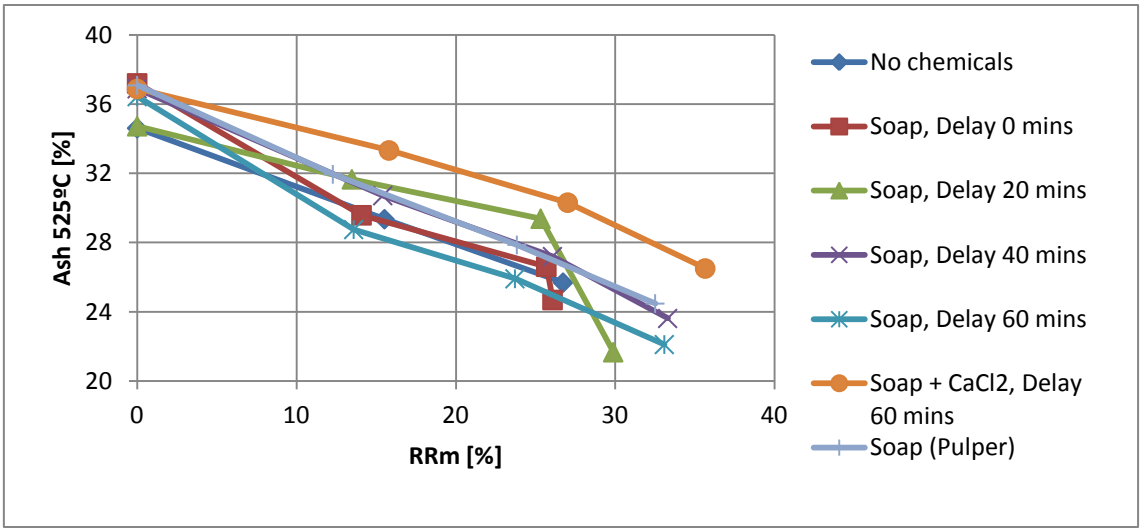


**Figura 16.** Contenido en cenizas para las flotaciones con reactivo a diferentes tiempos.

Según la Figura 16, la muestra con menor contenido en cenizas fue la del test 15 (Soap, Delay 20 mins), con menor pérdida de material fibroso en comparación con muestras con mayor tiempo.

Basándonos en la cantidad de rechazo de cada flotación, se calcularon los valores de eliminación de cenizas y finos (fibra corta) y están representados en las Figuras 17 y 18. En la Figura 17, se obtuvieron valores similares de eliminación de cenizas para los últimas cuatro flotaciones, con diferencias en $RR_m$. En la Figura 18, las flotaciones con mayor tiempo de actuación de los reactivos son las de mayor pérdida de finos.

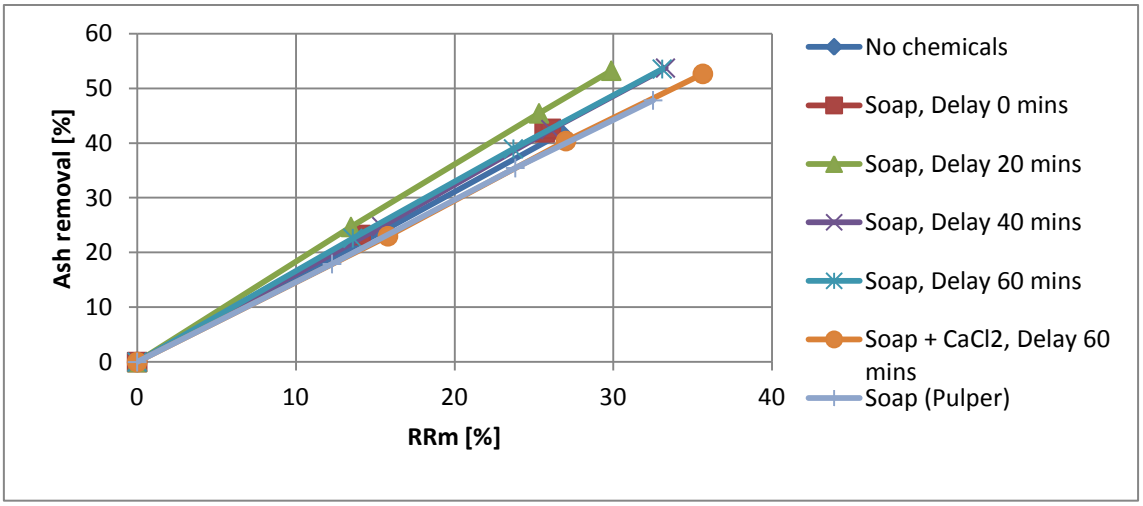


**Figura 17.** Eliminación de cenizas en las flotaciones con reactivo a diferentes tiempos.

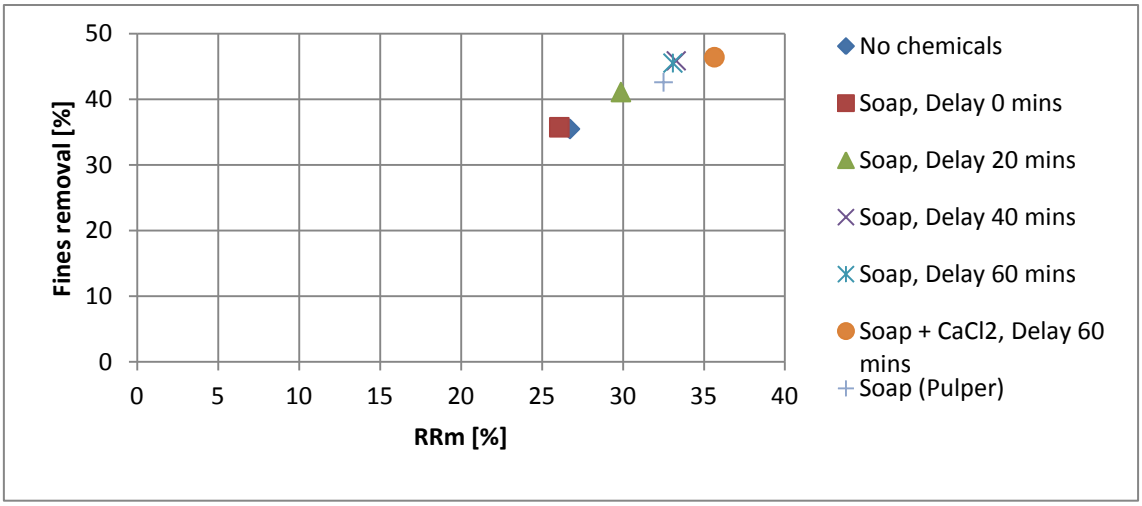


**Figura 18.** Eliminación de finos en las flotaciones con reactivo a diferentes tiempos.

Por tanto, la variable más afectada por los reactivos químicos fue la $RR_m$ ya que cuanto más tiempo actuaron los reactivos, mayor cantidad de fibra, finos y cenizas se perdió con el rechazo. Añadir el $CaCl_2$ junto con el jabón tuvo un efecto negativo en la flotación, ya que incrementó la pérdida fibra.

## 4.4 Efecto del pH en la flotación

*Comparación entre flotaciones de pasta sin fraccionar (Entire pulp)*

Los valores de brillo, contenido en cenizas, tinta residual y eliminación de cenizas se obtuvieron por medición óptica de hojas de mano grandes de cada flotación (muestras de alimentación y fracción de fibra corta). Se compararon estos datos con los obtenidos por las flotaciones de referencia y se representaron en las Figuras 19, 20, 21 y 22.

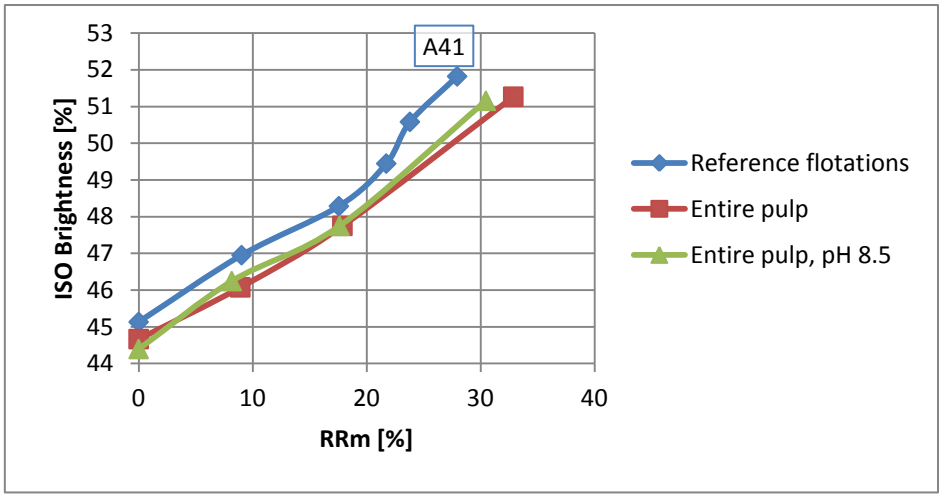


**Figura 19.** Brillo ISO para las flotaciones de pasta sin fraccionar con control de pH.

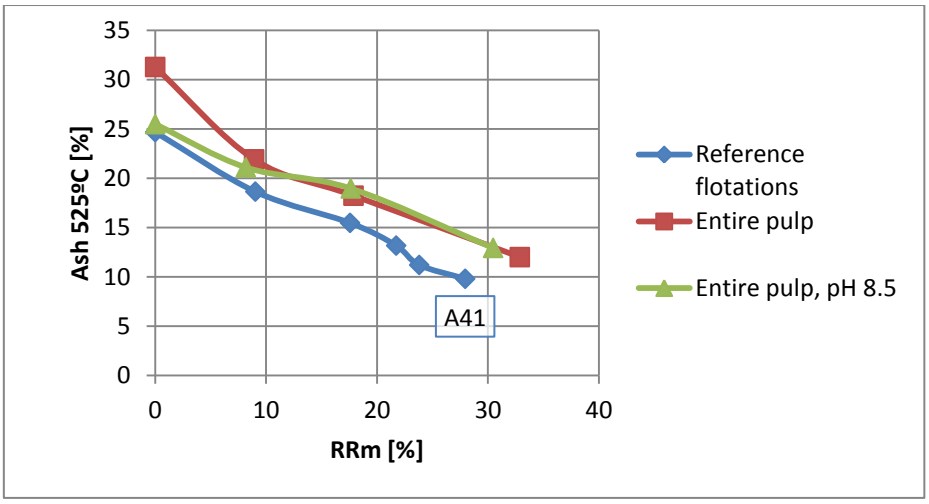


**Figura 20.** Contenido en cenizas de las flotaciones de pasta sin fraccionar con control de pH.

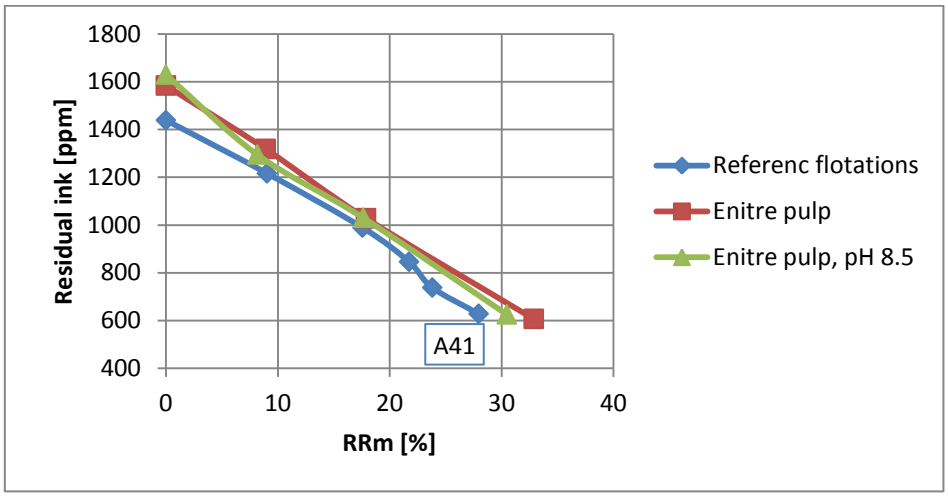


**Figura 21.** Tinta residual para las flotaciones de pasta sin fraccionar con control de pH.

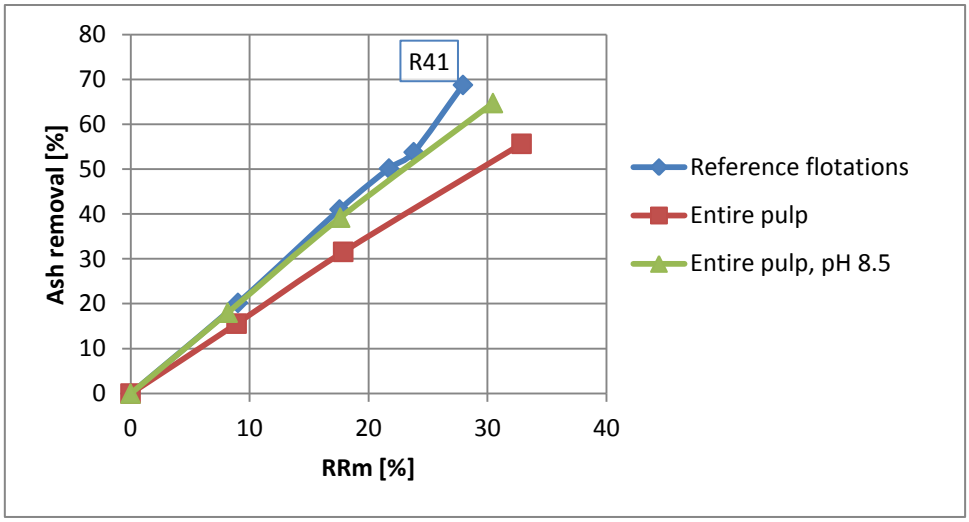


**Figura 22.** Eliminación de cenizas para las flotaciones de pulpa sin fraccionar con control de pH.

En la Figuras 19, 20, 21 y 22 se observa una pequeña diferencia entre las muestras de la flotación con control de pH respecto a las no controladas. Los valores finales de brillo, contenido en cenizas y en tinta residual fueron similares excepto para el test 20 (Entire pulp, pH 8,5) que obtuvo un valor de $RR_m$ máximo inferior. Comparando las flotaciones con control de pH con las flotaciones de referencia (Test 41), se observa que estas últimas obtuvieron valores de brillo levemente mayor (un 1% mayor), menor contenido en cenizas y similar contenido en tinta residual pero con menor ratio de masa de rechazo. Sin embargo, al añadir los reactivos químicos a la desintegradora se consiguió aumentar la flotabilidad de las partículas de tinta. Como se puede ver en la Figura 21, los valores de tinta residual tuvieron un drástico descenso desde 1630 hasta 625 en la muestra de la flotación con el pH controlado.

*Flotaciones de la fracción de fibra corta*

Se realizaron dos flotaciones de fracción de fibra corta, ajustando el pH de una ellas a 8,5. Los valores de brillo obtenidos gracias a mediciones ópticas de las hojas de mano pequeñas (filtros de membrana) de cada flotación (muestras de alimentación y fracción de fibras corta post-flotada), se representaron frente a $RR_m$ en la Figura 23. Otras variables como el contenido en cenizas, la eliminación de cenizas y la eliminación de finos también fueron estudiadas, pero se obtuvieron unas diferencias mínimas entre ambas flotaciones. Las gráficas de estas variables se pueden ver en las páginas 49 y 50 del Anexo 1.

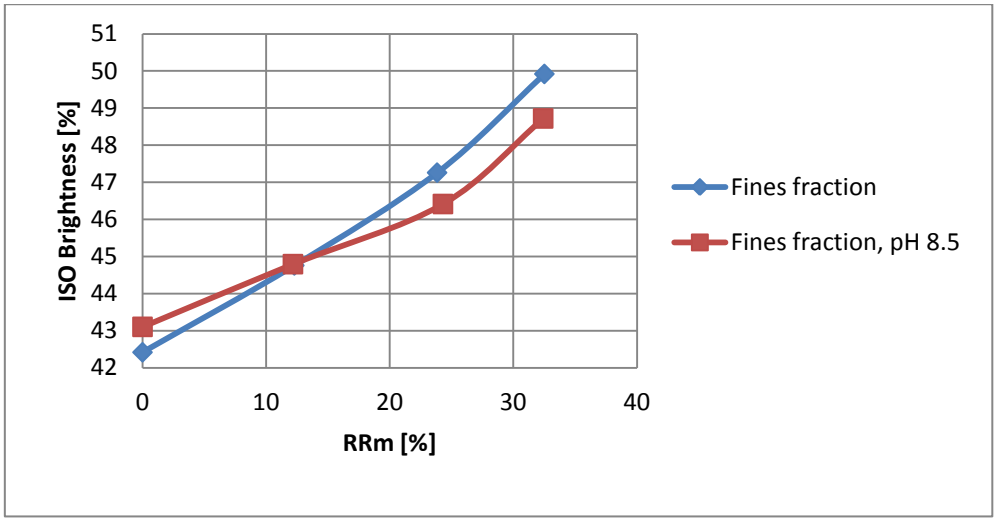


**Figura 23.** Valores de brillo para las flotaciones de fracción de fibra corta con control de pH.

Se observa que ajustar el pH no tuvo casi efecto en las flotaciones de la fibra corta. El $RR_m$ máximo de ambas flotaciones fue aproximadamente el mismo (32%), y la única diferencia fue en el valor de brillo de la muestra sin el pH controlado que resultó un 1% mayor que la muestra controlada, como se puede ver en las Figura 23. Comparando el valor máximo de brillo de estas flotaciones con los resultados obtenidos por las flotaciones de nivel de flotación y flujo de aire, se observa un aumento de 2% unidades de brillo ISO. Por lo tanto, al añadir los reactivos en la desintegradora se consigue una mejor separación entre la tinta y las fibras de papel.

Sorprendentemente, ajustar el pH a 8,5 no tuvo ningún efecto sobre las propiedades estudiadas. Según el método INGEDE 11, al final del pulpeado el valor de pH debe de ser de 9,5 y se ha de emplear sosa para alcanzarlo (si fuera necesario), por lo que la bibliografía se sugiere que cuanto mayor es el pH se obtienen mejores resultados. Sin embargo, queda demostrado que las flotaciones se pueden llevar a cabo con condiciones neutras de pH.

## 4.5 Adición de diferentes reactivos químicos de flotación

El efecto de los reactivos químicos (jabón, surfactante #1 y #2) durante la flotación fueron estudiados conjuntamente para comparar los resultados en variables como el brillo y el contenido en cenizas. Estas variables se obtuvieron gracias a mediciones ópticas de hojas de mano pequeñas (filtros de membrana) obtenidas a partir de la pasta de cada flotación (muestras de alimentación y fracción de fibras corta post-flotada), y están representados frente a $RR_m$ en las Figuras 24 y 25.

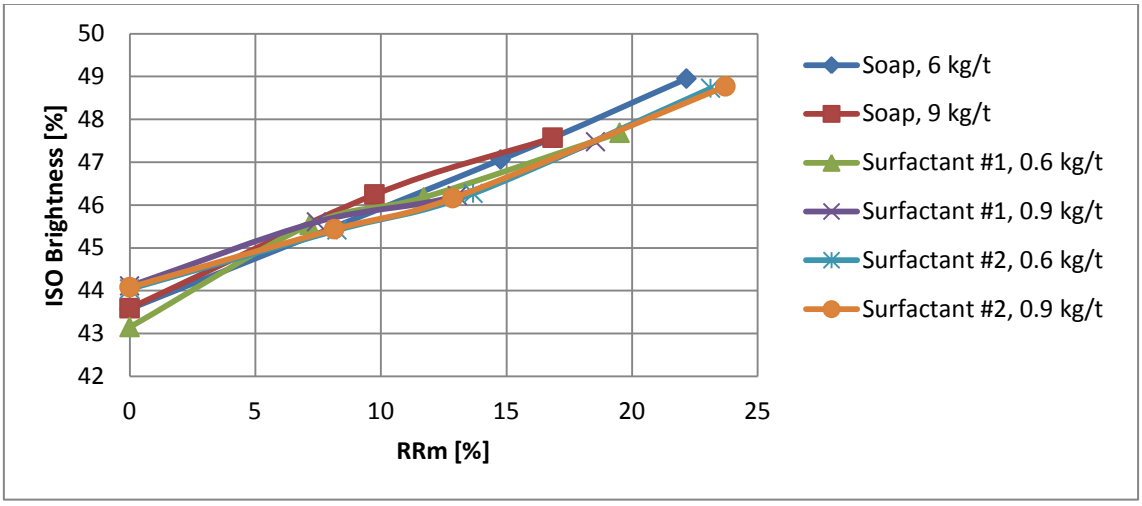


**Figura 24.** Valores de brillo para las flotaciones con diferentes reactivos químicos.

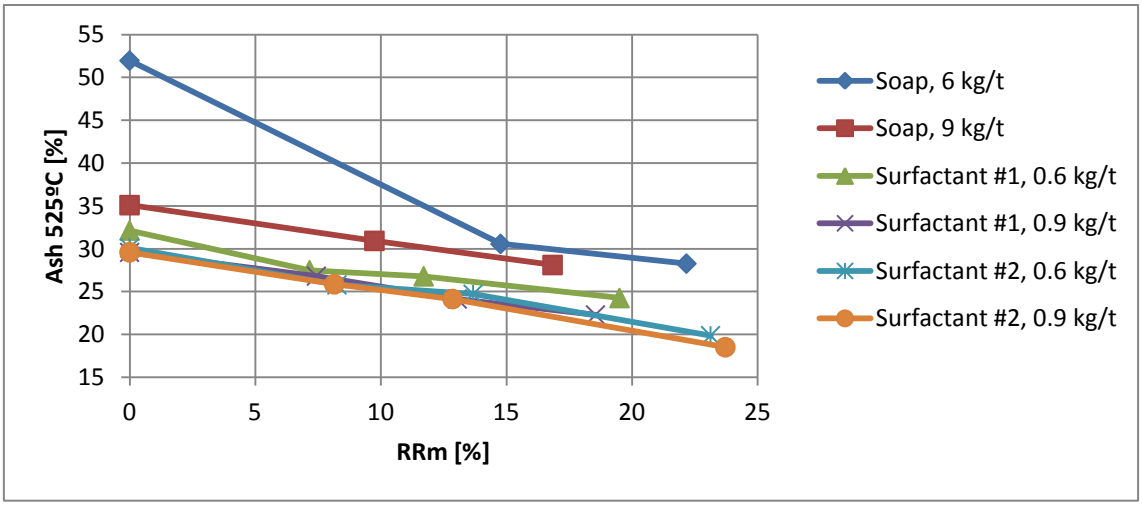


**Figura 25.** Contenido en cenizas para las flotaciones con diferentes reactivos químicos.

En la Figura 24 se observa que las muestras del test 23 (6 mg/g) y de las flotaciones con surfactante #2 obtuvieron los valores más altos de brillo. En la Figura 25 las flotaciones

con surfactante #2 obtuvieron los valores más bajos de contenido en cenizas. El primer dato de contenido en cenizas de la flotación con jabón (6 mg/g) es erróneo, debido a que la materia prima no puede variar tanto entre las distintas flotaciones.

Según Dorris *y cols.* (2011), los jabones de ácidos grasos se emplean como colectores de flotación para la eliminación de tinta para mezclas de ONP/OMG. En nuestras flotaciones, el valor más alto de brillo ISO se obtuvo en la flotación con la dosis de 6 mg/g de jabón. Una dosis de jabón baja consiguió una mejor aglomeración entre la tinta y el jabón creando agregados más hidrófobos. No hubo una gran diferencia entre las flotaciones con el surfactante #1 o el #2. Según Dorris *y cols.* (2011), la principal ventaja de usar surfactantes no iónicos es que se necesitan pequeñas cantidades para mezclas de ONP/OMG, por lo que se reduce la dosis. La velocidad de flotación fue mayor cuando se emplearon los surfactantes, debido a que la espuma se formó rápidamente en la superficie de la suspensión.

Basándonos en la cantidad de rechazo obtenido para cada flotación, se calcularon variables como la eliminación tanto de cenizas como de finos (fibra corta) y se representaron frente a $RR_m$ en las Figuras 26 y 27. En ambas figuras se observa que los valores más altos de eliminación de cenizas y de eliminación de finos fueron obtenidos por las flotaciones con surfactante #2.

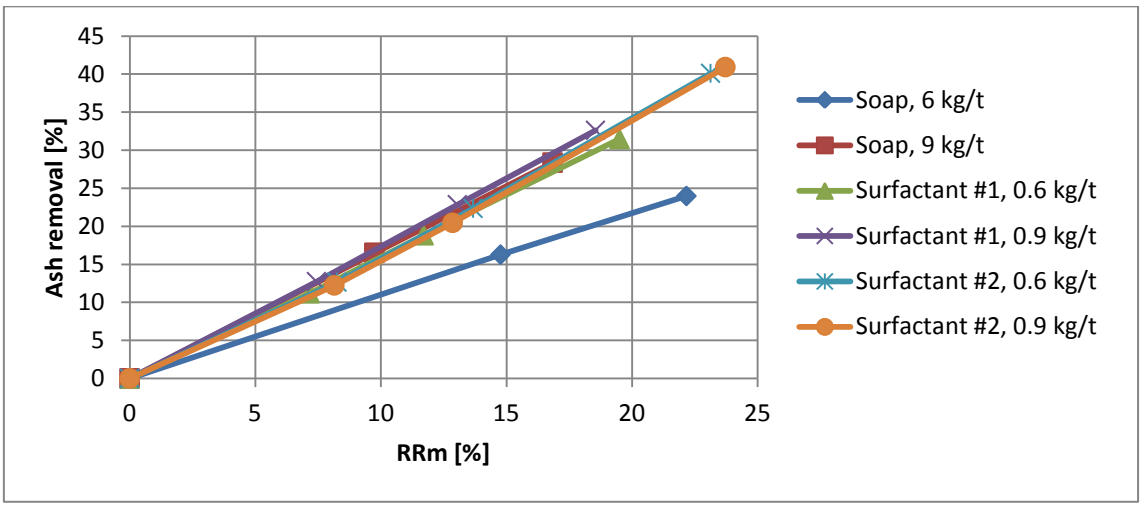


**Figura 26.** Eliminación de cenizas para las flotaciones con diferentes reactivos químicos.

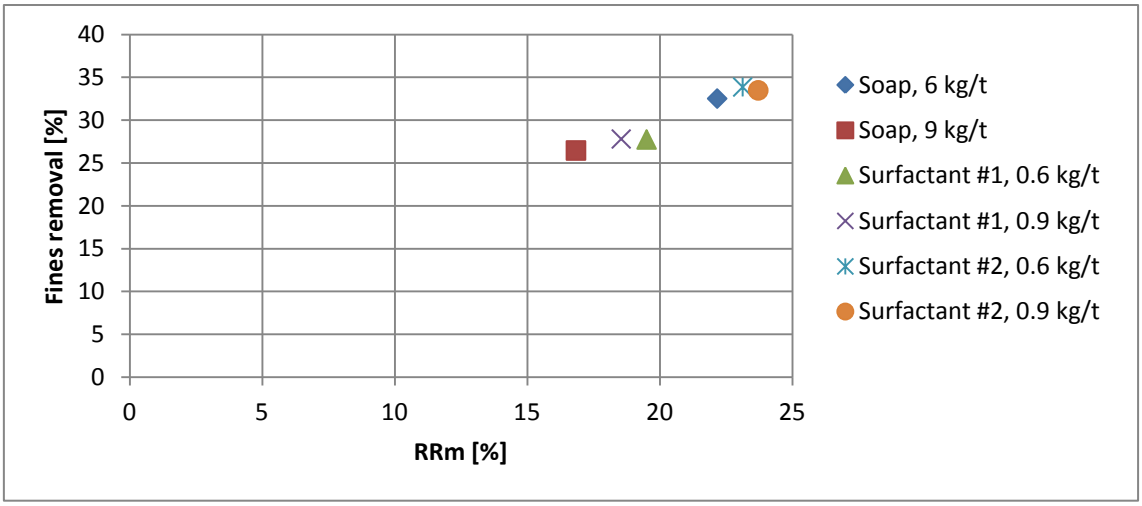


**Figura 27.** Eliminación de finos para las flotaciones con diferentes reactivos químicos.

Comparando todas las flotaciones químicas, el experimento con una dosis de jabón de 6 mg/g consiguió los mejores resultados según la Figura 24 (mismos valores de brillo pero con menor $RR_m$). No hubo gran diferencia al usar una dosis mayor o menor de surfactante, por lo que el uso de una dosis menor (0,6 mg/g) consigue ahorrar gastos.

# 5 CONLCUSIONES

Antes de comenzar las conclusiones, es necesario recordar que el uso de materia prima antigua (ONP/OMG) empeora el destintado de la pasta de papel y por consiguiente, los valores de propiedades como el brillo son peores que los obtenidos con materia prima fresca. Sin embargo, en nuestros experimentos, a través de la flotación de pasta de papel sin fraccionar (Entire pulp), el valor de brillo fue aumentando moderadamente cuanto mayor cantidad de rechazo se eliminaba de la suspensión. Por lo tanto, los experimentos de flotación funcionaron bien pese al uso de materia prima antigua.

Las conclusiones tras la realización de este proyecto son las siguientes:

El fraccionamiento empleado en este proyecto resultó ser adecuado para los posteriores experimentos. Los valores de brillo de la fracción de fibra larga fueron levemente inferiores debido a la presencia de tinta libre en la suspensión. La mayor presencia de cenizas en la fracción de fibra corta confirma que el fraccionamiento fue correcto.

Los resultados de las flotaciones de fibra corta muestran que cambiar parámetros como el flujo de aire o el nivel de flotación en el equipo no tuvo mucho efecto en las propiedades de la suspensión tras la flotación. Por otro lado, incrementar el tiempo de acción de los reactivos en la suspensión antes de la flotación sí que tuvo efecto en las propiedades de la fracción de fibra corta. A mayor tiempo, mayor valor tanto de brillo como de ratio de masa de rechazo, con la consiguiente pérdida de fibras.

Las propiedades de la pasta de papel no fueron modificadas por el uso de reactivos químicos para controlar el pH, por lo que aparentemente, estas flotaciones se pueden llevar a cabo en unas condiciones de pH neutras.

Basándose en estos resultados, se recomienda el uso de una dosis de jabón de 6 mg/g en el equipo de flotación. Gracias a esta dosis se consiguió una mejor aglomeración entre la tinta y el jabón creando aglomerados más hidrófobos. Se obtuvieron los datos más altos de brillo con menor ratio de masa de rechazo, en comparación con otros reactivos colectores como los surfactantes.

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# ANEXO 1

OULUN YLIOPISTO
UNIVERSITY of OULU

Department of process and environmental engineering

Fibre and particle engineering laboratory

Master's thesis

## Study of DIP fines fraction flotation

Oulu 15.9.2011

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**UNIVERSITY OF OULU** **Abstract of thesis**
**Faculty of technology**

| Department | Laboratory |
|---|---|
| Department of Process and Environmental Engineering | Fibre and Particle Engineering Laboratory |
| Author<br>Cisneros Barriga, Gabriel | Supervisor<br>Ämmälä, A. Adjunct Professor |

Name of the thesis

Study of DIP fines fraction flotation

| Subject | Level of studies | Date | Number of pages |
|---|---|---|---|
| Process Engineering | Master's thesis | September 2011 | 63 + 3 (1 app.) |

Abstract

An increasing use of recovered papers to produce, through deinking, white grades has developed deinking process lines in order to achieve the required final stock properties. For improving the deinking process, other deinking lines are being considered too. In this Master's Thesis, DIP fines fraction flotation was studied in detail, as part of a fractional deinking concept that has been considered as an alternative deinking process.

The objective of this study was to obtain more information about the less studied fines fraction flotation and to optimize its properties. The work was divided into five different flotation scenarios. Raw material used was a mixture of recovered paper with an ONP/OMG ratio of 50% ONP and 50% OMG. Flotation experiments worked very well despite of the old raw material. Fractionation was performed with a pressure screen with a long fibre share of 35% and short fibre 65%. Through fractionation free ink and ash drifted into the short fibre fraction.

Fines fraction flotation results suggest that changing either flotation cell level or air flow do not have so much effect on pulp properties after flotation. On the other hand, increasing the delay of chemicals prior to flotation has an effect on fines fraction properties: longer delay increases brightness but also mass reject rate which lowers the yield. Moreover, pH adjustment prior to flotation do not have an effect on pulp properties, so apparently fines fraction flotation experiments can be carried out in neutral pH conditions.

Based on the results presented here, the use of a soap dosage of 6 kg/t in the flotation is recommended in fines fraction flotation experiments. Highest value of brightness was reached with lower yield loss with 6 kg/t soap dosage compared to other chemicals tested.

Library location

University of Oulu, Science and technology Library Tellus

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Finally, I would like to thank my family, specially my parents, for their patience and for giving me the chance to study abroad, and my brother, for being my support and strength through the good and bad times. I would like to thank also all the friends I have made during my Erasmus in Oulu. This past year was full of unforgettable memories. Special thanks to my friends from school, from university and from my village Malanquilla for their love, friendship and for all the laughs we have shared.

Oulu, September, 2011

Gabriel Cisneros Barriga

# ABBREVIATIONS

| | |
|---|---|
| A | Accept |
| $c_f$ | Consistency of the feed pulp (%) |
| $c_r$ | Consistency of the reject pulp (%) |
| $Ca^{2+}$ | Calcium |
| DAF | Dissolved air flotation |
| DIP | Deinked pulp |
| ERIC | Effective residual ink concentration |
| F | Feed |
| FAS | Formamidine sulphinic acid |
| $g$ | Grammage |
| $H_2O_2$ | Hydrogen peroxide |
| HC | High consistency |
| HW | Hyper washed |
| INGEDE | International association of the deinking industry |
| $k$ | Light absorption coefficient |
| LC | Low consistency |
| LF | Long fibre fraction |
| LWC | Light weight coated paper grade |
| M | Molarity (mol/litre) |
| MC | Medium consistency |
| NaOH | Sodium hydroxide |
| R | Reject |
| RCF | Recycled fibre |
| $RR_m$ | Mass reject rate |
| $s$ | Light scattering coefficient |
| SC | Super calendared paper grade |
| SF | Short fibre fraction |

# TABLE OF CONTENTS

# 1 INTRODUCTION

Recycling is an old tradition of the paper industry. Recycling process was developed at the very beginning of the 20th century and in 1955 first recycling line was started (Kemper *et al.* 1999). In the early days, the processed waste paper was only used for low paper grades. Thanks to the consistent development of the machines and systems (such as screening, dispersing, and particularly flotation), the use of the processed waste paper was extended to papers with the highest quality requirements. Nowadays, more or less half of the paper and board produced worldwide are produced with recovered papers.

Since 15-20 years there has been a drastic increase in the use of recovered papers to produce, through deinking, white grades such as newsprint, tissue, market pulp and also more recently magazine papers (SC & LWC). By using the deinking technology, white grade papers can be produced from post-consumer, or post-industrial recovered papers. This means that the components which cause a reduction of brightness – the inks – must be removed, as well as all the contaminants of the pulp such as additives used during the paper converting and packaging: staples, plastic films, etc. (Carré and Galland 2007)

Due to the exacting demands on the final stock, the complexity of the machines increased constantly. Nowadays process lines are equipped with several screening and cleaning stages, pre- and post- flotation, and up to two dispersing systems with oxidative and reductive bleaching stages to achieve the required final stock properties. (Kraschowetz *et al.* 2008)

In order to improve the deinking process, other deinking lines are being considered. In this Master's Thesis, DIP fines fraction flotation is studied in detail, as part of a fractional deinking concept that is considered as an alternative deinking process. Fractional deinking concept in relation to fines flotation is shown schematically in Figure 1. Basically, recovered paper (a mixture between old newsprints and magazines) is pulped and then separated into short fibre fraction (also called fines fraction) and long fibre fraction in order to avoid excessive fragmentation and redeposition of ink. Pulping time used is very short and after fractionation long fibre fraction has a secondary

pulping. Flotation of the fines fraction is suggested to improve the yield for further combination of pulp streams that can be fed forward to the paper mill. (Körkkö *et al.* 2010)

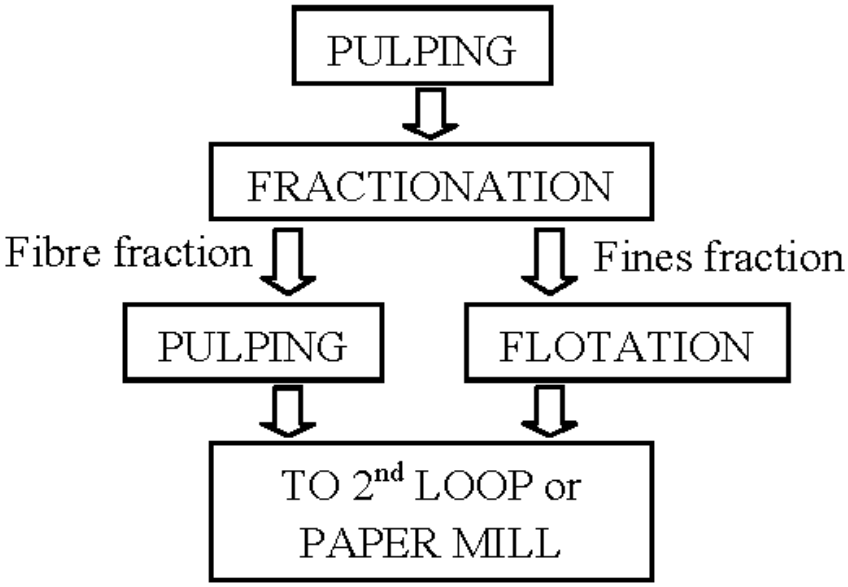


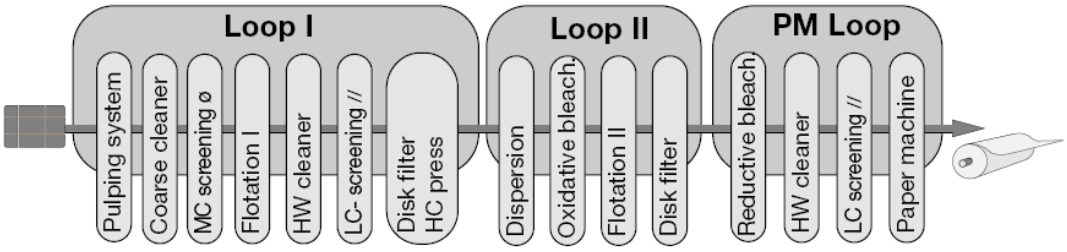

**Figure 1.** Block diagram of the studied fractional deinking (Körkkö *et al.* 2010).

The objective of this work was to obtain more information about the less studied fines fraction flotation and to optimize its properties. Theoretical part of this thesis is divided in three big chapters: in the first one, all the steps of a conventional deinking line are presented; in the second one, fractional deinking line concept is introduced; and finally fundamentals of flotation are viewed in detail.

# 2 CONVENTIONAL DEINKING LINE

The purpose of deinking is to remove printing ink and other substances that might affect the papermaking process or final properties of paper. For that reason, recycled fibres used in a deinking process require more complex process systems than virgin fibres because the recovered paper usually is a mixture of various paper grades and usually, it also contains a certain proportion of contaminants. The final stock properties depend on the removal of contaminants from the pulp. Unsatisfactory separation of contaminants results a low brightness and quality for the end product. (Lassus 2000 pp. 241, Holik 2000 pp. 91)

Some of the ink, like newsprint ink, is relatively weakly bonded to the fibres, and can be detached rather easily. On the other hand, toner or UV-cured ink may be strongly bonded to the fibres and cannot be detached easily in a pulper. Mechanical dispersion units or kneaders are needed to handle these inks. The production of newsprint and improved paper grades are nowadays usually carried out by using the process concept presented in Figure 2.




| | |
|---|---|
| Product: | Newsprint, and improved grades, approx. 40-90 g/m$^2$ |
| Furnish: | News, magazines |
| Yield: | Approx. 83 % |
| Effluent: | Approx. 8 L/kg |
| Ash: | Feed approx. 22 %, finished paper approx. 13 % |
| Brightness: | Finished paper approx. 63-66% ISO |

**Figure 2.** Concept for newsprint and improved paper grades (Schwarz 2000 pp. 213).

The unit operations of a deinking line are the following ones: pulping, screening, cleaning, flotation, dispersion, bleaching and dewatering. In pulping, mechanical, chemical and thermal forces are used to detach impurities from the fibres, such as ink.

This is usually carried out in pulpers where strong agitating provides shear force. Steam or hot water and deinking chemicals are added to help dislodge ink from the fibres. Screening, cleaning and flotation are separation stages. The detached ink is removed from the stock by screening, cleaning, flotation and washing stages. (Eul *et al.* 1990, Doshi 1997 pp. 3)

## 2.1 Pulping

The purpose of pulping is to slush the raw material into individual fibres as much as possible to form a suspension that can at least be pumped. During pulping, large, solid contaminants such as strings, plastic, stickies and printing ink, are removed from the pulp suspension. The disintegrating forces applied in the pulper must be greater than the strength of the raw material or the adhesion force of contaminants to the fibres. But pulping forces should not be excessive or contaminants will break excessively which leads to a less efficient removal. For that reason, it is desirable to remove solid contaminants at an early stage. To save energy, the first step is often limited to coarse slushing and heavy particle separation. (Holik 2000 pp. 95-97)

Chemicals are most often used in the pulping stage in order to improve ink release from the fibres even if there is a recent trend to move to neutral pulping conditions. For instance, NaOH swells fibres and increase slushing rate during pulping. Bleaching chemicals, such as hydrogen peroxide are also often used in this stage. The most important and oldest peroxide application in context of deinking is at the pulping stage. In industrial deinking operations, this peroxide application invariably occurs in an alkaline environment. To prevent yellowing of the mechanical fibres of the recycled pulp, peroxide has to be used in alkaline conditions. (Carré & Galland 2007, Ackermann 2000 pp. 322)

During pulping, a good detachment of the particles from the fibres is needed for further efficient treatment of the pulp suspension. Mechanical forces remove the ink through friction between fibres, and chemicals make some particles hydrophobic. Pulping variables such as pulper type or pulper chemistry influence the ink detachment most, because ink is mainly separated from the fibres during pulping. The aim is to free the

ink from the fibre and to prevent reattachment and fragmentation of the ink particles. The lower the ink fragmentation during pulping is, the higher will be the brightness after flotation. Chemicals during pulping and pulp pre-treatment using kneaders or dispersers improve ink detachment. (McKinney 1999 pp. 107, Beneventi *et al.* 2005, Holik 2000 pp. 151-153)

The presence of metal ions catalyses the undesired peroxide decomposition. The consequence is an increased addition of stabilizing chemicals such as water glass and possibly chelating agents. For an efficient use of hydrogen peroxide in pulper, the standard conditions are 15% to 18% consistency, 40 °C and 20 minutes pulping time. (Ackermann 2000 pp. 322, Eul *et al.* 1990)

Pulpers operate at high consistencies HC (up to 19%), at medium consistencies MC (up to 12%), and at low consistencies LC (up to 6%) depending on raw material. LC pulpers and HC drum pulpers usually operate continuously. HC pulpers (Helico) operate in batch mode. (Holik 2000 pp. 99)

## 2.2 Screening

The purpose of screening is to remove debris and solid contaminants from the recycled fibre pulp. Screening uses appropriate screen configurations according to particle size, shape, and deformability of the contaminants. Nondeformable contaminants whose dimensions exceed the screen opening size are retained while fibres go through the openings (slots or holes) of the screen. At the beginning, the screening devices were open vibrating screens, today the screens are closed and pressurised to increase capacity (Holik 2000 pp. 109-111, Carré & Galland 2007). The principle of screening is shown in Figure 3.

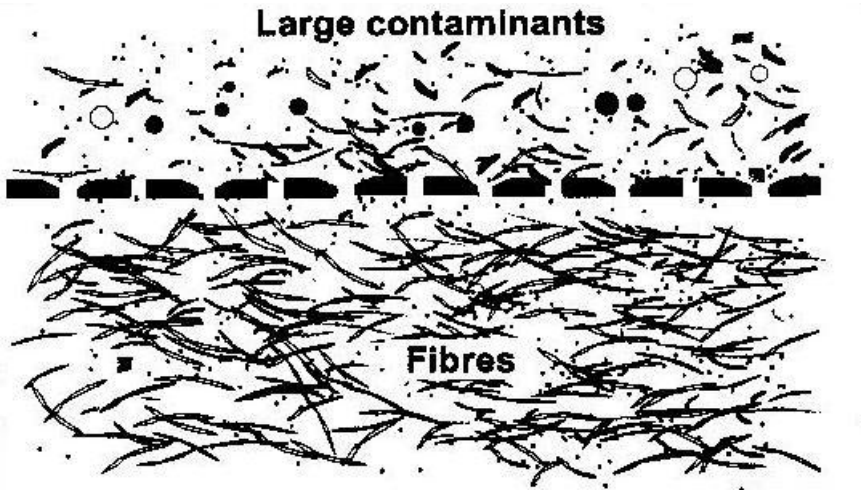



**Figure 3.** Principle of screening (Carré & Galland 2007 pp. 3).

Complete screening of recycled fibre pulp in a single stage is normally not possible. Avoiding fibre loss during screening is impossible as well. For these reasons, rescreening the first stage screen rejects in a second, third, or even fourth stage can reduce such losses significantly and improve the screening of recycle fibre pulp. The last stage (tailing screen) determines the overall fibre losses of the screening system. (Holik 2000 pp. 109)

Different types of screening machines have use at different points in the system depending on raw material, debris particles, flake content, and stock consistency. Coarse screening is used for pulp slurries with a high trash and flake content. Fine screening occurs in the LC range and partially in the MC range following coarse screening. For instance, large particles are removed by coarse screening while small particles (such as stickies) are removed by fine screening. Screen surfaces can be flat (disk screens) or cylindrical (cylindrical screens). Hole perforations are about 0.8 - 1.5 mm diameter for cylindrical screens and 2.0 - 3.0 mm diameter for flat screens. Slotted screens are generally in the cylindrical form with slot widths from 0.1 - 0.4 mm. (Holik 2000 pp. 119-120, 124)

## 2.3 Cleaning

Centrifugal cleaning is a separation process that complements other separation methods such as screening, and its purpose is to remove particles from the suspension that affect

paper quality or cause excessive wear in subsequent processing machinery. These contaminants may include heavy weight particles such as sand, metal pieces, and shives or light weight particles including plastic foam or other plastic material. (Holik 2000 pp. 134-135)

For an efficient removal of contaminants, their density must differ from water. Centrifugal cleaning can remove smaller particles than screening. The centrifugal field generated in these machines forces the heavy particles outward while the light ones move to the centre. The flow streams containing primarily heavy or light contaminants are separated from the main flow. (Holik 2000 pp. 134-135)

## 2.4 Flotation

Flotation is the most common method used for separating ink and fibres from each other. It is a separation process for suspension cleaning based on probability using air bubbles that attach themselves to the particles and transport them to the surface of the suspension. Deinking flotation has been used in recycled pulp processing for removing contaminants from the suspension, thanks to the different surface hydrophobicity of the particles to be removed and the fibres to be retained (Holik 2000 pp. 151-152, 241). The principle of flotation is shown in Figure 4.

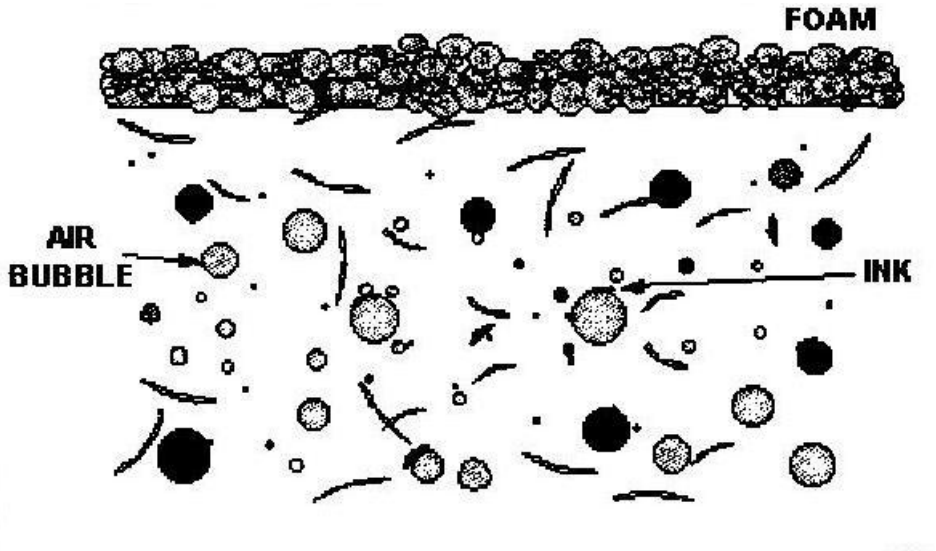



**Figure 4.** Principle of flotation (Carre & Galland 2007 pp. 4).

Once a good detachment of the particles from the fibres is achieved through pulping, air is introduced into the suspension and the water-repelling particles attach to the air bubbles and rise to the surface forming a foam layer at the top of the suspension, while the hydrophilic fibres remain in the water phase. These hydrophobic particles include printing ink, stickies, fillers, coating, pigments and binders. The foam can be removed mechanically, by overflow or by a vacuum extraction. (Holik 2000 pp. 151-153, 244)

In flotation systems air is introduced into a diluted fibre suspension of 0.8 - 1.5% consistency. The relative air load is mostly about 300% or more, expressed as total air volume flow to total suspension volume flow. pH should be about 7 - 9 when using fatty acid surfactants and temperatures of 40 – 70 ºC. The main application of selective flotation in the paper industry is deinking. This improves pulp brightness by removing ink particles and enhances cleanliness by removing dirt specks (Holik 2000 pp. 151, 157, 244). Flotation is discussed more deeply in Chapter 4.

## 2.5 Dispersion

The purpose of dispersion is to break down contaminants to a size where they usually no longer interfere or detach them from the fibres if necessary to remove them in a subsequent process. Dispersion makes dirt specks and stickies smaller and floatable, mixes in bleaching agents and detaches ink still adhering to the fibres. Two kinds of dispergers are used in the dispersion stage: disk and kneading dispergers. (Holik 2000 pp. 185-186)

Recycled pulp is dispersed at consistency of 25 - 30% and temperatures of 60 - 95 °C. These process conditions and the high shear environment are ideal for admixing of peroxide. Dispersion can be done also without NaOH and hydrogen peroxide. (Ackerman 2000 pp. 232)

## 2.6 Bleaching

Bleaching contributes decisively to the optical characteristics of DIP such as brightness and luminance. The bleached deinked pulp is usually used in the production of printing

papers such as super calendared (SC) and office papers. Especially with hygienic papers the microbial activity can be stopped with hydrogen peroxide.

Bleaching chemicals are divided into two groups: lignin preserving such as hydrogen peroxide ($H_2O_2$), sodium dithionite ($Na_2S_2O_4$) and formamidine sulphinic acid (FAS), and lignin degrading such as chlorine ($Cl_2$), chlorine dioxide ($ClO_2$), sodium hypochlorite (NaOCl), ozone ($O_3$) and oxygen ($O_2$). Lignin preserving chemicals are normally used for wood containing papers, and lignin degrading chemical (if used) for wood free papers. (Ackermann 2000 pp. 307)

The choice of bleaching chemicals and processes depends on the demanding quality of the final product. Besides fibre composition, this also includes different types and proportions of contaminants and detrimental substances still present in the deinked stock. (Ackermann 2000 pp. 307)

The bleaching of deinked pulp at the end of stock processing is post-bleaching. The deinked pulp is thickened and heated with steam to a temperature of approximately 60 °C. After adding bleaching chemicals, the pulp is stored in a bleaching tower at a consistency of about 15% for 1 - 3 hours. (Ackermann 2000 pp. 324)

## 2.7 Dewatering

Dewatering is a filtration process with mechanical pressing and its purpose is to remove process water from the pulp slurry and increase the stock consistency simultaneously. Dewatering makes subsequent processes at the production level more cost-effective and saves space in further storage. HC dispersion is an example of these subsequent processes that can be improved thanks to dewatering. Stock dewatering characteristics are normally expressed in the paper industry as Schopper-Riegles value (SR) or Canadian Standard Freeness (CSF). Dewatering without further mechanical pressing is often called thickening. The most common dewatering machines used in recovered paper processing are belt filters, drum filters, disk filters and screw presses. During filtration, a filter cake or filter mat is accumulating on the filter base such as a wire or screen. (Holik 2000 pp. 168, 169, 171, 172)

# 3 FRACTIONAL DEINKING LINE

Fibre fractionation and separate bleaching of the fractions (long and short fibre) could be an efficient way to upgrade deinked pulp and its properties. In fact, not only cleanliness of the stock with regard to stickies and cubic impurities is important, but optical properties such as brightness, dirt specks and bleachability of the stock are a challenge for deinking process to reach the exacting demand of the final stock. (Eul *et al.* 1989)

Fractional deinking lines are a development of conventional deinking lines. In fractional deinking lines, deinked pulp is separated into a long fibre fraction and a short fibre fraction (or fines fraction) through fractionation. Fractionation is a special screening application that splits a given fibre quality into two fractions, so these fractions can be treated separately and used for different paper grades. Fractionation can also be done by washing. The principle of fractionation is shown in Figure 5.

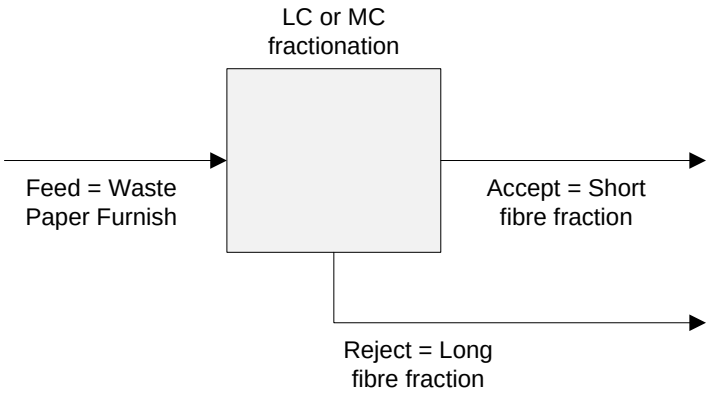



**Figure 5**. Principle of fractionation (Kraschowetz *et al.* 2008).

The fractionation concept has been introduced successfully in deinking lines. The main reason of this development and adjustment of fractionation into the deinking process is a worldwide increasing use of recovered paper to produce paper and board for environmental reasons. Increased collection of recycled material has resulted in increased quantities of non-fibrous materials or contaminants. Therefore, fractionation creates a superior long fibre fraction and minimizes the cleaning and screening effort by concentrating contaminants in one stream. (Kraschowetz *et al.* 2008, Meltzer 1999 pp. 153)

The efficiency of every fractionation experiment is focused on the flow characteristics of the screen surface. Based on fractionation efficiency, three different groups of factors or parameters can be identified.

- First group are the properties of the individual fibre such as length, thickness, coarseness, stiffness, and surface characteristics. The flow characteristics are influenced by these fibre properties and by the concentration of fibres in the suspension.

- The next group is the machine factors. Screen basket is a determining parameter because it is the physical barrier for all the particles. Fractionation efficiency depends on screen basket properties such as the type of aperture, hole or slot, the aperture's size and the surface profile. Other parameters regarding the housing and the rotor have gained importance in fractionation efficiency. Rotor design, the clearance between rotor and screen, the number of pulsation elements, and the rotor speed determine frequency, size, and shape of the pressure and suction pulses. Housing can be pressurized or at atmospheric pressure.

- Finally, the third group includes operating parameters. Consistency, reject rate, system design and rotor speed influence aperture velocity and flow characteristics. (Meltzer 1999 pp. 156)

## 3.1 Differences between screening and fractionation

The basic purpose of screening is the removal of disturbing components with a minimum loss of usable fibres. On the contrary, fractionation focuses on the separation of the stock flow into two or more fractions. Though fractionation and screening are based on the same principles, their focus is quite different.

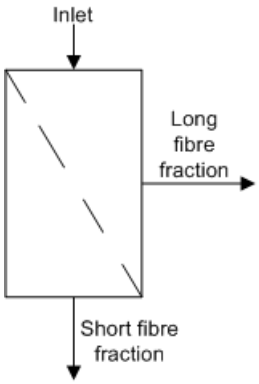
The performance of a screening system is determined by accept quality with several contaminants removed as reject. However, a fractionation system is typically evaluated by properties of the long fibre fraction. The main differences between screening and fractionation are explained in Figure 6.

**Fractionation:**

Separation of fibre stock into
stock fractions with different fibre characteristics

Applications:

- Further selective treatment of the two stock fractions
- Different fibre stock properties for multi-layer and multi-ply concepts or individual paper qualities




**Screening:**

Mechanical removal of undesired disturbing components

- High separation efficiency
- Minimum reduction in size of disturbing components
- Low fibre loss

Applications:

- RCF screening, approach flow, broke screening

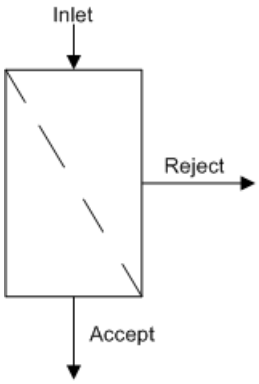



**Figure 6.** Fractionation and screening concepts (Meltzer 1999 pp. 153).

Fractionation uses normal screening machinery with some differences regarding equipment and operation conditions. Fractionation can use disk screens and cylindrical screens, with smaller holes than for screening. Hole perforations are about 0.8 - 1.5 mm diameter for cylindrical screens and 2.0 - 3.0 mm diameter for flat screens. Slotted screens baskets have narrower slots, zero-angle profile, or both, and are generally in the cylindrical form with slot widths from 0.1 - 0.4 mm. (Holik 2000 pp. 109, 127)

## 3.2 Properties of the fibre fractions

Fractionation separates fibres according to characteristics such as their length or flexibility. It is not possible to achieve a strict separation of long and short fibres with fractionation, usually an enrichment of long fibre concentration in one line is possible. (Holik 2000 pp. 127)

The two fractions have different characteristics:

The short-fibre fraction (SF):

- enrichment of fines, ash and short fibres
- ink particles already detached from the fibres
- bleach-inhibiting substances (ash and fines).

The long-fibre fraction (LF):

- enrichment of long fibres
- ink particles still sticking onto the fibres
- stickies.

Separate bleaching stages for long and short fractions could result in better brightness values than applying these chemicals to entire pulp fraction. In entire pulp fractions, fines leads to an over/consumption of bleaching agents. Due to fractionation, optimum bleaching conditions can be reached. The long fibre (LF) fraction can be bleached very efficiently with hydrogen peroxide ($H_2O_2$), and the short fibre (SF) fraction can be bleached with reductive bleaching agents, if necessary. (Eul *et al.* 1989, Eul *et al.* 1990, Lapierre *et al.* 2003, Kraschowetz *et al.* 2008)

Fibre fractionation improves brightness of both fractions. The LF fraction bleached with peroxide reached an ISO brightness of 75%, while the fines fractions bleached with hydrosulphite reached an ISO brightness of only 56%. (Floccia *et al.* 1996, Lapierre *et al.* 2003)

## 3.2 Place of fractionation

Fractionation can be implemented in DIP lines for newsprint as well as for higher paper grades, such as SC or LWC. The goal of fractionation is to reduce the number of deinking loops of a conventional deinking system. The DIP system for newsprint presented in Figure 7, consists of two-loops with oxidative bleaching and an optional reductive bleaching.

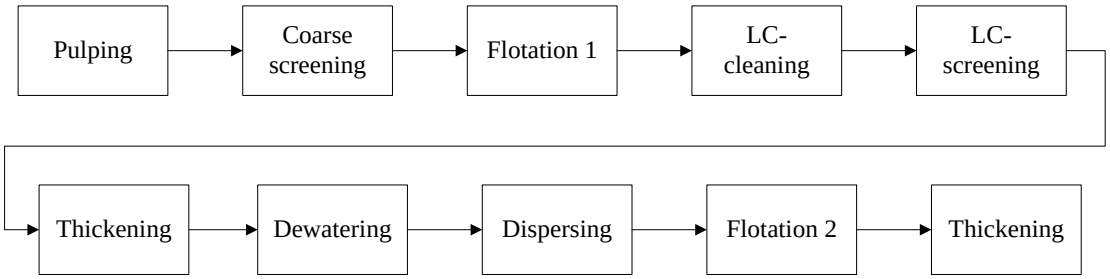



**Figure 7.** Conventional DIP system for newsprint (Kraschowetz *et al.* 2008).

Two new system configurations can be developed depending on the position of the fractionator in the DIP system for newsprint previously shown: fractionation at low consistency when the fractionation is positioned after the pre-flotation, and fractionation at medium consistency when the fractionation is placed right after the pre-thickening.

Figure 8 shows DIP system with fractionation at low consistency. Two separate disc filters (one for the long, one for the short fibre) are required. Several cost saving are achieved with fractionation at low consistency. Separating the long fibre fraction requires smaller equipment for high consistency thickening and dispersing. Savings in electric energy, steam consumption and bleaching chemicals are also achieved.

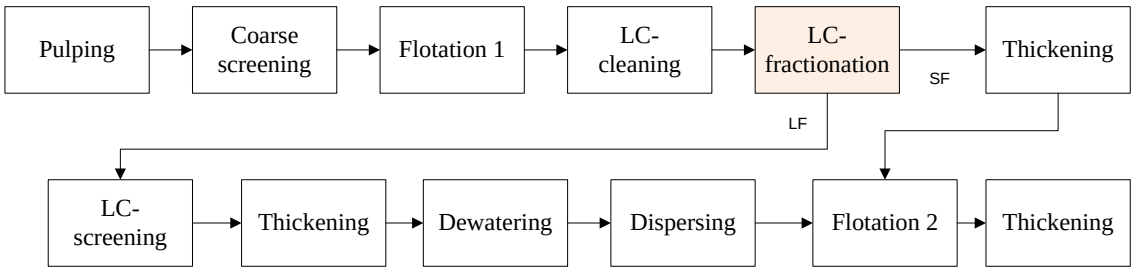



**Figure 8.** Fractionation at low consistency (Kraschowetz *et al.* 2008).

Figure 9 shows DIP system with fractionation at medium consistency. There are not as much savings advantages as in fractionation at LC, because one additional screen (fractionators is required) and long fibre fraction consistency values are quite low. In the other hand, thickening of long fibre fraction is much easier than entire pulp or short fibre fraction thickening. (Kraschowetz *et al.* 2008)

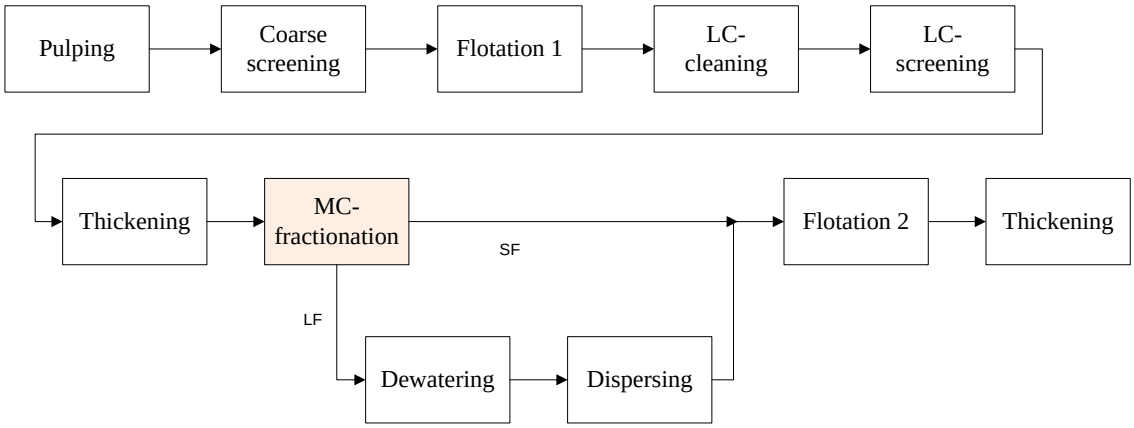



**Figure 9.** Fractionation at medium consistency (Kraschowetz *et al.* 2008).

Recent studies are focused on the use of fractionation during pulping. The goal of fractionation during pulping is to separate the detached ink particles from the fibres as early as possible and continue pulping of the fibre fraction with undefibered material. This fractionation has been achieved through several washing stages. Usage of multiple washing stages and continued pulping of fibre fraction resulted in the best quality of stock in terms of attached residual ink content and the highest ink detachment efficiency. (Kemppainen *et al.* 2010)

# 4 FUNDAMENTALS OF FLOTATION

Froth flotation is a separation process applied to separate ink from recovered paper stock. This separation is based on probability using air bubbles that attach themselves to the particles and contaminants of the pulp slurry and its migration to the surface of the suspension where they form a froth that can be removed by suction or overflowing. The efficiency of flotation is strictly related to the ink particle/air bubble hydrodynamic and physicochemical interactions. In recovered paper application, dispersed ink particles from fibres are made hydrophobic and are agglomerated to improve the selectivity of the attachment to the air bubbles. (Somasundaran 1998 pp. 83, Holik 2000 pp. 151, Beneventi *et al.* 2005, Dorris *et al.* 2011)

Selectivity of the air bubbles is the main difference between froth flotation and dissolved air flotation (DAF). The objective of dissolved air flotation is to remove all suspended solids, rather than separate different types of solids, especially ink and contaminants, which is the aim of froth flotation. In froth flotation, chemicals are added to make ink particles more hydrophobic and improve the attachment between ink and air bubbles (McKinney 1998 pp. 99-100). In this thesis, froth flotation is used.

During flotation deinking, ink and other contaminants are removed, but also flotation removes some valuable material such as ash, fillers, fibres and fibre fines. For a better understanding of the components removal during flotation deinking, the different mechanisms of particle separation from the pulp should be presented. Four main transport mechanisms describe the particle separation from the pulp suspension thanks to air bubbles (Beneventi *et al.* 2005). These four transport mechanisms are shown in Figure 10.

*i)* *True flotation.* Hydrophobic particles adhere themselves to the surface of air bubbles and are transferred to the froth.

*ii)* *Mechanical entrainment.* Hydrophobic and hydrophilic particles are both transported into the froth by air bubbles.

*iii)* *Water drainage.* Gravitational drainage of water from air bubbles in the froth into the pulp slurry

*iv)* *Solid drainage.* Water drainage can convey down to the pulp suspension solid particles of the froth.

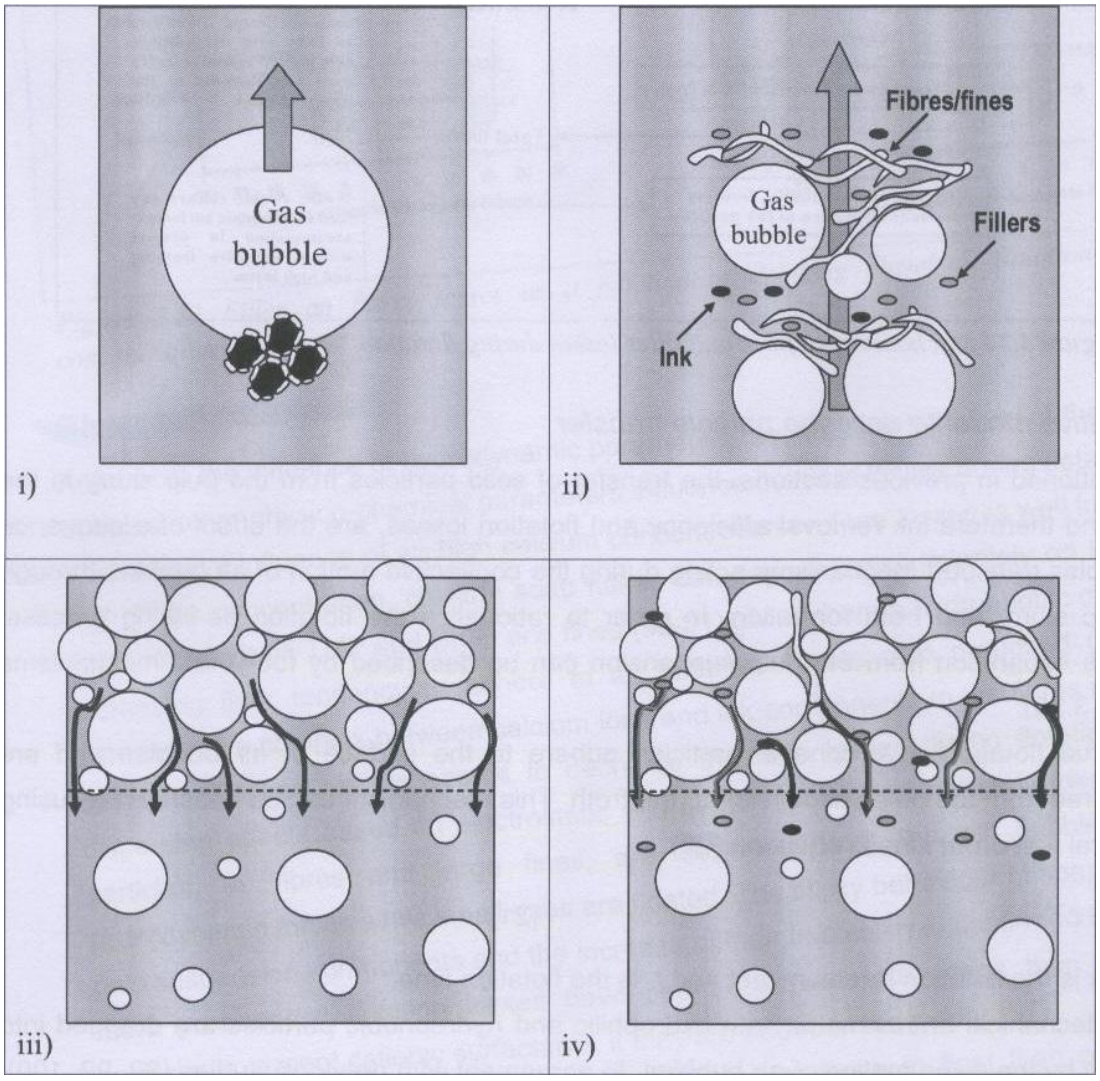



**Figure 10.** The four main solid transfer mechanisms used to describe the deinking flotation process. i) True flotation of hydrophobic particles, ii) mechanical entrainment of suspended solids, iii) water drainage, iv) drainage of solids dispersed in the froth (Beneventi *et al.* 2005).

## 4.1 Flotation phases

Many of the steps needed for a successful flotation are beyond the control of flotation deinking cell manufacturers. These steps are affected by several factors such as consistency, pH, temperature, size of air bubbles, air ratio, etc. A successful flotation requires:

- Ink detachment from fibre (shown in Chapter 2.1),
- a collision between an air bubble and a free ink particle,
- the formation of an air bubble-ink particle complex,
- migration of the complex to the surface,
- froth removal of air bubble-ink particle complex from the flotation cell.

### *4.1.1 Collision between an air bubble and a free ink particle*

Air bubble and ink particle has to be close enough to collide and form an air bubble/ink particle complex. A particle must be within the radius of the streaming tube, which is defined by the diameter of the air bubble and fluid streamlines. Particle and bubble size are factors that affect the probability of collision. The higher the particle size is and the smaller the bubble size is, the higher the probability of collision is. The effect of bubble size on flotation efficiency is shown in Figure 11. Flotation cell design influence the air bubble size and the degree of mixing and turbulence. (McKinney 1999 pp. 107-108, Julien Saint Amand *et al.* 2005)

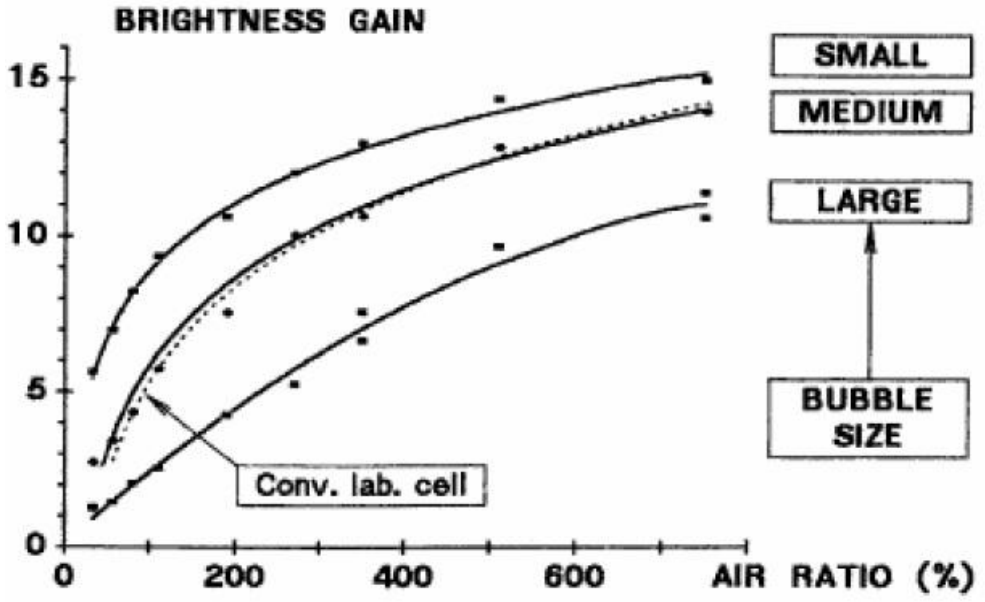



**Figure 11.** Effect of bubble size and particle size on flotation efficiency (Julien Saint Amand *et al.* 1999).

### *4.1.2 Formation of an air bubble-ink particle complex*

The most important factor that influences the formation of an air bubble/ink particle complex is the surface chemistry of the ink particle and the air bubble. Other factors are ink particle size and shape. Chemicals added are generally referred to as ink collectors and are surface active agents. It is important that small ink particles are agglomerated before flotation to improve the flotation efficiency. Traditional chemicals used are non saturated fatty acid soap, usually a blend of soaps with variations in chain length.

This soap reacts with the ink particles and calcium ions (determined by water hardness) to form a strongly hydrophobic complex that attached to the air bubble. Other surfactants are also used as ink collectors, with lower addition rates and less dependence of calcium hardness. The effect of the hydrophobic chain length of the soap in the pulp brightness is shown in Figure 12. Conditions for flotation should be alkaline with fatty acid soaps, typically in the pH range 8 - 10. (McKinney 1999 pp. 108-109, Beneventi *et al.* 2005)

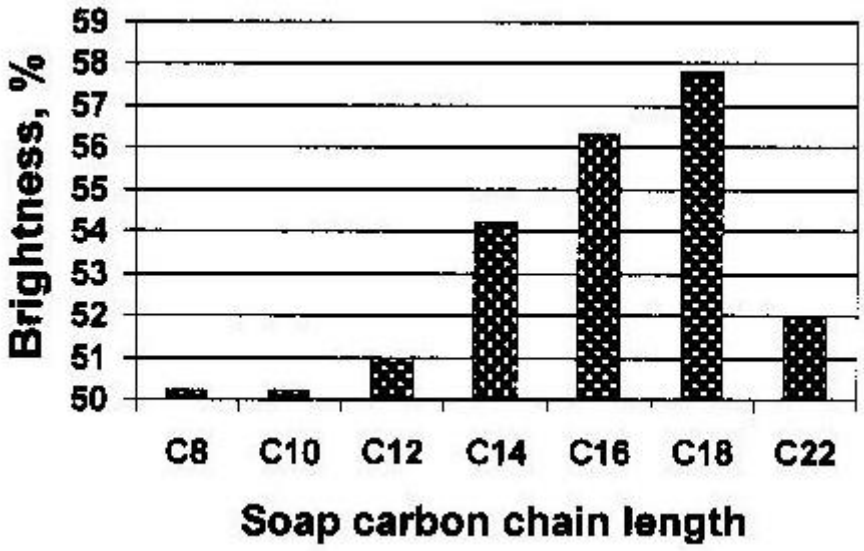



**Figure 12.** Effect of the hydrophobic chain length of the soap on the flotation efficiency (Beneventi *et al.* 2005).

Ink particle size is affected by the type of ink, age of ink and the treatment prior to flotation. Increasing ink particles size decreased the ink removal efficiency. For an efficient flotation of ink particles, their size range must be 10 – 250 mm. Regarding to

ink particle shape and according to Schmidt and Berg (1996), flat shaped particles did not float as well as spherical particle because of the low attachment probability of the disc shaped particles after collision between particle and bubble. (Holik 2000 pp. 153, McKinney 1999 pp. 108, Julien Saint Amand *et al.* 2005)

### *4.1.3 Migration of the complex to the surface*

Even if ink particle/air bubbles complexes are successfully formed, there is no guarantee that the ink will be removed from the flotation cell. It depends on factors such as the stability of the complex, consistency, turbulence, position relative to the surface, bubble complex rise rate, etc. Adhesion probability and migration to the surface is higher with smaller bubble rising velocity. Also small bubbles shown to be best, due to the higher amount of bubbles and surface area. (McKinney 1999 pp. 109, Beneventi *et al.* 2005)

### *4.1.4 Froth removal*

The air bubble/ink particle complex has to be stable enough to resist the forces applied by the froth removal system. Froth removal is accomplished by the overflow, sometimes assisted by a rotating mechanical scraper. The probability of aggregate stability depends on several parameters: gravity and centripetal acceleration which tend to separate heavyweight particles from the bubble, shear forces and attachment forces due to capillary force on air bubble/ink particle complex. For instance, large particles detach from the air bubbles especially at high turbulence. The presence of fibres in the pulp suspension is another factor that influences the probability of aggregate stability. (McKinney 1999 pp. 109, Beneventi *et al.* 2005)

## 4.2 Flotation yield

During flotation, valuable materials such as ink, ash, filler and fibres are also removed with the froth. Below is presented the variables affecting fibres and fines losses and an introduction of fines fraction flotation.

### *4.2.1 Fibres and fines losses*

Fibres and fines losses are influenced by several factors such as chemicals and air bubble size. Chemicals improve the selectivity of the flotation process making particles more hydrophobic attracting air bubbles. Without this selectivity, the flotation process would remove particulate matter in a non-selective manner (as in DAF) leading to the loss of valuable fines and fillers by entrainment rather than true flotation. This entrainment phenomenon in flotation deinking is particularly harmful. (Dorris *et al.* 2011)

A high calcium concentration has a negative influence on fibre losses. At high $Ca^{2+}$ concentration, calcium soap flakes can deposit on ink particles, fillers, fibres and fines, increasing their tendency to attach to air bubbles. Nevertheless, sodium silicate has a positive influence on fibre losses. Fibre losses decreased if sodium silicate is used during flotation, due to its dispersing effects based on electrostatic and steric repulsion. (Beneventi *et al.* 2005)

Air bubble size is a major factor affecting on pulp losses. If 0.01 mm to 0.1 mm sized air bubbles were created in flotation deinking cells, all the solids, including fibre, would float, and there would be no separation of ink from fibre. In froth flotation, air bubble sizes range from about 0.1 mm up to 5.0 mm, and differences in properties between the suspended solids (such as surface chemistry) assist the separation of solids (McKinney 1999 pp. 100).

According to Körkkö (2008), ash selectivity in removal was practically unaffected by bubble size and fibre fines loss was higher with larger bubbles. The effect of bubble size on pulp loss and fines and fillers loss are represented in Figures 13 and 14. According to this figures, the amount of flotation rejects increases as bubbles size is decreased.

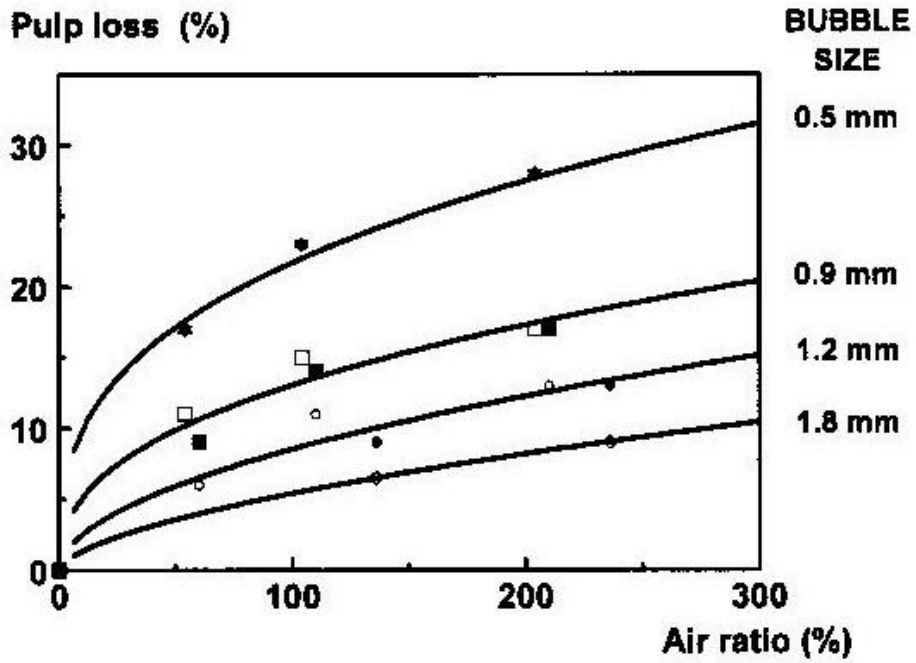



**Figure 13.** Pulp losses vs. air ratio for different bubble sizes (Julien Saint Amand *et al.* 1999).

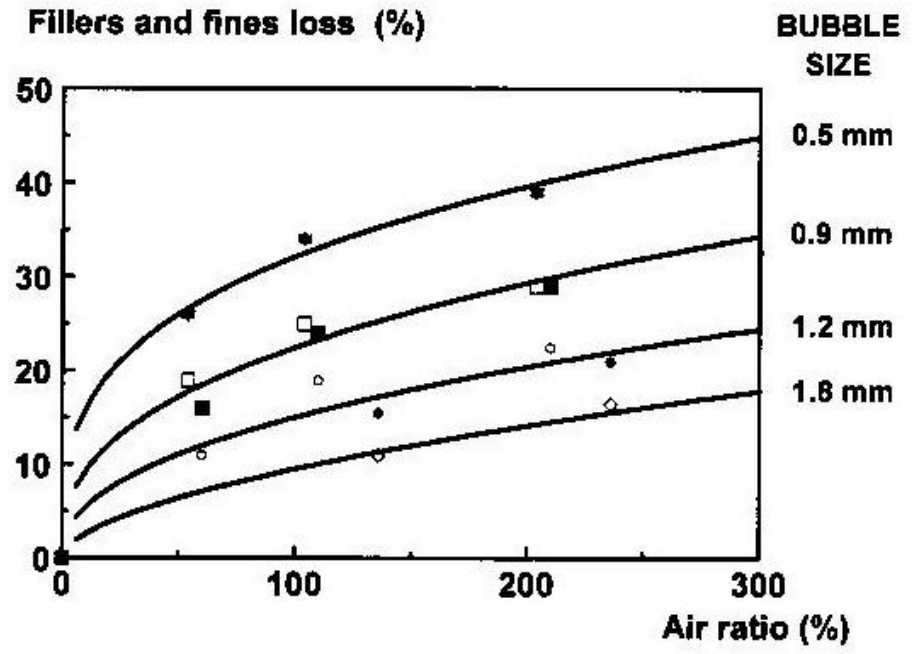



**Figure 14.** Fillers and fines losses vs. air ratio for different bubble sizes (Julien Saint Amand *et al.* 1999).

The mechanisms involving losses of fibre, filler, fines and inorganic components during flotation are represented in Figure 15.

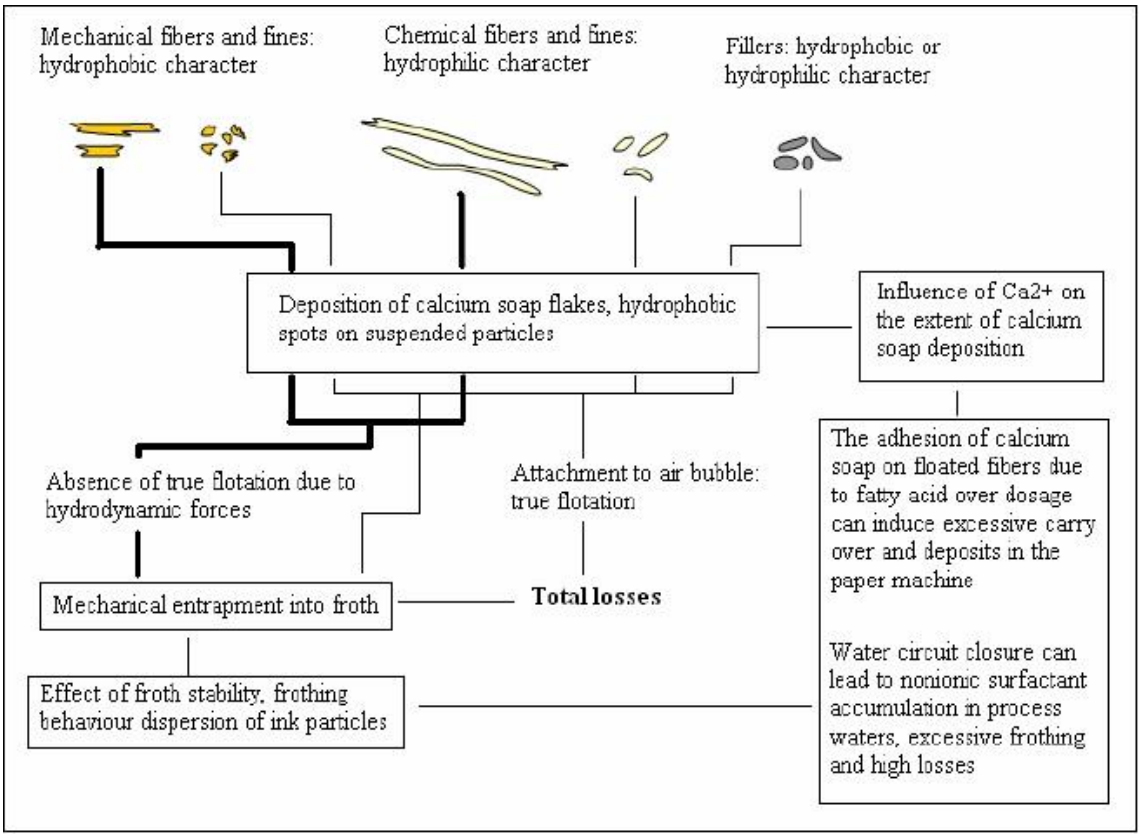



**Figure 15.** Mechanisms of fibre and filler losses during flotation (Beneventi *et al.* 2005).

### *4.2.2 Fines fraction flotation*

The study of flotation of deinked pulp samples without fibres could help for further developments in deinking line processes but nowadays there are not a lot of articles and information about this short fibre or fines fraction flotation.

According to Dorris (1995), flexographic inks, that are prone to float in normal deinking flotation conditions, were found to be floatable under various conditions when there was no fibre in the system, conditions such as low pH or high concentration of calcium ions. The use of conventional calcium soaps of fatty acids was effective as a flotation collector for oil-based inks and also for flexographic ink. According to Chabot (1997), the efficiency of flotation of flexo-printed paper decreased as fibre concentration increased. Fibres have an adverse role during the flotation of flexographic inks, affecting the formation and growth of flexographic ink-calcium soap aggregates.

Matzke (1996) proposed a new development in deinking and bleaching through flotation of washing filtrate. It was shown that flexographic inks can be floated from a wash filtrate because of its low viscosity and the absence of fibres. By this mean the yield could be increased while retaining a high brightness level.

# 5 EXPERIMENTAL

The experimental part of this work was divided in five different scenarios of flotation experiments. The first scenario was the reference flotation experiments with the whole pulp sample. The second scenario was performed to determinate the optimum variables for air flow and flotation level at the fines flotation. And the other three different cases were performed using several chemicals during the fines fraction flotation. Before a deeply description of the flotation experiments, an introduction about the materials and methods used is needed.

## 5.1 Materials

### *5.1.1 Raw material*

The recovered paper used in the experiments was a mixture of old Finnish newsprint (ONP) and old magazines (OMG). The feeding ratio for every pulping was 50% ONP and 50% OMG. The OMG feed consisted half of super calendared paper (SC) and half of light weight coated paper (LWC). The recovered paper used is considered as a very old raw material because it is older than three months. All papers used were sorted by hand in order to get batches of uniform quality for pulping.

### *5.1.2 Chemicals*

Three different commercial chemicals were used during the process: surfactant #1, surfactant #2 and soap. Surfactant #1 was a non-ionic surfactant, surfactant #2 was a slightly different non-ionic surfactant and the soap was a mixture of sodium soaps of fatty acids (i.e. anionic surfactant). Every experiment in which soap has been used, 7.2 g of $CaCl_2$ was also added to adjust the hardness. The dosing place of these chemicals and its dosage were different in several flotation experiments.

## 5.2 Methods

### *5.2.1 Process description*

*Pulping*

The pulp was produced using a Hobart (H600) pulper with a hook impeller as presented in Figure 16. Batch size in Hobart pulping was 2500 g. All the newsprints and magazines were torn into about two centimetre wide strips and mixed with warm tap water (45 °C) before pulping to adjust the consistency to 15%. Pulping time was 14 minutes while in every pulping experiment the first two minutes were performed at speed #1, the next two minutes at speed #2 and the last ten minutes at speed #3.


**Figure 16.** Hobart (H600) pulper.

*Fractionation*

Fractionation was performed using a PULA pilot that consisted of a mass tank of 300 litres, a pump and a screen basket with a slot width of 0.15 mm. The pulp was diluted in the feed tank with warm tap water (45 °C) into a consistency of about 0.8%. Experiments were performed with an accept flow of 0.05 litres per second and a screen rotor frequency of 50 hertz. At the end, the accept flow (short fibre fraction) of every fractionation experiment was mixed thoroughly for the short fibre flotation.

*Flotation*

Flotation was performed using the Voith Delta 25 flotation cell equipped with level box to maintain constant fluid level by injecting tap water into the cell during flotation as presented in Figure 17. Fines fraction samples from every fractionation experiment were used as input in the flotation cell. An electric heater was used to keep the fines fraction samples at 45 °C.


**Figure 17.** Voith Delta 25 flotation cell.

### *5.2.2 Analyses*

*Consistency and ash*

The consistency of pulp samples was determined according to SFS EN ISO 4119 standard. The ash content was done on ignition at 525 ºC according to ISO 1762 standard. Consistency and ash content values were used to calculate mass reject rate and ash removal with the following equations.

$$RR_m = \frac{c_{reject} \cdot m_{reject}}{c_{feed} \cdot m_{feed}} \cdot 100\%$$

where

$c_{reject}$ is consistency of the reject [%],

$m_{reject}$ is the mass of reject [g],

$c_{feed}$ is consistency of the feed sample [%] and

$m_{feed}$ is the mass of feed sample [g].

$$\text{Ash Removal} = \frac{c_{reject} \cdot m_{reject} \cdot Ash_{reject}}{c_{feed} \cdot m_{feed} \cdot Ash_{feed}} \cdot 100\%$$

where

$c_{reject}$ is consistency of the reject [%],

$m_{reject}$ is the mass of reject [g],

$c_{feed}$ is consistency of the feed sample [%],

$m_{feed}$ is the mass of feed sample [g].

$Ash_{reject}$ is the ash content of the reject [%] and

$Ash_{feed}$ is the ash content of the feed [%].

*Pads*

Pulp pads were prepared to determine the optical properties of the pulp. Cellulose nitrate membrane filter (from Sartorius, 50 mm of diameter), with a porosity of 0.45 µm to avoid the loss of ink, were used to prepare the pads. Opaque pads were prepared to reach a basis grammage of 225 g/m$^2$ (INGEDE Method 11p, 2009).

*Low grammage sheets*

Low grammage sheets (30 g/m$^2$) were prepared on a sheet mould using a high retention filter paper (Körkkö *et al.* 2010). The prepared sheets were acclimatized in the paper laboratory conditions prior to residual ink measurement.

*Optical measurements*

Optical properties such as brightness and residual ink were measured from the pads and sheets on the following day using an L&W Elrepho spectrophotometer. The optical properties were determined by calculating the average of two pads measurements.

- *Brightness:* Defined as the reflectance of blue light, ISO brightness is an intrinsic reflectance factor determined with a brightness meter whose sensitivity to light agrees with ISO standard 2470. The centroidal wavelength for the brightness function is 457 nm and that is the origin of the abbreviation R457. (Crawford 1999 pp. 4 and Vaarasalo 1999 pp. 173)

- *Residual ink:* The presence of ink influences the brightness and colour of recycled paper. The concentration of ink is determined by effective residual ink concentration (ERIC) method. The residual ink content of each sample was determined by infrared reflectance measurement according to ISO 22754, but using a wavelength of 700 nm instead of 950 nm. ERIC is obtained using the following equations.

$$s_{950} = \frac{1000}{g} \cdot \frac{R_\infty}{(1 - R_\infty^2)} \cdot \ln \frac{R_\infty (1 - R_0 R_\infty)}{R_\infty - R_0}$$

where

$s_{950}$ is the scattering coefficient at 950 nm [m2/kg],
$g$ is the grammage of the sheet [g/m2],
$R_0$ is the single-sheet reflectance factor and
$R_\infty$ is the intrinsic reflectance factor.

$$k_{950} = \frac{s_{950}(1 - R_\infty)^2}{2R_\infty}$$

where

$k_{950}$ is the light absorption coefficient at 950 nm [m2/kg].

$$\text{ERIC} = 10^6 \left(\frac{k_{sheet}}{k_{ink}}\right)$$

where

ERIC is the ERIC number [ppm],

$k_{sheet}$ is the light absorption coefficient of paper containing ink [$m^2$/kg] and

$k_{ink}$ is the light absorption coefficient of the ink itself i.e. 10000 $m^2$/kg.

*Hyper washing (HW)*

Hyper washing was made by washing pulp with water flow of 8 L/min for 20 minutes with Sommerville-type screening device using a 150-mesh wire screen. Retention in hyperwashing was calculated using the following equation.

$$\text{Retention} = \frac{c_{HW} \cdot m_{HW}}{m_{pulp}} \cdot 100\%$$

where

$c_{HW}$ is consistency of the hyperwashed pulp [%],

$m_{HW}$ is the mass of pulp suspension [g] and

$m_{pulp}$ is the mass of dry pulp before hyperwashing [g].

Once retention was obtained, fines removal was calculated using the following equation.

$$\text{Fines Removal} = \frac{c_{reject} \cdot m_{reject} \cdot (100 - \text{Retention}_{reject})}{c_{feed} \cdot m_{feed} \cdot (100 - \text{Retention}_{feed})} \cdot 100\%$$

where

$Retention_{reject}$ is the retention of the reject sample [%] and

$Retention_{feed}$ is the retention of the feed sample [%].

## 5.3 General construct of experimental part

In this chapter, flotation experiments are explained in detail. Schematic diagrams of these flotation experiments are presented in Appendix 1.

### *5.3.1 Reference flotation*

After pulping, the pulp slurry was diluted with warm tap water (about 45 ºC) to a consistency of 1.2%. Five reference flotation experiments were done using entire pulp samples. A soap dosage of 6 kg/t was added in the flotation cell, with 7.2g of $CaCl_2$ to adjust hardness. In these five experiments, the flotation level was 19 litres and the air flow was 7.4 L/min, but in every flotation the mass reject rate was different. The variables of these flotation experiments are presented in Table 1.

**Table 1.** Reference flotation variables.

| *Test Run* | *Flotation level (L)* | *Air flow (L/min)* | *Chemicals (kg/t)* |
|---|---|---|---|
| 37 | 19 | 7.4 | 6 |
| 38 | 19 | 7.4 | 6 |
| 39 | 19 | 7.4 | 6 |
| 40 | 19 | 7.4 | 6 |
| 41 | 19 | 7.4 | 6 |

### *5.3.2 Fractionation*

The target of the fractionation was to have 35% long fibres and 65% short fibres. After pulping without chemicals, the pulp slurry was diluted with warm tap water (about 45 ºC) to a consistency of 1.2%. Samples from a fractionation experiment were taken to compare the data obtained by the different flows of the PULA fractionation device: feed, accept (short fibre) and reject (long fibre).

### *5.3.3 Flotation level and air flow during flotation*

After pulping and fractionation, twelve flotation experiments were carried out in four groups of three experiments each. In every group of three experiments, flotation level and air flow were changed in order to reach the optimal values of both variables. In every group of experiment conditions, the difference between the three flotation experiments was the amount of reject. No chemicals were used in these flotation experiments. The values of these variables are shown in Table 2.

**Table 2**. Flotation level and air flow values for the different flotation experiments.

| ***Test run*** | ***Flotation level [l]*** | ***Air flow [l/min]*** |
|---|---|---|
| 1, 2, 3 | 19 | 7.4 |
| 4, 5, 6 | 20 | 7.4 |
| 7, 8, 9 | 19 | 13 |
| 10, 11, 12 | 20 | 13 |

### *5.3.4 Conditioning prior to flotation*

After pulping and fractionation, a 9.5 kg/t dosage of commercial soap was added to the short fibre fraction. Flotation level and air flow was set to 19 litres and 7.4 litres per minute. Five different flotation experiments were carried out with a higher delay of the chemical in every experiment. The parameters for every flotation are shown in Table 3. Test 13 was used as a reference for the other experiments. In every flotation that commercial soap was used, 7.2 g of $CaCl_2$ were added to adjust the hardness. The dosing place of this amount of $CaCl_2$ was the flotation device, except for test 18 in which the $CaCl_2$ was added in the bucket at the same time as the commercial soap.

**Table 3.** Parameters for delay chemical flotation.

| *Test Run* | *Chemical* | *Delay [min]* |
|---|---|---|
| 13 | - | - |
| 14 | Soap | 0 |
| 15 | Soap | 20 |
| 16 | Soap | 40 |
| 17 | Soap | 60 |
| 18 | Soap + $CaCl_2$ | 60 |

### *5.3.5 Effect of pH in flotation*

Four different flotation experiments were carried out controlling the pH. During pulping, a chemical dosage of 6 kg/t of commercial soap were added into the pulper. In each of the first two flotation experiments, 1520 g of entire pulp (15% consistency) were diluted in the flotation device until the flotation level was 19 litres. The pH of the second flotation experiment was adjusted to 8.5, using 0.2M NaOH to reach it. The other two flotation experiments were done with the short fibre fractionation obtained after fractionation. The pH of the second flotation experiment was set at 8.5 using 0.2M NaOH. In every flotation experiment, three different samples of accept and reject were taken and 7.2 g of $CaCl_2$ were added into the flotation device at the beginning of every flotation experiment. The variables of pH flotation experiments are presented in Table 4.

**Table 4.** Variables of pH flotation experiments.

| *Test Run* | *Flotation level (L)* | *Air level (L/min)* | *Note* |
|---|---|---|---|
| 19 | 19 | 7.4 | Entire pulp |
| 20 | 19 | 7.4 | Entire pulp – pH 8.5 |
| 21 | 19 | 7.4 | Fines fraction |
| 22 | 19 | 7.4 | Fines fraction – pH 8.5 |

### *5.3.6 Comparison of flotation chemicals*

Three different chemicals were used in six flotation experiments. Commercial soap, surfactant #1 and surfactant #2 were used in three different groups of two flotation experiments each, in which the dosage was changed. After pulping and fractionation, the short fibre fraction was used for the flotation experiments. All the chemicals were added in the flotation cell before starting flotation experiments. For the commercial soap, dosages of 6 and 9 kg/t were used (adding 7.2g of $CaCl_2$ every time soap was used). For the surfactants, dosages of 0.6 and 0.9 kg/t were added respectively. The parameters for the chemicals flotation experiments are shown in Table 5.

**Table 5.** Parameters for chemicals flotation experiments.

| ***Test run*** | ***Chemical*** | ***Dosage [kg/t]*** |
|---|---|---|
| 23 | Commercial soap | 6 |
| 24 | Commercial soap | 9 |
| 25 | Surfactant #1 | 0.6 |
| 26 | Surfactant #1 | 0.9 |
| 27 | Surfactant #2 | 0.6 |
| 28 | Surfactant #2 | 0.9 |

# 6 RESULTS

The results are presented in the same five different scenarios of flotation experiments explained in Chapter 5.3. Several pulp properties were studied while brightness and ash content were the variables studied in every flotation experiment.

## 6.1 Reference flotation

Brightness, ash content and residual ink values were obtained by optical measurement of sheets from every sample, and the results are presented versus $RR_m$ in the following figures.

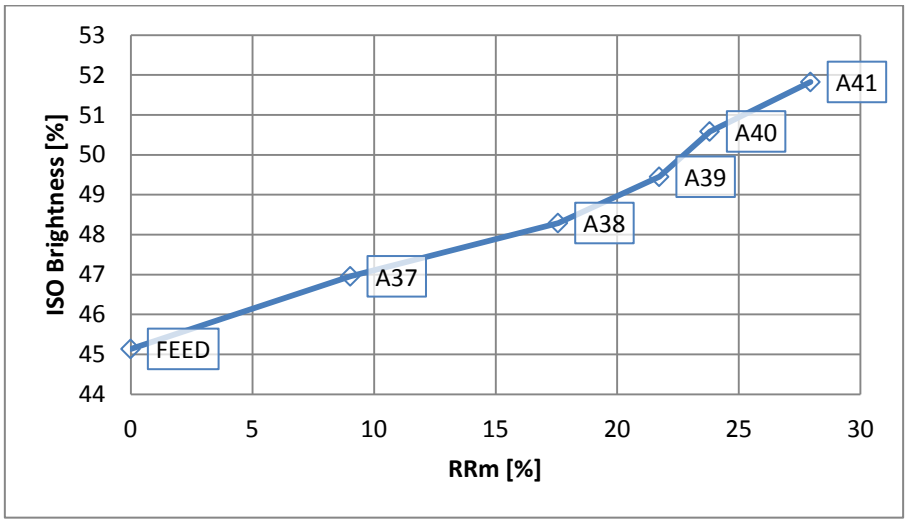



**Figure 18.** ISO brightness for entire pulp feed and accepts. A = accept.

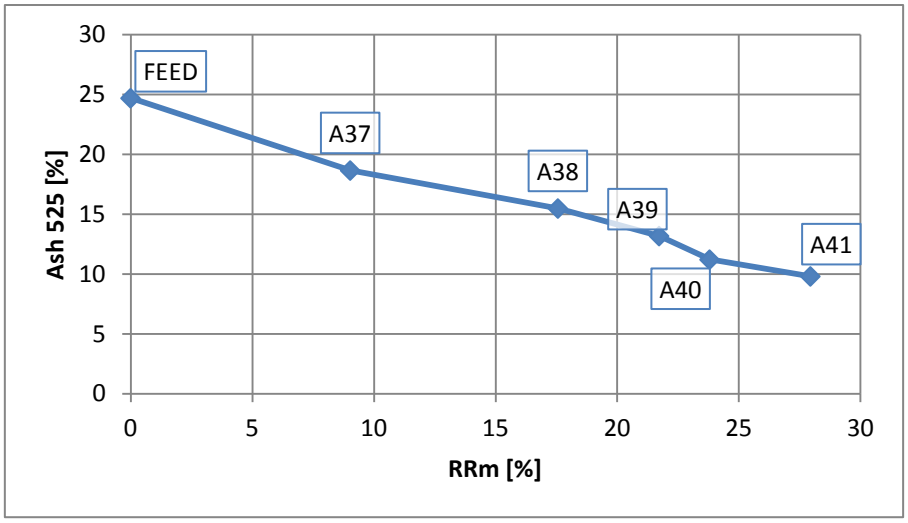



**Figure 19.** Ash content for entire pulp feed and accepts. A = accept.

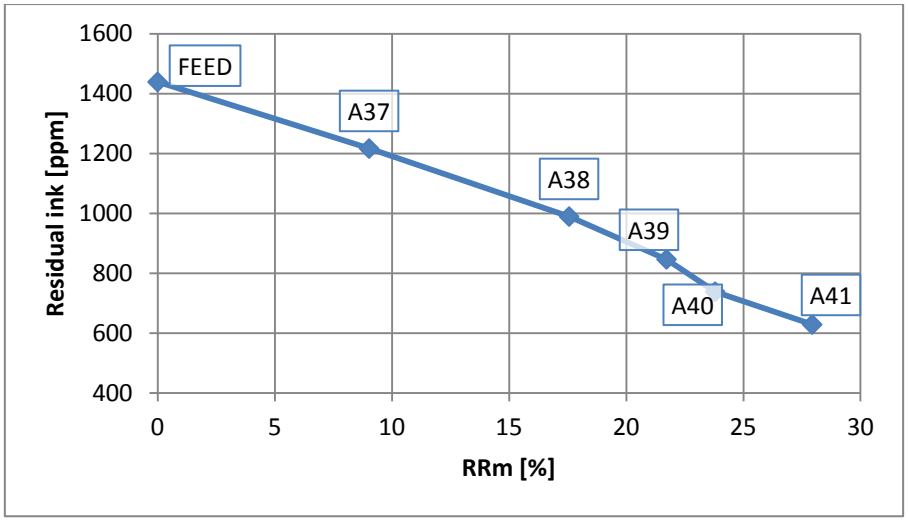



**Figure 20.** Residual ink content for entire pulp feed and accepts. A = accept.

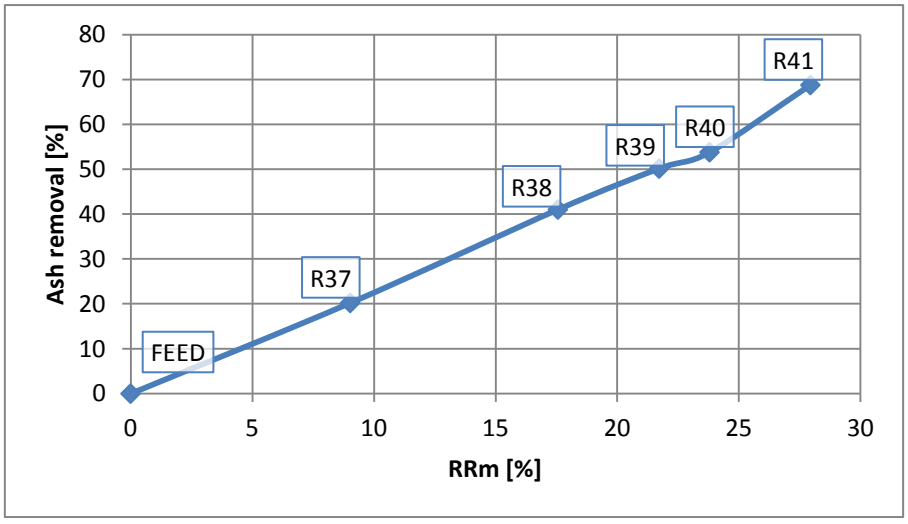



**Figure 21.** Ash removal for entire pulp feed and accepts. R = reject.

According to Figures 19 and 20, accept samples with a higher mass reject are the ones with less content in residual ink and ash. The higher the mass reject is the higher the brightness, despite losing some fibre because of higher mass reject rates.

## 6.2 Fractionation

The values from membrane pads of feed, accept (short fibre) and reject (long fibre) are presented in Table 6. After fractionation, brightness values were similar but the short fibre fraction had the highest ash content value. Feed and long fibre fraction samples had similar retention values. ISO brightness of hyperwashed pulps were 44.0% and 47.0% for feed and long fibre fraction, respectively.

**Table 6.** Brightness, ash content and retention of feed (entire pulp), accept (short fibre fraction) and reject (long fibre fraction) from fractionation.

| | ***Feed*** | ***Short fibre*** | ***Long fibre*** |
|---|---|---|---|
| ISO Brightness [%] | 44.3 | 44.0 | 42.7 |
| Ash 525 [%] | 24.0 | 32.9 | 21.9 |
| Retention [%] | 61.0 | 38.6 | 59.1 |
| HW Brightness [%] | 44.0 | NA | 47.0 |

## 6.3 Flotation level and air flow during flotation

Four groups of three flotation experiments in each were carried out in order to obtain the optimum values for flotation level and air flow during flotation. Optical properties were measured from pads of every flotation feed, accept and reject sample. Brightness and ash content values are presented in Figures 22 and 23.

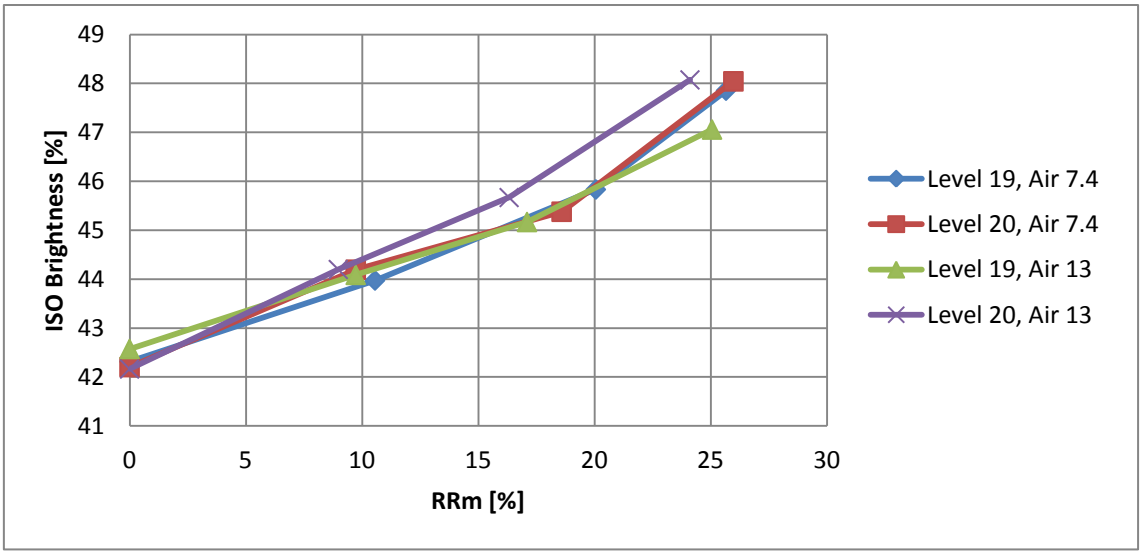



**Figure 22.** Brightness values for flotation level and air flow flotation experiments.

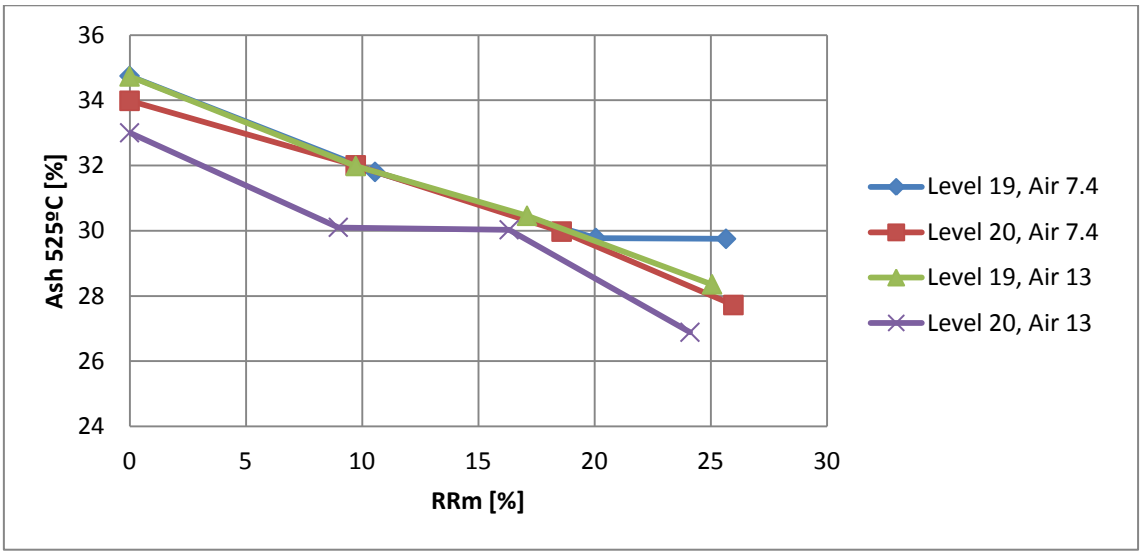



**Figure 23.** Ash content values for flotation level and air flow flotation experiments.

According to Figure 22, only 1.0% unit difference in ISO brightness was achieved with the studied range of air flow and flotation level. In Figure 23, there is a small difference in ash content between the experiments.

## 6.4 Conditioning prior to flotation

Three different amounts of reject sample were taken in every flotation experiment in order to observe the effect of chemical delay on brightness and ash content in feed and accept samples. In Figures 24 and 25 these variables are represented vs. $RR_m$.

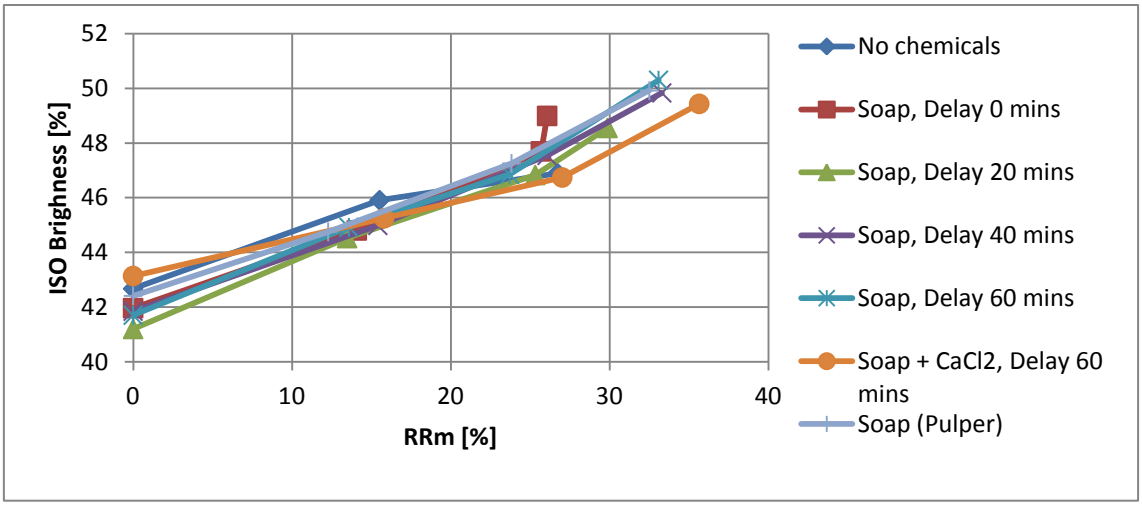



**Figure 24.** Brightness values for delay chemical flotation experiments.

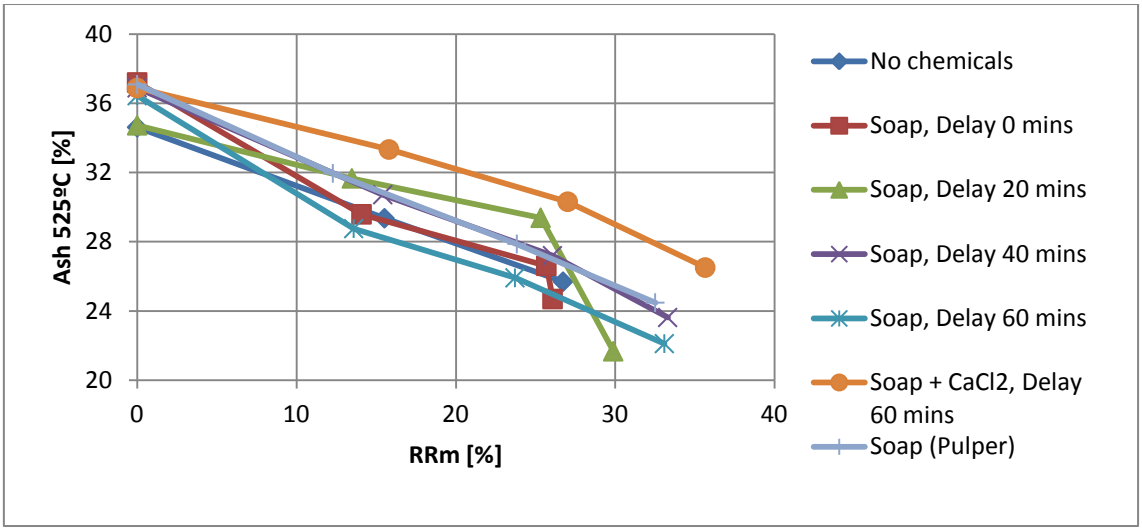



**Figure 25.** Ash content for delay chemical flotation experiments.

Highest values of brightness are obtained by the samples with higher chemical delay but also with the higher reject mass rate which leads to a loss of fibre on the yield. In Figure 25, lowest ash content was achieved by test 15 (Soap, Delay 20 mins), with lower fibre loss on the yield compared to samples with higher delay. Values from test 21, in which soap was added in the pulper and there was no delay, were added to these figures to compare results.

Based on the amount of reject obtained in every flotation experiment and further calculations, ash and fines removal values of all delay chemical flotation experiments were calculated. Ash removal and fines removal are shown in Figures 26 and 27.

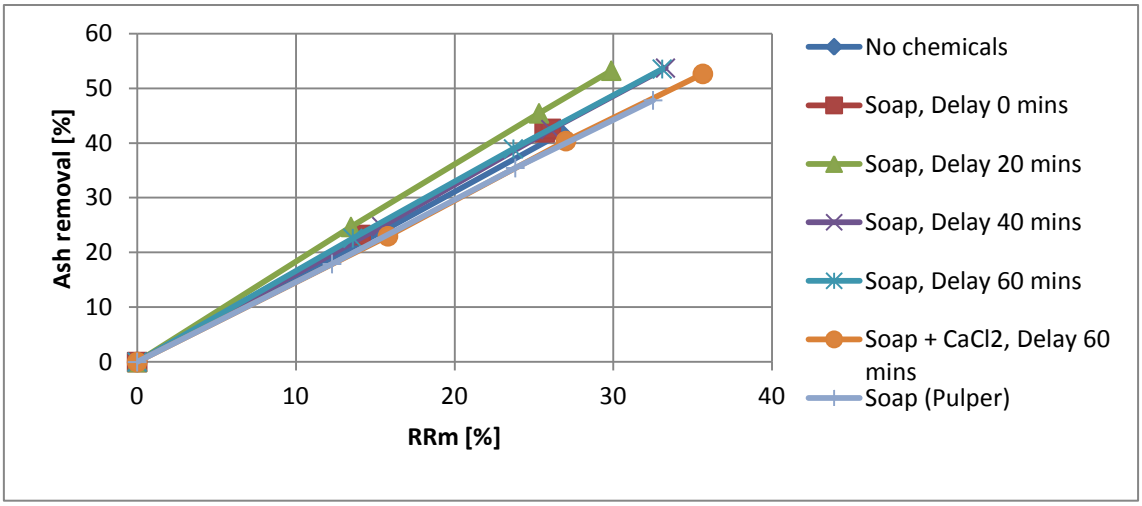



**Figure 26.** Ash removal for delay chemical flotation experiments.

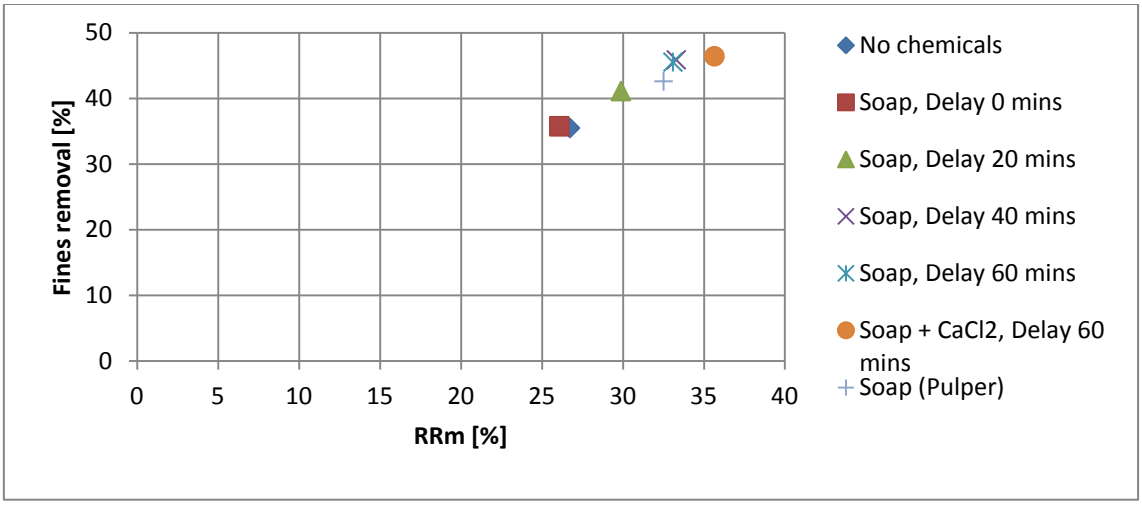



**Figure 27.** Fines removal for delay chemical flotation experiments.

In Figure 26, a similar value of ash removal was obtained by the last four flotation experiments, with differences in mass reject rate. In Figure 27, flotation experiments with higher chemical delay were the ones with higher yield loss of fines.

## 6.5 Effect of pH in flotation

*Comparison between entire pulp pH flotation and reference flotation experiments*

Brightness, ash content, residual ink and ash removal values were obtained by optical measurement from sheets of every flotation experiment (feed and accepts samples), and represented in Figures 28, 29, 30 and 31.

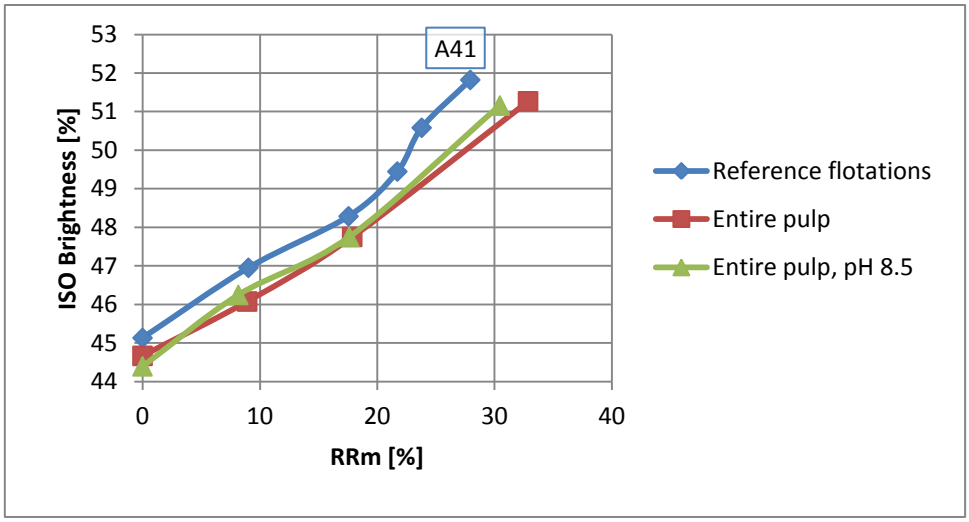



**Figure 28.** Brightness values for entire pulp pH flotation experiments. A = accept.

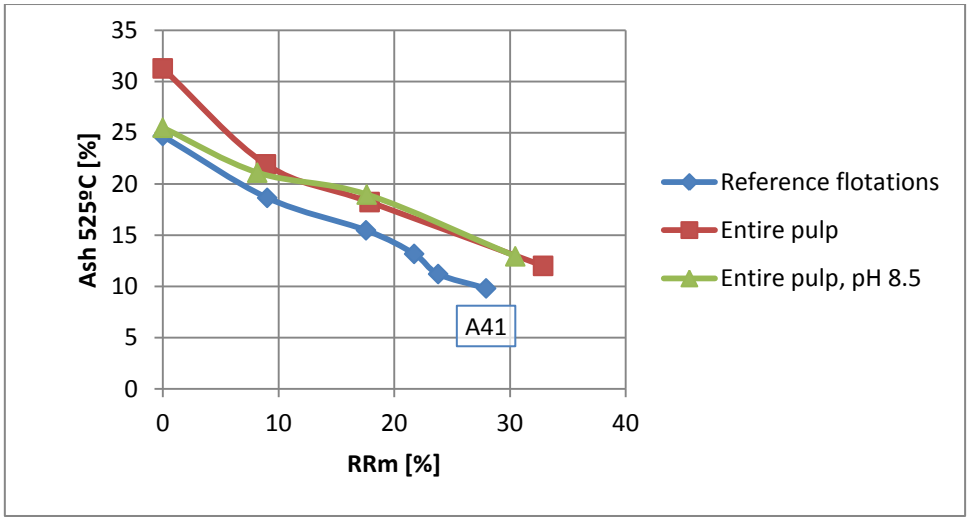



**Figure 29.** Ash content for entire pulp pH flotation experiments. A = accept.

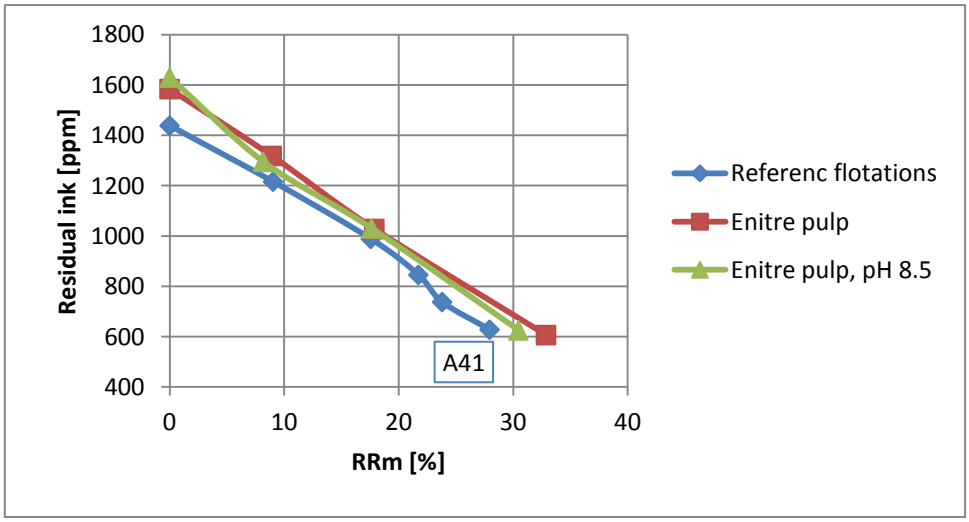



**Figure 30.** Residual ink values of entire pulp pH flotation experiments. A = accept.

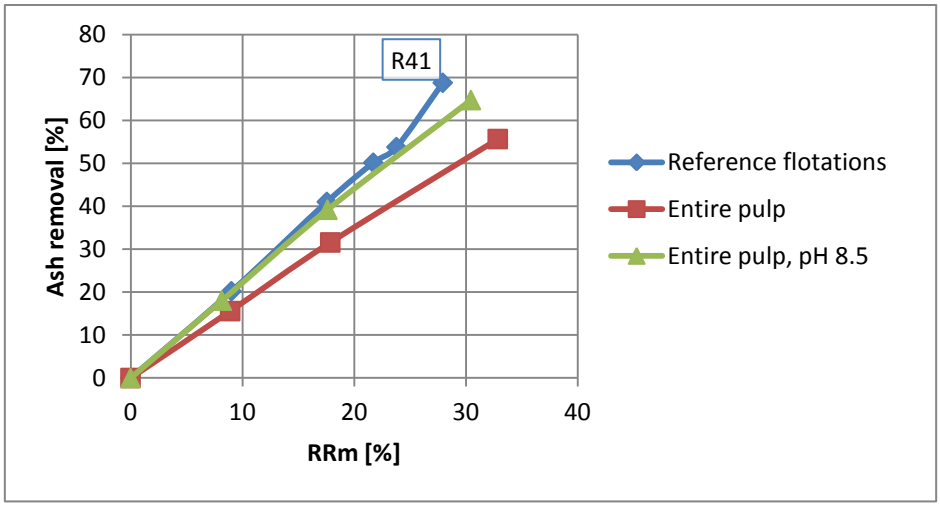



**Figure 31.** Ash removal for entire pulp pH flotation experiments. R = reject.

In Figures 28, 29, 30 and 31 there was a small difference between the pH controlled sample and the non-controlled one. Final values for brightness, ash content and residual ink content were similar but flotation test 20 (Entire pulp, pH 8.5) had a lower mass reject rate. Comparing pH flotation experiments with reference flotations, higher brightness, lower ash content and similar residual ink values were obtained by the reference flotation experiments but with lower mass reject rate.

*Fines fraction flotation experiments*

Two fines fraction flotation experiments were carried out, one of them adjusting the pH of the flotation feed sample to 8.5. Brightness and ash content values were obtained by optical measurement from pads of every flotation experiment (feed and accepts samples), and were represented versus $RR_m$ in Figures 32 and 33.

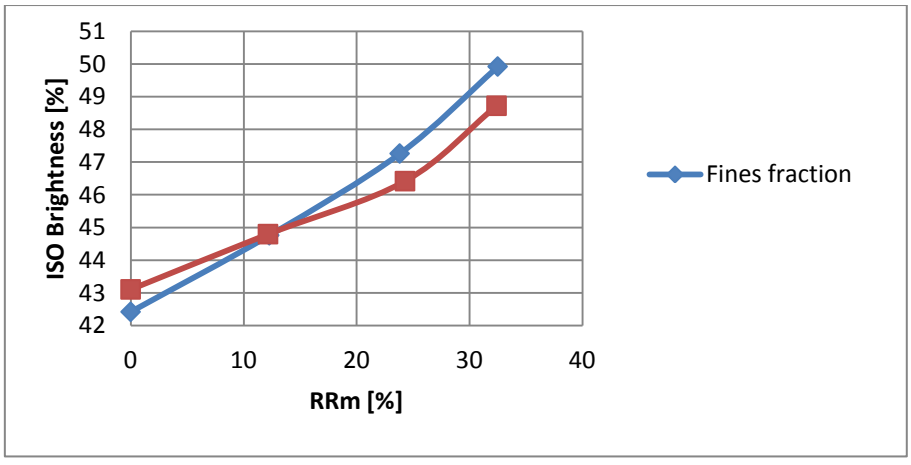



**Figure 32.** Brightness values for fines fraction pH flotation experiments.

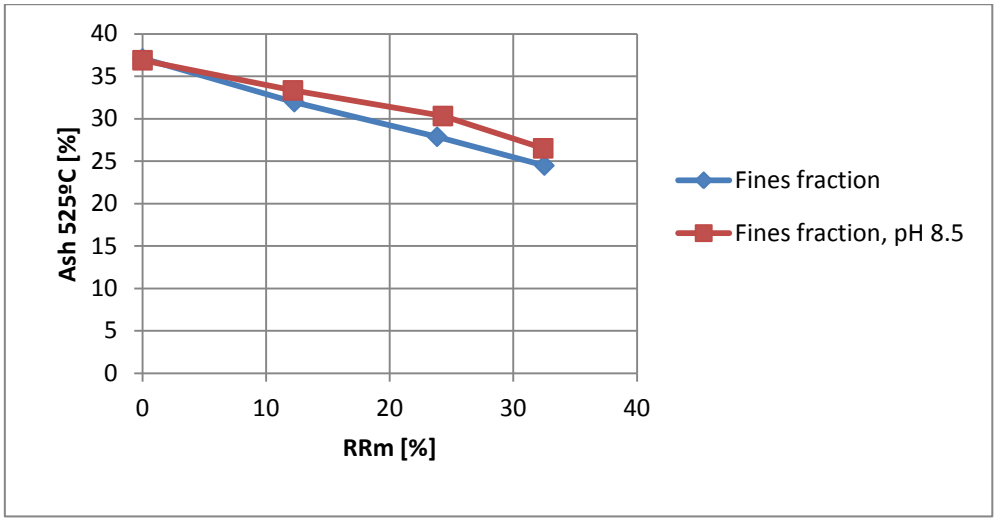



**Figure 33.** Ash content for fines fraction pH flotation experiments.

In these flotation experiments both had the same mass reject rate, around 32%, as shown in Figures 30 and 31. The biggest difference is that non pH controlled flotation test ISO brightness was 1% unit higher than the pH controlled one.

Based on the amount of reject obtained in every flotation experiment and further calculations, ash and fines removal values of the entire pulp pH flotation experiments were calculated. Ash removal and fines removal are shown in Figures 34 and 35.

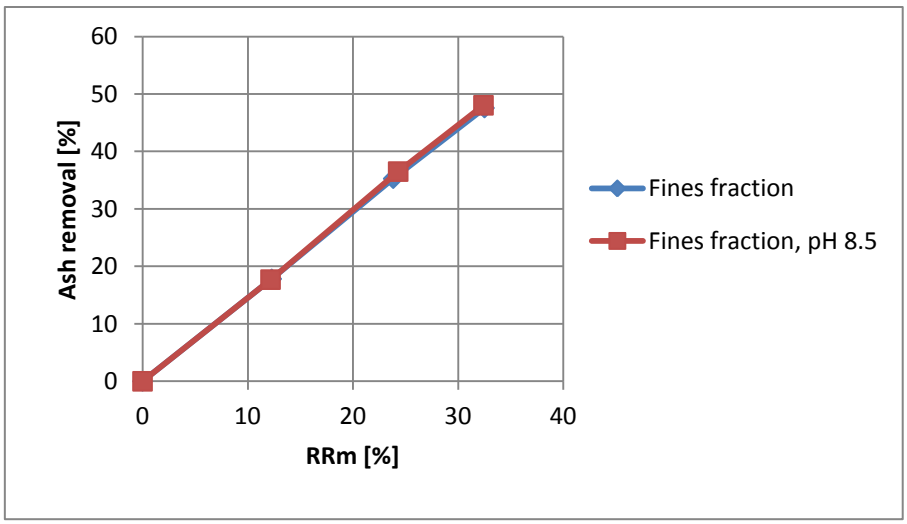



**Figure 34.** Ash removal for fines fraction pH flotation experiments.

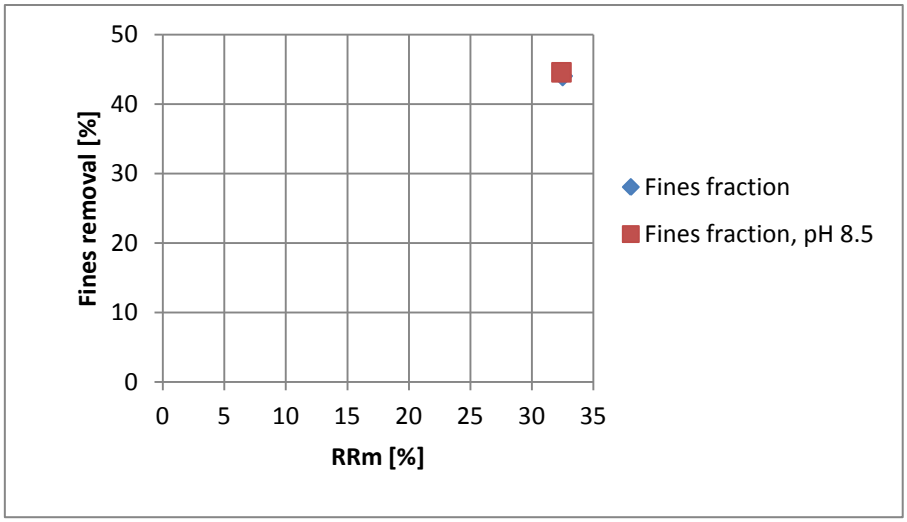



**Figure 35.** Fines removal for fines fraction pH flotation experiments.

In Figure 34 ash removal is the same for both flotation experiments. In Figure 35 there was no difference between the fines fraction pH flotation tests. Adjusting the pH to 8.5 had no effect on fines removal and mass reject rate values.

## 6.6 Comparison of flotation chemicals

The effect of all chemicals (soap, surfactant #1 and surfactant #2) during flotation was studied together to compare results from the three chemicals used. Brightness and ash content values were obtained by optical measurement from pads of every flotation experiment (feed and accepts samples). These values are shown in Figures 36 and 37.

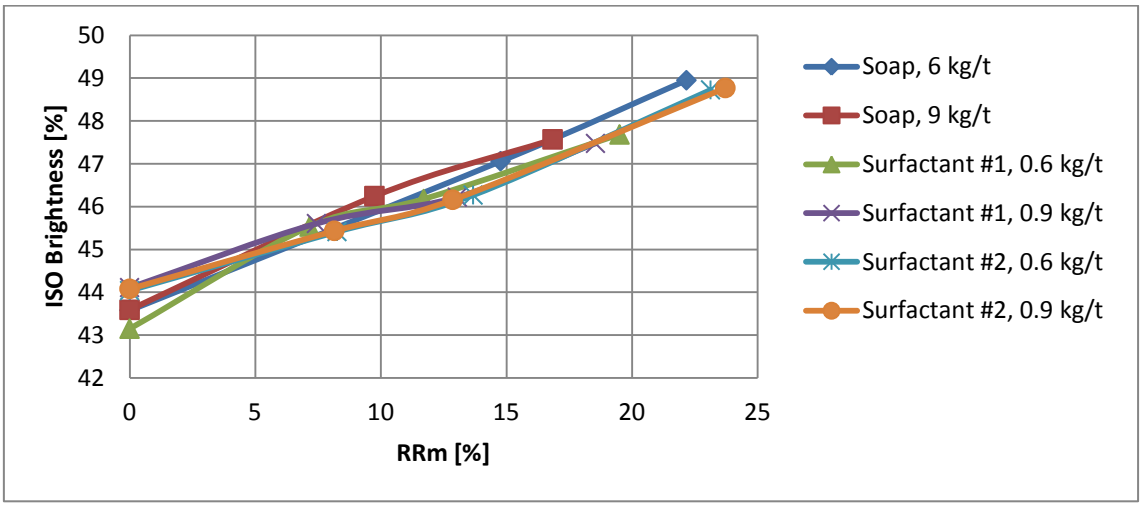



**Figure 36.** Brightness values for chemicals flotation experiments.

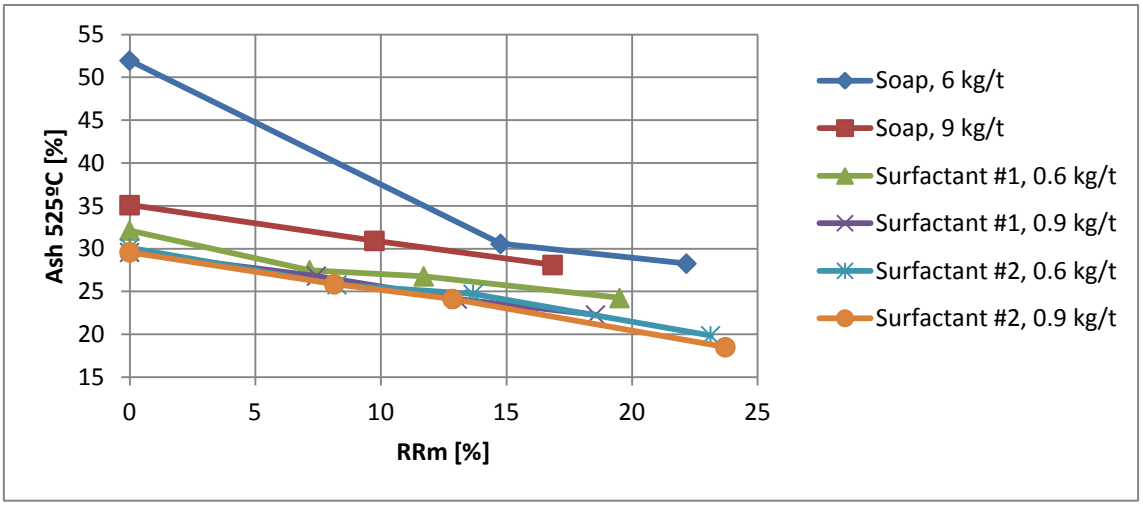



**Figure 37.** Ash content values for chemicals flotation experiments.

In Figure 36 higher values of brightness and mass reject rate were obtained by flotation test 23 (6 kg/t of commercial soap) and by surfactant #2 flotation tests. In Figure 37 lower values of ash content were obtained by surfactant #2 flotation tests. The first ash content value of Soap, 6 kg/t is wrong in all probability, because the raw material cannot change that much between experiments.

Based on the amount of reject obtained in every flotation experiment and further calculations, ash and fines removal values of the different chemicals flotation experiments were also calculated. Ash removal and fines removal values are represented versus mass reject rate in Figures 38 and 39.

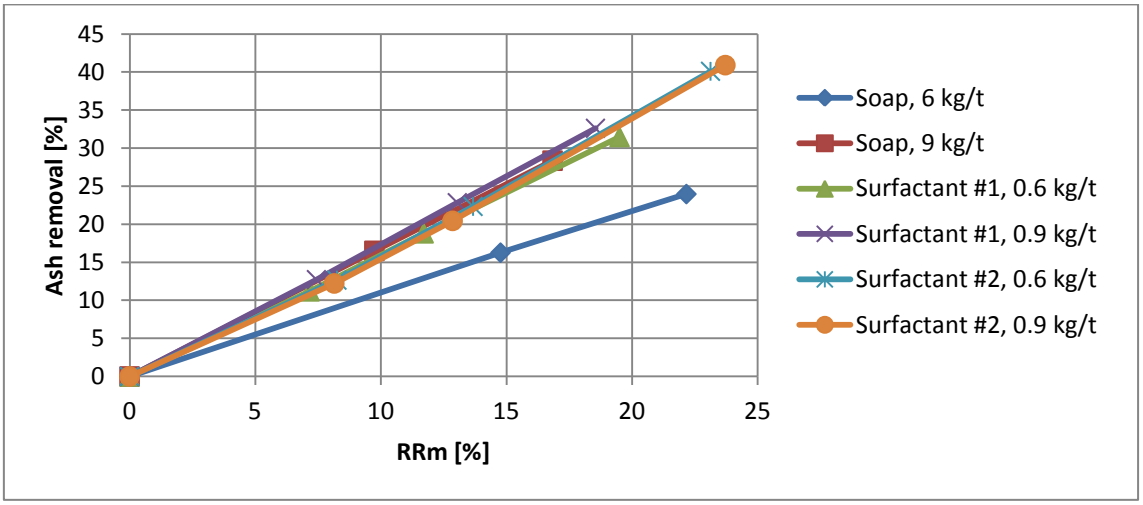



**Figure 38.** Ash removal values for chemicals flotation experiments.

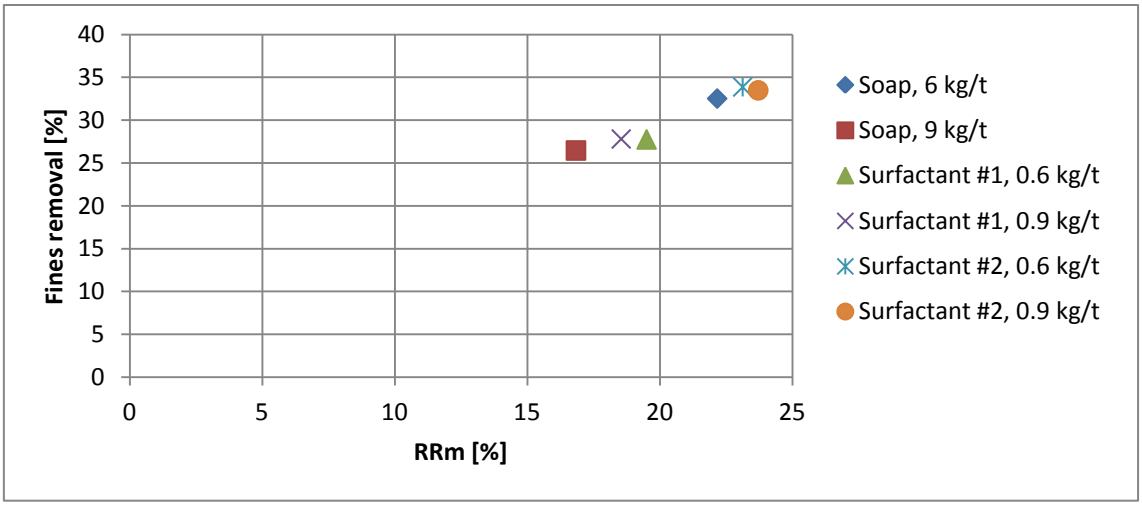



**Figure 39.** Fines removal values for chemicals flotation experiments.

In Figure 38 higher values of ash removal were obtained by surfactant #2 flotation experiments. For fines removal tests 23, 27 and 28 had similar values as shown in Figure 39.

# 7 DISCUSSION

## 7.1 Reference flotation

Before starting the discussion part, it should be reminded that the raw material used in these flotation experiments was quite old. The storage period of the recovered paper can influence their deinkability (poor ink detachment, high speck content, high ink fragmentation and poor ink removal). Because of the use of old newspapers and old magazines, brightness values of all the samples were lower than experiments done with fresh raw material. According to Vahlroos (2008), ISO brightness values around 60% can be reached with fresh raw material, instead of values around 50% that were obtained with old newspapers and magazines.

Since flotation removes inks and fillers, the brightness gain induced by the removal of the inks might be overshadowed by the removal of fillers if the brightness of the fillers is higher than the brightness fibres, such as with ONP/OMG mixtures. During entire pulp flotation experiments, brightness values increased while the more amount of reject were removed from the sample. According to Figure 18, the increase in ISO brightness between feed sample and the final accept was 6.7% unit. Ash content and residual ink values decreased while the amount of reject mass increased in every flotation test. Ash removal values obtained in reference flotation were quite high (around 70%) compared to ash removal shown in Körkkö (2008).

## 7.2 Fractionation

During fractionation, the feed sample was separated into two fractions, short and long fibre fraction. According to Table 6, brightness values were similar for feed and fines fraction, but long fibre fraction had a little lower value. However, brightness of hyperwashed long fibre fraction was the highest which suggest that there was a lot of free ink in the sample. Short fibre fraction had the highest ash content value after fractionation. Ash content in the long fibre fraction was higher compared to ash content values obtained by Mäkinen (2010), but also ash content of the feed sample was higher.

Comparing ISO brightness values from entire pulp flotation experiments and fractionation samples, a higher increase was observed through flotation. A difference of 7.1% units was shown between accept 41 and short fibre fraction sample according to Table 6. Thanks to flotation, samples lose a higher amount of ash and ink particles, increasing the final brightness values. Fractionation was not perfect but enough amount of fines fraction was achieved for the flotation experiments.

## 7.3 Flotation level and air flow during flotation

Flotation cell level and air flow did not have so much effect, although the increase in flotation cell level and air flow might increase the speed of flotation. All the ISO brightness values are in a margin of 1% unit according to Figure 22. A maximum value of 48% ISO brightness was obtained by flotation without chemicals, lower than the ISO brightness value of 52% obtained by the reference flotation experiments. Flotation level of 19 litres and air flow of 7.4 litres per minute were set for the rest of flotation experiments.

## 7.4 Conditioning prior to flotation

The delay of chemical during flotation affected the mass reject rate the most. The higher the delay was the higher amount of fibre, fillers and ash were lost with the reject sample. Compared with entire pulp reference flotation experiments, ISO brightness values were slightly lower (2% units less). The use of chemicals improved the brightness values: delay chemical flotation results were better than results of flotation level and air flow flotation experiments. So longer delay increases brightness, but also $RR_m$ which lowers the yield. According to Figure 24, higher values of brightness were obtained by tests 16 and 17 (Soap, Delay 40 mins and 60 mins), with values similar to the ones from entire pulp flotation (around 50 %).

Adding $CaCl_2$ at the beginning of the delay time increased the mass reject rate by making fillers, ash and fines more hydrophobic as shown in Figures 24, 25, 26 and 27. Comparing these values with the ones obtained by test 21, in which the soap dosage was added into the pulper, there was no effect on where the chemical was added. Adding $CaCl_2$ at the beginning of the delay had a negative effect on flotation, because the yield loss was higher.

## 7.5 Effect of pH in flotation

*Entire pulp flotation experiments*

According to Figure 28, ISO brightness values around 51% were obtained by pH flotation experiments, fairly similar values compared to reference flotations (only 1% unit difference). Adding chemicals in the pulper made ink particles more floatable. Residual ink values had a drastic decrease from 1630 to 625 ppm in the pH controlled flotation experiment, according to Figure 30.

*Fines fraction flotation experiments*

Adjust of the pH to 8.5 did not have so much effect in fines fraction flotation. Ash content, ash removal and fines removal of both experiments had very similar values as shown in Figures 33, 34 and 35. According to Figure 32 the highest value of brightness is obtained by flotation test 21, 1% unit higher than the pH controlled test 22. When adding chemicals to pulping, a better separation between ink and fibres was achieved: an increase of 2% units in ISO brightness can be seen compared to flotation level and air flow flotation experiments carried out without chemicals.

Surprisingly, adjusting the pH to 8.5 did not have an effect in the properties measured. There is a widespread belief that the higher the pH is, the better results are achieved. According to INGEDE method 11, at the end of pulping pH value has to be 9.5 and adjust using sodium hydroxide has to be done if the pH after pulping is too low. However, this flotation experiments can be carried out in neutral pH conditions.

## 7.6 Comparison of flotation chemicals

Commercial soaps of fatty acids are used as flotation collectors for ink removal in ONP/OMG blend for newsprint production (Dorris *et al.* 2011). The highest value of brightness was obtained with lower soap dosage (6 kg/t). Lower dosage of commercial soap led to a better agglomeration of ink and soap and made the ink-soap aggregates more hydrophobic.

There was not a big difference between flotation experiments using either surfactant #1 or surfactant #2. The main advantage of using non-ionic surfactants in ONP/OMG blends is that small quantities are needed, reducing the chemical dosage (Dorris *et al.* 2011). The speed of flotation was higher when surfactants were used because the froth was quickly formed and removed from the top of the sample.

Comparing all the chemicals flotation experiments, highest brightness value was obtained by the lower soap dosage (6 kg/t) flotation test according to Figure 36 (same brightness with lower $RR_m$). There were no big differences between higher or lower dosage of surfactants, so the best dosage was 0.6 kg/t which lead to cost savings. According to Dorris (2011), if too much surfactant is added, the adhesion of ink particle to air bubble will be too weak to permit flotation. It was supposed that the surfactants would have worked better than soap during flotation, however soap worked very well. For that reason lower soap dosage is recommended because higher value of ISO brightness was obtained compared to surfactants flotation tests.

After comparing all the different fines fraction flotation experiments carried out, the use of lower soap dosage (6 kg/t) in the flotation cell is recommended to improve pulp properties. Comparing this flotation with flotation level and air flow flotation (Figure 22) and with fines fraction pH flotation (Figure 32), a ISO brightness value of 48.9% is obtained with the lowest mass reject rate of all, losing less amount of fibres and fillers with the reject, and with no need of adjusting the pH.

# 8 CONCLUSIONS AND RECOMMENDATIONS

The use of old raw materials worsens the deinkability of the pulp and therefore pulp properties such as brightness values are lower than values obtained using fresh raw material. Through entire pulp flotation, however, the brightness value increased moderately while the more amount of reject was removed from the sample. Thus, flotation experiments worked very well despite of the old raw material.

Fractionation performed here was found to be adequate for these experiments. Brightness value of long fibre fraction was a little low because of the presence of free ink in the sample. However, short fibre fraction had the highest content of ash after fractionation, which indicates that the pulp was fractionated.

Fines fraction flotation results suggest that changing flotation cell level and air flow do not have so much effect on pulp properties after flotation. On the other hand, increasing the delay of chemicals prior to flotation has an effect on fines fraction properties. Longer delay increases brightness but also mass reject rate which lowers the yield.

The use of chemicals to adjust pH prior to flotation do not have an effect in pulp properties, so apparently this flotation experiments can be carried out in neutral pH conditions.

Based on these results, the use of a soap dosage of 6 kg/t in the flotation cell is recommended. Lower dosage of soap leads to a better agglomeration of ink and soap and made the ink-soap aggregates more hydrophobic. Highest value of brightness is reached with lower mass reject rate, compared to other flotation collectors such as surfactants.

# 9 SUMMARY

The target of this work was to obtain more information about the less studied fines fraction flotation and to optimize its variables. The work was divided in five different flotation scenarios in which a mixture of recovered paper was used as feed. The feeding ratio was 50% ONP and 50% OMG (OMG consisted in half SC and half LWC).

## 9.1 Reference flotation

Five entire pulp flotation experiments were carried out in the flotation device. The consistency of the feed was 1.2%, after diluting the pulp slurry from pulping with warm tap water. The flotation level and the air flow were set to 19 litres and 7.4 litres per minute, respectively. Residual ink and ash content decreased through flotation while ISO brightness increased from 45.1% to 51.8%.

## 9.2 Fractionation

Fractionation was performed with pressure screening in order to obtain 35% of long fibres and 65% of short fibres. Because high accept consistency was wanted due to the short fibre flotation right after fractionation, the feed consistency of the fractionation was around 0.8%. Three samples from a fractionation were taken: feed, short fibre fraction and long fibre fraction. Through fractionation, high free ink content was achieved in the long fibre fraction and high ash content in the short fibre fraction.

## 9.3 Flotation level and air flow during flotation

Flotation level and air flow values were changed in different fines fraction flotation experiments to obtain the optimum values. These changes did not have so much effect on pulp properties and therefore flotation level and air flow were set to 19 litres and 7.4 litres per minute for the rest of flotation experiments. Results showed that ISO brightness of the fines fraction increased to values around 48% through flotation.

## 9.4 Conditioning prior to flotation

Soap was added in this fines fraction flotation experiments to determine the effect of different delay time of chemicals in the pulp properties. The use of chemicals improved the brightness values: delay chemical flotation results were better than results of flotation level and air flow flotation experiments. Longer delay increases brightness, but also $RR_m$ which lowers the yield.

## 9.5 Effect of pH in flotation

pH of different flotation experiments were adjust to 8.5 to compare the difference between the controlled and the non controlled pH test. Controlling the pH was not an important factor for pulp properties. The results suggest that these flotation experiments can be carried out in neutral pH conditions.

## 9.6 Comparison of flotation chemicals

Three different chemicals were used during fines fraction flotation experiments in order to look for the best one and optimise its dosage based on pulp properties. Results suggest that lower soap dosage (6 kg/t) improve pulp properties, because highest ISO brightness value (48.9%) was obtained with the lowest mass reject rate of all, losing less amount of fibres and fillers with the reject, and with no need of adjusting the pH.

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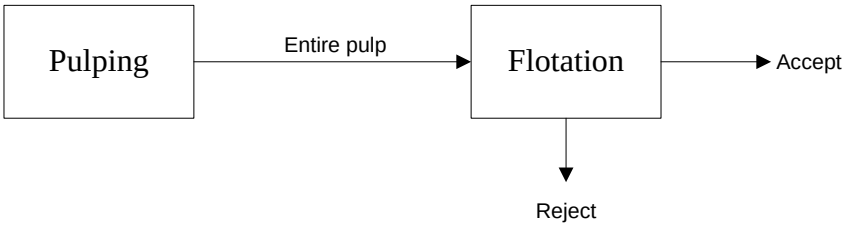
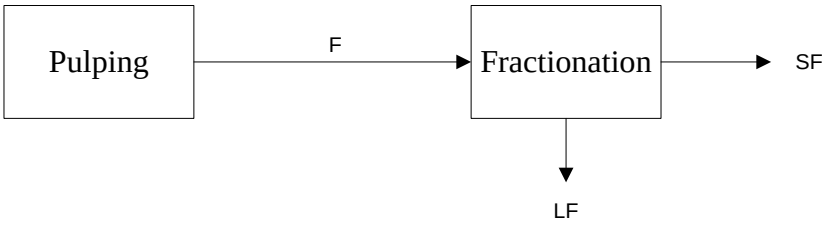
Block diagram of the flotation experiments APPENDIX 1 (1/3)

**Reference flotation**

- Flotation tests 37-41

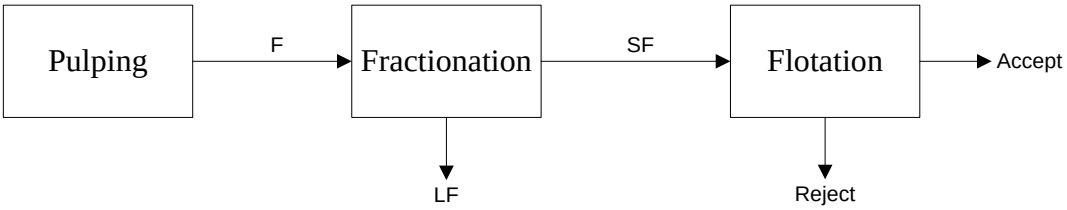



- Fractionation samples




**Flotation level and air flow during flotation**

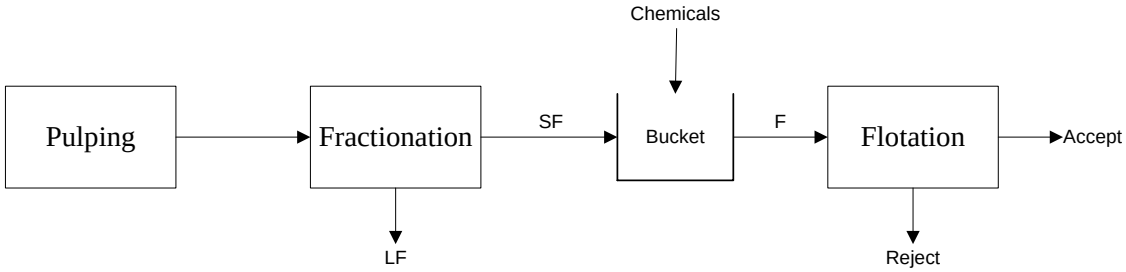
- Flotation tests 1-12



Block diagram of the flotation experiments APPENDIX 1 (2/3)

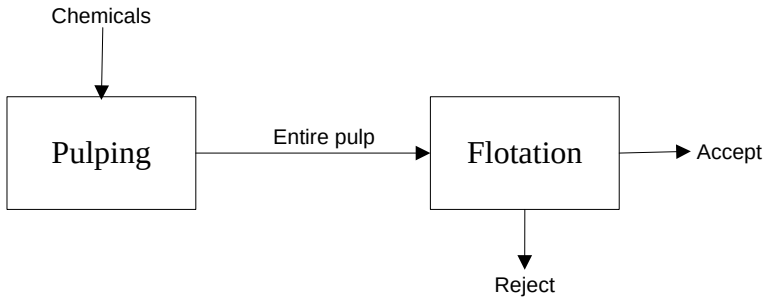
**Delay chemical flotation**

- Flotation tests 13-18

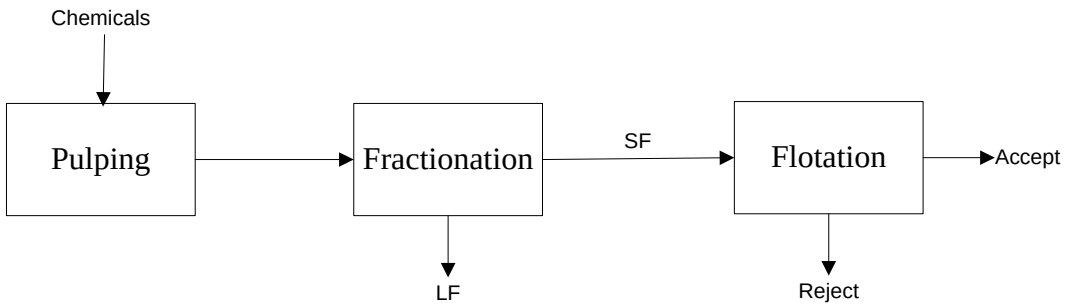



**pH flotation**

- Flotation tests 19-20




- Flotation tests 21-22



Block diagram of the flotation experiments APPENDIX 1 (3/3)

**Chemicals flotation**

- Flotation tests 23-28