

# Fractionation of High-Lysine Corn to Produce Edible By-Products

A. H. MISTRY,<sup>1</sup> M. P. STEINBERG,<sup>2</sup> and S. R. ECKHOFF<sup>1</sup>

## ABSTRACT

Cereal Chem. 69(4):433-435

A wet process for high-lysine corn was developed on a pilot scale with the primary objective of producing germ and soluble protein for food. Corn was steeped in lactic acid solution, and a hydraulic shear mill was used to remove whole germs. A floatation column separated the whole

germ from the coarsely ground corn. Whole, clean germs were recovered in 8.52% yield. Soluble protein was recovered by conventional tabling, with yields of 6.67%. The soluble protein was high in lysine content. The germs have potential for replacing confectionery nuts.

Sulfurous acid solution is used for conventional corn steeping to reduce disulfide bonds in matrix protein and maximize starch yield (Anderson 1970). Residual sulfur dioxide (SO<sub>2</sub>) imparts a bad taste to the germ and protein and is considered hazardous to health (NIOSH 1978). Unreacted SO<sub>2</sub> is not a problem in conventional wet milling since it is washed from the starch and is not soluble in oil. The germ is extracted to remove oil, and germ meal is not used for food.

In wet milling high-lysine corn, the germ is an attractive value-added food product because of its higher nutritional value (Bressani 1966, Clark 1966). To obtain edible germ, sulfur dioxide should be eliminated. Breakage of the germs must also be reduced to obtain pieces of maximum size and stability to oxidative rancidity.

Germs obtained in conventional wet milling are separated by the centrifugal action of hydroclones (May 1987). This equipment requires a high fluid velocity to achieve separation, which causes germ breakage and subsequent oil absorption by the protein (Watson 1984, May 1987).

Protein (i.e., corn gluten meal) from the conventional wet milling of yellow dent corn is deficient in the essential amino acids lysine and tryptophan and therefore is lower in nutritional value (Jimenez 1966) than protein from high-lysine corn. Corn gluten meal also has low functionality as a result of the partial denaturation of protein by sulfur dioxide. High-lysine corn has more protein with higher levels of lysine than dent corn. The germ of high-lysine corn, which is larger in size (Watson and Yahl 1967) than the germ of regular dent corn, contains more protein (Wichser 1966). The nonzein protein of high-lysine corn has better inherent functionality (more water-soluble protein) and is rich in nutritional value (Jimenez 1966). Separation of high-lysine protein for edible products could be of great commercial value.

Studies on wet milling of high-lysine corn using laboratory procedures including SO<sub>2</sub> have shown that separation of products (or fractions) is easier than with dent corn (Watson and Yahl 1967). The starch yield and recovery obtained from high-lysine corn were lower than those from dent corn (Dimler 1966, Watson and Yahl 1967), but the yields of gluten and steepwater solubles were higher. However, no efforts have been made to wet mill high-lysine corn to produce high-quality germ and protein products for food applications.

The objective of this research was to develop a wet fractionation method for high-lysine corn that: 1) uses no sulfur dioxide in the steeping solution; 2) has a high yield of whole, clean germs; and 3) obtains germ and protein suitable for food applications.

## MATERIALS AND METHODS

High-lysine corn (Crow's Hybrid Corn Co., Milford, IL) dried at 25°C in a forced-draft oven from an initial moisture content

of 20% to a final moisture content of approximately 12.5% (wb) was used. The corn had an initial protein content of 10.53% (db).

A flow sheet of the process used to fractionate the corn is given in Figure 1. The process was run in duplicate, and two sets of samples for each fraction were obtained.

### Steeping

High-lysine corn was steeped in a 40-L stainless steel can heated in a temperature-controlled jacketed tank. Corn (632.70 g per liter of steep solution) was steeped at 52°C for 48 hr. Steep solution was made by mixing 12.28 ml of 85% lactic acid and 3.12 g of potassium hydroxide in 1.0 L of water to give pH 3.8. The potassium hydroxide was used to adjust the final pH of the lactic acid steep to 3.8.

### Coarse Grinding

Steeped corn with steepwater was coarsely ground with added dilution water. Several mills such as the Buhr mill (Bauer Bros. Co., Springfield, OH), the Waring Blendor (Dynamic Corp. of America, New Hartford, CT), the FitzPatrick hammermill (W. J. FitzPatrick Co., Chicago, IL), and the Quaker City mill (Straub Co., Croyden, PA) were tested, but all ground the germs to fragments. A Fryma mill (model MK/95-R Fryma-Maschinen AG, Rheinfelden, Switzerland) was tried after being modified by reversing the blade and omitting the screen. This gave the proper hydraulic shear needed to free the germ without breakage. This was a batch operation; about 5 min was needed for complete release of germ. However, multiple Fryma mills connected in series could be used to obtain continuous grinding of corn.

### Floatation Column

The coarsely ground corn was fed into a floatation column to separate the germs from the remainder of the slurry. The floatation column consisted of a Plexiglass column (25 cm diameter × 122 cm height) with an overflow 7.5 cm from the top and a discharge port at the bottom. Ground corn was fed to the balance tank (Fig. 1), where it was mixed with underflow from the wedge-bar screen (progressive screen openings 0.51–1.02–1.52 mm, municipal sewage model, Wells Corp., Roscoe, IL) and pumped to the column at 38 cm from the top. A mixer (Cole-Parmer Instruments, Chicago, IL), having a 61-cm long shaft with a propeller in the column and installed at the top of the column, was used at 200 rpm to disperse the ground corn. To float the germ in the column, the density of the suspension had to be controlled between 7 and 8° Bé. The ground corn could be diluted to this density; however, for ease of operation during startup, the column, which held 68 L of liquid, was filled with a starch suspension at the desired density. During an extended run, this starch suspension would be gradually displaced by the diluted ground corn.

Germ, being less dense than the other corn components, floated to the top of the column, was carried out with the overflow, and was retained on a 16-mesh screen. The germ-free ground corn was removed from the bottom by a discharge pump and was passed through a wedge-bar screen to separate fiber and pieces of endosperm from the liquid. The liquid was recirculated to the column. Germs retained on the screen were rinsed free of endosperm and air-dried.

<sup>1</sup>Research associate and associate professor, respectively, Department of Agricultural Engineering, University of Illinois, Urbana 61801.

<sup>2</sup>Professor (deceased), Department of Food Science, University of Illinois, Urbana 61801.

### Fine Milling and Sieving

The underflow from the column was passed over the wedge-bar screen and the liquid sent back to the column. The overs were fine milled in an ordinary Fryma mill. The fine-milled slurry from the Fryma mill was again passed over the wedge bar screen, with the fiber being discarded and the liquid sent to the starch table.

### Tabling

Six lengths of 3-m aluminum trough 10 cm wide were fixed in cascade arrangement so that the overflow from one landed onto the next. The incline was 2.5 cm in 3 m, and the flow rate was 180–200 ml/min. The overflow from the last table was collected and stored in a cold room for further processing.

### Gravity Separation

The underflow fraction was collected in 40-L milk cans and placed in a cold room at 4°C for 24 hr. The supernatant liquid was pumped out and freeze-dried to obtain soluble protein. The sediment was air-dried at 25°C to obtain the gluten fraction.

### Analyses

All the fractions were collected separately and weighed for material balancing. A 100- to 150-g representative sample was obtained from each well-mixed fraction and dried to determine solids content, using the vacuum oven method (method 14.002, AOAC 1984). The dried samples were ground in a coffee grinder and sealed in plastic bags. These samples were sent to a commercial laboratory for determinations of starch (Ewers 1908), protein (Kjeldahl method 7.015, AOAC 1984), and lysine (method 43.224, AOAC 1984). In addition, germ was analyzed for oil content

(method 7.063, AOAC 1984) and the solubles fraction for ash (method 7.009, AOAC 1984), lactate (Cristina Gancedo and Luh 1986), sugars (method 31.034, AOAC 1984), fiber (method 7.066, AOAC 1984), and fats or oil (method 7.063, AOAC 1984).

Preliminary sensory testing of the raw germ and soluble protein was done with the objective of evaluating the acceptability of the products for possible commercial product development. A group of 10 faculty members and graduate students from the Department of Food Science of the University of Illinois at Champaign-Urbana were presented a sample of dried germ and protein to test for qualities acceptable in a food product. The group had experienced the usual sulfur dioxide odor and taste from the normal wet-milled yellow dent corn out of a commercial plant. The germs were presented as is (dried to 10% moisture content, wb) and the protein as a 1% solution in distilled water.

## RESULTS AND DISCUSSION

Solids recovery data indicated a 97% recovery after coarse grinding, 99% after germ separation, over 98% after tabling, and over 96% after gravity separation. Over 99% of the incoming starch (corn plus added starch) could be accounted for in the product streams. A similar balance for protein showed that 99% could be accounted for in the product. The germ balance presented a problem because the germ could not be weighed in the intact corn. To estimate germ recovery, a kernel count was obtained by counting a weighed aliquot. Mass balance showed over 97% recovery of the germ fraction.

On a dry weight basis, the germ yielded 8.52% of the corn feed solids (Table I). This was less than that reported by Watson and Yahl (1967). The discrepancy was probably due to extraction of solubles from the germ during steeping and differences in hybrids. Starch yield was lower and starch protein content was higher (Table I) than previous reports by Watson and Yahl (1967)

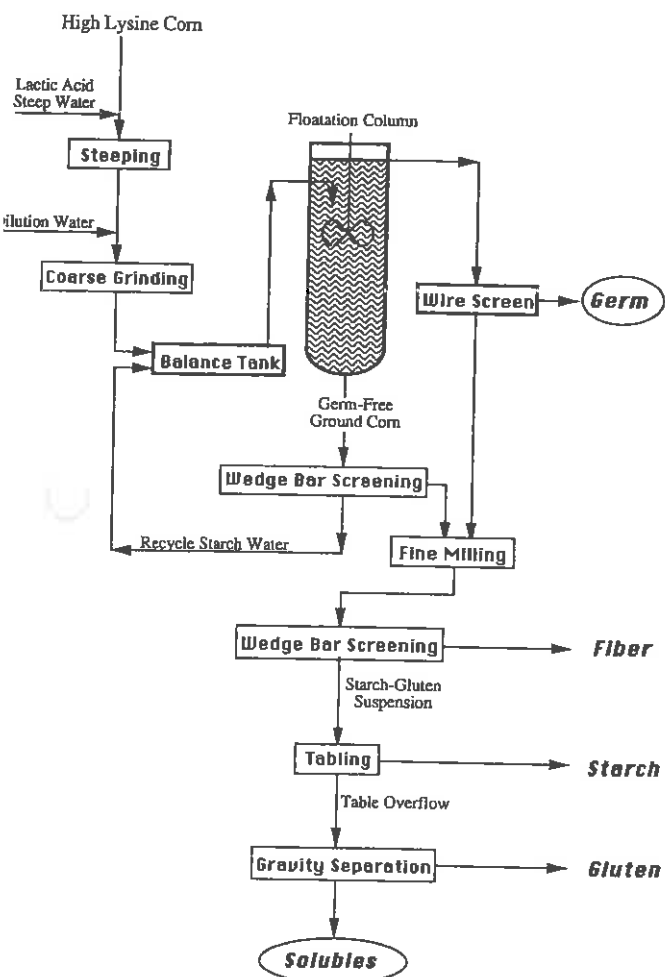


TABLE I  
Comparison of High-Lysine Corn Composition  
and Results from Wet Processing of High-Lysine Corn  
with the Literature Data on High-Lysine and Yellow Dent Corn

	Present (High-Lysine)	Literature Data		
		High- Lysine <sup>a</sup>	High- Lysine <sup>b</sup>	Yellow Dent <sup>c</sup>
Whole kernel				
Starch, % db	69.35 ± 0.92	64.4	64.0	71.4
Protein, % db	10.53 ± 0.04	13.6	13.0	10.4
Lysine, % db	4.20 ± 0.16	3.9	3.8	2.7
Starch				
Yield, % db	60.73 ± 1.32	61.3	59.0	67.4
Recovery, % db	87.50 ± 1.80	95.1	91.0	94.4
Protein, % db	1.28 ± 0.04	...	0.5	...
Germ yield, % db	8.52 ± 0.17	9.2	...	7.1
Fiber yield, % db	12.21 ± 0.62	10.2	19.0	9.4
Gluten yield, % db	11.57 ± 0.88	4.6	9.0	6.4
Solubles <sup>d</sup> yield, % db	6.67 ± 0.23	11.2	12.0	6.6

<sup>a</sup>Watson and Yahl (1967).

<sup>b</sup>Dimler (1966).

<sup>c</sup>Percent of protein.

<sup>d</sup>Steepwater plus process-water solubles.

TABLE II  
Starch, Protein, and Lysine Contents of Products  
from Wet Processing of High-Lysine Corn

Fractions	Starch (%, db)	Protein (%, db)	Lysine (% of protein)
Corn	69.35 ± 0.92	10.53 ± 0.04	0.44 ± 0.16 <sup>a</sup>
Germ	9.75 ± 0.50	22.00 ± 0.32	1.31 ± 0.04
Fiber	28.30 ± 0.35	7.35 ± 0.34	0.26 ± 0.02
Starch	96.10 ± 0.41	1.28 ± 0.04	...
Gluten	48.16 ± 0.91	40.94 ± 1.02	0.16 ± 0.01
Solubles	6.51 ± 0.37	39.80 ± 0.92	1.13 ± 0.03

<sup>a</sup>Percent of corn.

Fig. 1. Wet fractionation process for high-lysine corn to obtain edible germ and soluble protein.

TABLE III  
Composition of the Soluble Protein Fraction  
from Wet Processing of High-Lysine Corn

Analysis	Amount (%, db)
Protein	39.80 ± 0.92
Ash	17.10 ± 0.20
Lactate	15.30 ± 0.41
Sugars	14.00 ± 0.50
Starch	6.51 ± 0.37
Lysine <sup>a</sup>	1.13 ± 0.03
Fiber	0.84 ± 0.06
Fat	0.12 ± 0.00

<sup>a</sup>Percent of protein.

and Dimler (1966). This was expected since no sulfur dioxide was used in steeping. The majority of the remaining starch was retained in the fiber and gluten fractions (Table II). Since starch separation was not the primary objective of this study, no efforts were made to improve starch yield and purity. However, combined starch and fiber fractions could be used for fermentation to produce fuel ethanol and other chemicals.

Total protein in the corn was 10.53 g of protein per 100 g of dry corn (Table I). The soluble protein yield was 6.67% (Table I), for an apparent recovery of 63% of the total protein. Calculation based on data in Table II showed that the protein losses were about evenly divided between the starch, fiber, and gluten fractions. The starch contents of the fiber and gluten fractions were high (Table II).

The soluble protein stream (Table III) contained only 40% protein, with most of the remaining solids about equally divided among ash, lactate, and reducing sugars. This stream was obtained by freeze-drying the liquid leaving the system so that it contained all the solubles. A more sophisticated approach such as isoelectric precipitation would have yielded a product containing much more protein. The lysine content of this freeze-dried fraction was 1.13%. This was two and a half times more than that of the high-lysine corn (0.44% corn basis, Table II) and more than three and a half times more than that in yellow dent corn protein (0.31% corn basis, Wilson 1987).

The germs obtained using the floatation column were whole and free of adhering or admixed endosperm particles and tip caps. The comments from the preliminary sensory test on the germ included "good nutty flavor" and "crispy texture" with "slight tart" aftertaste. The whole germ might be used as a nut substitute in baked products, as a snack food, etc. The oil content of the germ was 44.2%. Treatment of the germ with an antioxidant and storage in a controlled atmosphere, as is commonly practiced with other nut products, may be needed to improve shelf life and stability to oxidative rancidity.

Preliminary sensory evaluation of the soluble protein fraction indicated an off-flavor to the product. Additional purification or processing to improve the organoleptic qualities of the soluble protein would probably be needed before it could be used for food or as a food ingredient.

### CONCLUSIONS

A modified steeping procedure without SO<sub>2</sub> was successfully used to steep high-lysine corn for subsequent separation of germ

and protein. A reversed-blade Fryma mill released the germ from kernels with minimum breakage. By use of a floatation column, continuous recovery of clean, whole germ (8.52% yield) was possible. The soluble protein yield was approximately 40%. This protein needs further improvement to enhance its organoleptic characteristics. The lysine content of the separated soluble protein was two and a half times more than that of the high-lysine corn and three and a half times more than that of yellow dent corn.

### ACKNOWLEDGMENTS

This research was sponsored by Crow's Hybrid Corn Company, Milford, IL.

### LITERATURE CITED

- ANDERSON, R. A. 1970. Corn wet milling industry. Pages 151-170 in: *Corn: Culture, Processing and Products*. G. E. Inglett, ed. AVI Publishing Co.: Westport, CT.
- ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. 1984. *Official Methods of Analysis*, 14th ed. The Association: Washington, DC.
- BRESSANI, R. 1966. Protein quality of opaque-2 maize in children. Pages 34-39 in: *Proceedings of the High Lysine Corn Conference*. T. M. Edwin and E. N. Oliver, eds. Corn Refiners Association Inc.: Washington DC.
- CLARK, H. E. 1966. Opaque-2 corn as a source of protein for adult human subjects. Pages 40-44 in: *Proceedings of the High Lysine Corn Conference*. T. M. Edwin and E. N. Oliver, eds. Corn Refiners Association Inc.: Washington DC.
- CRISTINA GANCEDO, H., and LUH, B. S. 1986. HPLC analysis of organic acids and sugars in tomato juice. *J. Food Sci.* 51:571.
- DIMLER, R. J. 1966. Report on kernel structure and wet milling of high lysine corn. Pages 121-127 in: *Proceedings of the High Lysine Corn Conference*. T. M. Edwin and E. N. Oliver, eds. Corn Refiners Association Inc.: Washington DC.
- EWERS, E. 1908. Erläuterungen Der vorstehenden Standard Methode Liegt die Veröffentlichung. *Z. Öffentliche Chim.* 14:150.
- JIMENEZ, J. R. 1966. Protein fractionation studies of high lysine corn. Pages 74-79 in: *Proceedings of the High Lysine Corn Conference*. T. M. Edwin and E. N. Oliver, eds. Corn Refiners Association Inc.: Washington DC.
- MAY, J. B. 1987. Wet milling: Process and products. Pages 377-397 in: *Corn: Chemistry and Technology*. S. A. Watson and P. E. Ramstad, eds. Am. Assoc. Cereal Chem.: St Paul, MN.
- NIOSH. 1978. National Institute of Occupational Safety and Health, U.S. Department of Health Education and Welfare: NIOSH Occupational Health Guidelines for Sulfur Dioxide. HEW publication 78-123. U.S. Govt. Printing Office: Washington, DC.
- WATSON, S. A. 1984. Corn and sorghum starches: Production. Pages 417-468 in: *Starch: Chemistry and Technology*. R. L. Whistler, J. N. BeMiller, and E. F. Paschall, eds. Academic Press Inc.: Orlando, FL.
- WATSON, S. A., and YAHL, K. R. 1967. Comparison of wet milling properties of opaque-2 high lysine corn and normal corn. *Cereal Chem.* 44:488.
- WICHSER, W. R. 1966. Comparison of the dry milling properties of opaque-2 and normal dent corn. Pages 104-116 in: *Proceedings of the High Lysine Corn Conference*. T. M. Edwin and E. N. Oliver, eds. Corn Refiners Association Inc.: Washington DC.
- WILSON, C. M. 1987. Proteins of the kernel. Pages 273-310 in: *Corn: Chemistry and Technology*. S. A. Watson and P. E. Ramstad, eds. Am. Assoc. Cereal Chem.: St Paul, MN.

[Received August 1, 1991. Accepted January 22, 1992.]