

# Flour Containing Protein and Fiber Made from Wet-Mill Corn Germ, With Potential Food Use

H. C. NIELSEN, J. S. WALL, and G. E. INGLETT, Northern Regional Research Center, Federal Research, Science and Education Administration, U.S. Department of Agriculture,<sup>1</sup> Peoria, IL 61604

## ABSTRACT

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Flour containing 30% protein, 5.9% lysine, generous amount of food fiber, and a good balance of other essential amino acids was prepared from wet-mill corn germ. It represented the upgrading of a present feed by-product to a product with potentially valuable food uses. To prepare the flour, dried wet-mill corn germ was first aspirated to remove hull and, after

flaking, fat was removed by solvent extraction. A second extraction of the flakes with 82:18 hexane/ethanol azeotrope via refluxing achieved very low fat levels in the final product. The ground flour contained approximately 2% ash, 18% starch, 26% pentosans, 12% cellulose, and 0.6% lignin, but starch content may vary, depending on processing conditions.

Hand-dissected corn germ contains about 35% oil, 19% protein, and 10% minerals (Earle et al 1946). The protein has a good balance of essential amino acids (Mertz et al 1966, Wall et al 1971). Both wet and dry corn milling industries separate germ-rich streams, which are now a valuable source of food oil. In both industries, germ material remaining after oil removal is combined with other fractions and used as animal feed. Methods of oil removal usually employ expeller processes that result in loss of nutritional quality and unfavorable color and organoleptic changes in the germ product.

A flour high in protein and minerals was prepared from dry-mill corn germ (Blessin et al 1974, Garcia et al 1972) and tested as a nutrient fortifier for bread (Tsen et al 1974). This flour is now available commercially. In addition, a protein isolate was prepared from dry-mill corn germ (Nielsen et al 1973, 1977).

This article describes the preparation from wet-mill corn germ of a flour rich in food fiber and quality protein, the chemical composition and physical properties of which indicate potential for food uses.

## MATERIALS AND METHODS

### Preparation of Flour

Three samples of wet corn germ were obtained from the Pekin, IL, plant of CPC International on October 1975, June 1976, and October 1976. The corn grain had been steeped and milled in water containing sulfur dioxide according to standard wet milling practice (Anderson 1970) to produce a germ stream contaminated with some hull (pericarp) and with a moisture content of about 55% at the sampling point. The wet-mill germ samples were promptly spread out in the laboratory to air dry at room temperature, which required from 48 to 136 hr depending on sample size. The hull, which represented 21–34% of the dry weight of each wet-mill germ sample, was removed by aspiration on a Eureka bean cleaner. The cleaner also removed some incompletely ground kernels as well as fines that represented 2–3% of the dry weight of each sample. The cleaned germ fraction, which passed through a sieve with 5.5-mm round openings and remained on a sieve with 1 × 6 mm openings, appeared similar to intact corn germ. At this point in the flour preparation, the cleaned germ contained about 48% lipid.

Most of the lipid then was removed from the three samples of full-fat germ material by batchwise extraction with either hexane or 82:18 hexane/ethanol. Sample 1 was cracked on corrugated rolls

before extraction, sample 2 was flaked on smooth rolls before extraction, and sample 3 was flaked after initial lipid extraction with hexane. Final lipid removal was accomplished by refluxing the cracked or flaked samples with an 82:18 hexane/ethanol azeotrope (Eldridge et al 1971) until no more lipid was extracted for several hours. This process took 48 hr with a Soxlet apparatus with a 9 × 41 cm chamber. Extracted samples were then spread out to allow residual solvent to evaporate, the samples being tempered to about 10% moisture to help displace solvent. After solvent removal, the germ material was ground to a fine meal or flour using a Wiley, hammer, or pin mill.

A fourth flour sample was prepared from a wet-mill germ sample obtained October 1976, which had been dried at the CPC Pekin mill using air at about 70°C. This is the standard material from which corn oil is produced. Flour was prepared from this sample by aspirating to remove hull or pericarp, extracting batchwise with 82:18 hexane/ethanol, flaking, refluxing with hexane/ethanol azeotrope in a Soxlet apparatus, and grinding. The procedure was the same as that used to prepare sample 3 except that the flakes were somewhat thicker because lighter flaking rolls were used.

### Analytical Procedures

Moisture content was determined by drying to constant weight at 105°C or by using a moisture balance equipped with an infrared lamp. Ash content was determined by adding magnesium nitrate to the sample to fix organic phosphate, then ashing at 600°C according to AACC Method 08-02.

Protein content was determined by multiplying semimicro Kjeldahl-N values by 6.25. Samples for amino acid analysis were hydrolyzed for 24 hr under reflux with 2 ml of 6*N* hydrochloric acid per milligram of sample. Amino acids were then determined by the method of Benson and Patterson (1965) and calculated by the automated procedure of Cavins and Friedman (1968). This procedure adds methionine and methionine sulfone and reports the sum as methionine. Tryptophan was determined on samples hydrolyzed by alkali (Wilkinson et al 1976).

Lipid content was determined by Butt extraction, using pentane-hexane, AACC method 30-25, and also by a scaled-down version (Nielsen et al 1977) of the gas-liquid chromatography (GLC) procedure of Black et al (1967). The method was further modified by adding 0.4 g of a neutralizing mixture that contained two parts by weight of sodium bicarbonate, one part of sodium carbonate, and two parts of sodium sulfate to each sample after overnight reaction, then mixing, and allowing to settle before injection.

Neutral detergent fiber was determined by AACC method 32-20 (Schaller 1977) with the  $\alpha$ -amylase steps before the detergent extraction. Acid detergent fiber was determined by Van Soest's procedure (1963), as described in AOAC procedure 7.055 (1975), and pentosans were estimated by Sloneker's GLC procedure (1971). Crude fiber was determined by AACC method 32-15 to allow comparison with earlier results. Starch was determined polarimetrically after extraction with dimethyl sulfoxide (Garcia

<sup>1</sup>Mention of firm names or trade products does not imply their endorsement or recommendation by the U.S. Department of Agriculture over other firms or similar products not mentioned.

and Wolf 1972) and also by polarimetric measurements of hot calcium chloride extracts of the flour (Clendenning 1945).

Protein dispersibility index was performed by a fast stir procedure, AACC method 46-24, and hydration capacity by AACC method 56-20. Protein efficiency ratio was determined by the Wisconsin Alumni Research Foundation using AOAC procedure 43.183-7, (1975). Flavor and odor were assessed on 2% dispersions in charcoal-filtered tap water by the 15-member taste panel at the Northern Regional Research Center trained to assess cereal and soy products (Kalbrener et al 1971, Nielsen et al 1977, Rackis et al 1972).

## RESULTS AND DISCUSSION

### Preparation

Preparation of flour from wet-mill corn germ involves three major steps: drying, cleaning, and removing fat.

Drying was best achieved by the commercial process of passing the moist germ material through a stream of air heated to about 70°C. Germ thus dried was slightly darker than air-dried germ and had a slight toasted flavor, but these differences were not observed in the final deoiled and ground product. Air drying of more than 5 kg of germ was slow, especially when the ambient air was humid, and the germ thus processed developed a rancid odor during the process and probably some microbial contamination.

The dried wet-mill germ was readily cleaned by aspiration and sieving. The aspiration removed hull or pericarp containing about 30% readily extractable oil. (The high oil content of aspirated hull was due in part to adhering germ.) The sieving removed a small amount of incompletely ground kernels and fines. The cleaned germ had the appearance of dissected intact germ from whole corn kernels.

TABLE I  
Yields of Fractions Obtained During Preparation  
of Wet-Mill Corn Germ Flour (Dry Basis)

Fraction	From Air-Dried Germ	From Heat-Dried Germ
	(Samples 1-3)	(Sample 4)
	(%)	(%)
Flour	29-31	39
Aspirated hull	34-37	9
Fractions removed by sieving	2-3	10
Oil	27-29	36
Bound lipid	2-3	4

TABLE II  
Composition and Properties of Wet-Mill Corn Germ Flour

	Sample			
	1	2	3	4
Proximate analysis (% dry basis)				
Protein (N × 6.25)	28.4	24.8	31.7	31.0
Pentosans	28.8	22.1	26.0	24.4
Acid detergent fiber	12.7	11.2	...	...
Lignin	0.6	0.6	...	...
Neutral detergent fiber	35.3	26.9	34.0	31.0
Crude fiber	10.0	8.3	10.3	10.2
Starch	15.7	35.5	19.4	20.2
Fat	0.4	0.5	0.3	1.5
Ash	2.0	1.8	2.1	2.1
Properties				
pH of water suspension	4.4	4.3	4.6	4.4
Water binding (g H <sub>2</sub> O/g flour)	7.4	6.9	8.0	7.4
Protein dispersibility index, %	38	34	30	28
Protein efficiency ratio	...	...	2.44	2.34

Most of the germ oil was initially removed by batchwise extraction with hexane or hexane/ethanol azeotrope. Bound lipid, which constitutes about 10% of the lipid in corn germ (Weinrauch and Matthews 1977), was removed by refluxing thinly flaked or ground germ with warm 82:18 hexane/ethanol azeotrope. The lipid thus extracted was dark brown and had a varnishlike odor. Removal of bound lipid was necessary to improve flavor stability of the germ flour (Eldridge et al 1971, Nielsen et al 1977).

Yields of flour, hull, crude oil, and other fractions obtained during the preparation of wet-mill corn germ flour are summarized in Table I.

### Properties of the Flour

The proximate composition of the four samples of wet-mill corn germ flour is given in Table II. (The four samples contained 6.0-8.7% moisture; however, values in the table are on a moisture-free basis.)

Three of the four samples of wet-mill corn germ flour have protein contents in the narrow range of 28.4 to 31.7%, which is higher than the typical 25% protein content of dry-mill corn germ flour (Garcia et al 1972). Corn germ feed material reportedly contains 25% protein (Reiners et al 1973). Sample 2 of wet-mill corn germ flour contained less protein, 24.8%, and proportionally more starch.

The amino acid composition of the protein in wet-mill corn germ flour is given in Table III. The amino acid composition of wet-mill corn germ flour is quite similar to both dry-mill corn germ flour (Blessin et al 1973) and protein isolate prepared from dry-mill germ material (Nielsen et al 1973). Table III also lists the 1973 FAO/WHO Provisional Amino Acid Scoring Pattern. Comparison shows that wet-mill corn germ flour has an excellent balance of essential amino acids. The flour is notably high in lysine in contrast to most cereal grain proteins. The excellent nutritional quality of the protein in the flour is verified by its protein efficiency ratio (PER) of 2.44 normalized to a PER of 2.50 for casein. Flour from the heat-dried wet-mill flour (sample 4) had a slightly lower PER of 2.34 (Table II).

Wet-mill corn germ flour is rich in food fiber. The four samples had neutral detergent fiber values from 26.9 to 35.3%. The four

TABLE III  
Amino Acid Composition of Wet-Mill Corn Germ Flour  
Compared with FAO/WHO Scoring Pattern for Essential Amino Acids  
(g/100 g of protein)

Amino Acid	Wet-Mill Corn Germ Flour	Scoring Pattern <sup>a</sup>
Essential Amino acids		
Histidine	3.6	1.7
Isoleucine	3.8	4.0
Leucine	8.5	7.0
Lysine	5.9	5.5
Methionine	2.2	
Cystine	1.4	
Total S-amino acids	3.6	3.5
Phenylalanine	5.2	
Tyrosine	4.1	
Total aromatic amino acids	9.3	6.0
Threonine	4.6	4.0
Tryptophane	1.3	1.0
Valine	6.6	5.0
Other amino acids		
Alanine	6.9	
Ammonia	1.8	
Arginine	8.3	
Aspartic acid	8.2	
Glutamic acid	17.1	
Glycine	6.6	
Proline	6.5	
Serine	5.7	

<sup>a</sup>FAO/WHO (1973).

flour samples contained 22.1–28.8% pentosans as measured by GLC, part of which were soluble in the neutral fiber determination. The acid detergent fiber content of the flour, which is an indication of cellulose content (Van Soest 1963), was 11.2–12.7%, and the lignin content was 0.6%. (The crude fiber content of the flour ranged from 8.3 to 10.3%.) However, the relative value of the fiber found in corn germ flour, compared with other sources of food fiber, requires further evaluation. Dry-mill corn germ flour only contains 4.2% crude fiber.

The fat content of samples 1–3 of wet-mill corn germ flour was 0.3–0.5% as measured by pentane-hexane extraction (AACC method 32-15) and by a GLC procedure that involves acid hydrolysis (Black et al 1967). Flour sample 4 had a lipid content of 1.5% because the flakes that were extracted with hexane/ethanol azeotrope were thicker.

The mineral content of wet-mill corn germ flour was only about 2%, compared with 10% for corn germ (Earle et al 1946) and dry-mill corn germ flour (Blessin et al 1974). Wet-mill corn germ flour contains little if any free sugars, whereas dry-mill corn germ flour contains 12% free sugars, the major one being sucrose (Garcia et al 1972). Most of the mineral constituents and free sugars are removed during steeping in the wet-milling process; thus wet-mill corn germ flour contains less minerals and free sugars than does dry-mill corn germ flour.

Samples 1, 3, and 4 of wet-mill corn germ flour contained 15.7–20.2% starch as measured by polarimetry after extraction of starch with dimethyl sulfoxide (Garcia and Wolf 1972). Sample 2 flour had a higher starch content of 35.5%, which was offset by the lower protein content of this sample, as mentioned. Starch contents of the flours determined by polarimetry on calcium chloride extracts (Clendenning 1945) were 18–30% lower than those determined on dimethyl sulfoxide extracts. Both methods for determining starch were developed for whole corn, and their accuracy in this application is not known.

Table II also lists the water-binding capacity and the protein dispersibility index of wet-mill corn germ flour. The water-binding capacity was in the 6.9–8.0 range, which is quite high. The protein dispersibility index was 28–38%, which is relatively low. Comparative values for dry mill corn germ flour were 4.2 for water-binding capacity and 75% for protein dispersibility index. The fat-binding ability of the wet-mill flour was poor.

The flavor characteristics of wet-mill corn germ flour were evaluated by a trained taste panel. Their conclusion was that sample 4, made from heat-dried germ, was similar to a "good" soy flour having weak cereal-grain, bitter, and astringent flavors. Sample 1, which was made from air-dried wet-mill germ, was judged to be more like an unheated soy flour having, weak, grassy-beany, and rancid-paint notes, in addition to the above flavors.

Large amounts of raw material now going into animal feed are available for making wet-mill corn germ flour and, with the exception of the azeotrope extraction step for removal of bound lipid, the flour can be readily prepared. The flour may be useful as a protein-fiber enrichment for foods. Work is underway to evaluate wet-mill corn germ flour in foods.

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