Microfiltration of gluten processing streams from corn wet milling

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Abstract

In corn wet milling, dry matter can be separated from liquids in process streams with centrifuges or vacuum belt filtration (VBF). Because separations usually are not complete, dry matter can be lost in the liquid streams (overflow from the gluten thickener centrifuge and filtrate from VBF). This represents a loss of nutrients, especially protein, to low valued coproducts and reduces quality of water for recycling within the process. The objective was to compare microfiltration of light and heavy gluten process streams to conventional separation methods. Batches of light and heavy gluten were obtained from a wet mill plant and processed by microfiltration. Samples of permeate and concentrate from microfiltration were analyzed and compared to corresponding streams from wet milling. Microfiltration of light gluten resulted in concentrate and permeate streams similar in composition to conventionally processed light gluten using a centrifuge, suggesting that microfiltration is as effective as centrifugation in partitioning solids and water in light gluten. Dewatering of heavy gluten found that conventional VBF caused dry matter concentrations in gluten cake to be higher than concentrate from microfiltration. Permeate from microfiltration of heavy gluten had higher concentrations of ash and lower soluble nitrogen than filtrate from VBF. Microfiltration was able to remove more ash from concentrate, which may improve the value of wet milling coproducts. These data demonstrated microfiltration has potential for separation of light and heavy gluten streams, but more data are needed on effectiveness and practicality.

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1. Introduction

Wet milling is a major technology for processing corn. In wet milling, corn is steeped for 24–36 h, germ and fiber are removed, and the resulting slurry is separated into two streams (Blanchard, 1999; Johnson and May, 2003). One stream contains highly concentrated starch. The second stream (light gluten) is dilute (2–6% dry matter) and consists mainly of proteins. Light gluten is separated by centrifugation (gluten thickener) into a heavy gluten stream (12–17% dry matter) and an overflow stream (defined as “overflow”, 2–3% dry matter). Heavy gluten is separated with a vacuum belt filter (VBF) into gluten cake and filtrate (defined as “filtrate”). Gluten cake is dried to form corn gluten meal, a high protein (67% db) coproduct used in animal diets (Blanchard, 1999).

These two separation steps (centrifugation and VBF) are not 100% effective in recovery of dry matter and protein, and considerable amounts are in the overflow from the gluten thickener centrifuge and filtrate from VBF,
which are recycled in the wet milling process. Rausch et al. (2002) reported that about 40% of protein in light gluten was found in the overflow and that about 10% of the protein in heavy gluten was found in filtrate. More effective separation would increase recovery of protein (which is marketable as animal feed) and improve quality of process water. Also, water removal using current technology is costly; alternative approaches could have significant economic impacts.

Technological advances in membrane design, such as use of stainless steel and ceramic materials in construction, have resulted in filtration systems that are more cost effective and more efficient. However, limited data have been reported on the effectiveness of membrane filtration systems to process gluten streams. Singh et al. (1998) found that a laboratory scale microfiltration system could increase dry matter content of a light gluten stream from 5.2 to 9.2 g/100 g; they recovered 20% more soluble material in the concentrate. Recently, we reported when light gluten is processed in wet milling, elements were concentrated in the resulting liquid streams (overflow and filtrate), while proteins and other organic materials were concentrated in the corresponding solid streams (heavy gluten and gluten cake, Rausch et al., 2003). Data are needed to evaluate the effectiveness of microfiltration to process gluten processing streams. The objectives were to: (1) determine effectiveness of microfiltration systems to process light and heavy gluten streams, and (2) compare characteristics of microfiltration streams to corresponding wet milling streams.

2. Methods

2.1. Description of membranes and equipment

Two membranes systems were compared. Membrane modules with nominal length of 1.50 m (0.35 m² membrane area, model 2.5-750A-5P, Graver Technologies, Glasgow, DE) or 3.05 m (0.70 m² membrane area, model 2.5-750A-10P, Graver Technologies, Glasgow, DE). The first membrane system (MF1) used a single 1.50 m stainless steel tubular membrane module with a membrane area of 0.35 m². The second membrane system (MF2) used two 0.70 m² and one 0.35 m² membrane modules in series, for a total membrane area of 1.75 m². Each membrane module had tube and shell configurations containing four tubes; each tube had an internal diameter of 1.90 cm, and a nominal pore size of 0.1 μm. All modules were constructed using the same porous stainless steel material.

Each system (MF1 and MF2) included a batch tank, pump, heat exchanger and membrane modules; these were configured in a loop to concentrate test materials (Fig. 1). A rotary lobe pump (P0399155, Waukesha Pumps, Waukesha, WI) was connected to a 7.5 Hp electrical motor and a digital variable frequency drive (AF-300 P11, Fuji Electric, Yokosuka City, Japan) to maintain 280–300 L/min and a crossflow velocity within the membrane module of 4.5 m/s in the loop, according to membrane manufacturer’s recommendations. Membranes were configured so that material was pumped through the tube interior and permeate passed through to the outside of the tubes and collected. Preliminary testing over a range of transmembrane pressures (TMP) prior to filtration tests indicated that a TMP of 200 kPa provided permeate flux rates that were sufficient for subsequent filtration experiments. For MF1, the batch tank was 200 L (nominal capacity). For MF2, the batch tank was 400 L to allow larger volumes of test materials and higher concentration of dry matter when filtering.

2.2. Experiment 1. Comparison of membranes

There were no published data available on membrane processing of gluten streams. The goal of this experiment was to obtain basic performance data, such as permeate flux rate, dry matter separation, dry matter concentrations and membrane fouling for MF1 and MF2. Batches of light gluten (~400 L) were obtained from the light gluten storage tank in a commercial wet milling plant. These were stored at 4–6 °C while experiments were being carried out. MF1 was evaluated first, followed by MF2. Microfiltration using MF1 lasted 9–10 h; microfiltration using MF2 lasted 2–3 h. For each replicate, light gluten was added to the batch tank, and pumping and filtration started. Permeate flux rate was determined by measuring the volume of permeate generated over a 1 min period, then calculating permeate flux rate by dividing volumetric flow rate (L/h) by membrane area of 0.35 and 1.75 m², for MF1 and MF2, respectively, to determine L/m²h (LMH). Each microfiltration evaluation was done twice; for each replicate, 1 L samples of permeate and concentrate were collected at 3–5 time intervals during filtration and analyzed in duplicate for dry matter based on change in permeate flux rate during filtration of each batch. As
microfiltration progressed, permeate was accumulated and concentrate was recycled to the batch tank. During each filtration test, light gluten temperature was allowed to increase from storage conditions (2–6 °C) by recirculating gluten until it reached 37–46 °C, similar to temperatures used in wet milling during gluten filtration. Permeate flux rates were initially high (80–140 LMH) and then quickly decreased in the early stages of the experiment to steady flux rates. Because permeate flux values were high and often decreased rapidly during this initial period, data obtained during the first 2 h of filtration were not used to calculate average permeate flux rates. Effects of treatments (MF1 and MF2) on flux parameters (LMH, initial and final dry matter of concentrate) were determined using a general linear model (SAS, 1985). Means were compared as least squares means when treatment effects were significant.

2.3. Experiment 2. Microfiltration of light gluten

Data from Experiment 1 indicated that performance characteristics (permeate flux rate, final dry matter in concentrate) of MF2 exceeded those of MF1; larger membrane area of MF2 resulted in shorter testing periods as well as higher permeate flux rates. Therefore, MF2 was used in Experiments 2 and 3 for processing of light and heavy gluten, respectively.

The goals of Experiment 2 were (1) to determine effectiveness of microfiltration to partition light gluten and (2) to compare composition of microfiltration streams (concentrate and permeate) to corresponding streams from wet milling (heavy gluten and overflow, respectively). Four batches (~400 L each) of light gluten were obtained from a commercial corn wet milling plant and transported to the UIUC campus. At the time each batch of light gluten was obtained, corresponding subsamples (~1 L) of overflow and heavy gluten were taken for later analyses. Each batch of light gluten was filtered using MF2 and used transmembrane pressure of 200 kPa, flow rate of 280–300 L/min, crossflow velocity of 4.5 m/s and temperatures of 37–46 °C. During each microfiltration, 1 L samples of permeate and concentrate were collected every 30–45 min of filtration and refrigerated. Microfiltration was stopped when material in the batch tank was depleted or became too viscous for pumping. Permeate, concentrate and original samples of light gluten, heavy gluten and overflow taken at the plant were sent under ice to the University of Missouri for analyses. These included dry matter, total N (TN), soluble N (SN), ash and total suspended solids (TSS). Dry matter was determined as weight loss overnight at 105 °C (AOAC, 1984). TN was determined by thermal conductivity (Leco, 1994). Ash was determined by heating over night at 550 °C (AOAC, 1984). SN was determined by filtering a sample through a 0.45 μm microdisk and measuring N in the filtrate. Ash and TN content were corrected to a dry basis. TSS was determined as proportion of dried solids retained on fiberglass filter paper having 1.2 μm openings. Permeate flux rates were measured every 30 min as described in Experiment 1. Average permeate flux rates were calculated using flux rates observed after 30 min of initial filtration. Analysis of variance was determined using a general linear model (SAS, 1985) for a simple block design. Means were compared as least squares means when main effects were significant.

2.4. Experiment 3. Microfiltration of heavy gluten

The goals of this experiment were to (1) determine capability of microfiltration to partition components in heavy gluten and (2) compare concentrate and permeate from microfiltration of heavy gluten to corresponding wet milling streams (gluten cake and filtrate, respectively). Three batches of heavy gluten (~400 L) were obtained from a wet milling plant. During collection of each batch, corresponding samples of gluten cake and filtrate also were obtained for analyses. Microfiltration was carried out using MF2; conditions, procedures, sampling and analyses were similar to those described in Experiment 2. Data were analyzed using a block design and a SAS (1985) general linear model; means were compared as least squares means when treatment effects were significant.

2.5. Cleaning of membranes

After filtration of each batch, membrane systems were cleaned to above 80% of manufacturer’s clean water flux rate prior to additional filtration. Warm tap water (200 L, 50–65 °C) was flushed through the systems. Sodium hydroxide (2–4% NaOH w/w) was added to hot water (93 °C) to raise pH to 12.5. Sodium hypochlorite (NaOCl) also was added at 500 ppm (as OCl⁻) to aid protein removal. Cleaning solution was recirculated for 45–60 min at a TMP of 200 kPa. The system was flushed with warm tap water (300 L, 50–65 °C). Permeate flux rate was measured with tap water (30 °C, 130 kPa TMP) to measure clean water flux rate; if flux rate was above 80% of the clean water flux rate prior to filtration, cleaning was considered completed.

3. Results and discussion

3.1. Experiment 1. Comparison of membranes

Performance data indicated that MF2 was more effective than MF1 in filtration of light gluten in terms of having a higher average permeate flux rate (65.3 vs 51.7 LMH, Table 1). Average permeate flux rate for MF2 could be due to variation in initial condition of
the membrane or cleanliness of the membrane. There were no differences detected between the two membranes in initial or final dry matter concentrations. There was a large amount of variability in dry matter concentrations among batches of light gluten (Experiments 1 and 2) and heavy gluten (Experiment 3).

During each experiment, initial composition of light and heavy gluten had a relatively large amount of variability. For example, light gluten varied from 2.9% to 4.94% dry matter (Tables 2 and 3, respectively). These variations were within the random variation in composition previously observed (Rausch et al., 2003). Representative flux characteristics are shown in Fig. 2 for LG and HG processing streams. Overall means of permeate flux rate were 65.3 and 51.3 LMH for LG and HG, respectively. Due to the variability observed in stream composition and filtration characteristics, more testing would be needed to determine filtration parameters for larger scale microfiltration.

### 3.2. Experiment 2. Microfiltration of light gluten

The gluten thickener centrifuge at the wet mill plant increased dry matter in light gluten (Table 2), which

<table>
<thead>
<tr>
<th>System</th>
<th>Permeate flux rate (LMH)</th>
<th>Initial DM (%)</th>
<th>Final DM (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MF1</td>
<td>51.7^a</td>
<td>2.90</td>
<td>12.35</td>
</tr>
<tr>
<td>MF2</td>
<td>65.3^a</td>
<td>5.25</td>
<td>15.35</td>
</tr>
<tr>
<td>SE</td>
<td>3.54</td>
<td>1.53</td>
<td>1.39</td>
</tr>
</tbody>
</table>

^ab Means differ at P < 0.01. Only flux had significant effect.

1 DM: dry matter.

* LMH: liters per m² membrane area per hour.

<table>
<thead>
<tr>
<th>Comparison</th>
<th>DM</th>
<th>TN</th>
<th>SN</th>
<th>Ash</th>
<th>TSS</th>
</tr>
</thead>
</table>
| Separation method
| Gluten thickener centrifuge | LG  | 4.94^b | 10.50^a | 5.53^b | 3.83 | 3.97^b |
|                         | HG  | 14.24^a | 11.40^a | 1.40^a | 2.54 | 10.32^a |
| Overflow              | 2.44^a | 8.87^b | 7.51^a | 6.97 | 1.12^b |
| SE**                  | 1.02 | 0.40 | 0.71 | 1.59 | 1.75 |
| Microfiltration (65.3 LMH average permeate flux rate)
| LG          | 3.65^b | 9.99 | 5.45^b | 5.34^b | 2.43^b |
| Concentrate | 14.70^a | 11.32 | 1.35^b | 2.21^b | 12.78^a |
| Permeate           | 1.98^b | 10.88 | 8.46^a | 16.27^a | 0.54^b |
| SE                  | 0.79 | 1.55 | 1.03 | 1.05 | 0.74 |

Comparison of streams

<table>
<thead>
<tr>
<th>Comparison</th>
<th>DM</th>
<th>TN</th>
<th>SN</th>
<th>Ash</th>
<th>TSS</th>
</tr>
</thead>
<tbody>
<tr>
<td>HG vs concentrate</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>HG</td>
<td>13.14</td>
<td>11.41</td>
<td>1.52</td>
<td>1.44</td>
<td>11.92</td>
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<tr>
<td>Concentrate</td>
<td>14.70</td>
<td>11.32</td>
<td>1.35</td>
<td>2.21</td>
<td>12.78</td>
</tr>
<tr>
<td>SE</td>
<td>2.30</td>
<td>0.14</td>
<td>0.40</td>
<td>0.32</td>
<td>2.13</td>
</tr>
<tr>
<td>Overflow vs permeate</td>
<td></td>
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<tr>
<td>Overflow</td>
<td>2.08</td>
<td>9.15</td>
<td>6.80</td>
<td>11.03</td>
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<tr>
<td>Permeate</td>
<td>1.98</td>
<td>10.88</td>
<td>8.46</td>
<td>16.27</td>
<td>0.54^b</td>
</tr>
<tr>
<td>SE</td>
<td>0.63</td>
<td>2.19</td>
<td>1.57</td>
<td>1.72</td>
<td>0.05^b</td>
</tr>
</tbody>
</table>

^abc Means within same column and effect having unlike letters differ (P < 0.01).  
1 LG: light gluten, HG: heavy gluten, Overflow: supernatant stream from gluten thickener.

2 Gluten thickener centrifuge located at a wet milling facility.

3 DM (dry matter) and TSS (total suspended solids): g/100 g sample; all others: g/100 g dry basis.

** SE: standard error.
was relatively similar to composition we reported in a previous study (Rausch et al., 2003). Dry matter content of the heavy gluten stream was greater than for the overflow stream (14.24 vs 2.44 g/100 g, respectively) and greater than the original light gluten stream (4.94 g/100 g). TN concentrations of heavy gluten were higher than in overflow (11.40 vs 8.87 g/100 g, respectively). Concentrations of SN decreased in heavy gluten and increased in overflow relative to the concentration in light gluten. Ash concentrations of the three streams (light gluten, heavy gluten, overflow) were not different. Concentrations of TSS were higher in heavy gluten.

The average permeate flux rate for microfiltration of light gluten was 65.3 LMH (Tables 1 and 2), indicating material that was filtered relatively easily. Microfiltration of light gluten produced two streams, concentrate and permeate, which are analogous to conventionally produced streams of heavy gluten and overflow, respectively (Table 2). Microfiltration affected dry matter partitioning similar to that of the gluten thickener; dry matter of concentrate was greater than permeate (14.70 vs 1.98 g/100 g) and greater than for original light gluten (3.65 g/100 g). TN concentrations were not different among streams (light gluten, concentrate, permeate) but SN had higher concentrations in permeate. Microfiltration increased the concentration of ash in the permeate; ash content of permeate was higher than concentrate (16.27 vs 2.21 g/100 g, respectively). The two concentrated streams (heavy gluten vs concentrate) were not different in composition; likewise, dilute streams (overflow and permeate) were similar except permeate was lower in TSS than overflow (0.54 vs 1.12, respectively). Variability of dry matter in overflow and permeate caused relatively high standard error and masked detection of differences (Table 2).

3.3. Experiment 3. Microfiltration of heavy gluten

VBF was effective in concentrating dry matter and TN of heavy gluten in the gluten cake stream (Table 3). Gluten cake had higher dry matter concentration than filtrate (40.79 vs 3.61 g/100 g, respectively) and original heavy gluten (14.24 g/100 g). Gluten cake also had higher TN concentrations than filtrate (11.53 vs 9.74 g/100 g, respectively). On the other hand, filtrate had higher ash concentration than gluten cake (6.97 vs 0.80 g/100 g, respectively).

Permeate flux rates for microfiltration of heavy gluten averaged 51.3 LMH (Table 3); these were lower than values for microfiltration of light gluten due to higher dry matter content. More experiments are needed to determine if flux rates observed during microfiltration of light and heavy gluten are economically feasible. Microfiltration of heavy gluten produced two streams, concentrate and permeate, which are analogous to the gluten cake and filtrate streams, respectively, from commercial wet milling. Concentrate from microfiltration contained higher dry matter concentration than permeate (20.81 vs 1.94 g/100 g) or original heavy gluten stream (12.34 g/100 g). TN concentrations were not different among streams; SN concentration of permeate was higher than concentrate. Ash concentrations in permeate also were higher than concentrate (16.24 vs 2.26 g/100 g). Dry matter content of gluten cake was greater than concentrate (41.68 vs 21.22 g/100 g); other parameters were similar for gluten cake and concentrate. Microfiltration was more effective than VBF in concentrating ash; ash content of permeate was greater than filtrate (16.24 vs 9.74 g/100 g, respectively). As VBF was a continuous commercial scale process while microfiltration was a batch pilot scale process; direct comparison of VBF and microfiltration may have some limitations.

Although concentrate from microfiltration did not reach dry matter concentrations of gluten cake, concentrate from microfiltration of heavy gluten resulted in dry matter concentrations above values achievable by conventional gluten thickener centrifuges (20.81 vs 12.34 g/100 g, respectively, Table 2). This would decrease the amount of water to be removed by VBF, which is often a processing bottleneck in wet milling facilities.

3.4. Partitioning of nutrients in light and heavy gluten

Stream compositions were affected differently by each separation method (Tables 2 and 3). For example, the gluten thickener and microfiltration concentrated dry matter in light gluten to similar extents in their respective concentrated streams (heavy gluten and concentrate, Table 2). The gluten thickener concentrated TN similar to microfiltration, while microfiltration was more effective in diverting ash and reducing TSS in the permeate stream. Dry matter concentrations were higher in concentrate (14.70 g/100 g, Table 2) than for heavy gluten (13.14 g/100 g), but this was not a statistical difference. Small increases in dry matter concentration in heavy gluten would have significant impact on wet mill operations, since it reduces the operating load on the VBF and gluten meal dryers. Reduction of solids in permeate or overflow, while small in this study, would have important implications since it would reduce the recirculation of solids in process streams that are used upstream in the wet milling process.

For dewatering of heavy gluten, VBF was more effective in concentrating dry matter in the gluten cake than microfiltration, but TN was partitioned similarly by the two methods. Additional work may show that microfiltration can concentrate to higher dry matter concentrations than those observed in the current study; upper limits of dry matter concentration during microfiltration were not tested since pump characteristics did not allow recirculation of gluten material at dry matter values above 21 g/100 g (Table 3). Concentrations of ash during dewatering of heavy gluten were more effectively
concentrated by microfiltration into the permeate stream. Increased ash in the permeate stream also was observed in microfiltration of light gluten to a smaller extent. More work is needed to determine whether microfiltration can reduce ash content in gluten meal. This may be an advantage, as use of phosphorus in animal diets has important environmental implications and may affect gluten meal value. We previously reported phosphorus levels in wet milling coproduct streams and found that they were high (up to 14,000 mg P/kg) relative to animal nutrient needs (Rausch et al., in press). Since differences in ash concentrations of permeate, overflow and filtrate are expressed on a dry basis, inferences could be limited because process flow rates could differ and affect absolute quantities of nutrients separated.

Another approach to evaluating effectiveness of separation methods is to estimate mass balances, which incorporate flow rates of streams as well as concentrations. An assumption of the estimated flows is that commercial scale continuous flow microfiltration will separate nutrients similarly to batch microfiltration used in this study. While no statistical inferences can be drawn, estimated flows can be useful to describe separation of nutrients on a mass basis. An estimated nutrient balance for 1000 kg of light gluten (49.4 kg dry matter) using the gluten thickener are compared to microfiltration in Fig. 3. It should be noted that continuous flow systems may have different mass balances.

In the conventional wet milling process, about 38% of dry matter from light gluten was recovered in overflow, compared to 62% in heavy gluten. Similarly, 31% of TN was recovered in overflow vs 67% in heavy gluten. For microfiltration, about 47% of dry matter from light gluten was diverted into permeate and 53% was diverted into concentrate. About half of the total flow, TN and ash were diverted into each process stream (permeate and concentrate); however, because of sampling or analytical errors, TN and ash balances were overestimated.

To compare VBF to microfiltration, estimated nutrient balances for 1000 kg of heavy gluten (142.4 kg dry matter) are presented in Fig. 4. When heavy gluten was dewatered using VBF, 82% of dry matter flowed into gluten cake (116.6 kg vs 25.8 kg into filtrate). Likewise, 83% of TN in heavy gluten was diverted into gluten cake rather than filtrate (13.5 vs 2.5 kg). When heavy gluten was dewatered by microfiltration, streams were more evenly separated on a wet basis (487 and 513 kg, respectively) for permeate and concentrate. However, on a dry basis, 93% of heavy gluten was diverted into concentrate (114.7 vs 8.7 kg into permeate).

The proportion of dry matter in light gluten concentrated by the gluten thickener into heavy gluten (0.67) was greater than by microfiltration into the concentrate (Fig. 3). On the other hand, microfiltration recovered a greater proportion (0.93) of dry matter from heavy gluten into concentrate than did the VBF into gluten cake (0.82), which could have significant economic impact for a wet milling facility (Fig. 4).

VBF was effective at concentrating dry matter and TN in the gluten cake and ash in the filtrate, and microfiltration was effective in diverting dry matter to the concentrate and ash to the permeate. Microfiltration did not affect TN partition. Dry matter concentration of gluten cake was greater that dry matter content of concentrate from microfiltration; ash content of the permeate was greater than ash content of filtrate. These data suggest that microfiltration and gluten thickener had similar abilities to partition dry matter and ash into concentrated gluten and process water streams, respectively, but the gluten thickener appeared more effective in concentrating TN in the heavy gluten stream. In dewatering of heavy gluten, the VBF was more effective in increasing dry matter and TN concentrations than microfiltration, while microfiltration was more effective in concentrating ash in the liquid streams.
4. Conclusions

Microfiltration of light gluten resulted in concentrate and permeate streams similar in composition to conventionally processed light gluten using gluten thickener centrifuge technology, suggesting that microfiltration was as effective as centrifugation in partitioning solids and water in light gluten.

During microfiltration of the heavy gluten process stream, conventional VBF caused higher dry matter concentrations in gluten cake than in concentrate that resulted from microfiltration; other components of gluten cake and concentrate were not different. Permeate from microfiltration of heavy gluten had higher concentrations of ash and lower SN than filtrate. The microfiltration system used in this study did not concentrate dry matter to values as high as conventional VBF, but microfiltration was able to remove more ash and inorganics, which may improve the value of wet milling coproducts. Lower concentrations of SN in permeate also indicated more protein was retained in concentrate. As membrane technology advances, methods of removing water and recovering nutrients in streams from wet milling may become an accepted alternative to centrifugation and belt filtration technologies.

References