# Fractionation of epidermal cells from wheat straw pulp by flotation

MIKKO KARJALAINEN, ARI ÄMMÄLÄ, PÄIVI ROUSU, AND JOUKO NIINIMÄKI

**ABSTRACT:** The wide availability of wheat straw makes it a promising raw material for various fiber products, but its heterogeneity makes it a challenging material to use. In addition to reducing paper properties, some cell types cause processing problems such as silicate deposition and poor dewatering. The aims of this work were to examine the fractionation tendency of wheat straw pulp cells in flotation and to determine the effect of fractionation on paper strength properties. A cell type categorization based on automatic optical fiber analysis was used to assess fractionation. The results showed that epidermal cells can be selectively enriched from unbleached wheat straw pulp by flotation and other short cells had a tendency to become enriched in the overflow fraction. The underflow fraction consisted mostly of fibrous material, as well as vessel elements and long parenchyma cells. Removal of the epidermal cells from pulp will reduce its silicate content and improve the strength properties of the paper.

***Application:*** Mills can use flotation to fractionate wheat straw pulp cells, thereby improving pulp processability and end use properties.

Wheat straw is an agricultural residue that constitutes one of the largest underused nonwood-fiber resources worldwide [1]. It has been long been used in papermaking and also has potential use in boards and construction materials or as a substitute for oil in products such as biofuels, plastics, and composites [1-8].

One challenge in the use of wheat straw is its heterogeneity. Wheat straw pulp contains a wide variety of cells [9,10], some of which have negative effects on pulp processability and paper properties. For example, the high fines content causes dewatering problems in pulp and paper production [6,11-15]. Fines include epidermal cells, a diverse group of cells covered with a waxy cuticle layer [16]. The hydrophobic waxy layer has low bonding ability and can cause picking in printing [17]. Another problematic property of epidermal cells is their high silicate concentration [16]. These silicates are dissolved in the alkaline processing stages and cause deposition problems in the pulping equipment [9,18].

Enhanced paper properties and better processability for wheat straw pulp could be achieved by fractionation; that is, division of the pulp into fractions that differ in their cell properties, cell length, or cell wall thickness. These fractions can be used in various paper grades to optimize paper quality or processed using different sequences, enabling processing cost savings [19]. Screens, wire washers, and hydrocyclones are typically used in pulp fractionation, but the use of froth flotation (or simply, flotation) to fractionate pulp constituents has also been studied. Flotation is a mechanical separation process used widely for the selective separation of hydrophobic and hydrophilic particles. Air is fed into a suspension and small hydrophobic particles become attached to the air bubble and rise to the surface of the flotation cell. The froth containing these hydrophobic particles is removed as an overflow fraction from the surface of the flotation cell, while the hydrophilic particles remain in the suspension and are removed as an underflow. In ink removal, flotation is efficient in the size range of a few micrometers up to a few hundred micrometers [20,21], but fibers can also float, causing fiber losses [22-24].

Flotation is a very complex process; it is even possible to find contradictory results concerning pulp fractionation by flotation in the literature. Schwinger and Hanecker [25], for instance, used various chemical and mechanical pulps but achieved flotation tendency only with thermomechanical pulp fibers. Other authors have found that both mechanical and chemical pulp fibers will float [22,26-28], although chemical pulp fibers have a higher tendency to do so [22,28]. In addition, it has been shown that flotation fractionates cells according to their length, with long fibers tending to become enriched in the froth at low consistencies but remaining in the underflow fraction at higher consistencies [22,26,27,29].

Flotation experiments have been performed previously using wood-based pulps, but there are no reports in which wheat straw pulp is used. On the basis of the theoretical background, flotation could be used as a fractionation method to classify small hydrophobic epidermal cells and anionic papermaking cells into separate fractions, thereby promoting pulp processability and improving paper properties. The fraction containing harmful cells could be used in other applications. We thus set out to investigate the possibility for using flotation to fractionate wheat straw pulp cells. Our aims were to assess whether it is possible to selectively fractionate wheat straw pulp cells into cell types by flotation and determine the effects of changing pulp composition on paper strength properties. Laboratory flotation cell and soap chemistry was used to fractionate unbleached wheat straw pulp delignified using a formic acid–based cooking method. This pulp was chosen because silicates and waxes are not dissolved in acidic cooking; the high silicate concentration and the hydrophobic nature of the epidermal cells remain after cooking [9]. A cell type categorization based on automatic optical fiber analyzer data was used to monitor the behavior of the various cell types in flotation.

## MATERIALS AND METHODS

### Materials

The wheat straw pulp used here was unbleached, never dried Finnish wheat straw (*Triticum aestivum L.*) pulp delignified with a formic acid-based cooking method. The washed and dewatered pulp was disintegrated in a vat pulper at a temperature of 20°C and a consistency of 1.5% for 6 min. Tap water was used for diluting the pulp at all stages in the processing.

### Methods

The wheat pulp was fractionated in a single-stage process using the University of Oulu pilot fractionation plant and flotation was performed using a Voith Delta 25 (Voith GmBH; Heidenheim, Germany) flotation unit with the batch size of 22 L. A commercial soap, Serfax MT90 (Stephenson Group; Leeds, UK), was used as an anionic surfactant. According to the manufacturer, Serfax MT90 is a mixture of fatty acid soaps. The standard dosage used in the experiments was 6 kg/ton; however, the effect of dosage on flotation was also studied by using dosages of 3 kg/ton and 9 kg/ton. The soap was dissolved in hot water and added to the flotation cell in solution. The manufacturer recommends a pH range of 8.5-9 for the soap, so pH was adjusted to 8.5 with sodium hydroxide. The solubility of silicates is low at that pH level when low temperatures are used [30]. A commercial soap, Fairy Original (Procter & Gamble; Weybridge, UK), was used as an additional frother (0.2 g/batch). According to the manufacturer, this soap is a mixture of surfactants. Calcium is used in soap chemistry to improve flotation [31,32], so calcium chloride was added to adjust the calcium concentration to 128 mg $Ca^{2+}$/L (equivalent of a German water hardness of 18°dH). The effect of calcium additions on flotation was also studied by using calcium concentrations of 28 mg $Ca^{2+}$/L (4°dH) and 78 mg $Ca^{2+}$/L (11°dH). Calcium chloride was dissolved in hot water and added to the flotation cell in solution. The feed consistency in the flotation was 3 g/L, and the effect of consistency on flotation was also studied by using consistencies of 6 g/L and 10 g/L. The flotation temperature was 24°C, the air flow was kept constant at 7.4 L/s, and the flotation time was 6 min. The following flotation procedure was used: pH was adjusted to 8.5, the chemicals were added and, after a 1-min conditioning time with mixing on, the air intake was opened and flotation was continued for 6 min. The design of the experiment is presented in **Table I**.

A fractionation index (FI), which is a comparison of an average fiber or pulp property X (e.g., the proportion of a given cell type) between the overflow (O) and underflow (U) fractions, was used to assess the enrichment of various cell types during flotation. An FI will vary in the range 0–1 if $X_O < X_U$; the closer it comes to 1, the more selective the fractionation. An FI can be also negative, however, if $X_U < X_O$, whereupon it varies in value from 0 to minus infinity. There is no linear correlation between positive and negative FI values. The advantages of an FI are that it is independent of the volumetric reject rate ($RR_v$, the total volumetric flow ratio between the overflow and feed streams) and the mass reject rate ($RR_m$, the solid material flow ratio between the overflow and feed streams) and that a value that differs from zero shows that cells are enriched into one or other of the fractions. The FI was calculated using Eq. (1) [33]:

$$FI(X) = 1 - \frac{X_O}{X_U} \tag{1}$$

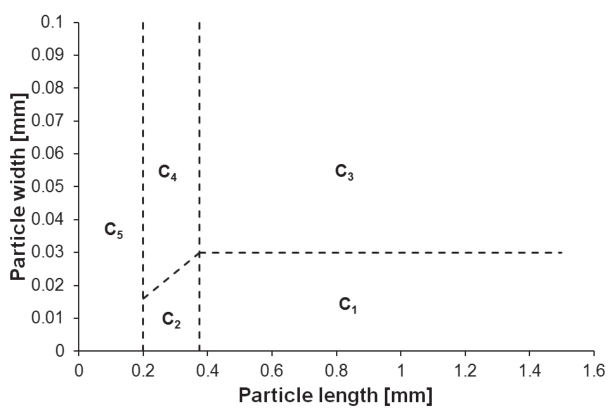
Removal efficiency, the percentage of the component *s* removed from the pulp, can be used to assess the fractionation result. This can be calculated using Eq. (2) [34]:

$$E_R = RR_m \cdot \left( \frac{s_O}{s_F} \right) \tag{2}$$

where *s* refers to the content of the component in the feed (F) and the overflow (O) fraction.

| Batch | Consistency, g/L | Soap Dosage, kg/ton | Water Hardness, °dH | pH | T, °C |
|---|---|---|---|---|---|
| 1 | 3 | 6 | 3 | 8.5 | 24 |
| 2 | 3 | 6 | 11 | 8.5 | 24 |
| 3 | 6 | 6 | 18 | 8.5 | 24 |
| 4 | 10 | 6 | 18 | 8.5 | 24 |
| 5 | 3 | 3 | 18 | 8.5 | 24 |
| 6 | 3 | 9 | 18 | 8.5 | 24 |
| 7 | 3 | 6 | 18 | 8.5 | 24 |

*I. Design of the experiment.*




***1. Cell type categorization.***

Samples were taken from the underflow, overflow, and feed, and consistency was analyzed using TAPPI T240 "Consistency (concentration) of pulp suspensions." Cell dimensions were analyzed using a FiberLab (Metso Automation; Helsinki, Finland) fiber analyzer. The samples were prepared according to TAPPI T271 "Fiber length of pulp and paper by automated polarized optical analyzer using polarized light," which recommends measuring 5000 cells in the analysis. The analysis involved preparing a sample of low consistency and photographing individual cells as they passed through a narrow capillary. Automatic image analysis was used to measure various cell properties (e.g., fiber length and width).

Cell dimension analysis data were used to categorize the cells according to their length and width to study the fractionation tendencies of the various cell types. A detailed description of the particle categorization procedure is presented elsewhere [35]. The wheat straw pulp cells were divided into five categories: (1) long, fibrous cells, including fibers and narrow vessel elements; (2) short, fibrous cells; (3) long, sac-like cells, including long vessel elements, parenchyma cells, and long cell bundles; (4) short, sac-like cells, including short vessel elements, parenchyma cells, epidermal cells, and short cell bundles; and (5) fines, consisting mostly of short parenchyma and epidermal cells. A schematic drawing of the categorization procedure is presented in **Fig. 1**, and the cell type proportions in the feed pulp, based on the number of cells, are presented in **Table II**. The error presented in Table II is calculated as the 95% confidence interval for a mean of 10 independent measurements.

| Cell Type | %, number |
|---|---|
| C1, Long fibrous cells | 26.8 ± 0.5% |
| C2, Short fibrous cells | 15.7 ± 0.4% |
| C3, Long sac-like cells | 2.4 ± 0.2% |
| C4, Short sac-like cells | 4.5 ± 0.1% |
| C5, Fines | 50.6 ± 0.4% |
| The error presented as 95% confidence interval for mean using ten replicates | |

***II. Cell types in the feed pulp.***

Laboratory handsheets were prepared from the feed pulp and the flotation underflow fraction. Because of the large amount of fines in wheat straw pulp, these sheets were prepared with a closed water system according to SCAN CM64 "Preparation of laboratory sheets for physical testing" and were conditioned according to TAPPI T402 "Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products." The following paper strength properties were tested using the respective standards: tensile properties (TAPPI T494 "Tensile properties of paper and paperboard [using constant rate of elongation apparatus]"), burst index (TAPPI T403 "Bursting strength of paper"), and tear index (TAPPI T414 "Internal tearing resistance of paper [Elmendorf-type method]"). In addition, CSF was measured from the feed and flotation underflow fraction according to ISO 5267-2 "Pulp—determination of drainability—part 2: 'Canadian Standard' freeness method." The silicate content of the samples was determined using TAPPI T244 cm-99 "Acid-insoluble ash in wood, pulp, paper, and paperboard."

## RESULTS

Average fiber lengths, cell width, and cell wall thickness (CWT); silicate content; and cell types in the U and O fractions were determined and fractionation indices were calculated for each test point. First, the effect of the operation parameters (i.e., pulp consistency, soap dosage, and water hardness) on the fractionation tendency was studied. The statistical significance of the variables studied was tested using MODDE 7.0 (Umetrics; Umeå, Sweden) software by fitting multiple linear regression models to the data. A 95% confidence level was used. A statistically sound model was not found and thus the effect of the operation parameters on the fractionation tendency of the wheat straw pulp constituents could not be shown with the design of experiments used. Consequently, batches were then studied as parallel test points to study whether fractionation occurred. SPSS 21.0 software (IBM; Armonk, NY, USA) with a 95% confidence level was used in the statistical testing of the fractionation. Data were first studied using the Kolmogorov-Smirnov test, which showed that the results are normally distributed in all cases; thus, the difference between the overflow and underflow fractions can be tested using a t-test. Next, a paired t-test was used in the statistical testing of means because the underflow and overflow are related; when a property in one fraction changes, the value in the other fraction changes too. Average cell dimensions, fractionation indices, and results from the paired t-test are presented in **Table III**. Cells fractionated according to fiber width and cell wall thickness but did not fractionate according to fiber length. Average cell width and cell wall thickness

| Dimension | F | U | O | FI | Significance | |
|---|---|---|---|---|---|---|
| L(n), mm | 0.30 | 0.31 | 0.28 | 0.08 | 0.112 | NS |
| L(l), mm | 0.65 | 0.66 | 0.65 | 0.02 | 0.687 | NS |
| Width, µm | 17.5 | 17.1 | 17.6 | 0.03 | 0.009 | S |
| CWT, µm | 4.3 | 4.2 | 4.5 | 0.06 | 0.002 | S |

L(n): average arithmetic fiber length; L(l): average length weighted fiber length; CWT: cell wall thickness; S: the difference between the overflow and underflow is significant at a confidence level of 95%; NS: the difference between the overflow and underflow is not significant at a confidence level of 95%; F: feed; U: underflow; O: overflow; FI: fractionation index.

***III. Average cell dimensions in feed (F) and in the underflow (U) and overflow (O) fractions.***

| Cell Type | F | U | O | FI | Significance | |
|---|---|---|---|---|---|---|
| C1, % | 26.8 | 26.8 | 22.2 | 0.17 | 0.017 | S |
| C2, % | 15.7 | 14.6 | 14.6 | 0.00 | 0.866 | NS |
| C3, % | 2.4 | 2.3 | 1.9 | 0.15 | 0.045 | S |
| C4, % | 4.5 | 4.1 | 4.7 | 0.13 | 0.014 | S |
| C5, % | 50.6 | 52.2 | 56.7 | 0.08 | 0.057 | NS |

S: the difference between the overflow and underflow is significant at a confidence level of 95%; NS: the difference between the overflow and underflow is not significant at a confidence level of 95%; F: feed; U: underflow; O: overflow; FI: fractionation index.

***IV. Cell types in feed (F) and in the underflow (U) and overflow (O) fractions. The proportion of cells is based on the number of cells.***

| F, % | U, % | O, % | FI | Significance | | $E_R$, % |
|---|---|---|---|---|---|---|
| 5.0 | 3.5 | 8.0 | 0.56 | 0.000 | S | 14 |

S: the difference between the overflow and underflow is significant at a confidence level of 95%; F: feed; U: underflow; O: overflow; FI: fractionation index.

***V. The content of silicates in feed (F) and in the underflow (U) and overflow (O) fractions, silicate fractionation index (FI), and removal efficiency for silicates.***

were higher in the flotation overflow fraction. The average mass reject rate in the flotation was 9% whereas the volumetric reject rate was 3%.

**Table IV** shows the results from cell type analysis. Long cells in $C_1$ and $C_3$ (i.e., long fibers and long sac-like cells) enriched to the U fraction whereas the short sac-like cells in $C_4$ had a higher tendency to enrich to the O fraction. The content of the finest cells in $C_5$ was higher in the O fraction but the difference was not significant at a 95% confidence level. The content of short fibrous cells ($C_2$) was similar in both the U and O fractions. The results also show that a small amount of cell agglomerates located in $C_2$ and $C_3$ was broken down during flotation; compared with feed pulp, the flotation O and U fractions had higher proportions of fines.

The average silicate content in the U and O fractions, silicate FI, and the removal efficiency of silicates are presented in **Table V**. The silicate content was higher in the O fraction and the FI was good. However, the removal efficiency was low, and only 14% of the silicates were removed on average. In addition, the mass balance of the silicate was calculated. On average, 14% of silicates enriched to the O fraction, 63% remained in the U fraction, and 23% dissolved during flotation.

Normalized paper properties (unfractionated feed = 1) for paper sheets prepared from the flotation U fractions are presented in **Table VI**. A t-test was used to determine the significance of the result. Fractionation improved most of the paper strength properties, yielding improvement of 16% in the tensile index and tensile energy adsorption index and a marked improvement in the tensile stiffness index. The burst index and breaking length increased slightly, but the tensile strain and tear index were at the same level as for the feed. CSF was lower in the U fraction than that in the feed pulp.

| Property | Normalized Value | Significance | |
|---|---|---|---|
| Freeness | 0.90 | 0.006 | S |
| Density | 1.04 | 0.001 | S |
| Bulk | 0.96 | 0.001 | S |
| Tear index | 1.01 | 0.399 | NS |
| Tensile index | 1.16 | 0.000 | S |
| Tensile strain | 1.00 | 0.857 | NS |
| Tensile energy absorption index | 1.16 | 0.002 | S |
| Tensile stiffness index | 1.11 | 0.000 | S |
| Breaking length | 1.06 | 0.003 | S |
| Burst index | 1.05 | 0.015 | S |
| S: the difference between the feed and the underflow is significant at a confidence level of 95%; NS: the difference between the feed and the underflow is not significant at a confidence level of 95%. | | | |

***VI. Normalized paper properties for the underflow fraction. Unfractionated feed = 1.***

## DISCUSSION

Flotation produced two fractions with distinct cell compositions. The U fraction was rich in long cells (i.e., fibers, vessel elements, and parenchyma cells) whereas short cells, rich in silicates, were enriched in the O fraction. The high silicate content in the O fraction showed that epidermal cells in particular were enriched. The evidence supporting this is the fact that the CSF in the U fraction was lower than the CSF in the feed pulp; thus, coarse material was removed in flotation. The low CSF of wheat straw pulp is caused by the fines fraction [11], which consists of thin-walled parenchyma cells with a high specific surface area, and coarse, thick-walled epidermal cells with low specific surface area. Because CSF is inversely related to specific surface area in chemical pulp [36,37], as demonstrated previously, low CSF in the U fraction indicates that the short parenchyma cells tended to remain in the suspension whereas the epidermal cells were enriched in the O fraction.

In addition to enhancing paper strength properties and reducing the silicate deposition problem, the selective removal of epidermal cells by flotation could be beneficial for printing as it would reduce picking and fiber rising problems, given the suggestion that epidermal cells with a hydrophobic surface have low bonding ability and cause picking in printing [17]. Support for this observation can be found in the results of Sood et al. [38], which show that picking was reduced when a fines fraction rich in epidermal cells was removed by screening and then placed in the middle layer of a triple-layered sheet. This also served to reduce fiber rising. Picking is connected with vessel elements, however [39], and it has been suggested that rising may be caused by coarse cells [40]. Therefore, picking and rising problems may well be reduced by the selective removal of epidermal cells but not necessarily eliminated.

Even though statistically significant effects were not found, it is likely that operational parameters have effect on the results but those are too complicated to analyze with the design of experiments used. For example, previous studies have suggested that fibers float in dilute suspensions by virtue of entrainment, whereas flocculation prevents them from floating when the consistency is increased [22,26,27,29,41]. Therefore, to prevent fiber floating, high consistency is recommended. In addition, flotation chemistry is complex. Clean water does not foam, for example, and surface active chemicals (surfactants) are needed to generate air bubbles and froth with the properties needed in flotation. A surfactant may have a negative effect on flotation selectivity, in that it can adsorb to the cell surfaces and render them hydrophobic, thus causing originally hydrophilic cells to adhere to bubbles and float [23]. The surfactant dosage also has an effect on both froth stability [23] and bubble size [27], which in turn affect the behavior of the cells in flotation [22,24,27]. In addition, it is reported that calcium used as a collector in flotation forms sticky precipitates and thus causes negatively charged cells to float and is a major contributor to stock loss in flotation [31,32]. Soap chemistry was not the optimal one for wheat straw pulp fractionation due to alkaline conditions that cause silicate dissolution, as was seen in the study. Soap chemistry used in this study is only one option in flotation, however, and a wide variety of surfactants can be found [31]. This was the first attempt to use flotation in wheat straw pulp fractionation and we intend to concentrate in subsequent work on the optimization of the flotation chemistry.

A marked improvement in paper strength properties were achieved even though a small proportion of the pulp was removed. The silicate removal efficiency was low, however, and fiber losses decrease the economic profitability of the flotation. Therefore, optimization and probably multistage fractionation are needed to make flotation economically feasible on an industrial scale. However, the purpose of the fractionation is to use both fractions in some application and small fiber losses are acceptable. One possibility for taking advantage of an overflow fraction containing epidermal cells rich in silicates and waxy cuticle could lie in using the fraction for the manufacture of co-products. Waxes account for approximately 1% of the dry weight of wheat straw [42] and silicates for 5%; therefore, the overflow fraction from flotation is a valuable raw material for wax and silicate products in mills that use hundreds of thousands of tons of straw annually. A desilicated and dewaxed overflow fraction could be combined with the underflow fraction or used as a raw material for other fiber products or in biofuel production, for example.

## CONCLUSIONS

Flotation was found to be capable of fractionating cells from a wheat straw pulp suspension. The overflow fraction was rich in short cells such as short parenchyma cells, epidermal cells, and epidermal cell plates, while the underflow fraction was rich in long, fibrous cells and long, sac-like cells (i.e., long, wide vessel elements and parenchyma cells). Flotation was not able to fractionate short fibrous cells. Compared with the underflow fraction, the cells found in the overflow fraction had higher silicate content. Even though only a small amount of pulp was removed, a marked improvement in the strength properties of the unbleached wheat straw pulp was achieved. **TJ**

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Our interest in this work was to find out whether it is possible to optimize wheat straw pulp composition by fractionation. Previous straw pulp fractionation studies conducted by others have been performed using laboratory scale screening equipment, such as Bauer McNett, for example. Our previous studies have demonstrated the feasibility of pressure screening and hydrocyclone in the fractionation of wheat straw pulp in an industrial scale. There are no previous studies of using flotation for this purpose.

Flotation is a complex unit process where chemistry plays a huge role. A large amount of chemicals are used, and thus the first jump into the empty map was a sophisticated guess. We used the chemistry widely used in pulp and papermaking. We thought it was fascinating to see the difference between fractions and have the first indications that flotation works in this application.

For mills, the benefits of fractionation are better pulp processability and paper properties. In addition, there is a possibility to use overflow fraction in other applications. Our next step is to discover other chemistries and thus optimize fractionation and removal efficiency.

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Leveraging Forest Biomass for Next Generation Fuels and Chemicals

## Value Added Products from Biomass

2015 International Bioenergy and Bioproducts Conference is focused on leveraging assets and feedstocks from within the pulp and paper industry. IBBC is scheduled for October 28-30 in Atlanta, Georgia. At IBBC you will hear the latest updates on:

- **Supply and Demand of Woody and Non-woody Biomass**
- **Collecting, Harvesting and Preparation of Biomass**
- **Torrefaction and Wood Pellets**
- **Thermochemical and Biochemical Conversion Pathways**
- **Production of Biochemicals from Biomass**
- **Pilot Plant/Demonstration Plant Case Studies**
- **Policies & Initiatives for Advancing Biorefinery Projects**

TAPPI 100 YEARS A CENTURY OF ACHIEVEMENT

Co-Located with: PEERS October 25-28, 2015 ATLANTA, GEORGIA

*Learn more and register at:* **www.tappi.org/15IBBC**