Oat Protein Concentrates from a Wet-Milling Process: Preparation[1]

J. E. CLUSKEY, Y. V. WU, J. S. WALL, and G. E. INGLETT, Northern Regional Research Laboratory, Peoria, Illinois 61604

ABSTRACT

A wet-milling process was developed to produce protein concentrates, starch, and residue fractions from dry-milled oat varieties having moderate- (Wyndmere) and high-protein contents (Garland). Different solvents and pH values were evaluated for their effectiveness in extracting an oat protein concentrate in good yield. The optimum yield of protein was obtained in dilute alkali solution (pH 9). Starch and protein were separated from bran by sieving the alkaline dispersion. After the fine suspension was centrifuged to separate pure starch (0.05% nitrogen), the protein solution was adjusted to pH 6 and freeze-dried. The protein content (nitrogen × 6.25) of the concentrate varied between 59 and 89%, depending on the dry-milled fraction and process used, and accounts for up to 88% of total protein in the starting material. This simple process for producing an oat protein concentrate may have commerical potential.

A 1972 survey established a total market potential for functional protein of approximately 3.1 billion lb. annually (1). Nonfat dry milk is by far the largest selling protein ingredient, while dry whole milk, soy protein, and casein are the next three. Freedom from objectionable taste is a significant factor for any ingredient. Although soy flours have a higher protein content, a higher or compatible degree of functionality, and a lower price than nonfat dry milk, sales of nonfat dry milk are more than ten times those of soy flour because the dry milk has a better taste. Since approximately two-thirds of the 3.1-billion-lb. potential protein market remains untapped there is considerable room for new protein ingredients with bland tastes.

Oats have good quality protein and a high-protein content (2,3,4). The increasing availability of high-protein oats and the favorable solubility characteristics of oat proteins (5) suggest that it may be feasible to make an oat protein concentrate having good nutritive value. An alkali process for preparing starch and protein from wheat and other cereal flours has been described by Dimler et al. (6). A wet-milling process is described here to produce protein concentrates, starch, and residue fractions from ground oat groats of moderate- and high-protein contents.

MATERIALS AND METHODS

The oats were purchased from Interstate Seed and Grain Co., Fargo, N. Dak.: Wyndmere (lot WO-494-495), grown in North Dakota in 1970, had a protein content (nitrogen × 6.25) of 14.2% d.b. and represented a variety with a moderately high-protein content; Garland (lot BH-474), grown in Minnesota in 1970, had a protein content of 17.2% d.b. and represented a high-protein variety. An Alpine pin mill at 1,445 r.p.m. was used to dehull the oats, and the resulting

[1]Presented at the 57th Annual Meeting, Miami Beach, Oct.-Nov. 1972. Contribution from the Northern Regional Research Laboratory, Agricultural Research Service, U.S. Department of Agriculture, Peoria, Ill. 61604. The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

groats were separated from hulls by screening and aspiration. The groats were ground in a hammer mill. Some of the groats were defatted with 1-butanol.

Ground oat groats were mixed with a solvent and stirred magnetically for 25 min. The slurry was then centrifuged for 10 min. at 5,000 r.p.m. (3,300 X g) in a Sorvall laboratory centrifuge. The supernatant was collected, analyzed for nitrogen by a micro-Kjeldahl method, and freeze-dried. To produce an oat protein concentrate, the slurry was passed through 100-mesh bolting cloth. When the dissolved protein and the starch that passed through the cloth were centrifuged, the starch solid remained at the bottom of the centrifuge tube while the protein remained in solution. The supernatant was decanted, brought to pH 6, and freeze-dried. The starch and the material that remained on the bolting cloth were adjusted to neutral pH and also freeze-dried.

RESULTS AND DISCUSSION

Solvent

Protein was extracted from Wyndmere groats with various solvents, at a 1:10 ratio of groats:solvent, to determine which would perform best (Table I). The best solvent for extracting protein is 0.015N NaOH, which extracted 88% of the nitrogen. When pH of the slurry drops to 7.00, the amount of nitrogen extracted drops to 57%, and at pH 6.25, only 19%. Apparently pH plays an important role in extracting groat protein, and the best solvent is NaOH with a slurry pH of around 9.0. The percentage of protein (nitrogen X 6.25) in the extract solids generally increases with the percentage of nitrogen extracted. Solids in the 0.015N NaOH extract have 72.1% protein, whereas those in 1M NaCl extract have only 8.5%. This low-protein content is partially caused by the NaCl present in extract solids because NaCl was not removed before freeze-drying.

Stirring

The effect of time of stirring on extraction of Wyndmere groat protein by 0.015N NaOH (1:10) is determined. The amount of nitrogen extracted increases

TABLE I. EFFECT OF VARIOUS SOLVENTS ON EXTRACTION OF WYNDMERE OAT PROTEIN (GROATS:SOLVENT RATIO 1:10)

Solvent	pH of Slurry	Nitrogen Extracted %	Protein (Nitrogen X 6.25) in Extract Solid %
0.05N HCl[a]	2.29	13	35.9
2N acetic acid	2.99	15	24.4
4N acetic acid	2.72	22	35.8
Water	5.96	17	31.4
1M NaCl	5.54	38	8.5
0.001N NaOH	6.25	19	27.7
0.005N NaOH	7.00	57	50.6
0.01N NaOH	7.83	83	71.2
0.015N NaOH	8.90	88	72.1
0.02N NaOH	9.53	84	64.5
0.03N NaOH	10.69	80	59.2
0.05N NaOH	11.71	85	54.9

[a]Garland groats.

from 64% with 4 min. of magnetic stirring to 86% after 60 min. The difference between 25 and 60 min. is not considered significant in percentage of nitrogen extracted (actual difference is 1.5% before rounding off); consequently, 25 min. of stirring was used for all subsequent extractions.

TABLE II. EFFECT OF GROATS:SOLVENT RATIO ON EXTRACTION OF WYNDMERE GROAT PROTEIN

Groats:Solvent Ratio	Solvent Recovered in Extract %	Solvent (NaOH) Normality	Concentration of Extract mg. Nitrogen/ml.	Nitrogen Extracted %
1:2	50	0.075	14.4	53
1:3	63	0.05	9.44	64
1:4	71	0.04	6.69	70
1:6	85	0.025	4.19	78
1:8	88	0.020	3.15	82
1:10	90	0.015	2.53	84
1:12	90	0.013	2.07	83
1:15	92	0.010	1.59	80
1:20	93	0.0075	1.18	81

Groats:Solvent Ratio

The effect of groats:solvent ratio (1:2 to 1:20) on extraction of Wyndmere groat protein is shown in Table II. The normality of NaOH is adjusted to maintain approximately the same pH value. The pH of the slurry varied between 7.8 and 8.4, which is near the optimum value. The percentage of nitrogen extracted increases to a maximum of 84 at a groats:solvent ratio of 1:10. A groats:solvent ratio of 1:6 with 0.025N NaOH was chosen for most of the oat protein concentrate work as a compromise between greatest recovery of nitrogen and smallest volume of solvent. The percentage of solvent recovered in the extract increases from 50 to 93 when groats:solvent ratio was increased from 1:2 to 1:20. Extract concentration decreases form 14.4 to 1.18 mg. nitrogen per ml. when groats:solvent ratio was increased from 1:2 to 1:20. These results indicate that the solubility of the protein is not exceeded at the lowest ratio; rather, an approximately constant volume of solvent is trapped in the centrifugate, which accounts for low recoveries at low ratio.

Product Yield from Groats

Comparison of groat protein levels and effect of defatting are given in Table III. A groats:solvent ratio of 1:6 with 0.025N NaOH was used. Garland protein concentrate with 75% protein (nitrogen X 6.25) accounts for 19% by weight and 70% total protein of the defatted groats, which had 20.6% protein.

A small yellowish layer was observed on top of the Garland starch after centrifugation. In a separate run on Wyndmere groats this small yellowish layer was scraped off and had 6.6% protein, while the starch fraction without this layer had 0.2% protein. The 2.4% protein value for defatted Garland groat starch includes this yellowish layer, which is rich in protein. If this yellowish layer is excluded from the starch, then the protein value for defatted Garland starch will be much below 2.4%.

Total products in Table III account for 96% of starting weight and 99% of total

TABLE III. PRODUCTS FROM GARLAND AND WYNDMERE GROATS (DRY BASIS, SOME DEFATTED)

Product	Weight %	Protein (Nitrogen X 6.25) of Solid %	Total Protein %	
		Defatted Garland		
Protein concentrate	19	75.0	70	
Starch[a]	53	2.4	6	
Bolting cloth residue	24	20.2	23	
Total	96		99	
		Defatted Wyndmere		
Protein concentrate	16	73.1	70	
Starch	46	0.8	2	
Bolting cloth residue	29	17.1	29	
Layer above starch	2			
Total	93		101	
		Wyndmere		
Protein concentrate	21	59.1	66.2	
Starch	37	0.2	0.4	
Screen (40-mesh) residue	15 } 30	19.6	15.2 } 17.1	
Bolting cloth residue	15 }	2.4	1.9 }	
Layer above starch	1	6.6	0.3	
Total	89		84.0	

[a]Includes a yellowish layer, above the starch, that has a considerably higher protein content than starch.

protein. Both residue and starch fractions included some protein solution so that the protein content of dried residue and starch in Table III is high. Protein content of residue and starch fractions will be significantly lowered by a water wash.

The yield of various products from defatted Wyndmere groats having 16.8% protein is also given in Table III. The defatted groats had a lower protein content than nondefatted, because 1-butanol removed 11% nitrogen. Protein concentrate from these groats had 73.1% protein, and accounted for 16% by weight, and 70% of total protein. Table III reveals that the high protein groats (Garland) gave a better yield of protein concentrate with a higher protein content, a better yield of starch but a poorer yield of bolting cloth residue. The starch and bolting cloth residues from the Garland groats are also high in protein, although part of the protein difference in the two starch fractions can be accounted for by the layer above the starch that has more protein than the starch fraction.

Table III also gives the yield of various products from Wyndmere groats having 18.9% protein. Protein concentrate with 59.1% protein accounts for 21% by weight and 66.2% of the total protein of Wyndmere groats. In a separate experiment where half the alkali extract of Wyndmere groats was precipitated isoelectrically and half the extract was freeze-dried with the supernatant, the increase in protein content of the precipitated protein concentrate solid was 5%. Starch from Wyndmere groats accounts for 37% by weight and has only 0.2% protein, the lowest protein content obtained in all the starch fractions without a water wash.

A screen residue (40 mesh) was also collected in addition to the usual bolting cloth of considerably finer mesh. The screen residue has 19.6% protein, whereas the bolting cloth residue has 2.4% protein. A comparison of the two Wyndmere groats reveals that the defatted ones give a lower yield of protein concentrate with higher protein content, a better yield of starch, and a higher total nitrogen recovery for protein concentrate, as well as total products. When Table III results are compared with corresponding data for Garland groats, the same difference is observed as described for Wyndmere groats.

Consecutive Extraction

One problem with an alkaline extraction is that the alkaline slurry is viscous and difficult to filter through the bolting cloth. Since two solvents used consecutively might eliminate this high viscosity problem, some extractions were carried out in this manner; namely, water-HCl, water-NaOH, and NaOH-HCl (Table IV).

Although too much nitrogen was left in the starch fraction, the HCl slurry after water extraction was not too viscous, and the solution and fine particles passed through the bolting cloth with ease.

To find out if low viscosity observed for the HCl slurry is a result of pH or the previous water extraction, defatted Garland groats were extracted with water and then 0.25N NaOH. Viscosity of the subsequent alkaline slurry was slight, and the solution and fine particles passed through the bolting cloth with ease. Apparently, water extraction had removed most of the oat gum. The sum of the water and NaOH extracts totaled 20% by weight and 68% of total protein and corresponded closely with the protein concentrate representing 19% by weight and 70% of total protein in Table III. The starch fraction after water-NaOH extractions had a higher protein content than that of defatted Garland groats in Table III, and may be the result of the higher protein content of the alkaline slurry from which the starch was separated.

TABLE IV. CONSECUTIVE EXTRACTIONS OF DEFATTED GARLAND GROATS (DRY BASIS)

Product	Weight %	Protein (Nitrogen X 6.25) of Solid %	Total Protein %
Water extract	7	27.9	9
HCl extract	8	71.3	29
Starch	62	12.4	38
Bolting cloth residue	17	25.8	22
Total	94		98
Water extract	7	26.6	9
NaOH extract	13	88.8	59
Starch	55	3.6	9
Bolting cloth residue	19	20.3	19
Total	94		96
NaOH extract	15	81.4	62
HCl extract	4	18.6	4
Starch	49	6.3	15
Bolting cloth residue	26	15.1	19
Total	94		100

The protein content of the starch fraction in Table IV can be greatly reduced by one or more water washes, because in a separate experiment a water wash of the starch removed 60% of the nitrogen from the starch. The protein content of the bolting cloth residue can probably be reduced substantially by another alkaline extraction, because in a separate experiment a water wash of the bolting cloth residue removed 38% of the nitrogen from the residue, and alkaline extraction was more efficient than water. The water wash can be recycled to reduce disposal problems and to recover more protein.

The result of 0.025N NaOH extraction followed by HCl extraction at pH 2.2 is also shown in Table IV for defatted Garland groats with groats:solvent ratio of 1:6. The combined NaOH and HCl extracts account for 19% by weight and 66% total protein, compared with the combined water and NaOH extracts accounting for 20% by weight and 68% total protein, and with the protein concentrate accounting for 19% by weight and 70% of total protein in Table III. Apparently there is no advantage in using HCl in place of water in the extractions including NaOH as one of the solvents.

GENERAL DISCUSSION

Dimler et al. (6) described an alkaline process to produce starch from flour and established conditions for the essentially complete dispersion of the protein of wheat flour. Although oat flour was included in their study, they did not try to find optimum conditions for it. We used ground oat groats and optimized all experimental conditions. They used a pH above 10.6 for oat flour, whereas our optimum pH for oat groats was around 8.9.

We selected ground oat groats because they are cheaper to produce than oat flour and have a higher protein content. Inclusion of bran and shorts, which have higher protein and larger particle size than flour, in the ground groats necessitated an additional screening step to separate starch from the bolting cloth residue. Ground oat groats include some gum, which gives a high viscosity slurry that is difficult to screen. The high viscosity, which is not encountered in other cereals, can be overcome by an initial water extraction.

The bolting cloth residue (bran fraction) has around 20% protein and may be used as either a food or a feed ingredient. The starch has food and industrial uses. If the starch and the bolting cloth residue are not separated by screening, the resulting mixture will have 8.0% protein (Table III, defatted Garland groats).

A number of such variables as solvents, pH, stirring time, groats:solvent ratio, defatting, and protein content of groats were evaluated to produce an oat protein concentrate and other products in optimal yield and composition. It is most encouraging that high-protein groats (Garland) give a better yield of starch and of concentrate with more protein than do moderate-protein groats (Wyndmere). Oats, higher in protein than the two varieties studied here, will probably yield an even better protein concentrate. Much research on high-protein oats is now being conducted in many locations.

Literature Cited

1. HAMMONDS, T. M., and CALL, D. L. Protein use patterns–current and future. Chem. Technol. 156 (1972).
2. JONES, D. B., CALDWELL, A., and WIDNESS, K. D. Comparative growth-promoting values of the proteins of cereal grains. J. Nutr. 35: 639 (1948).

3. HISCHKE, H. H., Jr., POTTER, G. C., and GRAHAM, W. R., Jr. Nutritive value of oat proteins. I. Varietal differences as measured by amino acid analysis and rat growth responses. Cereal Chem. 45: 374 (1968).
4. ROBBINS, G. S., POMERANZ, Y., and BRIGGLE, L. W. Amino acid composition of oat groats. J. Agr. Food Chem. 19: 536 (1971).
5. WU, Y. V., SEXSON, K. R., CAVINS, J. F., and INGLETT, G. E. Oats and their dry-milled fractions. Protein isolation and properties of four varieties. J. Agr. Food Chem. 20: 757 (1972).
6. DIMLER, R. J., DAVIS, H. A., RIST, C. E., and HILBERT, G. E. Production of starch from wheat and other cereal flours. Cereal Chem. 21: 430 (1944).

[Received October 12, 1972. Accepted February 23, 1973]