

# Recovery of Stillage Soluble Solids from Hard and Soft Wheat by Reverse Osmosis and Ultrafiltration

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ABSTRACT

Cereal Chem. 64(4):260-264

Ultrafiltration (UF) combined with reverse osmosis (RO) was used to separate wheat stillage solubles into small volumes of concentrated solutions and large volumes of permeates suitable for reuse. The UF permeate at 680 kPa (100 psi) accounted for 10–25% of total nitrogen and 20–36% of total solids of the stillage solubles. The UF permeate was used as the feed solution for RO. The RO permeate obtained at 1,360 kPa (200 psi) accounted for 4–4.8% of total nitrogen, 5.8–8.1% of total solids, and 77–90% of total volume of the UF permeate from wheat stillage solubles. Increasing RO pressure to 5,440 kPa (800 psi) reduced total nitrogen and solids of the permeate by about 40 times. The percentage of nitrogen and

solids from stillage solubles that can be economically recovered by combined UF and RO at 1,360 kPa (assuming only the amount of nitrogen and solids in the RO permeate is discarded) is 98.7–99.2% and 97.5–97.8%, respectively, whereas the corresponding value for combined UF and RO at 5,440 kPa is 100.0%. Conductivity of the RO permeate at 5,440 kPa was lower than that of tap water. The cost-effective UF and RO methods developed for wheat stillage solubles may encourage increased food use of wheat distillers' grains while reducing the total cost of the wheat alcohol process.

Corn is most commonly used for commercial ethanol fermentation in the United States, but a small amount of wheat is also used for this purpose (Morris 1983). Fermentation of ground cereal grains to make ethanol produces a protein-rich material (stillage) after alcohol is distilled. Optimum use of stillage is important for the commercial success of the overall ethanol process. Because fermentation of wheat creates a higher percentage of stillage solubles than corn (Wu et al 1981, 1984), the economics of the wheat process depend even more on recovery of stillage solubles at a minimal cost.

Stillage is first screened or centrifuged to yield a solid fraction (distillers' grains) and a soluble fraction (stillage solubles). Because stillage solubles from wheat and wheat flour contain approximately 5% dry matter, evaporation of water to recover solids is expensive. Ultrafiltration (UF) and reverse osmosis (RO) do not evaporate water, so large savings of energy and cost can be obtained through use of these methods. UF and RO can separate a large volume of dilute solution into a small volume of concentrated solution and a large volume of permeate that can be reused or safely disposed of. Preconcentration of apple juice by RO (Sheu and Wiley 1983), concentration of whole milk by RO for cheddar cheese production (Agbeve et al 1983), and UF and RO of oilseeds (Lawhon and Lusas 1984) have been reported. RO of stillage solubles from corn without UF to remove the larger molecules first resulted in fouling and leakage of the RO column (Wu et al 1983). The RO permeate from corn stillage accounted for 4.6% of total solids and 3.2% of total nitrogen. Stillage solubles from fermentation of whole ground wheat had lower protein but higher ash than those from wheat flour (Wu et al 1984). In this study UF combined with RO (at 1,360 and 5,440 kPa) were used to process stillage solubles from hard and soft ground wheats and their flours as an economic alternative to drying.

## MATERIALS AND METHODS

### Fermentation and Fractionation of Stillage

Figure 1 is a schematic diagram of the whole process including the fermentation, fractionation of stillage, and the UF and RO

recovery processes. Newton wheat, a typical hard red winter wheat, was grown in Kansas. Daws wheat, a soft white winter wheat, was from Washington. The dry weights used were 2,350, 1,838, and 1,811 g for hard wheat, hard wheat flour (70% extraction), and soft wheat flour, respectively. Ground wheat or wheat flour was dispersed in 5 L of tap water in a 20-L stainless steel, temperature-controlled, jacketed fermentor equipped with stirrers. The pH of each slurry was adjusted to 6.2, and 6 ml Taka-therm  $\alpha$ -amylase (Miles Laboratories, Elkhart, IN) was added. Adjustment of pH was made by 6N HCl and 12.5N NaOH. The temperature of the fermentor was maintained at 90°C for 1 hr to gelatinize and degrade starch to soluble dextrans. Then 1,328, 1,315, and 1,337 ml of tap water was added for hard wheat, hard wheat flour, and soft wheat flour, respectively. The fermentor was cooled to 60°C, the pH was adjusted to 4.0, and 18 ml of Miles Diazyme L-100 glucoamylase was added to hydrolyze dextrans to glucose. Yeast inoculum (500 ml containing 5 million cells per milliliter) made from Fermivin dry yeast (G. B. Fermentation Industries, Des Plaines, IL) was added to convert glucose to ethanol. Fermentation was carried out at 30°C for 66 hr. Ethanol was distilled from the fermentor by steam, and the residue (stillage) was filtered through cheesecloth under suction. The thin stillage that passed through the cheesecloth was centrifuged in a Sharples

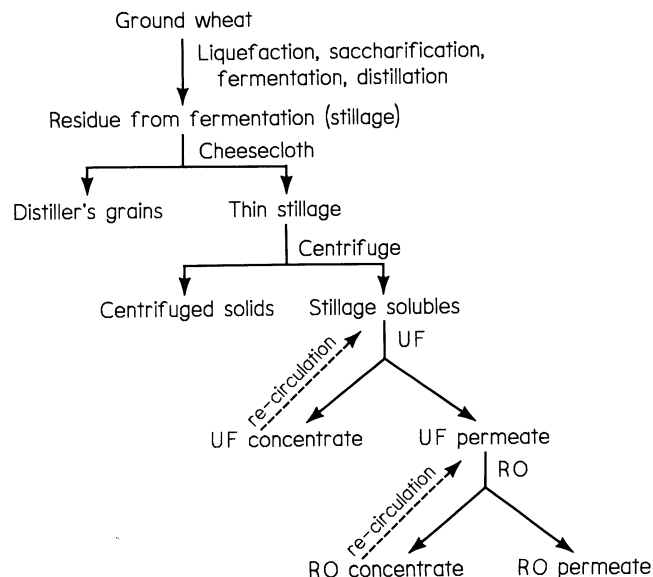


Fig. 1. Schematic diagram of the whole process including the fermentation, fractionation of stillage and the ultrafiltration and reverse osmosis recovery processes.

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continuous centrifuge at  $45,200 \times g$ . The solution that passed through the continuous centrifuge was called stillage solubles. Details of these procedures as well as ethanol yield and compositions of fermentation by-products are reported elsewhere (Wu et al 1984).

#### UF and RO

An OSMO Econo Pure RO unit (Osmonics, Inc., Minnetonka, MN) equipped with OSMO-112 Sepalators (1.0 m<sup>2</sup> membrane, hold-up volume about 600 ml) was used for UF at 680 kPa (100 psi) and RO at 1,360 kPa (200 psi). For UF, SEPA-0 Polysulfone (PS) or cellulose acetate (CA) membrane with a molecular weight cutoff of 1,000 for organics was used. For RO of UF permeate at 1,360 kPa, a SEPA-97 CA membrane with a molecular weight cutoff of 200 for organics was used. The solution that passed through the membrane was termed permeate, and the solution retained by the membrane was called concentrate. The concentrate stream was recirculated back to the initial solution. Samples of permeate and of concentrate plus initial solution (subsequently termed concentrate) were periodically taken for analyses to monitor the progress of RO runs. The flow rate of UF permeate at room temperature was 2.7 L per m<sup>2</sup> per hour for PS and 6.7–10 L per m<sup>2</sup> per hour for CA membranes. The flow rate of RO permeate was 3.8 L per m<sup>2</sup> per hour at 27°C. The hold-up fraction was obtained by draining the column. In addition, 4–6 wash fractions were collected by pumping approximately 1 L of distilled water through the system and draining each time. Material balance and percent recovery were based on the sum of permeate, concentrate, hold-up, and wash fractions.

A model UHPROLA-100 RO system (Village Marine Tec, Gardena, CA) equipped with a SW 30-2521 module with 1.1 m<sup>2</sup> polyamide (PA) membrane (Filmtec, Minneapolis, MN) with a molecular weight cutoff of 100 for organics was used for RO of UF permeate at 5,440 kPa (800 psi). Samples of permeate and concentrate were periodically removed for analyses to examine the progress of the RO runs. The flow rate of permeate was 20–25 L per

m<sup>2</sup> per hour at 20–26°C. The hold-up volume of the membrane module is 605 ml. The hold-up fraction was obtained by draining the RO system, and seven wash fractions were collected by pumping 2 L of distilled water through the system for 4 min and draining each time. The hold-up and wash fractions as well as permeate and concentrate fractions were used to calculate material balance and percent recovery.

#### Analyses

Nitrogen content was determined by micro-Kjeldahl in quadruplicate, and ash was determined in duplicate by heating to 600°C, as detailed in AACC methods 46-13 and 08-03 (AACC 1983). Solids content (dry matter) of solution was determined in duplicate by pipetting into a previously weighed crucible a known volume, which was dried overnight in an air oven at 100°C, then dried for three days in a vacuum oven at 100°C and weighed. Conductivity measurements were made at 24°C on stillage fractions by a Radiometer type CDM 2e conductivity meter with a CDC 104 NS cell.

## RESULTS AND DISCUSSION

#### Material Balance

For each UF and RO experiment, the amounts of nitrogen, solids, and ash in permeate, concentrate, hold-up, and wash fractions were determined and percent recovery was calculated based on the stillage solubles for UF and on the UF permeate for RO. Table I shows that recovery percentages of nitrogen and solids were relatively low for UF with PS membrane compared with CA membrane. For UF with CA membrane and RO with CA and PA membranes, recoveries of nitrogen, solids, and ash were close to 100% within experimental uncertainty. Apparently, some wheat proteins and solids were bound strongly by PS membrane and were not released after six water washes at the end of the UF experiment. The actual weights of wheat protein and solids bound by the PS membrane (weight of protein and solids introduced in stillage

TABLE I  
Material Balance for Ultrafiltration and Reverse Osmosis of Stillage Solubles

Stillage Solubles	Ultrafiltration				Reverse Osmosis			
	Membrane <sup>a</sup>	% Recovery			Membrane <sup>a</sup>	% Recovery		
		Nitrogen	Solids	Ash		Nitrogen	Solids	Ash
Hard wheat flour	PS	82	85	95	CA	100	99	94
	CA	100	89	103	PA	98	94	89
Hard wheat	PS	76	78	91	CA	90	85	89
	CA	96	91	101	PA	101	98	99
Soft wheat flour	PS	55	59	83	CA	99	91	103
	CA	87	90	104	PA	112	101	101

<sup>a</sup> PS = Polysulfone, CA = cellulose acetate, PA = polyamide.

TABLE II  
Ultrafiltration and Reverse Osmosis of Hard Wheat Flour Stillage Solubles<sup>a</sup>

Material <sup>b</sup>	Volume (ml)	Nitrogen (mg/ml)	Solids (mg/ml)	Ash (mg/ml)
Stillage solubles	4,770	4.32	46.4	2.02
Permeate (UF, PS)	4,460	0.926	15.2	1.72
Concentrate (UF, PS)	104	15.0	129	0.787
Permeate (RO, CA 1,360 kPa) <sup>c</sup>	3,970	0.045	1.37	0.138
range, 2 fractions	1,700–2,270	0.003–0.100	0.86–2.06	0.0826–0.207
Concentrate (RO, CA 1,360 kPa)	430	3.18	50.8	5.49
range, 2 fractions	50–380	1.27–3.43	22.1–54.5	2.35–5.88
Stillage solubles	4,230	4.84	53.1	1.77
Permeate (UF, CA)	3,845	0.525	11.4	1.39
Concentrate (UF, CA)	147	18.0	157	1.27
Permeate (RO, PA 5,440 kPa) <sup>c</sup>	3,054	0.000537	0.0317	0.0050
range, 10 fractions	226–335	0.00035–0.00081	0.0181–0.0460	0.0021–0.0083
Concentrate (RO, PA 5,440 kPa)	487	0.597	13.6	1.62
range, 10 fractions	37–50	0.351–0.968	8.49–22.9	1.15–2.63

<sup>a</sup> In addition to permeate and concentrate fractions, hold-up and wash fractions were also collected and analyzed for material balance and percent recovery.

<sup>b</sup> UF = Ultrafiltration; RO = reverse osmosis; PS = polysulfone membrane; CA = cellulose acetate membrane; and PA = polyamide membrane.

<sup>c</sup> 1,360 kPa = 200 psi; 5,440 kPa = 800 psi.

solubles minus total weight of proteins and solids recovered in permeate, concentrate, hold-up, and wash fractions) were 23, 26, and 46 g and 32, 62, and 102 g, respectively, for hard wheat flour, hard wheat, and soft wheat flour. If not removed, the bound protein and solids in the membrane fermented with bad odor and caused the membrane to be unsuitable for further use. Apparently, strong binding of wheat protein and solids by PS membrane was related to chemical composition of the polymer and not pore size of the membrane, because CA membrane of the same pore size did not bind wheat protein and solids. For comparison, PS membrane did not strongly bind corn proteins, sorghum proteins, or solids, because close to theoretical amounts of nitrogen, solids, and ash were recovered (Wu et al 1983, Wu and Sexson 1984).

#### UF and RO of Hard Wheat Flour Stillage Solubles

Table II summarizes the UF and RO results of hard wheat flour stillage solubles. The amounts of nitrogen, solids, and ash in hold-up and wash fractions are not included in Tables II-IV, but they are included in Table I. UF with PS or CA membrane will reduce the nitrogen and solids concentrations of stillage solubles by three- to ninefold but only reduce slightly the ash concentration in the permeate fraction. UF also removed large suspended particles from stillage solubles and resulted in a clear permeate solution suitable for subsequent RO. In general, the concentrations of nitrogen, solids, and ash in RO permeates and concentrates increased two to fourfold from the first to the last fractions, except that nitrogen concentration of RO permeate at 1,360 kPa (200 psi) showed a 33-fold increase as a result of the very low initial nitrogen concentration. The nitrogen, solids, and ash concentrations of RO permeate at 1,360 kPa (200 psi) decreased 11- to 21-fold from those of UF permeate, whereas those of RO permeate at 5,440 kPa (800

psi) decreased 278- to 978-fold from those of UF permeate. Almost 100% recovery of nitrogen, solids, and ash was achieved at 5,440 kPa compared with 92-96% recovery at 1,360 kPa.

#### UF and RO of Hard Wheat Stillage Solubles

Volumes and concentrations of nitrogen, solids, and ash of hard wheat stillage solubles and their UF and RO fractions are shown in Table III. Concentrations of nitrogen and solids in UF permeate were two to fivefold lower than those of stillage solubles. The nitrogen, solids, and ash concentrations of RO permeate at 1,360 kPa were nine- to 19-fold lower than those of UF permeate. When the RO pressure increased fourfold, from 1,360 to 5,440 kPa (200-800 psi), the nitrogen, solids, and ash concentrations of the permeate decreased by 69-, 62-, and 69-fold, respectively. Almost 100% recovery of nitrogen, solids, and ash was attained at the higher RO pressure of 5,440 kPa compared with 90-95% at 1,360 kPa.

#### UF and RO of Soft Wheat Flour Stillage Solubles

Table IV summarizes UF and RO results of soft wheat flour stillage solubles. For UF of soft wheat flour stillage solubles, CA membrane was more effective than PS membrane in reducing nitrogen and solids concentrations of permeate (three- to fourfold for PS versus four to eightfold for CA). Figure 2 shows the relationship between volume of concentrate remaining and concentration of solids in the permeate during RO of UF permeate from soft wheat flour stillage solubles at 1,360 kPa (200 psi). The concentration of solids in the permeate increased as the volume of concentrate remaining decreased, and a rapid increase in solids concentration of permeate was observed near the end of the RO experiment where the volume of concentrate remaining was low.

TABLE III  
Ultrafiltration and Reverse Osmosis of Hard Wheat Stillage Solubles

Material <sup>a</sup>	Volume (ml)	Nitrogen (mg/ml)	Solids (mg/ml)	Ash (mg/ml)
Stillage solubles	4,520	4.10	61.2	5.11
Permeate (UF, PS)	4,170	1.12	24.6	4.08
Concentrate (UF, PS)	146	12.6	163	5.35
Permeate (RO, CA 1,360 kPa)	3,680	0.060	1.60	0.440
range, 2 fractions	1680-2000	0.028-0.098	0.538-2.83	0.129-0.803
Concentrate (RO, CA 1,360 kPa)	275	3.48	71.6	11.8
range, 2 fractions	50-225	1.55-3.91	36.1-79.6	6.11-13.2
Stillage solubles	4,510	3.27	53.2	5.00
Permeate (UF, CA)	4,400	0.678	19.0	3.63
Concentrate (UF, CA)	156	9.73	115	5.12
Permeate (RO, PA 5,440 kPa)	3,620	0.000865	0.026	0.0064
range, 10 fractions	350-390	0.00053-0.00148	0.019-0.038	0.0005-0.0163
Concentrate (RO, PA 5,440 kPa)	450	0.806	22.3	4.35
range, 9 fractions	50	0.513-1.17	14.1-32.3	2.74-6.23

<sup>a</sup> UF = Ultrafiltration; RO = reverse osmosis; PS = polysulfone membrane; CA = cellulose acetate membrane; and PA = polyamide membrane.

TABLE IV  
Ultrafiltration and Reverse Osmosis of Soft Wheat Flour Stillage Solubles

Material <sup>a</sup>	Volume (ml)	Nitrogen (mg/ml)	Solids (mg/ml)	Ash (mg/ml)
Stillage solubles	5,470	3.26	45.8	1.70
Permeate (UF, PS)	5,000	0.844	17.8	1.30
Concentrate (UF, PS)	176	6.89	76.2	1.53
Permeate (RO, CA 1,360 kPa)	3,712	0.043	1.51	0.122
range, 10 fractions	112-400	0.017-0.129	0.38-3.07	0.035-0.488
Concentrate (RO, CA 1,360 kPa)	1,240	1.67	32.5	2.70
range, 10 fractions	100-340	0.816-2.59	17.3-48.3	1.30-3.91
Stillage solubles	5,710	3.32	43.1	1.78
Permeate (UF, CA)	5,520	0.445	11.7	1.40
Concentrate (UF, CA)	176	13.2	147	2.38
Permeate (RO, PA 5,440 kPa)	4,317	0.00071	0.022	0.00053
range, 10 fractions	415-440	0.00027-0.0019	0.002-0.031	0-0.0029
Concentrate (RO, PA 5,440 kPa)	997	0.704	17.1	2.00
range, 10 fractions	97-100	0.430-1.27	10.1-32.3	1.12-3.89

<sup>a</sup> UF = Ultrafiltration; RO = reverse osmosis; PS = polysulfone membrane; CA = cellulose acetate membrane; and PA = polyamide membrane.

## Efficiency of Various UF and RO Procedures

Efficiency of UF and RO can be measured by the percentages of original volume, nitrogen, solids, and ash in the permeate fractions. Ideally, a maximum percentage of original volume and minimum percentages of nitrogen, solids, and ash are in the permeate fraction. Table V shows the percentage of original material in permeate fractions, because percentages instead of absolute values normalize differences in starting materials. UF of stillage solubles from hard wheat flour, hard wheat, and soft wheat flour resulted in 91–98% of total volume, 10–25% of total nitrogen, 20–37% of total solids, and 70–80% of total ash being in the permeate fractions. The CA membrane was better than PS membrane, because the former had a faster flow rate, and the

**TABLE V**  
Permeate from Ultrafiltration and Reverse Osmosis as Percentage of Original Material<sup>a</sup>

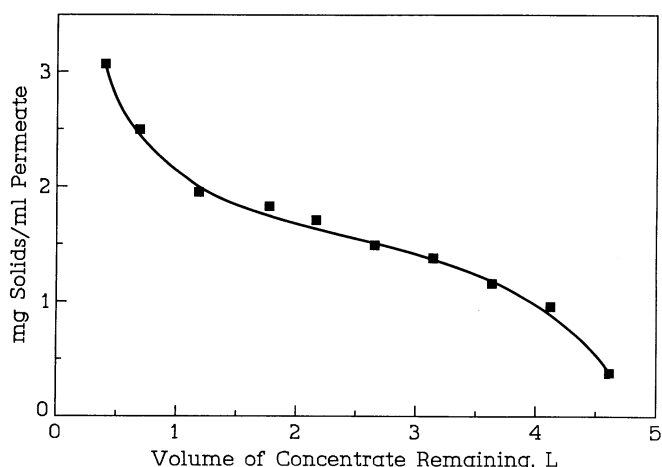
Stillage Solubles	Membrane <sup>b</sup>		Volume	Nitrogen	Solids	Ash
	(pressure)					
Hard wheat flour	UF-PS		94	20	31	80
	UF-CA		91	10	20	71
	RO-CA (1,360 kPa)		90	4.4	8.1	7.2
	RO-PA (5,440 kPa)		82	0.08	0.23	0.30
Hard wheat	UF-PS		92	25	37	74
	UF-CA		98	20	35	71
	RO-CA (1,360 kPa)		89	4.8	5.8	9.6
	RO-PA (5,440 kPa)		85	0.11	0.12	0.15
Soft wheat flour	UF-PS		91	24	36	70
	UF-CA		97	13	26	76
	RO-CA (1,360 kPa)		77	4.0	6.6	8.6
	RO-PA (5,440 kPa)		80	0.13	0.15	0.03

<sup>a</sup> Percent of stillage solubles for ultrafiltration; percent of ultrafiltration permeate for reverse osmosis.

<sup>b</sup> UF = Ultrafiltration; RO = reverse osmosis; PS = polysulfone membrane; CA = cellulose acetate membrane; and PA = polyamide membrane.

**TABLE VI**  
Correlation Coefficients of Conductivity Versus Ash and Conductivity Versus Solids Concentrations

Reverse Osmosis of Stillage Solubles, kPa	Conductivity versus	Correlation Coefficient
Hard wheat, 5,440	mg ash/ml concentrate	0.999
Soft wheat flour, 5,440	mg ash/ml concentrate	0.993
Hard wheat flour, 5,440	mg ash/ml concentrate	0.977
Soft wheat flour, 1,360	mg ash/ml concentrate	0.984
Hard wheat, 5,440	mg solids/ml concentrate	0.996
Soft wheat flour, 5,440	mg solids/ml concentrate	0.997
Hard wheat flour, 5,440	mg solids/ml concentrate	0.999
Hard wheat flour, 5,440	mg solids/ml permeate	0.967
Hard wheat, 5,440	mg solids/ml permeate	0.937
Soft wheat flour, 1,360	mg solids/ml concentrate	0.995



**Fig. 2.** Solids content of permeate versus volume of concentrate remaining during reverse osmosis of ultrafiltration permeate from soft wheat flour stillage solubles at 1,360 kPa (200 psi).

permeate had lower percentages of nitrogen and solids. The RO permeate obtained at 1,360 kPa (200 psi) accounted for 4–4.8% of the total nitrogen, 5.8–8.1% of total solids, and 77–90% of the total volume of the UF permeate from hard wheat, hard wheat flour, and soft wheat flour, respectively. However, the 5,440 kPa (800 psi) RO permeate had 0.08–0.13% of the total nitrogen, 0.12–0.23% of total solids, and 80–85% of the total volume of the UF permeate from hard wheat, hard wheat flour, and soft wheat flour. Thus, a fourfold increase in RO pressure reduced total nitrogen and total solids of the permeate fraction by about 40 times.

The nitrogen, solids, and ash concentrations of RO permeate and RO concentrate increased as the volume of the concentrate decreased. Solids and ash concentrations of RO concentrate were linearly related to conductivity (Table VI). RO permeates with much lower concentrations than RO concentrates show more scatter in Table VI. Thus conductivity can predict solids and ash concentrations of RO concentrates. Conductivity of cold tap water at room temperature (0.72 mS/cm) was close to that of RO permeate at 1,360 kPa (0.65–1.18 mS/cm), but much higher than that of RO permeate at 5,440 kPa (0.036–0.052 mS/cm).

## CONCLUSIONS

Gregor and Jeffries (1979) reported that the total cost for equipment, power, and labor (but not for interest) for a combination of UF and RO methods was \$3.53 per 1,000 gal of stillage treated, as compared to \$8.33 for fuel cost alone by the evaporative route. UF in combination with RO gave better results (a smaller percentage of solids, nitrogen, and ash in the permeate fraction) for wheat stillage than for recycled corn stillage with the same membrane and at the same pressure (Wu et al 1983). Ultrafiltration in combination with RO appears to be a practical method to process stillage solubles from wheat and wheat flour. A large volume of dilute solution can be separated into a small volume of concentrated solution and a large volume of permeate that can be reused or safely discarded. Cellulose acetate membrane was better than PS membrane for UF of stillage solubles from wheat and wheat flour. Reverse osmosis at 5,440 kPa (800 psi) was superior to that at 1,360 kPa (200 psi) because of the very large increase in efficiency.

The method described here to recover stillage solubles from wheat and wheat flour utilizing UF and RO may improve the economics of fermentation of wheat and wheat flour for ethanol and may provide valuable food-grade products. Tsen et al (1983) used corn distillers' dried grains flour as a bread ingredient, and barley distillers' dried grains flour was added to quick bread to build up fiber and protein content (Eidet and Newman 1984). Wheat distillers' grains may be more suitable than corn distillers' grains for food uses, because wheat is traditionally used for baked products and has a higher lysine content (Wu et al 1984). Wheat distillers' grains contain more protein and less fat than distillers' grains from corn (Wu et al 1981, 1984). The cost-effective UF and RO methods developed for recovery of wheat stillage solubles recovery may, therefore, encourage increased food use of wheat distillers' grains while reducing the total cost of the wheat alcohol process.

## ACKNOWLEDGMENT

I gratefully acknowledge technical assistance of N. E. Harrison, K. R. Sexson, J. P. Anderson, and A. A. Lagoda.

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[Received August 11, 1986. Revision received February 18, 1987. Accepted February 20, 1987.]