Total Oil Analysis of Soybeans by Simultaneous Grinding and Solvent Extraction[1]

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A new, rapid method for total oil analysis of soybeans is proposed in which ground soybeans are mixed with solvent and further ground in a closed homogenizer until the oil is completely dissolved. A sample of the slurry can then be filtered and the oil content determined by evaporating the solvent from a measured amount of miscella. Parameters studied included times and speeds needed to produce sufficiently small particles so that all the oil was dissolved. The new analysis compared favorably with conventional Goldfisch extraction for total oil of soybeans. Preliminary experiments indicated that rapeseed could be extracted by the new procedure without preliminary grinding.

KEY WORDS: Grinding, oil analysis, solvent extraction, soybeans.

Recent research has indicated that the usual analytical procedure for total oil in soybeans can be improved by extracting fine flours (100 mesh) by a rapid equilibrium procedure (1,2). The improvement is more oil extracted in a shorter time. However, to prepare 100-mesh full-fat soybean flour, it is necessary to sieve with a special device (Alpine Air Jet sieve, Alpine American Company, Natick, MA).

If an initial rough grinding could be done followed by breakup into fine particles in the presence of solvent, the sieving problem could be eliminated. The Swedish method, in which steel balls in steel centrifuge tubes are used to breakup soybeans in solvent, is one such approach (3,4), but the time involved is 1 hr, and there was no measure of particle size achieved.

We attempted to solve this problem by roughly grinding soybeans and then simultaneously grinding and extracting the soybean tissue in a closed homogenizer. This paper reports the results of experiments with that procedure.

MATERIALS AND METHODS

The soybeans used in all experiments were of the cultivar Forrest. Solvents used were high performance liquid chromatography (HPLC)-grade hexane and ligroine (boiling range 90–110°C).

Soybean preparation. To facilitate dehulling, soybeans were heated at 70°C for 1 hr. After cooling, the soybeans were broken up in a blender, and the hulls were removed by aspiration. The dehulled soybean pieces were ground in a UDY Cyclone mill without a screen (UDY Corp., Fort Collins, CO). The flour from this grinding was used as the sample for simultaneous grinding and extraction. For Goldfisch extraction, the ground flour was sieved in an Alpine Air Jet sieve, and the fraction passing 100 mesh was used.

Soybean extraction: Simultaneous grinding and extraction. A 1-g flour sample was added to hexane in the closed homogenizer vessel (Omni mixer homogenizer, Omni International, Inc., Waterbury, CN) and homogenized at the appropriate speed and time. Speeds were designated on a scale of 1 to 10 with 10 being 20,000 rpm. After homogenization, the slurry was allowed to settle for 10 min. The miscella was poured into the barrel of a 50-mL syringe and pushed through a 0.8-μm Millex-PF filter (Millipore Corp, Bedford, MA) into an Erlenmeyer flask and immediately stoppered.

Samples of 5 mL of filtered miscella were pipetted into tared aluminum pans and the solvent evaporated at 120°C for 15 min. The recovered amount of oil was used to calculate the oil extracted. A correction was made for the increase in total volume resulting from oil dissolving in the solvent.

Particle size was determined by evaporating solvent from the flour remaining in the homogenizer vessel and sieving a weighed sample through a 100-mesh sieve with the Alpine Air Jet sieve. The percentage of the sample not passing the sieve was determined. The original soybean flour from the UDY Cyclone mill had 30% of particles larger than 100 mesh.

The procedure for rapeseed did not involve any seed preparation. The homogenization was at speed 5.5 for 3 min and at speed 8 for an additional 5 min. The solvent for rapeseed was ligroine.

Goldfisch extraction. The AOCS official method AC 3-44 (5) was used for soybeans. For rapeseed, the United Oilseed Product Inc. (UOPI) method was used, which is a 4-hr extraction followed by regrinding and a second 4-hr extraction. The rapeseed for the UOPI procedure was ground in a coffee mill for 1 min.

All extractions were done in triplicate, and mean values are reported. Statistical evaluations for least significant differences were done by Statistical Analysis System (SAS).

Moisture adjustment. The flour coming from the UDY Cyclone mill was adjusted to different moisture contents by incubating it in a closed desiccator over water for varying times.

RESULTS AND DISCUSSION

The Omni homogenizer can be fitted with a blade or a rotor generator shaft (rotor-stator arrangement with the suspension of particles circulated between the rotor and stator). In preliminary experiments we found that the rotor generator shaft was much better at breaking up soybean particles than the blade, and so all subsequent experiments were done with the rotor generator shaft.

In the rapid equilibrium method for soybean analysis, maintaining the original volume of hexane is essential to accurate results (2). This means that evaporation has to be controlled, and the temperature difference between the original pipetting of solvent and the final pipetting of sample should be minimal. To determine the extent of

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evaporation of solvent and the extent of temperature changes resulting from the use of the homogenizer, a control experiment was done. Approximately 0.2 g of oil was measured accurately and dissolved in solvent to simulate extraction from 1 g of soybean flour. The miscella was placed in the closed homogenizer vessel and treated as if an extraction were taking place.

The results shown in Table 1 indicated that a temperature increase of 5°C was possible over 8 min of homogenizer operation. However, the 5°C increase gave the same percentage increase in oil as a negligible temperature increase (25.3 to 25.5°C). Therefore, pipetting errors due to temperature differences did not cause fluctuations in the oil measurement. The 0.25% increase that was found for a known amount of oil was assumed to be due to evaporation of solvent during the homogenization and subsequent analysis. The comparison of hexane (boiling point 69°C) to ligroine (boiling range 90-110°C) showed significantly less increase in percentage oil with ligroine as solvent. The lower percentage increase with ligroine was probably due to less evaporation. This would make ligroine the preferred solvent if evaporation were a serious problem, but we chose to use hexane as the solvent for the remaining experiments because of its almost universal use in soybean oil extraction.

To determine the optimum volume of solvent to use for extracting oil and for breaking up soybean tissue, a series of experiments was done at a homogenizing time of 3 min, and the results from speeds 4.5, 6, and 8 were pooled. Particle size reduction was measured as the percentage of particles that would not pass a 100-mesh screen. Earlier results (1) showed that particles smaller than 100 mesh rapidly equilibrate with solvent to release oil. As shown in Table 2, a volume of 20 mL was optimal with respect to oil extracted and with respect to particle size reduction.

The optimum time for extraction and for reduction of particle size was investigated by using 20 mL of solvent, speed 8, and 1 g of soybean flour. The results shown in Table 3 indicated that at 5 min maximum oil had been extracted and maximum breakup of particles had been achieved.

The speed of rotation of the rotor generator shaft was another variable that needed to be investigated with respect to oil release and particle reduction. Table 4 shows that at low and high speeds there is less oil release and less particle reduction. A speed of 8 was chosen as optimal for oil release.

The moisture content of fine flours can vary considerably based on the relative humidity in the laboratory (6). We extracted soybean flour that had been adjusted to different moisture contents from 4.2% to 8.3%. The results show that percentage oil increased with increasing moisture (Table 5). The likely cause of the increased oil is increased phospholipid extraction (2).

All of the results presented to this point were with dehulled soybeans. For comparison of the simultaneous homogenization procedure with the traditional Goldfisch extraction, both dehulled and nondehulled soybeans were used. Table 6 shows results comparing the two types of analyses. For dehulled soybeans the analyses were very close: 22.7% *vs* 22.5% for the Goldfisch and simultaneous homogenization procedures, respectively. For nondehulled soybeans there was 1.6% difference between the two types of analyses. A possible explanation for this discrepancy is that the ground soybeans were sieved to give a 100-mesh fraction for Goldfisch analysis. The sieving will tend to exclude hull particles from the fine flour (1), and thus there may not have been as much hull

TABLE 1

Effect of Temperature and Solvent on Oil Released (homogenization for 8 min at speed 8 in 20 mL solvent)[a]

Solvent	Solvent temperature (°C)	Miscella temperature (°C)	Increase in % oil
Hexane	22	27	0.25 a
Hexane	25.3	25.5	0.25 a
Ligroine	24.1	27	0.18 b
Ligroine	26	27.4	0.16 b

[a]Means with the same letter are not significantly different at the 5% level.

TABLE 2

Effect of Solvent Volume on Oil Release and Particle Size[a]

Volume (mL) of hexane	% Oil	% Particles (> 100 mesh)
10	21.5 b	7.8
15	21.9 a,b	2.9
20	22.2 a	2.0
25	22.1 a	3.2

[a]Means with the same letter are not significantly different at the 5% level.

TABLE 3

Effect of Time of Homogenization on Oil Release and Particle Size (speed 8, 20 mL hexane)[a]

Time (min)	% Oil	% Particles (> 100 mesh)
2	20.8 a	13.3 d
3	21.8 b	1.9 e
4	21.5 b	7.7 e
5	22.3 c	0.5 f
6	22.3 c	0.3 f
7	22.0 c	0.4 f
8	22.5 c	0

[a]Means within vertical columns with the same letter are not significantly different at the 5% level.

TABLE 4

Effect of Rotor Speed on Oil Release and Particle Size Reduction (time of homogenization 8 min; 20 mL hexane)[a]

Speed	% Oil	% Particles (> 100 mesh)
4		9
4.5	21.7 b	
5.5		2.5
6	22.0 a	
7.5		1.1
8	21.9 a	
9		7.8
9.5	21.5 b	

[a]Means with the same letter are not significantly different at the 5% level.

TABLE 5

Effect of Moisture on Oil Release and Particle Size Reduction (time 8 min; speed 8; 20 mL hexane)[a]

% Moisture	% Oil	% Particles (> 100 mesh)
4.2	21.6 a	0
5.6	22.2 b	0
6.1	22.5 b	0
7.4	22.6 b	0.03
8.3	22.8 c	0.06

[a]Means with the same letter are not significantly different at the 5% level.

TABLE 6

Effect of Hulls on Oil Release by Two Procedures[a]

Procedure	% Oil with hulls	% Oil dehulled
Goldfisch	21.0	22.7
Homogenization	19.4	22.5

[a]Parameters for homogenization: speed 8; time 8 min; 20 mL hexane.

material in the flour analyzed by Goldfisch as in the flour analyzed by simultaneous homogenization.

The simultaneous homogenization procedure was tried on rapeseed without prior grinding. The small size of the seed allowed grinding and extraction in the homogenizer. Triplicate analyses gave a mean value of 40.9% oil for simultaneous homogenization compared to 40.4% oil by the United Oil Seed Product Inc. (UOPI) method (an 8-hr conventional extraction with regrinding at 4 hr).

In conclusion, it was found that simultaneous grinding and extraction of preground soybean flour in a closed homogenizer was a successful alternative to grinding and sieving soy flour. Conditions for simultaneous homogenization were speed 8, 20 mL of hexane with 1 g of flour, and 5-min extraction time. If soybeans are not dehulled, care should be taken to assure an even distribution of hulls in the sample analyzed. It was possible to analyze rapeseed by the simultaneous homogenization without pregrinding.

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