Hydrocyclone Separation of Dry-Milled Corn[1]

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ABSTRACT

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Corn milled to <1 mm using a screen mill was sieved and the particles >590 µm were soaked for 1 hr in solutions with a range of glucose concentration providing liquid specific gravities above and below that of the lighter fraction (corn germ). A suspension of these particles was pumped through a hydrocyclone to separate the germ particles and to rinse water-soluble compounds from the corn. The specific gravity of the hydrocyclone streams (suspensions), as well as liquid phase (after solids settling) was measured, and product compositions and particle sizes were determined. This work shows that a germ-enriched fraction of corn ground to <1 mm, can be separated with a hydrocyclone. To enable computer simulation of the corn-treatment process, the experimental data was used to construct a two-component model of the hydrocyclone separation of the milled corn. In the model, the milled and sieved corn is virtually separated into germ and endosperm streams that are fed to paired hydrocyclone models. Hydrocyclone bypassing streams that simulate particles entrained to the contrary outlet (underflow for germ and overflow for endosperm particles) were included in the simulation to bring the model into agreement with the experimental product compositions.

Myers-Betts and Baianu (1990) and Hojilla-Evangelista et al (1992) have recently noted the need for less costly processes to extract the protein constituents from corn gluten. An attractively priced protein coproduct would support the overall return for starch and ethanol production and provide an outlet for corn protein other than animal feed, which is sensitive to political influence because of the significant export market. About half of the protein content of corn, called zein, is insoluble in water and aqueous salt solutions but soluble in mixtures of ethanol and water. Zein has been extracted and used as a photographic film substrate and to form textile fibers (Moncrieff 1963, Balmaceda and Rha 1974). Currently, zein, purified by a sequence of alcoholic solution extraction steps, supplies a small, relatively high-priced market for edible coatings for hygroscopic foods and is getting increased attention as a fast-food package coating material (Yamada et al 1996). Its alcohol-solubility and water-insolubility recommends zein for wider, nonfood, coating uses, if it can be extracted at a sufficiently low cost.

In response to this opportunity to enable better use of a corn-derived material in potential oversupply, the USDA-ARS has begun developing a lower cost, corn protein extraction method. Unlike the usual zein extraction methods, this process will use corn rather than corn gluten as its feed. High starch purity is not necessary for ethanol production, therefore removing the protein before chemically attacking the corn structure may be acceptable, even though very pure starch may not be produced. Unsteeped corn can be milled more easily to a size that optimizes the extraction of the zein; the steeping cost will be avoided and inhibitory lactate will be kept out of the fermentor feed.

Examination of existing methods shows that the major fraction of zein extraction cost is due to the alcohol recovery. Therefore, minimal use of alcoholic extraction solution will be a feature of any new, large-scale process. Low solvent use can be achieved by reducing the solvent holdup in the starch into which the solvent diffuses while dissolving the zein. Reduced holdup can be obtained by grinding the corn to increase the specific zein dissolution rate. Experiments have shown that the zein extraction rate will also improve if the ground corn is rinsed with a nonalcoholic aqueous solution to remove soluble sugar and protein first. Finally, after extraction and precipitation, the extraction solution can be filtered from the precipitated protein and recycled at a modest cost using the ethanol plant's distillation facilities.

Because corn oil is a valuable constituent, economical separation of the germ fraction and recovery as a fairly pure, extractable product is an important requirement of a new corn-to-ethanol process. A recent report (Jain and Bal 1997) of an attempt to pearl millet to separate low-fat grits showed that sieved fractions of milled (millet) grains had quite different fat content. Analysis of a variety of corn types and their roller-milled products (Peplinski et al 1992), showed that 37–50% of the oil in corn can be recovered. Although not steeped, finely dry-milled corn should be separable in a hydrocyclone using an aqueous fluid of density intermediate between the density of the germ and endosperm. To achieve some of the benefits of steeping by removal of soluble species that might interfere with zein extraction, the ground corn can be soaked and suspended in sufficiently dense dextrose solutions. Dextrose is preferable to salt for increasing the liquid density because it has minimal interference with fermentation, the ultimate destination of the corn starch. After separation, the germ fraction can be subsequently dried and the oil removed by pressing and solvent extraction. The zein is distributed throughout the corn endosperm quite uniformly in 1-µm diameter "zein bodies" within 5–35 µm starch granules (Duvick 1961). The corn can be ground to starch granule size and the protein extracted in two steps: 1) a heated alkaline solution, to remove glutelins, 2) an ethanol extraction of zein (Kampen 1992). Corn fractionation starting with a dry-milling step appears to have economic promise as part of an alcohol plant, especially if larger particles can be used. However, although there are recent reports on the separation of wet-milled corn using hydrocyclones (Singh and Eckhoff 1995), insufficient experimental information is publicly available describing the separation of the germ fraction of a larger than granule size powder to use as part of a credible zein extraction cost estimate. This article describes the experiments made to enable preliminary estimation of the cost of corn grinding and treatment before extraction.

MATERIALS AND METHODS

Milling and Sieving

Corn purchased from a local feed supplier was milled <1 mm using a Wiley screen mill (model 1, Thomas Scientific Co.,

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Copies of the input file for the hydrocyclone model, with or without product filtration and recycle of the liquid to the hydrocyclone inlet, will be E-mailed by the authors to interested readers on request.

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Swedesboro, NJ) driven at 775 rpm with a 1-hp motor. The milled corn was separated with screens of sizes 840, 710, 590, 420, and 250 T (U.S. standard screens 20, 25, 30, 40, and 60). Seven milling trials were made, all producing corn flour with a bimodal size distribution, with maxima at 0.71–0.84 mm and 0.42–0.59 mm. Table I shows the variation of composition with particle size for one particular sample.

Chemical Analysis

The protein content was determined by micro-Kjeldahl (AACC 1995) and the conversion factor 6.25 was used to convert nitrogen to protein (AOAC 1984).

Moisture was determined gravimetrically by oven-drying at 103°C overnight. Oil content was determined by drying the sample overnight in an oven at 110°C. Approximately 100–300 mg of the dry sample is then packed into a weighed glass wool-plugged pipette (the glass wool is rinsed with hexane and chloroform and dried before use) and the filled pipette is weighed. The microcolumn is eluted with 5 mL of hexane, followed by 5 mL of chloroform. The eluate is collected in a tared vial and subsequently evaporated to constant weight with nitrogen. The weights of the hexane and chloroform extracts divided by the sample weight was taken to be the weight fraction of oil in the sample.

The starch content of corn samples was determined after they were ground in a Wiley mill to a uniform size using the 20-mesh screen. Moisture was measured to determine the sample dry weight. Samples (100 mg) were then hydrolyzed to glucose with 2 mL of 1*N* trifluoroacetic acid (TFA) at 120°C for 1.5 hr. After hydrolysis, the samples were cooled to 25°C, the TFA evaporated with nitrogen, and the dry residue washed into a 100-mL volumetric flask with 10 mL of water. After filtration through a 0.45-μm filter, the glucose content of the hydrolysate was determined using an HPX-87H HPLC column with refractive index detection.

Physical Tests

One set of samples containing 123.6 g of milled corn with 11.2% moisture, with particle size >590 μm, were mixed with 1,000 g of solutions containing dextrose from 13 to 15 wt% (1:9 solid to liquid) and allowed to soak with mild stirring for 1 hr. A second set of samples (52.6 g) were mixed with 1,000 g of solutions with 18.4–27% glucose (1:19 solid to liquid). The solid-to-liquid ratios were selected to represent likely maximum values that could be used with a hydrocyclone. The liquid density, viscosity, and absorbance all increased slightly after exposure to the milled corn, indicating dissolution of a significant amount of material. The increase in density of the product solutions increased with increase in the original solution density, suggesting that the corn swells during immersion. After soaking, the slurries were centrifuged (model 80120-B International Equipment Co., Boston) at 1,960 rpm (790 × *g*) for 5 min. The liquid and buoyant particles were filtered and rinsed with 1,000 g of distilled water using an aspirated Buchner funnel with Whatman no. 1 paper, freeze-dried, and weighed. The measured glucose content of the final solid was used to determine the weight of corn solids buoyant in the solutions and, consequently, the calculated fraction of the original corn mass and the lipid content of the fraction. As evident in Table II, for solutions with a specific gravity <1.05, taken to be that of germ particles, a low density (unprecipitated) fraction of the corn survived the centrifugation. Apparently, possibly because of the high solids loading, most of the germ particles were entrained by endosperm particles.

TABLE I
Lipid and Protein Content of Size Fractions of Dry-Milled Corn

Particle size, mm	Mass Fraction in Size Intervals	Lipids, wt%	Protein, wt%
1.0–0.84	14.9	2.11 ± 0.10	8.45 ± 0.17
0.84–0.71	54.8	2.98 ± 0.15	8.80 ± 0.08
0.71–0.59	7.60	3.50 ± 0.17	9.08 ± 0.07
0.59–0.42	15.8	4.11 ± 0.20	9.27 ± 0.13
0.42–0.25	6.07	4.66 ± 0.23	8.61 ± 0.10
0.25–0	0.80	4.81 ± 0.24	8.08 ± 0.07

Hydrocyclone Separation

Batches of sieved and milled corn were added to a measured quantity of dextrose solution in a 50-gallon kettle with two counter-rotating agitators powered through concentric shafts (Hamilton Kettles, Fairfield, OH). The mixtures were agitated for 1 hr while being pumped from the bottom kettle outlet through a loop containing an oscillating tube densitometer (Automation Products, Houston) and a return line emptying into the top of the kettle. After the mixing period, a three-way valve was turned to divert the return flow to a small hydrocyclone (Centri-cleaner 600-N-3, Sprout-Bauer, Inc., Muncy, PA) with the polyurethane bottom section trimmed to form a 0.25-in. outlet diameter. The solution and corn specific gravities shown in Table III were determined from the density measurement of the suspension fed through the densitometer tube to the hydrocyclone. The soaked corn specific gravity was 1.294 for all tests. The value was determined by flotation tests in solutions of known specific gravity. The streams from the hydrocyclone were collected on filters, drained, and dried in a vacuum drier.

The dried product from each outlet was weighed, and a sample of these dry products along with a sample of the feed corn were analyzed for starch, lipid, and protein content. The experimental data shown in Tables IV and V were derived from these measurements.

TABLE II
Variation of Dry-Milled Corn Buoyancy with Liquid Density

Original Liquid Composition (Glucose wt%)	Liquid Specific Gravity After Soaking	Viscosity (cP)[a]	Corn Fraction Buoyant[b]	Lipid wt% of Buoyant Fraction[c]
13	1.0415	1.56	0.00353	4.9
13.5	1.0423	1.53	0.00338	4.5
14	1.0462	1.56	0.00399	3.4
14.5	1.0474	1.59	0.00386	4.3
15	1.0489	1.62	0.00396	4.9
18.4	1.0721	2.05	0.0138	6.4
20.5	1.0790	2.29	0.0263	9.3
22.1	1.0882	2.44	0.0177	8.4
24.8	1.0975	2.56	0.0241	11.1
27.0	1.1064	3.04	0.0167	7.6

[a] Temperature in bath = 19 ± 1°C

[b] Final corn weight corrected for glucose content in the final solid.

[c] Lipid content was 2.9 ± 0.4 for the first five samples of milled, sieved corn and 2.4 ± 0.2 for the last five samples of milled, sieved corn.

TABLE III
Inlet Stream Corn Content and Liquid Specific Gravity for Hydrocyclone Tests

Test	Feed Mass%	Liquid Specific Gravity[a]	Effluent Rates (lb/hr)	
			Overflow	Underflow
53a	4.01	1.044	· · ·	· · ·
53b	1.90	1.044	· · ·	· · ·
53c	1.023	1.044	· · ·	· · ·
53d	0.514	1.044	892	3,060
57a	6.25	1.080	· · ·	· · ·
57b	0.67	1.075	· · ·	· · ·
57c	0.29	1.075	· · ·	· · ·
57d	0.13	1.075	· · ·	· · ·
57e	0.084	1.075	846	3,336
58a	5.445	1.067	3,522	703.5
58b	0.567	1.065	· · ·	· · ·
58c	0.222	1.067	542	3,686
58d	0.101	1.066	· · ·	· · ·
58e	0.096	1.067	· · ·	· · ·

[a] Specific gravities were corrected to 25°C (Paljk et al 1990). $Y = (180.157\ \text{mol} + 1{,}000)/(994.764 + 110.88\ \text{mol} + 0.0760\ T\text{m})$ where mol is moles of glucose/kg of water, Y is in g/cm^3 and T is temperature in °C.

The solid particle mass in the feed was determined from the weights of solids collected on filters. The solids were dried and the dextrose content measured and used to calculate the solid mass in each hydrocyclone outlet stream. The solids weight fraction in the hydrocyclone feed stream was calculated using the recovered corn mass and the measured suspension density of the feed stream. The specific gravity of the liquid was checked by weighing 25 mL from the top of filtrate samples held until the next day. Samples of the filtered underflow and overflow were rinsed, dried, and analyzed for moisture, glucose, protein, and lipid content. Results are listed in Table IV.

Simulation

To conserve dextrose, it will be necessary to recycle most of the liquid effluent from the hydrocyclone directly to a mixing point ahead of the inlet. Thus, dextrose solution must be separated from the outgoing streams using appropriately sized filters or centrifuges capable of handling the rather substantial flows needed for even a small hydrocyclone. To examine the process without using a large filter, batch runs in which the corn suspension is fed to the hydrocyclone for a short period were used, along with a simulation program. The simulation program can predict the steady mass balances of flow sheets containing hydrocyclones, if the unit models for the process elements are valid.

The ASPEN PLUS simulation package was used to prepare a flow sheet of the corn grinding, sieving, soaking, and hydrocyclone separation process. Simulation is the calculation of stream flow rates and compositions that constitute a steady balance of the mass and energy at each block, consistent with the block and input stream specifications. The blocks are computational models of the unit operations and the program converges the stream values in the network connecting the blocks. For the milling process, the CRUSHER block, and for the sieving process the SCREEN block, were used. These units matched the experimental particle size distribution (PSD) produced by corn milling and sieving experiments.

TABLE IV
Corn Component Distribution to Hydrocyclone Outlets[a]

	Overflow %			Underflow %		
Test	**Protein**	**Lipid**	**Starch**	**Protein**	**Lipid**	**Starch**
53a	15.69	5.68	75.38	9.05	1.48	81.10
53b	14.04	15.27	45.93	8.72	1.22	76.46
53c	15.39	15.77	40.71	9.20	0.905	66.98
53d	13.95	6.66	48.37	9.46	1.30	76.08
57a	10.52	4.04	63.47	8.50	1.16	72.14
57b	11.14	7.54	38.77	8.55	3.54	57.19
57c	11.31	6.19	36.17	8.33	2.38	62.79
57d	10.30	8.11	37.16	7.89	2.26	62.71
57e	9.20	11.81	41.75	9.33	3.78	83.40
58a	10.52	4.24	63.47	8.50	0.84	72.14
58b	11.14	18.23	38.77	8.55	4.03	57.19
58c	11.31	16.61	36.17	8.33	3.99	62.71
58d	10.30	16.64	37.16	7.89	5.15	62.71
58e	9.20	18.62	41.75	9.33	2.67	83.40

[a] Final corn weight was corrected for the glucose content in the final solid.

The hydrocyclone block model is based on several published correlations: the separation efficiency, defined as the ratio of solids underflow rate to the hydrocyclone solids feed rate, as a function of the diameter of the solid particle being separated (Yoshioka and Hatta 1955); and the particle diameter, for which half the feed passes out the underflow, d_{50}. The parameter d_{50}, is calculable using a correlation involving the liquid viscosity, density and flow rate, and the dimensions and characteristics of the hydrocyclone (Bradley 1965). The hydrocyclone characteristics, flow rate ratio (underflow rate to feed rate) and power of the tangential velocity loss coefficient can be calculated from empirical correlations reported by Moder and Dahlstrom (1952). Although the density difference between the liquid and solid particle is part of the relation to determine d_{50}, the HYDROCYCLONE block model is not suitable for simulating the use of a hydrocyclone to sort particles on the basis of their specific gravity. To produce product streams with different fractions of light and heavy particles, the simulation must separate the mixture before it is fed to the hydrocyclone and then show how each fraction is separated in the hydrocyclone. One method is to use separate HYDROCYCLONE blocks for each fraction. In the case of corn, there are two fractions. A virtual separation of the milled and sieved (>590 µm) corn stream into two streams representing light (germ) and heavy (endosperm) particle fractions was made before feeding them to (paired) HYDROCYCLONE blocks. The output of the paired blocks was combined to give the simulated output of a single hydrocyclone fed with the particle mixture.

Separate streams were generated from the coarse corn stream, with flow rates in proportion to the (experimentally measured) germ and endosperm fractions of the corn. The separate streams were created by converting a fraction of the coarse stream to a soluble compound (glucose), using an RSTOIC block and filtering it from the rest of the (corn) solids in the coarse stream, using a FILTER block. The corn solid fraction was renamed endosperm and suspended in an aqueous solution, determined by a FORTRAN block to give a solids loading equal to the actual feed to the hydrocyclone. The flow rate of the germ feed stream was specified to match the rate of the total germ recovered from the hydrocyclone outlets. The solids and sugar content, and thus liquid density, were controlled by adding liquid feed streams added to the streams going to the hydrocyclone inlets. These added streams were varied to match experimental conditions using a FORTRAN block.

TABLE V
Hydrocyclone Model Fits[a]

	Germ (% of solid mass recovered)		Corn Mass Ratio	Feed Flow Entrained (solid mass fraction)	
Test	**Underflow**	**Overflow**	**Over/Under**	**Germ**	**Endosperm**
53a	3.00 (2.99)	11.4 (11.6)	0.0053 (0.0053)	0.975	0
53b	2.40 (2.40)	30.5 (29.7)	0.0068 (0.0068)	0.90	0.0005
53c	1.8 (1.8)	31.5 (31.5)	0.0070 (0.0071)	0.82	0.0007
53d	2.6 (2.6)	13.3 (11.6)	0.0100 (0.0100)	0.945	0.005
57a	2.3 (2.3)	8.1 (8.1)	0.0849 (0.0850)	0.77	0.064
57b	7.1 (7.07)	15.1 (15.2)	0.0343 (0.0344)	0.931	0.021
57c	4.8 (4.77)	12.4 (12.4)	0.0400 (0.0401)	0.906	0.0262
57d	4.5 (4.5)	16.2 (16.3)	0.0465 (0.0463)	0.857	0.0297
57e	7.6 (7.6)	23.6 (23.4)	0.0761 (0.0761)	0.810	0.050
58a	1.7 (1.7)	8.48 (8.49)	0.0695 (0.0696)	0.742	0.0509
58b	8.06 (8.06)	36.5 (36.5)	0.0259 (0.0259)	0.895	0.0082
58c	8.0 (8.0)	33.2 (32.8)	0.0316 (0.0316)	0.885	0.0132
58d	10.3 (10.3)	33.3 (33.3)	0.0266 (0.0265)	0.921	0.0100
58e	5.34 (5.34)	37.2 (37.3)	0.0201 (0.0201)	0.877	0.0038

[a] Calculated values using model in parentheses.

The particles in these streams were given the PSD produced by sieving the milled corn, with specific gravities of 1.05 for the germ, consistent with the germ separation liquid density used in wet milling (Blanchard 1992), and 1.30 for the endosperm. A perfect separation would be predicted using a two HYDROCYCLONE block model with solid streams suspended in a liquid of specific gravity only 0.0003 higher than the 1.0503 of the germ fraction (equivalent to a 14.31% dextrose solution). In these simulations the measured ground corn PSD was used.

As shown in Table II, only 0.4% of the corn is buoyant in a liquid with specific gravity of 1.05. Some of the corn dissolves during the 1-hr soak in dextrose solution, and although not experimentally determined (in fact this is difficult to determine without equipment to measure the particle sizes in suspension), a PSD change could be inferred from the modeling results. The model, even when modified as described later, predicted more endosperm mass in the overflow than was observed for the liquid specific gravity of 1.0503 and the PSD. With this liquid, only particles in the smallest size interval could be found in the overflow when the simulation was run with the presoaked PSD. Reducing the corn mass fraction in the smallest interval from 0.16 to 0.015 was the least change that enabled the model to match the experimental results. This smallest interval reduction is consistent with the observed 10–20% (filtered solid) corn mass lost as a result of slurry preparation. More particles would be reduced below the filter size by dissolution, and thus out of the smallest size range, than would fall into it by reduction of larger particles because of the large fraction originally in the smallest size interval.

To give each HYDROCYCLONE block a mass flow equal to the experimental (mixed) masses, the simulated feed stream masses were increased by a factor (using a second FORTRAN block) equal to the total corn fed to the hydrocyclone divided by the component (germ or endosperm) mass that the model was separating. For example, the germ hydrocyclone feed stream was increased by the inverse of the germ mass fraction of the corn feed to the real hydrocyclone. The outlet streams from that model were reduced by the same factors in a third FORTRAN block so that the outlet streams formed from the two model hydrocyclones would sum to the recovered mass flows.

We took the germ particle mass to be equal to twice the lipid mass determined by analysis of each sample. If the heavy particles consisted only of endosperm, hull, and tip cap, and the light particles only of germ, then the ratio of the two fractions should be much higher (87:13) (Wilson 1983) than we observed. To improve the fitting flexibility we extended the two-hydrocyclone model to include the effect of interparticle resistance to separation. The effect is simulated by bypassing streams from the endosperm (virtual) hydrocyclone inlet to the overflow and from the germ hydrocyclone feed to the underflow. These bypassing streams flow counter the direction expected on the basis of particle buoyancy when the liquid density is between that of the two particle densities. In the case of the germ fraction, bypassing might be caused by germ particles being trapped by the underflow, entrained by the more numerous endosperm particles. This type of particle interaction is absent for low solids feeds on which the ASPEN PLUS hydrocyclone block model is based. Separations with liquid density lower than the nominal germ density gave low yields of overflow product as well as low overflow germ content (only 0.67% of the recovered corn, with a germ content of 18.5%, when a 2.5% corn suspension in 14% glucose solution was fed). As shown in Table V, the best model fit for liquids with low buoyancy shows almost no endosperm bypassing, with most of the germ bypassing to the underflow. As the liquid density increases, more endosperm bypasses and less germ. The high solids feed tests with a dense liquid (57a and 58a) show a significantly greater endosperm bypassing, reducing the germ weight fraction of the overflow solids. This effect seems to be the main problem to be overcome if a high germ content fraction is to be economically recovered with a hydrocyclone from corn suspensions with a relatively high solids content. A flow diagram of the hydrocyclone model incorporated in a process to separate ground corn into endosperm and germ fractions without recycle of the glucose solutions is Fig. 1. A similar flow diagram with recycle is Fig. 2.

RESULTS AND DISCUSSION

This article describes a process consisting of milling, sieving, soaking, and hydrocyclone separation intended to produce a germ-enriched fraction suitable for hexane extraction and remaining corn fraction (grits) suitable for subsequent extraction with ethanolic solution. Combined dry-wet milling processes claim advantages accruing from elimination of the long steeping times and large amounts of process water needed for wet-milling (Powell and McGeorge 1975, Chwalek and Olson 1980). More recently, milling corn before steeping has been studied as a means of reducing steeping time (Eckhoff et al 1993). These methods were

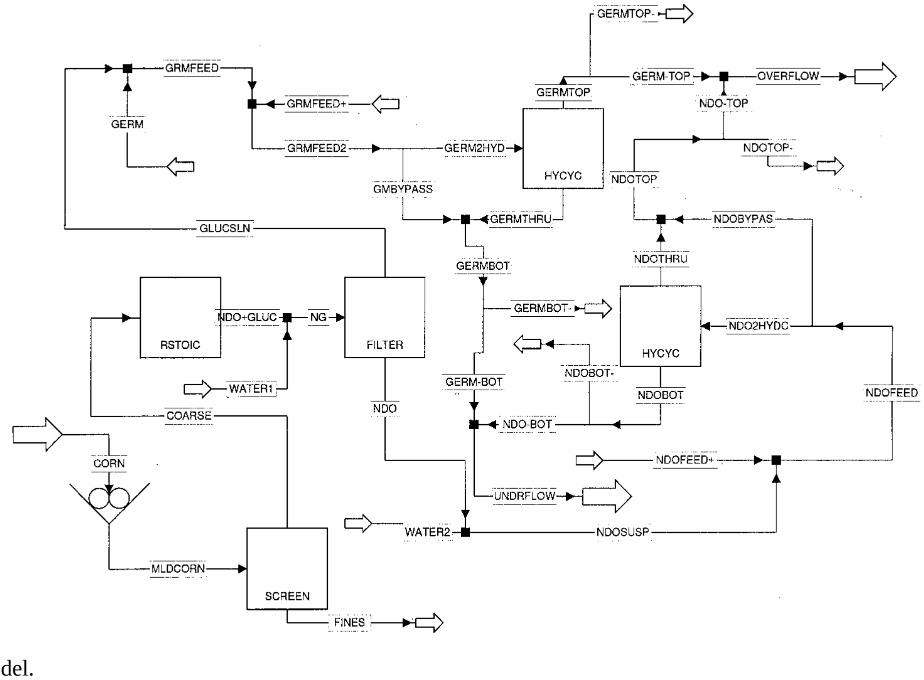

Fig. 1. Hydrocyclone model.

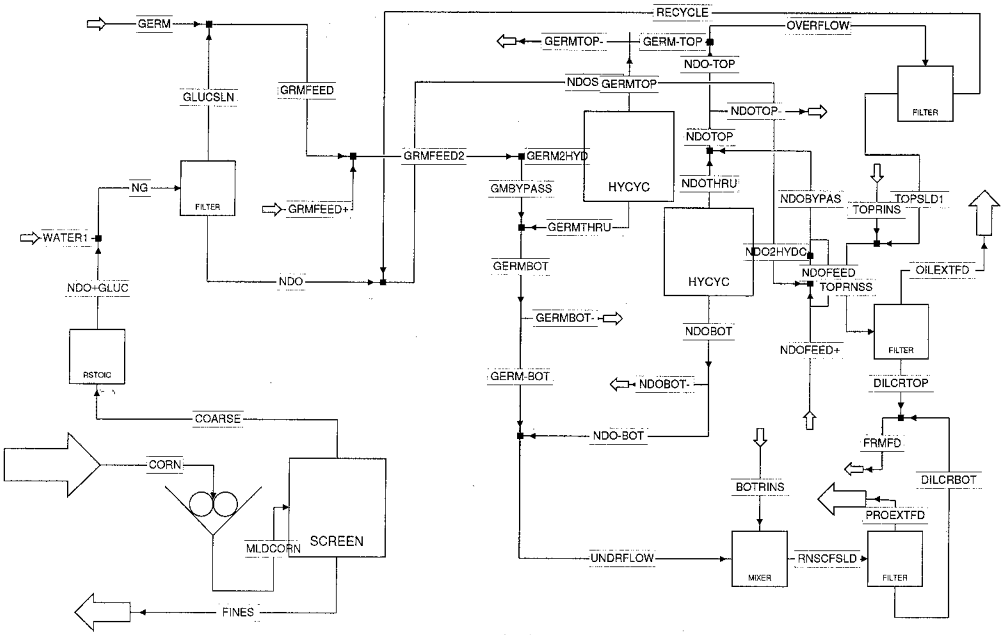

Fig. 2. Hydrocyclone model with liquid recycle.

not concerned with separating germ and preparing it for oil extraction. This work shows a slight germ enrichment of corn ground <1 mm with a single hydrocyclone. This size fraction is substantially finer than powder generated by usual milling processes, in which only ≈20% is <2 mm (Mistry and Eckhoff 1992). The germ fraction separated using a series of hydrocyclones from the 0.59- to 1-mm size fraction can be mixed with the dry <0.59-mm fraction separated by sieving before germ separation and potentially further separated to corn oil and starch or, ultimately, ethanol by conventional wet-milling processes. Conventional wet-milling practice (Blanchard 1992) separates a 45–50% lipid germ product by a sequence of primary grinding, primary hydrocyclone separation, screening, secondary grinding, secondary hydrocycloning, recycle to the primary grinding, and final screening. Germ recovery based on the procedure described here would include more types of equipment and loops if developed to commercial scale. Steady hydrocyclone capability to separate a solid mixture of particles of differing density can be estimated using experimental batch measurements to set the parameters for a simulation model consisting of hydrocyclone blocks (unit models) incapable, by themselves, of simulating particle sorting on the basis of specific gravity. Combining available, but inadequate, blocks and virtual separation of streams and blocks to simulate effects not entailed in the original blocks may be useful in other processes (especially solids handling) for which blocks are not yet available or are functionally incomplete.

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