Differential Settling Test for Evaluation of Liquid Cyclone Classification Performance

R. J. HRON, Sr., Southern Regional Research Center, Agricultural Research, Science and Education Administration, U.S. Department of Agriculture,[1] New Orleans, LA 70179

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ABSTRACT

A differential settling test (DST) developed by the Southern Regional Research Center utilizes small quantities of sample to assess the protein-classification efficiency of a commercial-sized liquid cyclone. The test can be applied to any grain or oilseed. In this study, cottonseed, corn germ, and unblanched peanut samples were comminuted, slurried with hexane, and liquid classified. Protein-classified and recovery data are shown comparing dry-pin and wet-stone milling methods as evaluated by the DST and a subsequent liquid cyclone process (LCP) test on identically treated material. Results of the DST showed a maximum protein concentration for cottonseed, corn, and peanuts of 68.67, 24.06, and 75.79%, respectively. The results of LCP tests were 67.30, 28.41, and 69.06%, respectively. LCP-processed unblanched peanuts, in addition to yielding a skin-free product approximating a protein concentrate, had a recovery rate of 73.8%, which suggests that the LCP is a possible commercial alternative to conventional solvent extraction methods for peanuts.

The differential settling process (DSP) is a procedure (Spadaro et al 1948; Vix et al 1949, 1951) for separating toxic gossypol-containing pigment glands from cottonseed protein. It led directly to the development of the continuous liquid cyclone process (LCP) (Gardner et al 1976a, 1976b; Gastrock et al 1971).

When the LCP was recognized as a potentially workable, economic process for preparing a protein concentrate from glanded cottonseed, the DSP, with refinements, was also recognized as an excellent means to simulate, and hence predict, large-scale LCP results. This article presents a comparison of results obtained with the refined bench-top differential settling test (DST) and with the large-scale LCP for cottonseed, dry milled corn germ, and unblanched peanuts.

The original DSP, as described by Vix et al, was designed for cottonseed flakes comminuted in a Waring Blendor in the presence of hexane. In the DST, grinding may be carried out either wet or dry, depending on the sample being tested. Samples with equilibrium (8%) or higher moisture content and lipids exceeding 45% usually plug most dry-milling equipment, necessitating a wet comminution. Oven-dried samples with moisture and lipid contents less than 3 and 45%, respectively, are generally friable and can be either dry or wet milled. To obtain efficient comminution, samples having hard hulls or husks must be physically cracked before being either wet or dry comminuted.

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MATERIALS AND METHODS

Materials

Cottonseed. Two types of cottonseed, glanded and glandless, were tested. The glanded cottonseed was obtained from Yazoo Valley Oil Mill, Greenwood, MS. The seeds were hulled in the pilot plant and dried from 7.4 to 1.6% moisture content with 82°C air for 80 min. Dried kernel samples containing approximately 2% hulls were prepared for the DST by being comminuted on an Alpine 160 Z laboratory model Kolloplex pin mill operated at a speed of 11,750 rpm and on a commercial 250 CW Alpine Contraplex pin mill operated at a door pin disk speed of 2,500 rpm and mill-side pin disk speed of 7,350 rpm.

The hulled, glandless cottonseed (Cot-n-nuts) was obtained from Rodgers Seed Co. through the Plains Cooperative Oil Mill, Lubbock, TX. The kernels were comminuted both on an as-is basis (6.19% moisture) and after being dried to 2.0% moisture by heating for 2.5 hr in a forced draft oven at 82°C. Comminution before applying the DST was done both on a Kolloplex pin mill at speeds of 11,750 and 14,600 rpm and on a Contraplex mill operated at 2,500 rpm on the door disk and 9,500 rpm on the mill-side disk. Double-pass milling of glandless seed was also evaluated with the

DST.

Pilot plant LCP runs were performed on 80-lb and 120-lb samples of glanded and glandless kernels, respectively. The dried kernels milled on the Contraplex pin mill (single pass) were slurried with hexane to produce a solids content of approximately 19% before feeding into the cyclone.

Corn Germ. Dry-milled corn germ (3.2% moisture) obtained from the Northern Regional Research Center, Peoria, IL, was dried for 35 min at 82°C to a moisture content of 2.0% and both wet and dry comminuted before being differentially settled. The wet comminution procedure consisted of flaking the germ to 0.006 in. in a Ross roller mill, fluidizing the flakes with hexane to produce a 40–45% solids slurry, and then passing the fluidized slurry through a Morehouse horizontal stone mill with a 0.003-in. clearance between stones. Dry comminution for both the DST and LCP was performed on the Alpine 160 Z Kolloplex pin mill operated at its maximum speed of 14,600 rpm.

Peanuts. Shelled, unblanched, full-fat Southeastern Runner peanuts were provided by Gold Kist, Inc., Atlanta, GA. The Pet Division of Quaker Oats, Co., Barrington, IL, provided shelled, unblanched, full-fat Southwestern Spanish peanuts. The Runners, containing 45.75% lipids, were dry comminuted in a Grindmaster model 490 PB peanut butter grinder with and without a discharge chute. Peanut samples were dry comminuted as is (8.0% moisture) and after drying for 4 hr at 82°C (3.6% moisture). Before wet comminution, the Southwestern Spanish peanuts, containing 45.35% lipids, were dried on a continuous pilot-plant belt dryer from 7.6% moisture to 2.6% with 82°C air. They were then cracked on Allis Chalmers cracking rolls at a setting of 0.090 in. and flaked to between 0.008 and 0.010 in. on a 18 × 24 Ross roll flaker. After hexane addition to produce a 45% solids slurry, wet comminution was performed using a model 530 Morehouse stone mill with a 0.002-in. clearance between stones. Two representative 118-cm^3 "ointment jar" samples were taken for the DST tests before dilution of the slurry to 19.1% solids with hexane and subsequent processing by the liquid cyclone.

Hexane. Hexane used in both the DST and LCP was Skellysolve B, a commercially available solvent high in normal hexane.

Methods

DST. The refined test was performed in duplicate. A portion (100 ± 0.1 g) of the prepared sample was weighed out (in the case of Morehouse-milled slurries, the sample size was an amount that would yield approximately the same total DST recovered solids (±10%) as a 100-g dry-prepared sample would yield) and transferred to a 1-L graduated cylinder with a stopper. Hexane was added to bring the level in the cylinder to approximately 500 cm^3 and the sample was shaken until thoroughly wetted. Additional hexane was added to bring the level of slurry to 1 L. The slurry was then mixed (Fig. 1) by slowly rotating the cylinder on its axis and simultaneously describing a larger elliptical pattern with the cylinder base as an apex. Mixing was continued for 3 min and the mixture then allowed to settle for 20 min. Settling time was varied from 15 to 25 min, depending upon the nature of the material being tested.

Samples that were previously solvent extracted tend to settle faster, probably because of lowered lipid content and partial denaturization and/or agglomeration of protein. At the end of the settling period, the supernatant later containing a suspension of finely divided solids was carefully siphoned, with suction into a 4-L filter flask, down to a level of 1 cm above the settled solids layer, using low-density polyethylene tubing with 3/16 in. ID. The procedure was repeated three times. The supernatant then approximated the "overflow" (OF) from a liquid cyclone. The coarse solids remaining in the cylinder were similar to "underflow" (UF).

The supernatant accumulated from the four settlings was reslurried and filtered with suction through tared Whatman No. 4 filter paper on a 90-mm Buchner filter funnel, and the resultant cake was washed with hexane three times. Each wash was approximately equal to the volume of the cake. The seeping of superfine particles under the edge of the filter paper into the filtrate was prevented by using a tared stainless steel ring to seal the edges. Rings 3/8 in. thick, cut from a stainless steel pipe 3-in. in diameter, were polished and made excellent seals. The cake, filter paper, and ring were air dried on a tared pan to evaporate excess solvent (approximately 2 hr in a vented hood), then oven dried at 101°C for 1 hr. The coarse solids remaining in the graduate were filtered, washed with hexane, and dried in the same manner to recover the UF solids. The percentage of OF and UF material was calculated.

Liquid Cyclone Process. Large-scale batch runs (80–120 lb samples) were conducted in the LCP equipment shown in Fig. 2. The process consisted of mixing the prepared material with metered hexane in the feed tank and pumping the resulting slurry, under pressure, tangentially into the cyclone. The slurry was split in the cyclone into OF and UF fractions, which were then directed to drum receivers. A series of OF and UF samples reflecting a broad cyclone-classifying range were taken simultaneously. The solids

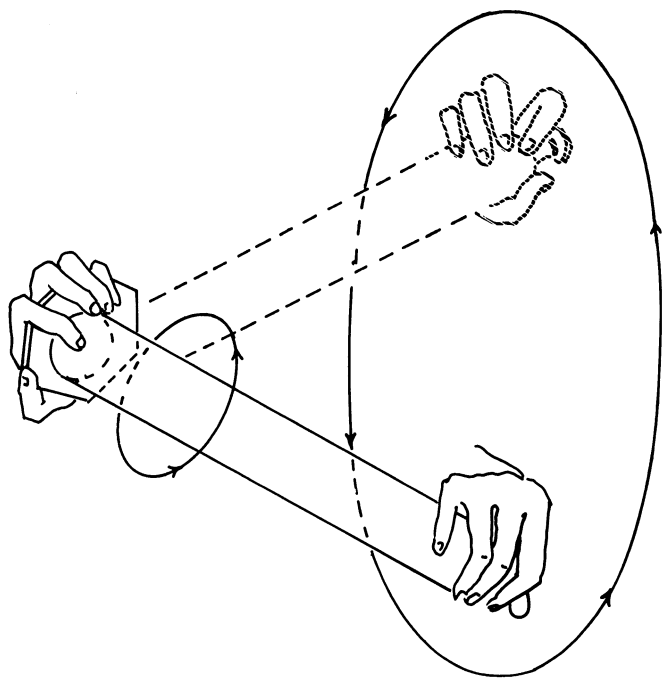

Fig. 1. Method of mixing a sample in a laboratory differential settling test.

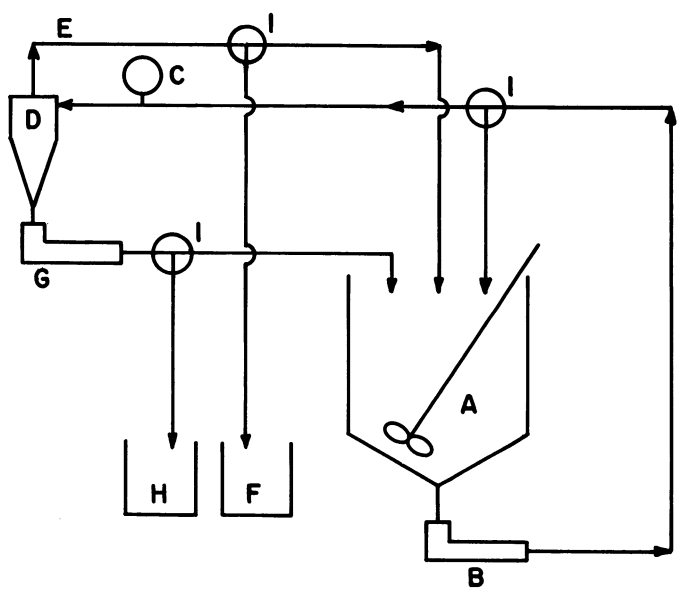

Fig. 2. Pilot-plant liquid cyclone process flow sheet. **A,** Feed tank with mixer; **B,** feed pump; **C,** feed pressure gauge; **D,** 3-in. diameter liquid cyclone; **E,** overflow fraction; **F,** overflow collection drum; **G,** underflow pump; **H,** underflow collection drum; **I,** three-way valves.

from these slurry samples were recovered in the laboratory by the same technique as for the DST. In this manner, predictive data obtained by the DST could be compared with actual on-stream LCP operation on identical material.

Analytical Methods. Moisture, lipids, free and total gossypol, nitrogen, fiber, and ash were analyzed according to official AOCS methods (1965).

RESULTS AND DISCUSSION

The most recent laboratory sedimentation or gravity classification techniques developed by Finley (1976), Cluskey et al (1978), and Clements (1979) produce protein concentrates from cereal products by using fluorinated hydrocarbons. Although these sovents are easily evaporated from resulting products, the products have not yet been proven safe to eat. In contrast, the LCP uses hexane, which also is easily evaporated and is approved for food processing operations (extractions of oilseeds). Because the methods of Finley, Cluskey et al, and Clements depend essentially upon density differences, their classification efficiencies can be seriously affected by high-lipid samples like oilseeds.

Although density differences exert some effect in both the DST and LCP, particle size and the attendant frictional resistance are the major factors in separating gossypol-pigment glands from cottonseed protein. The density of pigment glands (1.36 g/cm^3) is lighter than that of kernel tissue (1.41–1.44 g/cm^3) (Vix 1951), but in a hexane-lipid medium the glands settle out faster than the proteinaceous and/or cellular material. This settling is because of the high frictional resistance imparted by the large surface area per unit weight of both the mesophyll cells, 20–40 μm in diameter, and the individual aleurone grains or protein bodies, 4–6 μm in diameter. Pigment glands average 100–200 μm in diameter and, because of the size differential between them and the remaining tissue elements (Fig. 3), some mechanical fragmentation of glands can be tolerated. Yatsu et al (1974) found that pigment glands contain a matrix of pigment spherules 0.1–1.5 μm in diameter. He also found that if this matrix is maintained intact during processing and the spherules not dispersed, a successful separation of gossypol can be obtained. The relative effectiveness of the DST and the LCP in separating pigment glands and classifying protein can be seen in Table I.

Free gossypol levels of 0.03 and 0.04% for the overs protein concentrates obtained from the DST and liquid cycloning of glanded seed reflect a 96.9 and 95.8% reduction in free gossypol, respectively. Both the DST and the LCP produced an OF exceeding 66% protein composition in both glanded and glandless

TABLE I
Solids and Protein Recoveries (%) from Differential Settling Test (DST) and Liquid Cyclone Processing (LCP) of Glanded and Glandless, Full-Fat Cottonseed Kernels

Sample and Process	Classified Fraction	Solids Recovery	Protein		Gossypol	
			Content[a]	Recovery	Free	Total
Glanded cottonseed kernels	...	...	60.16	...	0.96	1.05
DST						
Kolloplex milled	Overs	19.6	67.58	21.8	0.03	0.04
	Unders	80.4	59.12	78.2	1.82	2.09
Contraplex milled	Overs	25.8	67.75	28.0	0.04	0.07
	Unders	74.2	60.42	72.0	1.82	2.28
LCP[b]	Overs	44.3	66.53	48.3	0.04	0.05
	Unders	55.7	56.49	51.7	2.45	2.52
Glandless cottonseed kernels	...	...	59.69	...	0.01	0.02
DST						
Kolloplex milled						
Single pass	Overs	34.7	68.67	37.4	0.00	0.01
	Unders	65.3	61.11	62.6	0.06	0.07
Double pass	Overs	75.7	67.48	80.4	0.00	0.01
	Unders	24.3	51.70	19.6	0.18	0.20
Contraplex milled						
Single pass	Overs	38.0	67.80	41.2	0.00	0.00
	Unders	62.0	59.47	58.8	0.09	0.11
Double pass	Overs	43.2	67.72	...[c]	0.01	0.01
	Unders	56.8	...[c]	...[c]	...[c]	...[c]
LCP[d]	Overs	55.9	67.30	62.2	0.01	0.01
	Unders	44.1	51.72	37.8	0.12	0.17

[a] Percent protein on a moisture-free and oil-free basis, N × 6.25.
[b] 18.8% Contraplex-milled feed slurry solids.
[c] Data not available.
[d] 19.1% Contraplex-milled feed slurry solids.

TABLE II
Solids and Protein Recoveries (%) from Differential Settling Test (DST) and Liquid Cyclone Processing (LCP) of Dry and Wet Comminuted Corn Germ

Sample and Process	Classified Fraction	Solids Recovery	Protein		Fiber	Ash
			Content[a]	Recovery		
Corn germ	...	...	22.87	...	3.5	7.05
DST						
Kolloplex dry milled	Overs	52.1	23.98	55.6	3.6	9.43
	Unders	47.9	20.88	44.4	6.1	4.90
Morehouse wet milled	Overs	41.4	24.06	43.7	3.2	13.92
	Unders	58.6	21.98	56.3	6.3	6.20
LCP[b] Kolloplex dry milled	Overs	36.0	28.41	46.2	4.0	16.10
	Unders	64.0	18.60	53.8	5.2	5.04

[a] Percent protein on a moisture-free and oil-free basis, N × 6.25.
[b] 20.7% Kolloplex-milled feed slurry solids.

seed. Solids and protein recoveries for overs or concentrate fractions obtained from the DST and liquid cycloning of glanded seed are considerably lower than those for glandless seed. This difference is because of the prime restriction of minimizing gland breakage during comminution of glanded seed in order to produce an edible concentrate. Once this restriction can be ignored, as in the case of the glandless seed, multiple milling produces significantly higher overs solids and protein recoveries. The reporting of small amounts of gossypol in glandless kernels and various fractions reflects glanded kernel contamination in the original sample. The lipid content of glanded and glandless kernels was 32.14 and 32.69%, respectively. Because lipid levels for the various classified fractions were below 1%, they were not reported.

Data from initial differential settling of dry and wet (in hexane) comminuted corn germ and from a liquid cyclone run are given in Table II. Because of the fast-settling nature of the wet-milled and dry-milled test samples, the DST was run at 15-min settling times. Because dry Kolloplex milling yielded essentially the same protein content as wet milling but with higher product recoveries, dry milling was chosen as the method of comminution for the liquid cyclone. Liquid cycloning yielded a solids recovery of 36.0% with a small but definite protein classification of 28.4%. The fast settling rates experienced on the bench-top tests were also evident in the operation of the cyclone.

The cyclone was operated at the maximum flowable UF solids concentration of approximtely 54% for this product. Further increased OF solids recoveries could not be expected without major changes in cyclone operating conditions and the use of larger cyclones. Other or additional comminution methods would also have to be thoroughly investigated in order to improve solids recovery and protein classification simultaneously. The concentration of minerals, 16.1% ash in the OF LCP fraction, is not restricted to liquid classification. Stringfellow et al (1977) also reported it in air-classification studies of defatted corn germ flour. In the present study, the initial lipid content of 23.26% was reduced to less than 0.3% in all processed samples.

The ability of the bench-top DST to forecast liquid cyclone protein classification is best shown in Table III. The DST on a dried (2.6% moisture), wet-milled sample of Southwestern Spanish peanut kernels yielded essentially the same total overs or product solids recovery and protein concentration as did the pilot-plant liquid cyclone run. Peanuts contain approximately 45% oil and, because of lipid extraction, undergo an additional disintegration during the DST and liquid cycloning. The production of extra fines along with extracted lipids affects the settling resistance of the resulting slurry during the DST and liquid cycloning. Kernel drying along with wet comminution further accentuates the restricted settling effect to the point that even after a 25-min settling time only 26% of a wet-milled Southwest Spanish peanut sample had settled out.

The slow settling effect was also reflected in an excessively large fine solids concentration (+16%) in the OF slurry obtained from

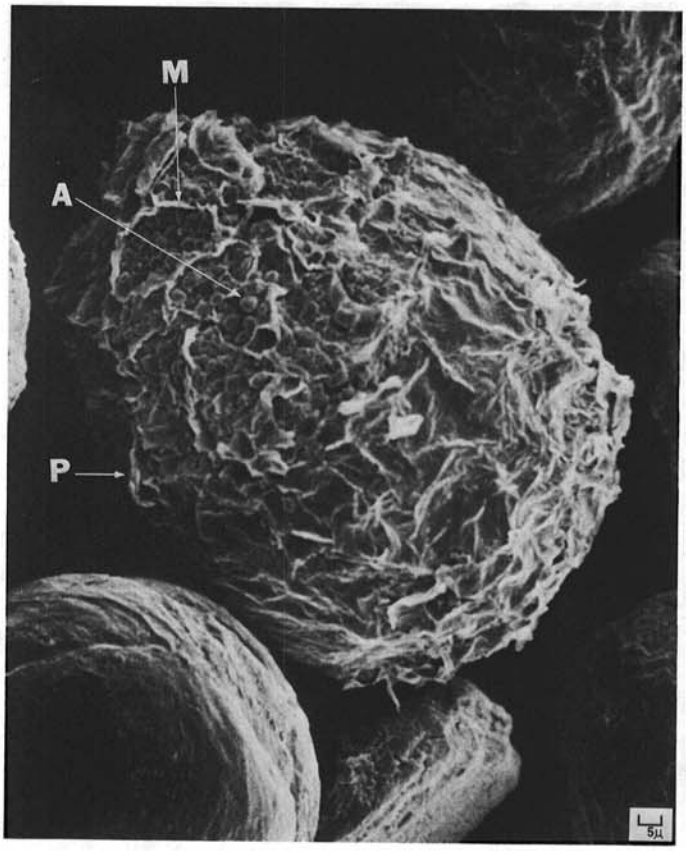

Fig. 3. Scanning electron micrograph showing relative sizes of a pigment gland **(P)**, mesophyl cell **(M)**, and aleurone grain or protein body **(A)**.

TABLE III
Solids and Protein Recoveries (%) from Differential Settling Test (DST) and Liquid Cyclone Processing (LCP) of Shelled, Unblanched, Full-Fat Peanut Kernels

Process	Sample	DST Settling Time (min)	Classified Fraction	Solids Recovery	Protein Content[a]	Protein Recovery	Fiber	Ash
DST	Southeast Runner kernels "As is"	...	...	...	59.70	...	4.5	2.95
	Dry grind[b]	15	Overs	46.9	74.44	56.6	1.1	5.84
			Unders	53.1	50.48	43.4	7.2	5.01
	Wet grind	20	Overs	33.7	75.79	41.7	1.2	5.94
			Unders	66.3	53.87	58.3	6.0	4.95
	Dried, dry grind[c]	20	Overs	78.7	66.91	85.2	3.1	5.77
			Unders	21.3	42.85	14.8	8.6	4.64
	Southwest Spanish kernels	...	...	...	59.45	...	5.4	2.38
	Dried, wet grind[d]	25	Overs	74.0	71.81	82.0	...[e]	...[e]
			Unders	26.0	44.88	18.0	...[e]	...[e]
LCP[f]	Southwest Spanish kernels Dried, wet grind	...	Overs	73.8	69.06	78.3	3.3	4.77
			Unders	26.2	53.94	21.7	7.5	4.58

[a] Percent protein on a moisture-free and oil-free basis, N × 6.25.
[b] 8.0%-moisture kernels ground through Grindmaster mill.
[c] 3.6%-moisture kernels ground through Grindmaster mill.
[d] Morehouse milled.
[e] Data not available.
[f] 19.1% Morehouse-milled feed slurry solids.

liquid cycloning of this product. This result compares to 10, 12, and 14% solids in OF obtained from cycloning roughly equivalent feed solids of corn germ and glanded and glandless cottonseed, respectively. The 33.7% overs solids recovery from the DST on as-is (8.1% moisture), dry-ground, Southeastern Runner peanuts shows the effect of moisture on overs recovery. Drying these kernels to 3.6% moisture more than doubled the overs solids and protein recoveries. However, some protein content was sacrificed. All of the overs samples contained less then 1% lipids and were white without a trace of skins.

Although the DST is normally run in duplicate, statistical analyses of 35 duplicate sets of DSTs run on different materials comminuted in various ways indicated a Fishers least significant difference for the method of 2.38 at the 95% confidence level.

The refined DST is a valuable laboratory technique for predicting the classification potential of the LCP. Generally the LCP results in both higher recovery and higher protein concentration than the DST on identically processed material. The difference is attributable to the high shear (increased comminution) and centrifugal (improved classification) forces developed within the liquid cyclone. Besides demonstrating the effectiveness of the LCP in concentrating protein and separating pigment glands in cottonseed, the DST showed that unblanched peanuts are also particularly suitable for the LCP. This result suggests the LCP as a possible alternative method for producing solvent-extracted peanut concentrates.

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