Mikko Karjalainen

STUDIES ON WHEAT STRAW PULP FRACTIONATION

FRACTIONATION TENDENCY OF CELLS IN PRESSURE SCREENING, HYDROCYCLONE FRACTIONATION AND FLOTATION
STUDIES ON WHEAT STRAW PULP FRACTIONATION
Fractionation tendency of cells in pressure screening, hydrocyclone fractionation and flotation

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Abstract

Plant fibres are an important part of modern daily life. The most obvious consumer products manufactured from them are paper, cardboard and the fibreboards used in the construction and furniture industries. Plants producing a woody stem are the most widely used raw materials for these fibre products but fibres originating from other plants, i.e. non-wood fibres, are used too. One of the most promising potential non-wood fibre resource categories is that of grasses, of which the cereals are the most important plants grown worldwide. A huge amount of straw is generated as an agricultural by-product annually, but the amount used as an industrial raw material is low because it contains components that are detrimental either to processability or to product quality.

The purpose of pulp fractionation is to divide pulp into fractions with distinct properties. Industrially feasible fractionation methods are pressure screening, hydrocyclone fractionation and flotation. In pressure screening, separation is based on a mechanical barrier and particles are fractionated according to their dimensions, while a hydrocyclone fractionates particles according to their density and specific surface area and flotation fractionates particles according to their surface chemistry. These methods are traditionally used for removing impurities from pulp but numerous reports on pulp fractionation can also be found. Previous fractionation experiments were performed using wood-based pulps, whereas no previous studies are available concerning the fractionation of pulps manufactured using grasses.

The aim of the present work was to determine whether it is possible to fractionate wheat straw pulp by methods that are feasible on an industrial scale. The experimental part was concerned with wheat straw pulp fractionation by pressure screening, hydrocyclone fractionation and flotation.

The results show that all these fractionation methods were able to divide the wheat straw pulp into fractions with different cell properties and cell types, e.g. distinct cell lengths, cell wall thicknesses or surface chemistries. Likewise, fractionation can be used to remove detrimental components or to optimize pulp properties according to their end use or to optimize pulp processing sequences. Due to the uniform structure of grasses, it is likely that the results can be generalized to other grasses than that employed here.

Keywords: automatic optical fibre analysis, flotation, fractionation, hydrocyclone, non-wood, pressure screening, silicates, Wheat straw
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Tiivistelmä


Tämän tutkimuksen tarkoituksena oli selvittää vehnämassan sisältämien solujen fraktiointia teollisuuden käyttöön soveltuvilla menetelmillä. Työn kokeellisessa osassa fraktioinnin vehnäsellullakin painelajittimella, hydrosyklonilla ja flotaatiolla.


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Oulu, October 2015

Mikko Karjalainen
List of original papers

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


The present author was principal author of all the papers. He was responsible for the experimental design, for performing the experiments and for writing the papers. The additional authors participated in the designing of the experiments and writing of the papers by making valuable comments.

Other related publications by the author:

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1 Background and outline of the thesis

Wood is a versatile renewable raw material used in the production of pulp and paper, building materials, furniture and energy, for example. Future scenarios do not show any reduction in the demand for wood, on account of the increasing population and rising standard of living. Much of the research aimed at reducing carbon dioxide emissions is concentrated on studying the possibilities for using wood to replace crude oil in the production of energy and various goods. The demand for wood is not distributed evenly around the world, however, any more than are the forests, and in the worst case the demand for wood may exceed the annual production in the forests and the use of wood may have undesirable consequences. A large number of tree species are threatened because of the great demand for wood, the high incidence of illegal logging and bad forestry management. In addition, trees are an important part of general biodiversity and the clear-felling of forests may damage local ecosystems. This means that alternative raw materials are needed for industrial use.

Huge amounts of cereals are produced worldwide annually. When these crops are harvested, the grain is collected and used in food production but the remaining part of the plant, the straw, is usually left in the field and burned where this is allowed. This non-food part of the plant is a renewable material, and large amounts of straw are available annually. Unfortunately, some features in this lignocellulosic biomass are detrimental as far as straw processability and the properties of possible end products, and thus non-wood plants make up a very small proportion of all industrial raw materials.

This work was focused on finding a solution to some of these problems and on optimization of the use of non-wood plants, especially grass plants, as industrial raw materials. Background information giving an insight into the industrially available fibre crops, especially wheat is presented first in chapter 2. Wheat straw was chosen as a raw material due to its general availability worldwide, and this is consequently reviewed more detailed. The fractionation methods available on an industrial scale are presented in section 2.4, and chapter 3 summarizes the research problem to be investigated and presents the aims of this thesis. The experimental part of the work is detailed in chapters 4 and 5, and the applicability of the results is discussed in chapter 6. Finally, chapter 7 presents conclusions based on the outcomes of the research. The original peer-reviewed papers published as a result of this work are included at the end of this thesis.
2  State of the art in wheat straw pulp fractionation

The literature survey in this chapter will be concerned with the structure of grasses and the fractionation of pulps. The aim is to present the background to the problems examined in this thesis.

2.1  Introduction – plant fibres in industrial use

Industrial fibres of plant origin can be divided into those originating from wood and other fibres, which may be called non-wood fibres. Wood as a botanical term refers to the xylem produced as secondary growth by a plant, and it is from this that wood-based fibres originate. The term ‘non-wood’ is used when fibrous material originates from some other part of a plant, so that non-wood fibres can be obtained from annual plants in which no secondary growth takes place and also from plants that produce a wooden stem. Non-wood fibres can be divided into four groups according to their origin: leaf fibres, bast fibres, fruit fibres and grass fibres (Ilvessalo-Pfäffli 1995). Leaf, bast and fruit fibres are typically used in the textile industry and in the making of ropes, but some of them are also used in the production of paper when certain particular properties are required.

Leaf fibres are cell bundles obtained from the vascular bundles of some monocotyledons growing very long leaves. The most important species used in their production include abaca and sisal. Leaf fibres are also called hard fibres on account of their high lignin content (David & Pailthorpe 1999, Ilvessalo-Pfäffli 1995, McKenna et al. 2004).

Bast fibres are fibre bundles derived from the phloem of perennial plants such as flax, hemp, sunn, kenaf, jute, ramie, paper mulberry, mitsumata and gambi. These are dicotyledons grown as shrubs and trees. Bast fibres are typically called soft fibres due to their low lignin content (David & Pailthorpe 1999, Ilvessalo-Pfäffli 1995, McKenna et al. 2004).

Fruit fibres grow in or around the seed of a plant. These can be divided into three groups: seed fibres (cotton), pod fibres (kapok) and husk fibres (coir) (David & Pailthorpe 1999, Ilvessalo-Pfäffli 1995, McKenna et al. 2004).

Grass fibres include agricultural residues such as the straw from cereals, rice straw, corn stalks and sugar cane (bagasse), and also naturally growing plants such as reeds, bamboo, sabai, albardine, esparto, papyrus and palms (Ilvessalo-Pfäffli 1995).
Grasses grow all over the world. Over 2300 million tons of grain from cereals is produced worldwide annually for food and cattle fodder (Anon. 2013a), and this generates large amounts of dry straw as a by-product, only a minor proportion of which is utilized. The utilization rate is especially low in the industrialized countries. It is not easy to estimate the total amount of straw which is available for use as an industrial raw material, however, partly because straw has an important function in the improving of soil quality and the amount of residual straw that can be collected varies from 0 to 50% depending on the climate, soil-specific characteristics, the previous use made of the soil and the tillage methods employed (Blanco-Canqui 2010, Hettenhaus 2006, Lal 2005, Lal 2009). The figures of $1120 \times 10^6$ tons and $1550 \times 10^6$ tons published by Lal (2005) and Kim & Dale (2004), respectively, have been widely cited in recent years as estimates of amount of straw collectable globally, both being calculated on the basis of a 40% removal rate. Even if the pulp yield were to be low (say 40%, when the amount of produced wheat straw pulp would be approximately $500 \times 10^6$ tons), that amount could have replaced a total of, which was approximately $174 \times 10^6$ tons of virgin wood pulp in 2011 (Anon. 2013b), for example.

The advantage of agricultural residues is that they can be harvested and collected with same effort as required for collecting the grain and no additional harvesting is needed, while other grasses can be grown on poor quality land that is unsuitable for cereal production (Blanco-Canqui 2010, Lal 2009, Paavilainen & Torgilsson 1994). The annual production of grasses may indeed be much higher than that of wood, which makes them a very attractive alternative. Eucalyptus grown in the tropics can produce 15 tons of dry matter per hectare in a year and typical annual growth rates for Scandinavian hardwood and softwood are 3.4 and 1.5 tons per hectare, respectively (Paavilainen & Torgilsson 1994), whereas much higher productivity can be achieved using grasses. Aleman grass (Echinochloa polystachya), for instance, is among the fastest-growing plants and it can produce 100 tons of dry matter per hectare per year (Piedade et al. 1991), while annual figures of 30 tons of dry matter per hectare have been achieved in Europe with giant mischantus (Miscanthus x giganteus) (Lewandowski et al. 2000) and more than ten tons with reed canary grass (Phalaris arundinacea) and hemp (Cannabis sativa) grown in Scandinavia (Paavilainen & Torgilsson 1994, Pahkala et al. 2008, Saijonkari-Pahkala 2001).

Due to their wide availability and high productivity, grasses are a promising raw material for use in various fibre products. They nevertheless differ in structure and chemical composition from the wood species used in industry, and
this reduces their attractiveness as industrial raw materials. These issues will be taken up in the following sections.

2.2 The structure of grass

2.2.1 General structure

A grass contains four distinct parts: the root, stem, leaves and flowers. The root is the part of the grass that grows below the ground and has the functions of binding the plant to the ground and supplying it with water and nutrients from the soil. The roots of grasses are of no value as an industrial raw material nowadays.

The above-ground parts of a grass consist of the stem, leaves and flowers (see Figure 1 on the following page). The stem comprises nodes and internodes. The nodes are typically solid, while the tendency for the cells to break away from the centre of the stem means that the internodes in most mature grasses are typically hollow tubes, although some species can be found which have solid internodes. The length and the diameter of the stem can vary greatly between species. All the cells in grass stems are axially oriented, so that there are no cells providing radial transportation (Evert 2006, Hubbard 1968, Ilvessalo-Pfäffli 1995, Metcalfe 1960).

The leaves are connected to the stem at its nodes, with each leaf consisting of a sheath and a blade. The sheath is the cylindrical or compressed part of the leaf that attaches it into the stem, while the blade is a relatively long, narrow, flat or v-shaped organ, except in some tropical grasses in which the leaves may be greater in width than in length (Evert 2006, Hubbard 1968, Metcalfe 1960).

The dry surplus residue from cereal production is loosely referred to as straw. Technically speaking, the term ‘straw’ applies only to the dry, coarse stems (Anon. 2003), whereas the agricultural residues from cereals usually consist of both leaves and stems, but the term ‘straw’ is accepted parlance in industrial circles even though in some cases, the leaves and stems may be separated and only one or the other used (Ilvessalo-Pfäffli 1995).

The type of flower head in a grass can vary, even between varieties of the same species. A common feature of grasses is that the flowers are arranged as spikelets that can include one or more flowers, each of which will produce a fruit. A dry fruits of plants in the grass family are called seeds or grain (Hubbard 1968, Metcalfe 1960). Grain is an almost perfect source of human nutrition and thus should be used primarily as food, although in practise large amounts of cereals are
also used as animal fodder and for biofuel production, for example. Meanwhile
the husk (or hull) that covers the grain and protects it is a residue left over from
food production that could be a valuable raw material for industrial use

Fig. 1. The stem and leaves make up the major part of the above-ground dry matter in
cereals. The dried-up straw could be a useful industrial raw material.
2.2.2 Tissues and cells in grasses

The tissues in plants can be divided into the meristem, epidermis, ground tissue and vascular tissue. The meristem is the tissue that produces all the cells in a plant and is to be found in growing plants. It will not be discussed in any more detail here as it is mature or dead plants that are usually used in industry.

The epidermis is the outermost cell layer of the grass that protects it from external threats and controls gas exchange. Due to their protective function, the cells are tightly attached into each other and are typically arranged in longitudinal rows. It has also been reported that epidermal cells focus light in order to maximize the amount of light available for photosynthesis. The shape of the cells in the epidermis varies according to the grass species, but five types of cell can be found: long cells, short cells, the guard cells of stomata, bulliform cells and trichomes. Long cells are elongated and may have undulated or serrated margins to increase the contact area between adjacent cells, while short cells include cork cells and silica cells. One distinctive feature of cork cells is their thick suberized walls, whereas silica cells are filled with a body consisting of transparent silica (SiO$_2$), although silica also occurs in the cell wall. Short cells are moulded by the adjacent cells and thus vary in shape. The guard cells are a group of oblong short cells that form the stomata which control gas exchange and water evaporation in the plant, while bulliform cells are colourless, large, thin-walled cells arranged in rows along the longitudinal axes of the leaves. It has been suggested that these cells cause the leaf to fold, thus reducing the surface area exposed to sunlight and minimizing evaporation under dry conditions. Finally, the protective cells, or trichomes, exist in the epidermis of all parts of the plant and include hairs, i.e. cells growing outwards from the epidermis, and papillae, direct outgrowths of epidermal cells. The epidermis in the aerial part of a grass is covered by a waxy layer, cuticle (Evert 2006, Ilvessalo-Pfäffli 1995, Metcalfe 1960), consisting of cutin, cutan and waxes. This highly hydrophobic layer forms an efficient barrier controlling the evaporation of substances from the plant and the entry of substances into the plant, making plant surface in effect self-cleaning and providing protection from UV light and pathogens (Barthlott & Neinhuis 1997, Evert 2006, Riederer 2006). Epidermal cells from wheat straw are shown in Figure 2.
Fig. 2. Cells contained in wheat straw pulp: an unbroken plate of epidermal cells (top left), sac-like parenchyma cells (top right), fibres (bottom left) and a vessel element (bottom right). Scale bar is 0.5 mm (bottom left) or 0.2 mm (otherwise).

The major part of the primary body of a grass consists of ground tissue. This takes care of storage and basic metabolism and supports the plant body. Ground tissue is composed of three cell types: parenchyma, collenchyma and sclerenchyma cells. Parenchyma cells are the most numerous cells in the plant and are located in all of its parts, almost entirely filling the stem and leaves. The easiest way of defining parenchyma cells is to say that they are cells that cannot be categorized into any other type. They constitute the most abundant and versatile cell type in plants and thus vary considerably in shape and dimensions, so that they can be sac-like (see Fig. 2), rounded, rectangular or rod-like. Parenchyma cells typically have thin walls, but these may also be thickened. The ground tissue of leaves is composed of mesophyll. The photosynthetic (chlorenchyma) cells found in the leaf and stem form the most important tissue in the plant, and another important function of parenchyma cells is the storage of water and organic substances. Collenchyma cells are long, elongated support cells.
located next to the epidermis in young plants. Their walls are unevenly thickened and non-lignified when the plant is growing, but these cells may transform into sclerenchyma cells and become lignified upon maturity. Sclerenchyma cells can be divided into fibres (see Fig. 2) and sclereids. The fibres in a stem are located in vascular bundles or in a sclerenchyma cylinder near the epidermis. In leaves, fibres can be found in vascular bundles or as separate strands. Grass fibres are typically long, narrow and thick-walled, although thin-walled fibres also exist. Sclereids are small, markedly lignified cells with a supporting function in the plant. They can be found in all parts of the plant and may be isodiametric, rod-like, elongated or irregular in shape. The cell wall of a sclereid is thick and highly lignified (Evert 2006, Ilvessalo-Pfäffli 1995).

The vascular tissue contains xylem, which transfers water and nutrients from the roots to other parts of the plant, and phloem, which transports sugars and other assimilation products from the leaves to other parts. The vascular bundles form continuous tubes from the roots to the leaf tips and the top of the stem (Evert 2006, Ilvessalo-Pfäffli 1995) and those in the stem may be scattered throughout its cross-section or arranged in two circles near the epidermis (Ilvessalo-Pfäffli 1995). The vascular bundles in grass leaves run in parallel longitudinally from the node to the tip of the leaf and are typically connected by transverse veins (Metcalf 1960). The cell types in vascular bundle are vessel elements, sieve tube members and companion cells, fibres, tracheids, parenchyma cells and sclereids. The vessel elements (see Fig. 2) and tracheids in the xylem are water-conducting cells, the former having annular, spiral, netlike or pitted walls. The annular and spiral vessel elements are long and narrow and are usually broken down or unwound during pulping, while the pitted vessel elements are thin-walled and vary greatly in width according to species. The sieve tube members are solute transporting cells in the phloem and typically have non-lignified cell walls of variable thickness, those forming early having thin walls, whereas the walls of the later-formed cells are relatively thick. The sieve tube members are accompanied by companion cells, parenchymatous cells that work with them to transport of organic substances in phloem (Evert 2006, Ilvessalo-Pfäffli 1995).

Grasses are monocotyledonous plants belonging to the extensive Poaceae family. The members of this family are closely related to each other and the cells vary between the species mainly in terms of size. Cells of a similar morphology may nevertheless belong to different cell types, and cells of the same type may differ in morphology according to the location of the tissue or organ in the plant.
It may therefore be hard to distinguish between the cell types (Ilvessalo-Pfäffli 1995, Jayme & Harders-Steinhauser 1941, Metcalfe 1960).

### 2.2.3 Cells in wheat straw

The chemical composition of wheat straw varies between the parts of a single plant and with the variety of wheat, the growing site and growing conditions, in addition to which the timing of the harvest have an effect on its chemical composition (Jacobs 1999). The primary chemical components of wheat straw are cellulose, hemicelluloses and lignin (39%, 33% and 14%, respectively) (Xu 2010). Cellulose makes up the strong body of the cell wall, while lignin is typically found mainly in the supporting and conducting cells (Evert 2006), although the parenchyma cells (Donaldson et al. 2001, Lloyd 1921, Zhai & Lee 1989) and sieve cells (Kuo & O’Brien 1974) of mature wheat straw can also be lignified. The hemicellulose content of wheat straw is high compared with that in wood, whereas the lignin content is low. Minor structural components found in the cell walls are pectins, suberin, callose and proteins (Carpita 1996, Liepman et al. 2007). The main types of extractives are fatty acids, resin acids, waxes, sterols, steryl esters and triglycerides. Depending on the delignifying chemistry, extractives may be hard to remove when dispersed during processing and thus may cause deposition problems (Peng et al. 2010).

The inorganic content of straw is higher than that of wood, and the major part of the ash consists of silicate, which is concentrated especially in the epidermal cells but can also be found in other cells, where it strengthens the wall structure (Ilvessalo-Pfäffli 1995, Sangster & Hodson 1986). The macronutrients present in wheat straw are N, P, S, K, Mg and Ca, accompanied by the micronutrients Al, Ba, Na, Sr, Fe, Mn, Zn, Cu, B, Mo, Cl and Ni (Jacobs 1999, Mehra & Farago 1994).

The basic structure of the cell wall is similar that observed in hardwoods and softwoods: middle lamella connects adjacent cells to each other, the primary cell wall is the outermost cell wall layer and the secondary cell wall, which can be divided into three layers (S₁, S₂ and S₃), is the innermost layer surrounding the lumen. The S₁ layer in wheat straw fibre is relatively thick (Donaldson et al. 2001, Zhai & Lee 1989). The microfibrils in a primary cell wall have a random net-like structure (Hua & Xi 1988, Zhai & Lee 1989). Hua & Xi (1988) attribute the fact that parenchyma cells do not shrink during drying due to this structure. Zhai & Lee (1989) note that the microfibril angle in the secondary wall is low,
being almost perpendicular to the longitudinal fibre axis in the \( S_1 \) layer and in the range 20° to 30° in \( S_2 \), but Hua & Xi (1988) maintain that the orientation in \( S_1 \) is a lateral cross-helix and that the angle in \( S_2 \) varies from 30° to 40°, thus being higher than in softwoods. The walls of the elongated epidermal cells are thick and the microfibrils are arranged parallel to the longitudinal axis of the cell (Liu et al. 2005).

The proportions of the various cell types found in wheat straw pulp depend on the parts of the plant used for pulping, since the leaves contain higher numbers of parenchyma and epidermal cells than the internodes (Jacobs 1999). In addition, the degree of refining affects the proportion of cells, since refining breaks down the plates of parenchyma and epidermal cells that are typically left unbroken during defibering (Roy et al. 1994, Subrahmanyam et al. 1999, Zhao et al. 1992). The proportions of the various cell types to be found in wheat straw pulp are summarized in Table 1. In addition to these numerical proportions, Müller (1960) has demonstrated that the cross-sectional area of a wheat straw stem contains 26% sclerenchyma cells, \( i.e. \) fibres, 68% parenchyma cells and 6% epidermal cells.

### Table 1. Proportions of the various cell types in wheat straw pulp.

<table>
<thead>
<tr>
<th>Fibres [%]</th>
<th>Parenchyma cells [%]</th>
<th>Epidermal cells [%]</th>
<th>Vessel elements [%]</th>
<th>Fibrous cells [%]</th>
<th>Non-fibrous cells [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>62</td>
<td>38</td>
</tr>
<tr>
<td>52.2</td>
<td>28.4</td>
<td>13.7</td>
<td>5.7</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>62.1</td>
<td>29.5</td>
<td>2.3</td>
<td>4.8</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Hua & Xi (1988)
Jayme & Harders-Steinhauser (1941)
Cheng et al. (1994)

The morphology and dimensions of the cells in wheat straw pulp vary greatly. The mechanical supporting cells in wheat straw consist of the fibrous cells found beneath the epidermis and the supporting cells in the vascular bundles. The mechanical tissue near the epidermis consists of thick-walled, elongated cells with a narrow lumen, whereas the fibres in the vascular bundles are more slender (Hayward 1938, Percival 1921).

The vessel elements in the vascular bundles are either narrow with annular or spiral thickenings or wide and pitted (Hayward 1938, Percival 1921). It is the primary walls of the annular and spiral vessel elements that are broken during pulping, and thus only the secondary walls, \( i.e. \) doughnuts and serpentine lines originating from these cells, can be found in the pulp (Ilvessalo-Pfäffli 1995). The sieve cells and companion cells in the phloem are short, narrow and thin-walled
(Hayward 1938), leading Lloyd (1921) to claim that these are dissolved during pulping.

The characteristic epidermal cells in wheat are short and elongated with undulated margins, although long, elongated cells with smooth edges can also be found. These cells cannot be distinguished from other fibrous cells in terms of their appearance (Lloyd 1921). Hairs are unicellular and vary in length from 20 μm to one millimetre. The short epidermal cells vary greatly in their dimensions and morphology (Percival 1921).

The morphology of the parenchyma cells in wheat straw varies greatly depending on the tissue in which they are located and the function they have. They vary in width from very narrow (4 μm) up to 350 μm, the cells in the ground parenchyma being short but varying in width from 35 μm to 100 μm, while wider ones are located in the centre of the stem (Hayward 1938, Percival 1921). Both thin and thick-walled ground parenchyma cells exist, and the cell wall may also be layered (Donaldson et al. 2001, Hayward 1938, Percival 1921, Zhai & Lee 1989). The assimilating tissue in wheat straw consists of cells that decrease in length from the top to the lower levels in the plant and possess lateral protuberances (Chonan 1965, Parker & Ford 1982).

The dimensions of the various cell types found in wheat straw are summarized in Table 2. Typically only the average cell length for the whole cell population is quoted for industrial purposes. As the cell type composition of pulp can vary, the average cell length varies too, and pulps with similar average cell lengths may have different cell length distributions.
Table 2. Measured dimensions for the various cell types present in wheat. Either the range or the average value is given, depending on the reference.

<table>
<thead>
<tr>
<th>Cell type</th>
<th>Width [µm]</th>
<th>Length [mm]</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epidermal cells</td>
<td>20–32</td>
<td>0.030–0.585</td>
<td>Lloyd (1921)</td>
</tr>
<tr>
<td></td>
<td>0.15</td>
<td></td>
<td>Wettstein (1962)</td>
</tr>
<tr>
<td></td>
<td>&lt; 0.5</td>
<td></td>
<td>Ilvessalo-Pfäffli (1996)</td>
</tr>
<tr>
<td></td>
<td>11–44</td>
<td>0.044–0.242</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>0.132</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td>Elongated cells in leaves</td>
<td>19–37</td>
<td>0.2–1.0</td>
<td>Beemster &amp; Masle (1996)</td>
</tr>
<tr>
<td>Other long cells</td>
<td>16–30</td>
<td>0.08–0.4</td>
<td>Beemster &amp; Masle (1996)</td>
</tr>
<tr>
<td>Elongated cells</td>
<td>9–20</td>
<td>0.15–0.3</td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Square cells</td>
<td>9–20</td>
<td>0.009–0.02</td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Bulliform cells</td>
<td>17–20</td>
<td>0.1–0.25</td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Hairs</td>
<td>up to 1 mm</td>
<td></td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Fibres</td>
<td>0.5–4.5</td>
<td></td>
<td>Lloyd (1921)</td>
</tr>
<tr>
<td></td>
<td>1.32</td>
<td></td>
<td>Wettstein (1962)</td>
</tr>
<tr>
<td></td>
<td>8–34</td>
<td>0.4–3.4</td>
<td>Ilvessalo-Pfäffli (1996)</td>
</tr>
<tr>
<td></td>
<td>5.5–44</td>
<td>0.385–4.565</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>16.5</td>
<td>1.144</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td>Parenchyma cells</td>
<td>0.4</td>
<td></td>
<td>Wettstein (1962)</td>
</tr>
<tr>
<td></td>
<td>33–110</td>
<td>0.055–0.407</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>70.4</td>
<td>0.209</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>20–100</td>
<td>0.10–0.35</td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Vessel elements</td>
<td>0.3</td>
<td></td>
<td>Wettstein (1962)</td>
</tr>
<tr>
<td></td>
<td>&lt; 60</td>
<td>&lt; 1.0</td>
<td>Ilvessalo-Pfäffli (1996)</td>
</tr>
<tr>
<td></td>
<td>5.5–55</td>
<td>0.176–0.902</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>24</td>
<td>0.443</td>
<td>Jayme &amp; Harders-Steinhauser (1941)</td>
</tr>
<tr>
<td></td>
<td>&gt; 20</td>
<td></td>
<td>Percival (1921)</td>
</tr>
<tr>
<td>Mesophyll</td>
<td>36</td>
<td>0.067</td>
<td>Parker &amp; Ford (1982)</td>
</tr>
<tr>
<td></td>
<td>20–50</td>
<td>0.030–0.150</td>
<td>Chonan (1965)</td>
</tr>
<tr>
<td>Sieve cells</td>
<td>0.25–0.30</td>
<td></td>
<td>Kuo &amp; O’Brien (1974)</td>
</tr>
</tbody>
</table>

2.3 The use of straw in industrial applications

At present only a small portion of all agricultural residues are utilized, mainly as bedding and feed for livestock, in pulp and papermaking in Asia, in building materials, in the automotive industry and in household energy supplies. Most residues are burnt in the fields, with the consequence that serious environmental pollution and health risks ensue on account of the smoke clouds (Magwood 2005, Yuan & Sun 2010).
2.3.1 Possible uses for straw

There are many research institutes, companies and organizations working for the more efficient use of agricultural residues. A major effort has put into the development of biorefineries, for example, the latest generation of which follows the principle of leaving grain for food production and using the non-food part of the plant for manufacturing various products. Biorefineries separate the chemical components of the straw into streams from which specialized chemicals, bioenergy and various types of biomaterials can be produced. These compounds may be used in the pulp and paper industry, the pharmaceuticals industry, water purification and the food industry, or in the manufacturing of cosmetics, plastics and construction materials, for example (Clark & Deswarthe 2009, Kamm et al. 2006). The possible applications proposed for wheat straw are summarized in Figure 3. Even though laboratory studies have shown that these products are possible, only a few of them are being produced on an industrial scale.

2.3.2 Challenges in the industrial use of straw

Despite the large amount of straw grown annually, the first challenge to overcome in the chain of production is related to raw material availability, in that the low bulk density of straw makes its transportation expensive. In addition, large storage areas are required for this bulky, seasonally produced material (Clark & Deswarthe 2009, Yuan & Sun 2010). It has been shown, however, that straw can be stored without quality degradation (Leponiemi 2011) and the development of compacting devices, e.g. pelletizing, briquetting and baling equipment will ease the transportation and storage problems (Clark & Deswarthe 2009).

In addition to the availability of the straw, the economics of the conversion processes used to separate the cells or chemical constituents from the plant body have to be considered. Even though the raw material is in many cases available practically free of charge, the price of petroleum is still sufficiently low that compensations and financial support are needed to make a biorefinery profitable today (Clark & Deswarthe 2009). Legislation, taxes, rises in petroleum prices and the development and optimization of conversion processes will in time reverse this situation (Satyanarayana et al. 2009). In addition, the new high-value end products obtainable from future biorefineries will make them more self-sufficient.

The heterogeneity of the raw material causes problems in industrial processes and product properties. Cells vary in their length, width and wall thickness and thus dimension distributions are wide, but the raw material should be consistent in quality to keep the product quality constant (John & Thomas 2008, Karade 2010, Satyanarayana et al. 2009). A high fibre length and length-to-width-ratio is recommended for fibres used as reinforcement in construction materials (de Lhoneux & Bordin 2011, Vinson & Huff 1988) and in biocomposites (Fowler et al. 2006), and thus the removal of fines from wheat straw could be beneficial. It is also reported that the fines fraction has a negative effect on the properties of medium-density fibreboards (Halvarsson et al. 2005). Variations in the chemical nature of the raw materials are detrimental in reinforcing fillers used in biocomposites, and the fact that cellulose fibres are hydrophilic whereas polymers used as the matrix in composites are typically hydrophobic may lead to problems in the compounding of these materials (Fowler et al. 2006, John & Thomas 2008, Kabir et al. 2012). In addition, the cuticle covering the epidermal cells is detrimental to the properties of construction panels manufactured from straw (Mo et al. 2005) and cause poor adhesion between wheat straw cells and the polymer matrix (Mo et al. 2005, Reddy & Yang 2005). Cell walls also absorb water and...
swell, so that composites strengthened with cellulose fibres are not dimension-stable under changing moisture conditions (Kabir et al. 2012).

A high fines content causes poor dewatering in pulp and paper making, (Cheng et al. 1994), and for this reason straw pulp beating is not recommended, because the plates of parenchyma and epidermal cells are broken down and the generation of fines can even detract further from the dewatering process (Roy et al. 1994, Sood et al. 2007, Zhao et al. 1992). Rising and picking are also reported in paper manufactured using wheat straw (Sood et al. 2007). When wood-based pulps are used, rising is caused by coarse fibres and can be observed as a loss of gloss and increasing sheet surface roughening. In this case fibre rising can be avoided by refining (Aspler & Bélard 1994). Vessel picking, manifested in the form of white spots in the printing ink, is caused by vessel elements when hardwood is used (Ohsawa 1987), and it seems that the fines fraction is a major contributor to both of these problems when wheat straw pulp is used (Sood et al. 2007). Hua & Xi (1988) reported that the cutin covering the epidermal cells causes poor adhesion and may thus be a possible cause of picking and rising. In addition, the silicate found mainly in epidermal cells may be dissolved and later deposited on surfaces during alkaline pulping (Ilvessalo-Pfaffli 1995) and will then complicate the chemical recovery. This issue is discussed in more detail by Tutuș and Eroğlu (2003).

2.4 Pulp fractionation

The idea behind pulp fractionation is to divide the pulp constituents, i.e. plant cells, into fractions with divergent cell properties, e.g. cell length, cell wall thickness or surface chemistry. An advantage can be gained from this fractionation in terms of better product properties or savings in energy or chemical consumption when the fractions are processed separately with sequences optimized according to the respective cell properties. In addition, the fractions can be used in separate products or in different places in a product, e.g. in separate layers of a multi-ply paper. The equipment available for use in fractionation on an industrial scale includes pressure screens, wire washers and hydrocyclones (Karnis 1997, Niinimäki et al. 2007, Seifert & Long 1974, Schabel 2010). In addition, the use of froth flotation (referred to below simply as flotation) in pulp fractionation has been studied.

The purpose of pressure screening, hydrocyclone cleaning and flotation is usually to remove impurities from the pulp, and the resulting fractions in these
applications are either acceptable or is to be rejected, we customarily refer to ‘accept’ and ‘reject’ fractions. Both fractions are meant to be used in some way in fractionation, however, so that naming them ‘accept’ and ‘reject’ fractions is not terminologically correct and an alternative nomenclature would be desirable. Pairs such as ‘underflow’ – ‘overflow’, ‘coarse’ – ‘fine’ and ‘concentrate’ – ‘tailings’ are used in industry for fractions of this kind, but they do not provide a universal description of them either. Therefore the terms ‘accept’ and ‘reject’ fractions will be used in this thesis to simplify the fractionation terminology.

2.4.1 Pulp fractionation by screening

Screening is a size-based separation method used widely in industry in which a material of mixed particle size is fed onto a screening surface that has apertures of a certain size. Particles with dimensions larger than the aperture remain on the screen, whereas small particles are able to pass through it, resulting in separation (Wills 1992). The separation principle in wire washing is similar to that in screening (Schabel 2010).

The mechanisms behind particle separation in a screen can be divided into barrier and probability screening. In barrier screening all the dimensions of a particle to be rejected must be larger than the smallest dimension in the screen aperture and thus there is no possibility at all of such a particle passing through the screen. The screening of elongated particles such as fibres, however, is a probabilistic matter, because it is possible for an elongated particle to pass through the screen even though not all its dimensions are smaller than the smallest aperture dimension. In the case of a fibrous material fractionated using a slotted screen, only one dimension needs to be smaller than the smallest aperture dimension, for example (Hautala et al. 2009, Steenberg 1953).

The screens used in pulp and paper production are pressure screens in which the pulp is fed into a casing containing a cylindrical screen. Small particles are able to pass through the screen into the accept line (often referred to as the fines fraction), whereas large particles remain on the screen and drift into the reject line (often referred to as the coarse fraction). Valves in the accept and reject lines can be used to control the splitting of the flow between them (Hautala et al. 2009). A schematic presentation of pressure screening is provided in Figure 4. Slotted screens with aperture widths as small as 0.10–0.25 mm and perforated screens with hole diameters of 1.0–3.0 mm are typically used in industrial applications (Hautala et al. 2009, Schabel 2010).
Screening is used in pulp and paper production for separating large impurities such as shives, fibre bundles, bark, sand and metal from the fibre fraction (Hautala et al. 2009), but the idea of using screens for fibre fractionation was patented as early as 1953 (Hill & Coghill 1953). A screen fractionates pulp constituents mainly according to particle size, i.e. short cells such as ray cells tend to become enriched in the accept fraction whereas long cells such as fibre tracheids tend to be enriched in the reject fraction. Fibre flexibility has an effect on fractionation too, however, as slender particles have a higher tendency to pass through the screen than do stiff fibres (Hautala et al. 2009). Screening can also be used to enrich specific cell types, e.g. to produce a fraction rich in vessel elements (Ogata 1978, Ohsawa et al. 1982).

There is an abundance of literature on wood pulp fractionation using pressure screens, but surprisingly, there is none on wheat straw pulp fractionation by this method. Even though the idea of non-wood pulp fractionation was patented in 1959 (Cusi 1959) and a current patent describes the use of screening as a pre-treatment method in the desilication of non-wood raw material (Chute & Vichnevsky 2008), all the existing studies of wheat straw pulp fractionation have been performed using flat laboratory screens such as the Bauer McNett model. These have nevertheless served to demonstrate the advantages of wheat straw pulp fractionation into short and long cell fractions, in that the removal of fines has been shown to be beneficial for water removal and the optical properties and tear strength of the paper (Aronovsky et al. 1947, Cheng et al. 1994, Ljusegren et al. 2006, Jacobs 1999, Rousu & Hytönen 2007, Rousu & Niinemäki 2007, Roy et al. 1994, Sood et al. 2007, Subrahmanyam et al. 1999, Vichnevsky & Chute 2001). It has also been shown that fines have poor bleachability and need different sequences from long fibres in the production of cellulose nanofibres by...
TEMPO-mediated oxidation (Heijnesson-Hultén et al. 2012). This process employing TEMPO (2,2,6,6–tetramethylpiperidine–1–oxyl radical) is a chemical treatment used to assist the liberation of individualized cellulose nanofibres from cell walls (Isogai et al. 2011). Fines also have a positive effect on the opacity and tensile strength of some pulps, and therefore not all fines should be removed (Jacobs 1999, Rousu & Niinimäki 2007) and various fractionation strategies should be considered. Sood et al. (2007) have shown that to obtain a better tensile index, burst index and folding endurance and to avoid fibre rising and vessel picking, triple-layered paper should be manufactured in which the fines fraction should be placed in the middle layer located between fibre fractions. Aronovsky et al. (1947), Jacobs (1999), Roy et al. (1994) and Subrahmanyam et al. (1999) have also demonstrated that fines should be removed prior to screening because undefibered cell plates are broken in refining, causing the amount of fines to increase further during refining, thus detracting from water removal. These studies also showed that the development of wheat straw pulp fibres in refining is faster when the fines are removed. Screens are used also in bagasse depithing, since the removal of the fines from bagasse pulp is beneficial for pulp processability and paper properties (El-Sharkawy et al. 2007, Rainey 2012).

2.4.2 Hydrocyclone fractionation

The hydrocyclone process is a means of classification in which particles are separated according to their settling properties, these being mainly affected by particle size, shape and density. In practice, a hydrocyclone can be used to fractionate particles of the same size but different densities, or alternatively, particles of similar densities but different sizes (Bradley 1965). A schematic presentation of the hydrocyclone fractionation process is contained in Figure 5. A suspension is fed tangentially into the cylindrical section of the hydrocyclone, whereupon the resulting rotational movement inside the hydrocyclone brings about centrifugal acceleration and a settling of particles. Since large, heavy particles have a higher settling velocity, they gravitate close to the hydrocyclone wall, where the vertical flow pattern towards the apex drives them into the reject fraction. Conversely, the vertical flow in the centre of the hydrocyclone carries the small, light particles having a low settling velocity towards the accept fraction (Bradley 1965).
The hydrocyclone method is widely used in pulp and paper production for separating either light or heavy impurities such as bark, plastics, metals and sand from the pulp (Hautala et al. 2009). Much research has been done into the behaviour of fibres and fines in hydrocyclone fractionation, and it can be stated in general terms that hydrocyclone fractionation produces an accept fraction that is rich in low density particles with a high specific surface area and the reject fraction consists of thick-walled, coarse particles (Niinimäki et al. 2007). Softwood fibres have been shown to be fractionated according to their cell wall thickness, with spring wood cells having a thin cell wall and large lumen tending to become enriched in the accept fraction whereas thick-walled summer wood cells are enrich in the reject fraction (Asikainen et al. 2011, Brännvall et al. 2007, Jones et al. 1966, Kure et al. 1999, Laine et al. 2004, Malm 1967, Paavilainen 1992, Pesch 1963, Shagaev & Bergström 2005, Vomhoff & Grundström 2003). The advantage of such fractionation lies in the contrasting paper-making properties of thin-walled and thick-walled fibres, the former producing smooth-surfaced paper whereas the latter produce bulky paper (Paavilainen 1992). In addition, thick-walled cells show a faster response in refining (Laine et al. 2004), so that fractionation can be used to optimize the refining stages. In addition to paper manufacture, fractionation and alternative refining strategies for the fractions can also be exploited in the manufacturing of cement-bonded construction materials (de Lhoneux & Bordin 2011, Vinson & Huff 1988). Hydrocyclone fractionation can also be used to separate pulp constituents according to their specific surface area (Wood et al. 1991, Wood & Karnis 1977, Wood & Karnis 1979), allowing unbeaten and beaten fibres to be enriched in separate fractions (Park et al. 2005).
Less attention has been paid to the fractionation of various cell types in a hydrocyclone, but fractionation studies performed using hardwood pulps have shown that this method can be used to remove vessel elements from pulp and thus reduce vessel picking (Asikainen et al. 2010, Ohsawa 1987, Ohsawa et al. 1982, Ohsawa et al. 1984, Panula-Ontto et al. 2007). It has been found that the bonding potential of vessel elements can be increased, and vessel picking thereby reduced, by breaking these cells up by means of high consistency refining (Marton & Agarwal 1965, Nanko et al. 1988, Ohsawa et al. 1984). The behaviour of vessel elements in a hydrocyclone depends on the shape of their cells, since vessel elements with a high length-to-width-ratio reportedly become enriched in the accept fraction whereas those with a low length-to-width-ratio have a higher tendency to be enriched in the reject fraction. If the ratio is in the range 5 to 10, it seems that cells do not fractionate at all (Asikainen et al. 2010, Mukoyoshi & Ohsawa 1986, Mukoyoshi et al. 1986). Even though a patent exists for non-wood pulp fractionation using a hydrocyclone (Coppic & Brown 1967), no research has been done into this use of the method.

2.4.3 Pulp fractionation by flotation

Flotation is a unit process in which particles are separated on the basis of their surface chemistry, i.e. hydrophobic and hydrophilic particles can be enriched in separate fractions. Flotation is widely used in the minerals industry and in waste water treatment. Basically, flotation is a simple unit process. A suspension containing hydrophilic and hydrophobic particles is fed into a flotation cell. Air bubbles injected into the bottom of the cell rise up and collect the hydrophobic particles, which adhere to them. A froth containing these hydrophobic particles is then collected from the surface of the flotation cell to constitute the reject fraction, whereas the hydrophilic particles remaining in the suspension are collected from the bottom of the cell to form the accept fraction (Nguyen & Schulze 2004). One advantage of flotation is that it can be used to fractionate particles of similar densities and dimensions. A schematic presentation of the flotation process is given in Figure 6.
Fig. 6. Separation of hydrophilic and hydrophobic particles by flotation.

Flotation is also used in the deinking of recovered paper to remove hydrophobic contaminants such as ink, stickies, fillers, coating pigments and binders from the suspension (Schabel 2010). It is efficient in removing particles in the size range of a few micrometres to a few hundred micrometres (Schabel 2010, Nguyen & Schulze 2004), as these small particles are subject to true flotation, i.e. flotation caused by the adherence of hydrophobic particles to air bubbles, whereas larger particles are too heavy to be raised to the surface by true flotation. Attempts have also been made to fractionate chemical and mechanical pulp cells by flotation, the hypothesis being that these can be separated due to their differences in surface chemistry, given that mechanical pulps have a higher lignin content than chemical pulps and are thus more hydrophobic (Hodgson & Berg 1986, Koljonen 2004) and have a higher tendency to float. The level of hydrophility is shown to increase with decreasing lignin content, and also with decreasing extractives content (Koljonen 2004). In their tests performed on various chemical and mechanical pulps, Schwinger & Hanecker (1991) found that only TMP fibres floated, whereas Muvundamina and Li (1997) found that chemical pulp fibres had a higher tendency to float into the froth, and Eckert et al. (1997, 2000) and Ajersch (1997) showed that both chemical and mechanical pulp cells can be floated. The chemistry of flotation is highly complex, and the surface chemistry of fibres may have role on their flotation, although it has been suggested that surface chemistry plays only a minor role in fibre flotation and that, contrary to the situation in true flotation, fibres are mainly floated by mechanical entrapment when the consistency is low, i.e. fibre agglomerates are raised when the flock that they form collects sufficient air bubbles to cause them to float, leading to fibre losses in deinking. The formation of a continuous fibre network causes the interlocking of fibre flocks as consistency increases and the bubbles are unable to lift the flocks but themselves rise through channels that form within the fibre network. The fines content of the flotation reject increases because the bubbles collect the
fine particles that are unable to participate flocculation (Ajersch 1997, Eckert et al. 1997, Eckert et al. 2000).

No research results are available concerning straw pulp fractionation using flotation, but it is evident that, together with the rough surface microstructure, the waxes covering a plant can produce superhydrophobic self-cleaning surfaces (Barthlott & Neinhuis 1997) and that hydrophobicity is higher in epidermal cells than in other cell types. Flotation could therefore be a suitable process for separating small epidermal cells from other cells found in wheat straw pulp.

2.4.4 Assessment of fractionation

Regardless of the method used, fractionation is a multivariable system in which the design parameters, operational parameters and feed quality all affect the outcome (Steenberg 1953). The comparing of different operational points and fractionation methods is not a straightforward matter, but some basic parameters can be used to define the point of fractionation. These include the volumetric and mass flow rates and parameters that measure fractionation selectivity.

The volumetric and mass flow rates (R_V and R_m, respectively) denote the proportion of the feed pulp that flows into a particular fraction. Typically it is the flow rate into the reject fraction, i.e. RR_V or RR_m, that is presented. The volumetric reject rate RR_V is equal to the total liquid flow rate into the reject fraction (in litres per minute, for example) and can be calculated using the equation

\[ RR_V = \frac{V_R}{V_F}, \]  

where \( V_R \) and \( V_F \) are the volumetric flow rates of the reject (R) fraction and feed (F). The mass flow rate (in grams per minute, for example) denotes the total flow rate of solids into a fraction. Thus the mass reject rate (the mass flow into the reject fraction) can be calculated using the equation

\[ RR_m = \frac{V_R c_R}{V_F c_F} = RR_V \cdot \frac{c_R}{c_F}, \]  

where \( c_R \) and \( c_F \) are the consistencies of the reject fraction and feed, respectively. (Anon. 1974)

When separating impurities from pulp, the mass flow rate into the accept fraction is taken as a measure of the fractionation capacity, as it is the accept fraction that is used as a raw material in the subsequent stages in the process and
the aim is to maximize that fraction. Both fractions are meant to be used, however, so that in general terms fractionation capacity is equal to the amount of feed. On the other hand, capacity alone does not define the success of fractionation, as the cleanliness of the fractions has to be considered too. Therefore, some effort should be made to maximize fractionation selectivity, whereupon fractionation capacity can be increased by investing in larger equipment.

The simplest way of determining whether fractionation occurs is to compare the average pulp or paper properties, cell dimensions or component contents between the fractions. The fractionation index (FI) presented by Karnis (1997) compares pulp or cell property X, e.g. average fibre length, between fractions I and II. This can be calculated using the equation

\[ FI = 1 - \frac{X_I}{X_{II}} = \frac{X_{II} - X_I}{X_{II}}. \] (3)

Thus FI indicates the percentage by which property X is higher in fraction I, so that a value differing from zero denotes that fractionation has occurred. The values are typically chosen in such a way that \( X_I < X_{II} \), whereupon the fractionation index varies in a range between zero (no fractionation at all) and one (towards which fractionation becomes progressively more selective). The fractionation index can also be negative, implying that \( X_I > X_{II} \). In that case it varies in a range between zero and minus infinity, with higher negative values denoting more selective fractionation.

The screening quotient \( Q \) presented by Nelson (1981) can be used to determine the fractionation selectivity for impurities found in pulp, but it can also be used to compare the mass proportions of a certain cell type or chemical component in the accept and reject fractions. Basically, the idea of the screening quotient is similar to that of FI and it can be calculated using the same equation but including the mass proportion \( Y \) of the component in the fraction instead of a cell property:

\[ Q = 1 - \frac{Y_I}{Y_{II}} = \frac{Y_{II} - Y_I}{Y_{II}}. \] (4)

The fractionation index and screening quotient can be used to indicate whether fractionation has occurred, whereas the removal efficiency \((E_R)\) denotes the proportion of a component by weight selected from the feed pulp into a given fraction. Where the component concerned is concentrated in the reject fraction, removal efficiency can be calculated using the equation
where Y is the mass proportion of the given component in the reject (R) and feed (F) (Anon. 1974).

The relationship between fractionation selectivity, removal efficiency and mass reject rate can be demonstrated by means of Equation 6 (Wahren 1979):

\[
E_R = RR_m \cdot \frac{Y_R}{Y_F},
\]  

(5)

\[
E_R = \frac{1}{1+(1-Q)(RR_m-1)}
\]  

(6)

The relationship presented in Equation 6 is illustrated in Figure 7. Here the dashed line \((E_R = RR_m)\) corresponds to the T-pipe and represents a situation in which fractionation does not occur at all \((Q = 0)\). The solid lines depict alternative instances of fractionation selectivity: the bigger the difference between \(E_R\) and \(RR_m\), the better the fractionation selectivity is. Thus a high \(E_R\) gained using a low \(RR_m\) or a low \(E_R\) gained using a high \(RR_m\) yield the purest fractions. In pressure screening, for example, an accept fraction that is rich in short cells can be produced using a high \(RR_m\) and small apertures, whereas a screen with large apertures and operated at a low \(RR_m\) will produce a reject fraction rich in long cells (Olson et al. 2001).
Fig. 7. Removal efficiency presented as a function of mass reject rate, illustrated using various degrees of fractionation selectivity (solid lines). The dashed line (fractionation selectivity $Q = 0$) represents a situation in which no fractionation occurs at all. Any point above that line represents a situation in which particles are enriched in the reject fraction, whereas points below the line denote cases in which fractionation into the accept occurs.
3 Aims of the present research

Based on the literature survey presented above, it was concluded that the heterogeneity of straw pulp hinders its use in industrial applications. Some cell types are detrimental to straw processability and end use properties and the removal of these cells would increase the attractiveness of straw as an industrial raw material. Fractionation could be a tool for modifying straw pulp composition. In addition to optimizing pulp properties, it would also enable new tailored properties to be achieved, thus creating new applications for straw pulps. The aims of the work reported in this thesis were thus:

1. to determine the fractionation tendency of wheat straw pulp cells in ultra-fine pressure screening,
2. to determine the fractionation tendency of wheat straw pulp cells in hydrocyclone fractionation, and
3. to find out whether it is possible to fractionate epidermal cells selectively by flotation.
4 Materials and methods

4.1 Materials

Two wheat straw pulps were used in the experimental part. Pulp 1 was a commercial bleached Chinese wheat straw (Triticum aestivum L.) pulp delignified by a soda method and received in the form of once-dried sheets. These sheets were soaked overnight in deionized water and disintegrated for two hours at a temperature of 50°C and a consistency of 0.7% in a vat pulper. Tap water with a hardness of 4°dH, was used for diluting at all stages in the process. This pulp was used in the wheat straw pulp dimension analysis (Paper I) and pressure screen (Paper II) and hydrocyclone (Paper III) fractionation experiments.

Pulp 2 was an unbleached, never-dried Finnish wheat straw (Triticum aestivum L.) pulp delignified using a formicofib™ technology and formic acid-based delignification. An unbleached pulp was chosen because silicates and waxes are not dissolved during acidic cooking (Iler 1979, Ilvessalo-Pfäffli 1995), and thus the epidermal cells retain their hydrophobic nature. The washed and thickened pulp was received at a consistency of 20%. It was then disintegrated in a vat pulper at a temperature of 20°C and a consistency of 1.5% for six minutes. Tap water with a hardness of 4°dH was used for diluting at all stages. This pulp was used in the flotation experiments (Paper IV) and in a supplementary trial for pressure screening and hydrocyclone fractionation.

4.2 Fractionation trials

The fractionations required for studying the behaviour of wheat straw pulp cells in pressure screening, hydrocyclone fractionation and flotation were performed in the Fibre and Particle Engineering Laboratory of the University of Oulu. A multi-stage fractionation experiment using pressure screening and a hydrocyclone was performed first as a preliminary trial in order to study whether fractionation would occur at all. The further purpose was to study the feasibility of using an automatic fibre analyser to determine the cell types present in wheat straw pulp. Secondly, single-stage fractionations were performed to study the behaviour of wheat straw pulp cells in pressure screening, hydrocyclone fractionation and flotation.
4.2.1 Preliminary trial

A multi-stage fractionation was performed to ascertain whether wheat straw pulp could be separated into fractions containing different cell types (Paper I). A diagram of this multi-stage fractionation is presented in Figure 8. A pressure screen equipped with a smooth, perforated screen plate with a hole diameter of 0.2 mm and a 2-foil rotor with a tip velocity of 16.3 m/s was first used to produce long and short cell fractions. The feed consistency, aperture flow velocity and mass reject rate were 0.5%, 0.45 m/s and 40%, respectively. The reject fraction was then fractionated with a 60 mm Cellco Cleanpac 270 hydrocyclone using a pressure difference of 3.7 bar, a mass reject rate of 45% and feed consistency of 0.2%, while the pressure screen accept fraction was fractionated with a 50 mm Mozley C124 hydrocyclone using a feed consistency of 0.15%, pressure difference of 4.0 bar and total mass reject rate of 50%. These short cell fractions were thickened first by settling and then by filtration using a 0.02 mm filter cloth. The pulp temperature at all stages in the fractionation was 21°C and tap water was used for dilution. Data were collected by means of pressure gauges and magnetic flow meters.

Fig. 8. Schematic diagram of the multi-stage preliminary fractionation trial performed to determine whether fractionation occurs at all. The feed (F) was first fractionated by pressure screening and then both fractions were fractionated in hydrocyclones. Modified from Paper I, published by permission of Karjalainen et al.
4.2.2 Primary experiments: Pressure screening

A single-stage fractionation was performed to study the effect of operation and design parameters on the behaviour of wheat straw pulp cells in pressure screen fractionation (Paper II). A diagram of this pressure screening is presented in Figure 9. Data collected from pressure gauges and magnetic flow meters were used to control the pump, and thus also the feed flow velocity. The splitting of the flow between the resulting fractions was controlled using valves in the accept and reject pipes. The accept and reject streams were recirculated to an open 300 litre feed tank and sampled when a steady-state had been reached.

Fig. 9. Single-stage fractionation by pressure screening. The accept (A) and reject (R) flows were recirculated to the feed tank (F). The whole flow was turned on and off in both the accept and reject lines simultaneously during sampling (S) so that this should not have any effect on feed quality. Modified from Paper II, published by permission of Karjalainen et al.

The fractionation was performed by means of a Metso FS 03 pressure screen, using a wedge wire and a smooth perforated basket. The wedge wire screen basket had a slot width of 0.06 mm, a profile height of 0.5 mm, a screening area of 0.03 m² and an open area of 3.3%. The mass reject rate varied in the range 29 to 80% and the volumetric reject rate in the range 20 to 50%. The slot flow velocity varied in the range 0.34 to 2.02 m/s. The smooth perforated screen basket had a hole diameter of 0.2 mm and an open area of 10%. The mass reject rate and volumetric reject rate varied in the ranges 73 to 92% and 20 to 27%, respectively. The aperture flow velocity varied from 0.40 to 0.43 m/s. A feed consistency of
0.5%, temperature of 21°C and a 2–foil rotor with a tip velocity of 16.3 m/s were used in all the experiments.

4.2.3 Primary experiments: Hydrocyclone fractionation

A single-stage fractionation was performed to study the effect of varying the operation parameters on the behaviour of wheat straw pulp cells in hydrocyclone fractionation (Paper III). A diagram of this hydrocyclone fractionation is presented in Figure 10. A 60 mm Celleco Cleanpac 270 hydrocyclone with a feed pulp consistency of 0.17% and a temperature of 23°C was used. The pressure difference (dP) between the hydrocyclone feed and accept pipes is typically used to control the operation of the hydrocyclone (Bradley 1965, Jokinen 2007). The operation pressure range given by the hydrocyclone manufacturer, 1.8–3.7 bar, was used throughout. Since the pressure drop is proportional to the feed flow rate (Bradley 1965), it was controlled by adjusting the latter. The pressure difference range used here corresponded to feed flow velocities of 1.4 to 2.0 litres per second. Data collected from pressure gauges and magnetic flow meters were used to control the pump, and thus also the operation point of the fractionation. The accept and reject streams were recirculated into an open 300 litre feed tank and sampled when a steady state had been reached. Nine parallel test points were defined for each operation point and the run order was fully randomized.
4.2.4 Primary experiments: Flotation

Flotation was used to study the feasibility of fractionating the epidermal cells in wheat straw pulp (Paper IV). A soap chemistry typically used in deinking was adopted here, and the soap and calcium dosage and flotation consistency were employed as variables in the flotation experiment. The anionic surfactant was a commercial soap, Serfax MT90 (Stephenson Group; Leeds, UK), which, according to the manufacturer, is a mixture of fatty acid soaps. Since a pH range of 8.5–9 is recommended by the manufacturer, pH was adjusted to 8.5 using sodium hydroxide. A temperature of 24°C was used because the solubility of silicates at that pH is low at low temperatures (Iler 1979). A commercial soap, Fairy Original (Procter & Gamble, UK), was used as an additional frother. According to the manufacturer, this soap is a mixture of surfactants. The dosage of this flotation aid was 0.2 grams per batch. The concentration of calcium, which is typically used with a soap chemistry as a flotation aid (Ferguson 1992, Larsson et al. 1984), was adjusted with calcium chloride. The design of the experiment is presented in Table 3. The flotation was performed as a single-stage fractionation with a Voith Delta 25 flotation unit using a batch size of 22 litres and a constant
air flow of 7.4 litres per second. The calcium chloride and soaps were dissolved in hot water prior to the flotation. In the flotation itself, a sample of the proper consistency was prepared and its pH adjusted, after which the chemicals were added and the air intake opened after a 1 min conditioning time with mixing switched on. The resulting froth was collected from the top of the cell to form the reject fraction. The flotation was continued for 6 minutes, after which the air intake was closed and the pulp remaining in the flotation cell was collected to form the accept fraction.

Table 3. Design of the flotation fractionation experiments. The flotation temperature was 24°C and the pH 8.5.

<table>
<thead>
<tr>
<th>Batch</th>
<th>Soap dosage [kg/t]</th>
<th>Water hardness ['dH']</th>
<th>Feed consistency [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6</td>
<td>4</td>
<td>0.3</td>
</tr>
<tr>
<td>2</td>
<td>6</td>
<td>11</td>
<td>0.3</td>
</tr>
<tr>
<td>3</td>
<td>6</td>
<td>18</td>
<td>0.6</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
<td>18</td>
<td>1.0</td>
</tr>
<tr>
<td>5</td>
<td>3</td>
<td>18</td>
<td>0.3</td>
</tr>
<tr>
<td>6</td>
<td>9</td>
<td>18</td>
<td>0.3</td>
</tr>
<tr>
<td>7</td>
<td>6</td>
<td>18</td>
<td>0.3</td>
</tr>
</tbody>
</table>

4.2.5 Sampling

Samples of approximately 5 litres were taken from the accept and reject lines and feed tank in the pressure screening and hydrocyclone fractionation experiments. In the case of the accept and reject lines, automatic three-way sampling valves were used to turn the whole flow into the sample line and were switched on and off simultaneously so that despite the recirculation, the feed quality did not change during fractionation. The simple instrumentation and short piping also meant that sampling did not affect the operational point of the fractionation. In the flotation experiments the reject formed during flotation was collected as a single sample and the accept fraction consisted of the pulp remaining in the flotation cell.

4.3 Analyses

A method was developed for studying the proportions of the various cell types in wheat straw pulp. This method is presented in the following section. The other analyses used in the experimental part of the work are presented in section 4.3.2.
4.3.1 Analysis of cells in wheat straw pulp

The composition of wood-based pulps has been well studied and the analysis of the cells to be found in these pulps has been automatized. The cells found in softwood pulps are either long fibre tracheids or short ray cells, and an automatic fibre dimension analysis is available that divides cells according to their dimensions, those shorter than 0.2 mm (ray cells) been taken as fines and the others as fibres. In addition to these cells, hardwood pulps contain vessel elements with a much greater width than other cells, so that they are easily distinguishable. Thus the proportions of the cell types can be determined using automatic cell dimension analysis and it is easy to monitor their fractionation. The currently available automatic optical fibre length analysers have shown to be reliable (Guay et al. 2005, Turunen et al. 2005).

Characterization of the cell types found in wheat straw pulp is not so straightforward, however, because the whole plant is typically used and the pulp contains various cell types. No automatic analysis method exists for these cell types, and the standardized methods used in cell type analysis (ISO 9184, SCAN G3 and G4, TAPPI T401) are based on microscopic examination, which is time-consuming and calls for a high degree of expertise. Consequently, the analysis of samples from extensive fractionation trials is expensive.

In an attempt to find a fast, straightforward method for analysing the cell type composition of wheat straw, a microscopic study was performed in which cell dimensions were measured in order to define minimum and maximum dimensions for the various cell types (Paper I). A Leica MZ FLIII stereomicroscope equipped with a Leica DFC 320 digital camera was used to photograph the cells and Leica IM50 software to measure their lengths and widths. The Fiber Atlas of Ilvessalo-Pfäfl (1995) was used to identify the cell types and at least 155 particles were measured for each type.

The microscopic analysis thus revealed the variation in the dimensions of wheat straw pulp cells, which were in turn categorized according to their appearance into epidermal cells, parenchyma cells, vessel elements and fibrous cells. No assimilating cells, i.e. mesophyll cells, were found in the pulp. The easiest to identify were the short epidermal cells with undulating margins and the pitted vessel elements. The doughnuts originated from annular vessel elements, and thin strings originating from helical vessel elements were also found. Short sac-like parenchyma cells were also easily recognizable, but long, thin parenchyma cells could easily be confused with fibres, so that all the elongated
cells that couldn’t be placed in any other class were designated as fibrous cells. Thus collenchyma cells, fibres, sieve cells and epidermal cells that had a smooth cell wall were all included in this class. Maximum and minimum cell lengths and widths were measured for all of these types. The resulting dimensions and aspect ratios, i.e. length-to-width ratios, and the ranges that encompassed 95% of the cells in each case are presented in Table 4.

Table 4. Variations in the lengths and widths of the various cell types in Pulp I. Modified from Paper I, published by permission of Karjalainen et al.

<table>
<thead>
<tr>
<th></th>
<th>Fibrous cells</th>
<th>Pitted vessel elements</th>
<th>Parenchyma cells</th>
<th>Epidermal cells having undulated margins</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Length</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min-max</td>
<td>0.13–3.18</td>
<td>0.08–1.42</td>
<td>0.04–1.5</td>
<td>0.01–0.36</td>
</tr>
<tr>
<td>95%</td>
<td>0.2–2.4</td>
<td>0.2–1.1</td>
<td>&lt; 0.5</td>
<td>&lt; 0.3</td>
</tr>
<tr>
<td><strong>Width</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min-max</td>
<td>10–40</td>
<td>10–200</td>
<td>10–170</td>
<td>10–50</td>
</tr>
<tr>
<td>95%</td>
<td>10–30</td>
<td>&gt; 70</td>
<td>10–110</td>
<td>&lt; 30</td>
</tr>
<tr>
<td><strong>Aspect ratio</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min-max</td>
<td>5–133</td>
<td>2–120</td>
<td>1–30</td>
<td>1–20</td>
</tr>
<tr>
<td>95%</td>
<td>9–105</td>
<td>8–36</td>
<td>&lt; 12.5</td>
<td>&lt; 12.5</td>
</tr>
</tbody>
</table>

The cells were then divided into five categories according to their dimensions as determined in the microscopic examination or defined in the literature survey. The resulting dimensions for the categories and cell types belonging to each category are presented in Table 5 and a schematic diagram of the categorization in Figure 11. This categorization combined with automatic cell dimension analysis was used when characterizing the behaviour of the various cell types in wheat straw pulp.
Table 5. Cell categories used in this thesis.

<table>
<thead>
<tr>
<th>Category</th>
<th>Length [mm]</th>
<th>Width [µm]</th>
<th>Aspect ratio</th>
<th>Cells typical of the class</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₁: Long fibrous</td>
<td>≥ 0.375</td>
<td>≤ 30</td>
<td></td>
<td>Long fibres, collenchyma cells, the longest epidermal cells.</td>
</tr>
<tr>
<td>C₂: Short fibrous</td>
<td>0.2–0.374</td>
<td>≥ 12.5</td>
<td></td>
<td>Short fibres, sieve tube cells, medium-sized epidermal cells.</td>
</tr>
<tr>
<td>C₃: Long sac-like</td>
<td>≥ 0.375</td>
<td>&gt; 30</td>
<td></td>
<td>Pitted vessel elements, long parenchyma cells.</td>
</tr>
<tr>
<td>C₄: Short sac-like</td>
<td>0.2–0.374</td>
<td>&lt; 12.5</td>
<td></td>
<td>Short parenchyma cells, epidermal cells with undulating margins.</td>
</tr>
<tr>
<td>C₅: Fines</td>
<td>&lt; 0.2</td>
<td></td>
<td></td>
<td>The shortest parenchyma and epidermal cells.</td>
</tr>
</tbody>
</table>

Fig. 11. Categorization of wheat straw pulp cells into five classes by length and width. Paper I, published by permission of Karjalainen et al.

A preliminary fractionation trial was performed to produce pulp fractions rich in various cell types, and it was found that the above categorization method enabled us to monitor the behaviour of the various cell types in pressure screen and hydrocyclone fractionation, as the method showed good correlation with the standardized cell type analysis (for more detailed information, see Paper I). An example of the composition of the feed pulp used in Paper II is shown in Table 6. The measurement error is presented as the 95% confidence interval of the mean for 10 replicates.
Table 6. Composition of the feed pulp used in Paper II. The measurement error is calculated as the 95% confidence interval of the mean determined from 10 replicates.

<table>
<thead>
<tr>
<th>Category</th>
<th>Average [%]</th>
<th>± [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>35.3</td>
<td>0.5</td>
</tr>
<tr>
<td>C2</td>
<td>19.2</td>
<td>0.4</td>
</tr>
<tr>
<td>C3</td>
<td>3.8</td>
<td>0.2</td>
</tr>
<tr>
<td>C4</td>
<td>5.7</td>
<td>0.1</td>
</tr>
<tr>
<td>C5</td>
<td>36.0</td>
<td>0.4</td>
</tr>
</tbody>
</table>

4.3.2 Other analyses used in the experimental work

Consistency was analysed using the TAPPI T240 method, Canadian standard freeness according to the ISO 5267–2 standard, ash content according to the ISO 1762 standard at a temperature of 525°C and silicate content by the TAPPI T244 cm–99 method. An automatic analysis of cell dimensions was performed using a FiberLab (Metso Automation, software version 4.2.4) fibre analyser. Samples for cell dimension analysis were prepared according to the TAPPI T271 standard, which recommends the analysis of a minimum of 5000 measured particles. This method involves measurement of the dimensions of individual cells passing through a narrow capillary by automatic image analysis. The mass-based length distribution of wheat straw pulp cells was measured using a Bauer-McNett classifier according to the TAPPI T233 method, employing sieves of 28, 48, 100 and 200 mesh.

The reference measurements for the cell type analysis performed in the preliminary trials (Paper I) were made in the Finnish KCL pulp and paper analysis laboratory according to the KCL method and the ISO 9184 standard and using the Fiber Atlas of Ilvessalo-Pfäffli (1995) for cell type identification.

4.3.3 Statistical testing of the results

Statistical testing was performed to determine whether the accept and reject flows had statistically different values, i.e. that fractionation had occurred. The data for each fractionation method were studied as parallel test points and the means calculated from the accept and reject flows were compared. The results were analysed statistically using IBM SPSS 21.0 software and a confidence level of 95%. Initial use of the Kolmogorov-Smirnov test showed that the results were normally distributed in all cases. Next, the T-test was used to compare the means.
The paired T-test was used in the statistical testing of means, as the values for the accept and reject flows are obviously related, i.e. when a property changes in one fraction the value in the other fraction will change too. P-values less than 0.05 were considered to denote statistical significance.
5 Results

The main findings to arise from the fractionation experiments are presented in this chapter. The detailed data often referred to can be found in the original papers presented at the end of the paper version of the thesis. The average cell dimensions in the accept and reject fractions will be considered first, and after that the behaviour of the various cell types in fractionation.

5.1 Average cell dimensions in the fractions

The fractionation pattern for wheat straw pulp, expressed in terms of average cell dimensions, is presented in Table 7. This gives the range within which the FI varied, the average FI and the statistical significance of the result. A p-value of less than 0.05 shows that the content of the cell type in question is higher in one or other of the fractions, as indicated in the table.

Pressure screening, especially with a perforated screen, gave good fractionation in terms of cell length, the average cell length being lower in the accept fraction. In the case of hydrocyclone fractionation the average length-weighted cell length was higher in the accept fraction but the difference between the fractions was negligible. The average cell length was also higher in the flotation accept fraction, but the result was not statistically significant even though the fractionation index was as high as 0.2. This can be explained by the method used for statistical testing, in which the data for each fractionation method were assessed as a single dataset. Thus, since the average fractionation index in flotation was low, the difference in average cell length between the accept and reject fractions was low (for more detailed information, see Paper IV). At the same time, however, a much higher fractionation index was achieved at three operational points, and these caused a high standard deviation for the whole dataset, as a consequence of which the small differences between the accept and reject fractions were not statistically significant. By contrast, nine parallel tests were performed at each operation point in the hydrocyclone fractionation experiment and the operational range was narrow, so that the dataset was homogeneous and the standard deviation small, with the consequence that small differences between the fractions were statistically significant.
Table 7. Fractionation indices for the average cell dimensions. P-values smaller than 0.05 denote statistical significance and show that the cell dimension concerned is higher in one or other of the fractions.

<table>
<thead>
<tr>
<th>Dimension</th>
<th>Fractionation method</th>
<th>FI range</th>
<th>Average FI</th>
<th>P-value</th>
<th>Higher in</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Arithmetic fibre length</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Perforated screen</td>
<td>0.56–0.64</td>
<td>0.61</td>
<td>0.010</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.12–0.27</td>
<td>0.22</td>
<td>0.000</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.00–0.03</td>
<td>0.01</td>
<td>0.152</td>
<td>NS -</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.00–0.23</td>
<td>0.10</td>
<td>0.112</td>
<td>NS -</td>
</tr>
<tr>
<td><strong>Length-weighted fibre length</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Perforated screen</td>
<td>0.52–0.57</td>
<td>0.55</td>
<td>0.003</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.08–0.27</td>
<td>0.18</td>
<td>0.000</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.00–0.03</td>
<td>0.02</td>
<td>0.021</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.04–0.16</td>
<td>0.08</td>
<td>0.687</td>
<td>NS -</td>
</tr>
<tr>
<td><strong>Width</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Perforated screen</td>
<td>0.09–0.10</td>
<td>0.09</td>
<td>0.002</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.01–0.08</td>
<td>0.05</td>
<td>0.001</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.06–0.09</td>
<td>0.08</td>
<td>0.000</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.00–0.05</td>
<td>0.03</td>
<td>0.009</td>
<td>S Reject</td>
</tr>
<tr>
<td><strong>Cell wall thickness</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Perforated screen</td>
<td>0.14–0.21</td>
<td>0.19</td>
<td>0.016</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.01–0.15</td>
<td>0.08</td>
<td>0.011</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.11–0.15</td>
<td>0.13</td>
<td>0.000</td>
<td>S Reject</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.01–0.08</td>
<td>0.06</td>
<td>0.002</td>
<td>S Reject</td>
</tr>
</tbody>
</table>

FI range: the range within which the fractionation index varied
Average FI: average fractionation index
S: the difference between the accept and reject fractions is significant at a confidence level of 95%
NS: the difference between the accept and reject fractions is not significant at a confidence level of 95%

All the methods were able to fractionate cells according to their width, but the fractionation index was low. In addition, fractionation in terms of cell wall thickness was observed, in the sense that the hydrocyclone method produced fractions having the same cell length but differing in cell wall thickness (see detailed data in Paper III). The average cell widths and cell wall thicknesses were higher in the reject fraction in all the methods used.
5.2 Behaviour of cells in fractionation

The fractionation tendencies of the cell types were examined using the categorization presented in section 4.3.1. The data presented in Papers II, III and IV are summarized in Table 8, which gives fractionation indices for the various cell categories and the significances of the results. A p-value of less than 0.05 shows that the content of the cell type concerned is higher in one or other of the fractions.

The pressure screen was the most efficient in producing enrichments of the longest and shortest cells to separate fractions, with long cells, both narrow (C₁) and wide (C₃), enriched in the reject fraction and the shortest cells (C₅) in the accept fraction. In addition, the perforated screen gave a better fractionation result than the slotted screen. The hydrocyclone achieved the best fractionation of wide cells (C₃ and C₄), which were enriched in the reject fraction, whereas the fractionation indices for fibrous cells (C₁ and C₂) and fines (C₅) were negligible, even though the differences between the fractions were statistically significant. Flotation was able to concentrate the long cells (C₁ and C₃) in the accept fraction and the short, wide cells (C₄) in the reject fraction, but the fines (C₅) content was similar in both fractions. Only the pressure screen equipped with a perforated screen was able to enrich short fibrous cells (C₂), but their fractionation index was low.
Table 8. Fractionation indices for the various cell categories. P-values smaller than 0.05 were considered to denote statistical significance and thus showed that the cell type concerned was enriched in one or other of the fractions.

<table>
<thead>
<tr>
<th>Category</th>
<th>Fractionation method</th>
<th>FI range</th>
<th>Average FI</th>
<th>P-value</th>
<th>Higher in</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1</td>
<td>Perforated screen</td>
<td>0.64–0.72</td>
<td>0.69</td>
<td>0.010 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.09–0.32</td>
<td>0.21</td>
<td>0.001 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.00–0.04</td>
<td>0.02</td>
<td>0.044 S</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.01–0.34</td>
<td>0.17</td>
<td>0.017 S</td>
<td>Accept</td>
</tr>
<tr>
<td>C2</td>
<td>Perforated screen</td>
<td>0.21–0.32</td>
<td>0.25</td>
<td>0.023 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.01–0.21</td>
<td>0.10</td>
<td>0.362 NS</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.01–0.06</td>
<td>0.03</td>
<td>0.026 S</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.01–0.09</td>
<td>0.05</td>
<td>0.866 NS</td>
<td>-</td>
</tr>
<tr>
<td>C3</td>
<td>Perforated screen</td>
<td>0.80–0.83</td>
<td>0.82</td>
<td>0.006 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.18–0.54</td>
<td>0.36</td>
<td>0.001 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.26–0.34</td>
<td>0.30</td>
<td>0.000 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.05–0.34</td>
<td>0.19</td>
<td>0.045 S</td>
<td>Accept</td>
</tr>
<tr>
<td>C4</td>
<td>Perforated screen</td>
<td>0.01–0.10</td>
<td>0.07</td>
<td>0.248 NS</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.01–0.11</td>
<td>0.07</td>
<td>0.305 NS</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.24–0.33</td>
<td>0.29</td>
<td>0.000 S</td>
<td>Reject</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.00–0.26</td>
<td>0.12</td>
<td>0.014 S</td>
<td>Reject</td>
</tr>
<tr>
<td>C5</td>
<td>Perforated screen</td>
<td>0.57–0.79</td>
<td>0.69</td>
<td>0.007 S</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Slotted screen</td>
<td>0.12–0.45</td>
<td>0.25</td>
<td>0.000 S</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Hydrocyclone</td>
<td>0.00–0.02</td>
<td>0.01</td>
<td>0.016 S</td>
<td>Accept</td>
</tr>
<tr>
<td></td>
<td>Flotation</td>
<td>0.01–0.18</td>
<td>0.08</td>
<td>0.057 NS</td>
<td>-</td>
</tr>
</tbody>
</table>

FI range: the range within which the fractionation index varied
Average FI: the average fractionation index
S: the difference between the accept and reject fractions is significant at a confidence level of 95%
NS: the difference between the accept and reject fractions is not significant at a confidence level of 95%

Removal efficiencies were calculated for the cell types using Equation 6, with the exception that the fractionation index was used instead of the screening quotient, since it was not possible to measure mass-based proportions for the cell categories. The removal efficiencies are presented in Figures 12–15 as a function of the total mass reject rate in fractionation.
Fig. 12. Removal efficiency of the various cells in pressure screening with a perforated screen. The lines in the figure represent varying degrees of fractionation selectivity.

Fig. 13. Removal efficiency of the various cells in pressure screening with a slotted screen. The four operation points with the most pronounced fractionation are indicated. The lines in the figure represent varying degrees of fractionation selectivity.
Fig. 14. Removal efficiency of the various cells in hydrocyclone fractionation. The lines in the figure represent varying degrees of fractionation selectivity.

Fig. 15. Removal efficiency of the various cells in flotation. The lines in the figure represent varying degrees of fractionation selectivity.
Pressure screening resulted in high removal efficiencies in the case of long (C₁ and C₃) and short (C₅) cells, whereas only wide cells (C₃ and C₄) had marked removal efficiencies in hydrocyclone fractionation. The major components fractionated in flotation were long cells, but removal efficiencies were universally poor.

Other supplementary analyses in addition to automatic optical analysis were also used to monitor cell type fractionation. The most important finding to arise from the standardized microscopic analysis was that vessel elements tended to be enriched in the hydrocyclone accept fraction (for more detailed information, see Paper I). In addition, standardized microscopic analysis showed that the hydrocyclone drove the parenchyma and epidermal cells into the reject fraction whereas epidermal cells were enriched in the accept fraction in pressure screening. The freeness values (see Papers III and IV) were found to be lower in the hydrocyclone and flotation accept fractions, indicating that flexible, thin-walled cells were enriched in these.

In addition to the trials presented in Papers I–IV, a complementary fractionation trial was performed using unbleached wheat straw pulp, after which Bauer McNett analysis was performed to determine the size fractionation of the cells (see Table 9). Here it was found that pressure screening with a perforated screen caused the long cells to be enriched highly selectively in the reject fraction and the shortest cells in the accept fraction, whereas the hydrocyclone did not fractionate the cells in terms of length to any marked extent. Furthermore, silicate content was measured in order to monitor the behaviour of epidermal cells in fractionation, yielding the fractionation selectivity values for silicates and silicate removal efficiencies presented in Table 10. This trial showed that epidermal cells were enriched in the reject fraction in hydrocyclone fractionation and flotation but in the accept fraction in pressure screening. Approximately 50% of the silicates were removed in a single-stage pressure screening or hydrocyclone fractionation. Epidermal cells were enriched in the reject fraction in flotation with good selectivity, but because of the low mass reject rate, the separation efficiency was low.
Table 9. Bauer Mc-Nett fractions of wheat straw pulp fractionated using a pressure screen and a hydrocyclone. All the experiments were single-stage fractionations using Pulp 2 (unbleached).

<table>
<thead>
<tr>
<th>Fraction</th>
<th>P&lt;sub&gt;48&lt;/sub&gt; [%]</th>
<th>Q</th>
<th>P&lt;sub&gt;48-100&lt;/sub&gt; [%]</th>
<th>Q</th>
<th>P&lt;sub&gt;100-200&lt;/sub&gt; [%]</th>
<th>Q</th>
<th>P&lt;sub&gt;200&lt;/sub&gt; [%]</th>
<th>Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>32.4</td>
<td>24.2</td>
<td>16.6</td>
<td>26.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pressure screen*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Accept</td>
<td>0.4</td>
<td>0.99</td>
<td>10.5</td>
<td>0.60</td>
<td>28.8</td>
<td>0.37</td>
<td>60.2</td>
<td>0.71</td>
</tr>
<tr>
<td>Reject</td>
<td>38.1</td>
<td></td>
<td>26.3</td>
<td>18.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrocyclone**</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Accept</td>
<td>28.3</td>
<td>0.15</td>
<td>23.6</td>
<td>0.10</td>
<td>19.8</td>
<td>0.20</td>
<td>28.3</td>
<td>0.13</td>
</tr>
<tr>
<td>Reject</td>
<td>33.4</td>
<td></td>
<td>26.2</td>
<td>15.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flotation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The pulp on the 28 and 48 mesh screens was combined due to the small amount of P<sub>28</sub>

* Perforated screen, feed consistency 0.4%, passage ratio 0.28, RR<sub>v</sub> 64%, RR<sub>r</sub> 21%
** Feed consistency 0.2%, pressure difference 3.3 bar, RR<sub>v</sub> 32%, RR<sub>r</sub> 11%

Table 10. Enrichment of silicates in fractionation. All the experiments were single-stage fractionations using Pulp 2 (unbleached).

<table>
<thead>
<tr>
<th>Fraction</th>
<th>RR&lt;sub&gt;v&lt;/sub&gt; [%]</th>
<th>RR&lt;sub&gt;r&lt;/sub&gt; [%]</th>
<th>Q</th>
<th>Er</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure screening*</td>
<td>21</td>
<td>64</td>
<td>0.51</td>
<td>45</td>
</tr>
<tr>
<td>Hydrocyclone fractionation**</td>
<td>11</td>
<td>32</td>
<td>0.62</td>
<td>48</td>
</tr>
<tr>
<td>Flotation***</td>
<td>4</td>
<td>12</td>
<td>0.54</td>
<td>17</td>
</tr>
</tbody>
</table>

* Perforated screen, feed consistency 0.4%, passage ratio 0.28
** Feed consistency 0.2%, pressure difference 3.3 bar
*** Feed consistency 0.3%, soap dosage 6 kg/t, water hardness 11 °dH
6 Discussion

This discussion is divided into two parts: first a short discussion on the characterization of wheat straw pulp cells, and second a discussion of the behaviour of wheat straw pulp cells in fractionation and the applicability of fractionation.

6.1 The analysis of cells in wheat straw pulp

One of the biggest challenges in straw utilization is the heterogeneity of straw and the variations in pulp composition and properties that this causes. The average dimensions used in the characterization of wood-based pulps do not give any information about the cell types contained in straw pulp nor about the properties of paper produced from wheat straw pulp, as was demonstrated in our recent study (Rousu et al. 2013a). It was therefore necessary to develop an advanced method for characterizing wheat straw pulp that was based on cell dimension analysis and would be fast, straightforward and easy to use. The method developed here showed good repeatability and a good correlation with a standardized cell type analysis. Also, since the cells in grasses are very much alike, the method should be applicable to other grasses, too.

The work showed the advantage of cell type categorization when studying the fractionation performance of different cell types, in that average dimensions are dictated by the major components in a pulp, so that they cannot be used for monitoring the fractionation behaviour of minor components. For example, the wide cell content of wheat straw pulp was low, which meant that thin cells had a major impact on the average cell width. In consequence, fractionation according to cell width as determined using average cell dimensions was poor. On the other hand, the fractionation of wide cells became evident when cells were divided into categories and the fractionation behaviour of wide cells was studied alone.

The automatic cell analyser was equipped with two cameras, one for cell length analysis and the other for cell width and cell wall thickness. The optical resolution in the length analysis was 10 µm, and as the shortest cells in the wheat straw pulp were of approximately this length, the resolution should be sufficient for the analysis of such a pulp. However, the smallest particles, covering only one pixel, are usually ruled out of the analysis in order to exclude the small air bubbles, and therefore, the shortest particle visible in the image analysis was of
the order of 20 µm in length and the smallest and narrowest cells were not included. This meant that the amount of fines was underestimated.

The cell categorization was based on analysis of the dimensions of the cells. In addition to morphology, the appearance of the cells could be used to distinguish certain types, e.g. short epidermal cells have undulating margins and vessel elements can be identified by detecting their pitted areas. The analyser would nevertheless have to be able to measure micron-scale details in order to detect these features.

Good optics and high resolution images would be of little use if the cells were of similar dimensions and had no characteristic features, as is the case with long epidermal cells with straight margins, which can be confused with other elongated cells. In that case, one possibility for enhancing the analysis would be staining, in which cells with a given surface chemistry would be stained in order to distinguish them. The staining procedure is time-consuming, however, and dyes are useless when it is black-and-white images that are to be analysed. It would be best to use a simple staining procedure and to equip the analyser with a colour camera.

6.2 Wheat straw pulp fractionation

6.2.1 The fractionation tendency of wheat straw pulp cells

Fractionation produced fractions with distinctive cell properties, not only with respect to their morphology, i.e. dimensions and cell wall thickness, but also regarding differences in surface chemistry.

The pressure screen method fractionated cells mainly in terms of length, although cell width also played a role in pressure screen fractionation, in that wide cells (C₃ and C₄) had lower chances of passing through the screen than narrow ones of the same length (C₁ and C₂). In addition, the average cell wall thickness was higher in the reject fraction, indicating that stiff cells had poorer ability to pass through the screen. Fractionation in terms of cell wall thickness is hard to verify in pressure screening, however, as in the case of wood-based pulps the typical assumption is that wall thickness increases with cell length (which is not always true, as in the case of spring wood and summer wood, for example). If it were true, this would imply that fractionation according to cell wall thickness was correlated with fractionation by cell length. In practise, cell wall thickness
was higher in the reject fraction in every category studied here (for more detailed information, see Paper II), which indicates that thick-walled cells really did have a higher tendency to be rejected, but a more detailed study of cell morphology should be performed to verify this result.

The fractionation of cells according to their morphology resulted in differential fractionation of the cell types in pressure screening, since the long cells in classes C1 and C3, including fibres and tracheids, vessel elements and long parenchyma cells, were enriched in the reject fraction, while the higher cell wall thickness in the reject fraction indicated that it also contained an enrichment of long epidermal cells. The shortest cells found in C5 consist mainly of the shortest parenchyma and epidermal cells, and these were the main components in the pressure screen accept fraction. The incidence of short, wide cells (C4, comprising parenchyma cells, epidermal cells and also short vessel elements) was similar in both fractions. The majority of epidermal cells and parenchyma cells are short ones, but the results show that pressure screening is unable to produce a fraction that is free of these cells due to the large variation in cell dimensions.

The hydrocyclone was able to produce fractions with similar average cell lengths but varying cell wall thicknesses and cell widths. The thick wall and the presence of silicate inside the cell makes epidermal cells heavy, which means that these cells have a high settling velocity, resulting in their enrichment in the hydrocyclone reject fraction. This was also true of thick-walled fibres, which is consistent with the spring wood/summer wood fractionation in the processing of softwood pulps. In addition to fractionation by cell wall thickness, it seems that the hydrocyclone method fractionates wheat straw pulp according to cell width, since the incidence of wide cells (C3 and C4) was higher in the reject fraction, presumably on account of the enrichment of parenchyma and epidermal cells and large cell plates in the reject fraction. Consistent with previous results obtained with hardwoods, the long, relatively narrow vessel elements found in wheat straw pulp had a tendency to be enriched in the hydrocyclone accept fraction. The fines (C5) content was similar in both hydrocyclone fractions, but the high silicate content of the reject fraction and low freeness of the accept fraction showed that the finest cells of all differed in nature between these fractions those enriched in the accept fraction being the shortest parenchyma cells, with a high specific surface area, whereas those in the reject fraction were epidermal cells (for more details, see Paper III).

Although flotation is not a size separation method as pressure screening and hydrocyclone are, long cells (i.e. classes C1 and C3, including fibres, vessel
elements and long parenchyma cells) had a higher tendency to remain in the accept fraction. As in hydrocyclone fractionation, the incidence of the shortest cells (C3) was the same in both fractions, but they differed in nature, epidermal cells being enriched especially in the reject fraction due to the hydrophobic cuticle covering them. Removal of these coarse cells reduced the silicate content and dewatering ability of the pulp but improved its strength properties (see Paper IV).

### 6.2.2 On fractionation selectivity and removal efficiency

The fractionation index and removal efficiency were used to assess fractionation. The advantage of the FI is that a value different from zero shows that the property or quantity concerned is higher in one or other of the fractions, i.e. it shows that fractionation has occurred. But what level of separation efficiency would be high enough to make fractionation economically feasible? In terms of size separation, a length difference between hardwood and softwood fibres (1–2 mm), results in a fractionation index of only 0.5 although the properties of the paper produced using hardwood and softwood are markedly different. Thus the fractionation index does not need to be extremely high in order to achieve marked differences between the fractions.

The use of removal efficiency for estimating the numbers of cells removed from the feed is not unambiguous, however, as the cell type categorization is based on numbers of cells whereas mass-based proportions should be used to calculate removal efficiency. As the FI was used here to calculate removal efficiency, the latter entails the assumption that all cells are of same weight, and thus the removal efficiencies presented here are somewhat approximate.

A single-stage fractionation achieved good selectivity and removal efficiency in many cases, with pressure screening showing the highest fractionation selectivity and thus emerging as the most promising fractionation method for use on an industrial scale. Its fractionation indices for the longest (C1 and C3) and shortest cells (C5) were good, and a simple fractionation system would suffice to provide enrichment of these cells in separate fractions. Taking into account the fact that optical analysis underestimated the amount of fines and the actual fractionation selectivity is higher than that given in the optical analysis, the fractionation tendency of these cells in pressure screening was good. In addition, the fractionation tendency of epidermal cells was good in all the methods studied.
Wide cells (C\textsubscript{3} and C\textsubscript{4}) performed well in hydrocyclone fractionation, but more advanced fractionation systems should be used for short, fibrous cells (C\textsubscript{2}).

It should be noted that wheat straw pulp is a multi-component system and none of the methods was able to produce a fraction consisting mainly of just one cell type. In pressure screening, for example, the accept fraction consisted of short cells, but these included fibres, vessel elements, parenchyma cells and epidermal cells. To produce a fraction consisting mainly of one cell type, further fractionation should be undertaken using other methods. This means in turn that the production of fractions consisting mainly of one cell type might not be economically feasible due to the high investment and processing costs involved in multi-stage fractionation systems. Instead, fractionation would be more profitably used for optimizing the pulp properties. The economics of fractionation is a case-specific issue that depends on the application and the added value achieved.

6.2.3 Applicability of fractionation in wheat straw pulp processing

Even though the production of a fraction consisting mainly of one cell type would be economically unprofitable, fractionation may still have other applications. The present work was focused on the techniques of wheat straw pulp fractionation, and we have discussed its applicability elsewhere. It is nevertheless possible on the basis of the findings and literature review presented here to outline some prospects for using fractionation in connection with materials of this type.

Fractionation by cell length showed the highest selectivity and would thus be the most promising alternative for a fractionation step. Long cells enriched by pressure screening could be used in pulp and paper making and the manufacture of biocomposites or construction materials, for example. Pressure screening could take place as a pretreatment in connection with refining, because a high fines content is known to disturb the refining of wheat straw pulp. Refining also creates fines, as unbroken cell plates break up, so that fractionation could also be used after refining, to reduce the amount of fines.

More sophisticated fractionation sequences could also be used, however, to enhance certain pulp properties. In our recent study (Rousu et al. 2013a) the combined use of first pressure screening and then hydrocyclone fractionation produced four wheat straw pulp fractions with unique cell compositions and it was possible to customize the pulp properties by mixing these fractions in different proportions. Long cell fractions rich in fibres and vessel elements made a major contribution to paper strength, the hydrocyclone accept fraction,
containing more slender fibres compared with the coarse ones in the reject fraction, had better tensile properties. The removal of short cells was beneficial for most paper strength properties, with only the Scott bond increased with increasing short cell content. In addition, the dewatering ability of the short cell fractions, especially the hydrocyclone accept fraction, was inferior, and thus pressure screening could be used to ease water removal from straw pulp. Based on these findings, pressure screening could find a place as a means of reducing the amount of fines, and hydrocyclone fractionation could be used to divide long cells according to their cell wall thickness. The bonding ability of the hydrocyclone reject fraction, containing thick-walled cells, could then be increased further through refining.

Removing the short cells from straw pulp is beneficial for processability and paper properties, but these fines should not be wasted. The applicability of the wheat straw pulp fines fraction as a strength enhancer in wood-based pulps has been demonstrated by Heijnesson-Hultén et al. (2012), for example, and previous studies have shown that husks, mainly consisting of short epidermal and parenchyma cells (Evert 2006), are a good raw material for use in the manufacture of adsorbents or cellulose fibres and nanocrystals, for example (Bhatnagar & Sillanpää 2010, Chen et al. 2011, Johar et al. 2012). We have previously demonstrated (Rousu et al. 2013b) that the fines fraction can be fractionated further to yield enhanced properties, as addition of the accept fraction rich in fines, i.e. short parenchyma cells and fibres, obtained from wheat straw pulp using a hydrocyclone in the present work was added to eucalyptus pulp it promoted the Scott bond, tensile properties and paper smoothness of the latter and had only a minor effect on water removal, whereas the reject fraction from the hydrocyclone, containing high numbers of epidermal cells, had only minor impact on the paper properties. In another study performed in our laboratory, Upola (2012) enriched fines from wheat straw pulp by pressure screening and used a hydrocyclone to produce two fractions with distinct cell compositions, one rich in parenchyma cells and the other in epidermal cells. These fractions were first microfibrillated by homogenization and then treated chemically with periodate and bisulphite (for a more detailed description of the chemical treatment, see Liimatainen et al. 2013) to yield sulphonated microcellulose, after which the adsorption of lead (Pb(II)) from water was studied. For this purpose the microcellulose produced from the hydrocyclone reject fraction had a better adsorption capacity. The difference in adsorption capacity is probably explained
by the different chemical compositions of the fractions. These examples show the potential applications of wheat straw pulp fines in various uses.

All the fractionation methods studied here were able to enrich cells containing silicate, i.e. epidermal cells, and a marked improvement in paper properties was seen in our flotation experiment when the epidermal cell content of the pulp was reduced (see Paper IV). The fraction rich in epidermal cells could be used to obtain various valuable co-products, since the silicates in plant tissues are generally in an amorphous form (Prichid et al. 2004) and are soluble under alkaline conditions (Iler 1979), so that the fraction rich in epidermal cells can be desilicated by a simple chemical treatment. Amorphous silica has various industrial applications. It can be used as a pigment, filler, abrasive, absorbent and catalyst support, for example (Patnaik 2003), and thus applications could be found for silicates derived from wheat straw, too. Alternatively, the solubility of silicates can be reduced and they can be bound to the pulp by means of an additional treatment with lime (Yılmaz 1995) or aluminium and magnesium oxides (Tutuş & Eroğlu 2003), whereupon they behave like fillers. In addition, the waxes covering the epidermal cells could be removed by supercritical carbon dioxide extraction, for example, and used in the manufacturing of cosmetics, personal care products, polishes and coatings, for example (Deswarte et al. 2006).

The advantage of using fractionation in these cases would lie in the reduced amount of pulp needing to be processed if the epidermal cells enriched in one fraction.

6.2.4 Comments on the operation parameters

The fractionation tendency of cells is affected by the equipment design, furnish and operational parameters. When a single pulp is fractionated in a given apparatus, as was done here, the operational parameters can be used to control pulp fractionation. The most important findings in this respect will be discussed briefly below.

The main operational parameters of interest in pressure screening are the slot flow velocity, feed consistency and volumetric reject rate (Jokinen 2007), all of which may be combined into a single parameter, the passage ratio. The present results showed the fractionation tendency of wheat straw pulp cells to be to a very considerable extent a function of the passage ratio (for more details, see Paper II), and it is this that should be used to control the pressure screening of wheat straw pulp.
In hydrocyclone fractionation it is the pressure difference between the feed and accept lines and the feed consistency that are the most important operational parameters (Jokinen 2007) and the economics of fractionation are closely related to the costs of pumping, since the formation of a high pressure difference requires a great amount of energy. Increasing the pressure difference had only a minor effect on the fractionation tendency of wheat straw pulp cells, however (see Paper III), and thus use of a low pressure will facilitate low operational costs. On the other hand, the low consistency used in hydrocyclone fractionation means that the pulp contains a large amount of water, which increases investment and processing costs.

The relevant operational parameters in flotation include the chemistry, temperature, pH and amount of air used, the bubble size and the mixing of the suspensions (Schabel 2010). Probably the most challenging task is to find the optimal flotation chemistry in order to maximize the separation efficiency. At least a surfactant has to be added to create powerful bubbles and froth, but this may be detrimental to fractionation selectivity, because surfactants and other chemicals also interact with, or may be deposited on, the cell surface. Calcium, for example, which is widely used in flotation, forms calcium soaps and bridges hydrophilic cells with air bubbles, causing them to float (Drabek et al. 1998, Ferguson 1992), and may thus be a source of fibre losses in flotation.

### 6.3 Suggestions for further research

The present work was focused on finding possibilities for fractionating wheat straw pulp cells, and the results showed that these cells can indeed be divided into fractions with distinct properties. Wheat straw is only one widely available agricultural residue, however, and even though grasses are alike and fractionation should be applicable to other cereals too, this should be verified before attempting to make more thorough use of agricultural residues.

Further research could be concentrated on increasing fractionation selectivity and enhancing its economic viability. One way of improving fractionation selectivity could be through changing the design parameters. In pressure screening this could be done by changing the screen geometry, *i.e.* by reducing the aperture dimensions (hole size or slot width) or by changing the profile height, slot width or wire geometry (Jokinen 2007, Julien Saint Amand & Perrin 1998, Niinimäki 1998, Sloane 1999, Wakelin & Corson 1997, Wakelin et al. 1994, Ämmälä 2001). In hydrocyclone fractionation, greater effectiveness could be
achieved by altering the hydrocyclone geometry, \textit{i.e.} the diameter or length of the cyclone (Jokinen 2007). In flotation, the design parameters are not so crucial, and it is the operation parameters that should be optimized first.

In addition to optimizing each unit process, the use of combinations of processes for wheat straw pulp fractionation could be investigated. For example, long cells do not participate in true flotation and may only disturb the flotation process, so that flotation could be tried for the pressure screen fines fraction alone, with the aim of separating the parenchyma cells from the epidermal cells.

Among the furnish parameters, the feed consistency has a considerable impact on the economics of pulp processing. We used a low feed consistency to find out whether it is possible to fractionate wheat straw pulp cells. Increasing the feed consistency would lower the fractionation efficiency in pressure screening and hydrocyclone fractionation (Jokinen 2007; Jones \textit{et al.} 1966; Ohsawa \textit{et al.} 1984), while reducing the feed consistency would increase the water processing, pumping and storage costs. It would therefore be interesting to explore the possibility of using a higher feed consistency in fractionation in order to render the process more economic.
7 Conclusions

Straw pulps produced using agricultural residues contain high numbers of cells with a variety of properties. Fractionation provides a tool for dividing pulp into streams with distinct cell compositions and properties.

A pressure screen is capable of fractionating short and long cells into separate streams. The long cells, comprising long fibres, vessel elements and parenchyma cells, become enriched in the pressure screen reject fraction in ultra-fine pressure screening, whereas the short parenchyma cells and epidermal cells are enriched in the reject fraction. The cells are fractionated mainly according to their length, but wide cells have a lower tendency to pass through the screen than narrow ones of same length. In addition, cell wall thickness is higher in the reject fraction. The results also show that the passage ratio is a good parameter for predicting the fractionation behaviour of wheat straw pulp cells in pressure screening.

In a hydrocyclone the cells are divided according to their width, the wide parenchyma cells and unbroken cell plates gravitating to the reject fraction, as also do the cells containing silicates, i.e. epidermal cells, whereas vessel elements are enriched in the accept fraction. The cells are also fractionated according to their cell wall thickness, as the hydrocyclone produces an accept fraction that is rich in thin-walled cells while the thick-walled cells and wide cells are enriched in the reject fraction.

In flotation performed using conventional soap chemistry, the epidermal cells are enriched in the reject fraction whereas the long cells, comprising fibres, vessel elements and parenchyma cells, tend to be enriched in the accept fraction.

Since the removal efficiencies of the various cell types were low in single-stage fractionation, multi-stage fractionation has to be used to achieve marked levels of enrichment. Considerable differences in pulp properties can be brought about with simple fractionation sequences, however, enabling optimization of the pulp properties. In addition, all the methods are able to produce a fraction that is rich in epidermal cells and can thus be used to reduce silicate deposition. The most promising fractionation method for use on an industrial scale is pressure screening, which demonstrated the highest selectivity and removal efficiency in one-step fractionation. The most promising application for hydrocyclone fractionation lies in the fractionating of cells according to cell wall thickness, thus to optimizing refining, but it is also possible to use a hydrocyclone for removing vessel elements from a pulp, thereby reducing picking.
References


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Original papers


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