Letters to the Editor

Extraction and Diffusion of Corn Germ and Corn Germ Press Product by a New System

Sir:

Gulbaran (1-3), in his research on solid materials extraction, has developed a different method which can be applied to the countercurrent principle and to the conventional presses which are still in use. Using this new method, he has obtained a higher yield and better technical advantages.

DESCRIPTION AND MECHANISM OF THE EXTRACTOR USED

The extractor used for the extraction of solid materials is shown in Figure 1. It is made of the glass cylinders (E,E_1), one inside the other, the heights of which are 40 cm. The diameter of the cylinder containing the material is 3.5 cm, and the diameter of the exterior cylinder which functions as a water jacket is 5.0 cm. A condenser (g) is placed at the top. There is a round plate (c) perforated like a sieve and made of stainless steel at the lower part of the column carrying the material. The plate is covered with a fine sieve cloth. The function of this cloth is to avoid the passing of solid particles below. There is a ring (b) on top of this plate. The function of this ring is to press down the plate (c). This ring is filled with glass ball bearings (e) with a diameter of 2-3 mm. Raw material (f) to be extracted is filled into this column by pressing slightly with a glass pipe which is flat and wide on the lower part. Again, glass ball bearings (e) of a diameter of 2-3 mm are put on top of the material. Then a perforated, flat plate shaped like a wheel (d) is placed on top of the ball bearings. The function of this plate (d) is to spread the solvent dripping from the top homogeneously on the material and to avoid the formation of cavities within the material which can bulge out from the pressure of gas formed during extraction.

A condenser (g) is placed on the extractor to return the evaporated solvent. The solvent tank (h) with the condenser (g) at the top is heated with a thermostated heater (i). In order to hold the system at the desired temperature, a thermostat (T) sends the water heated at a certain temperature to the extractor by means of path no. 1. Taps are shown in the figure with the symbol (J). Miscella is collected through the lower tap (J) into a graduated cylinder (k).

OPERATION OF THE EXTRACTOR AND CARRYING OUT THE EXPERIMENTS

Corn germ (75 g) obtained from material ground and sifted or pressed from corn germ is mixed with 7 cc of technical hexane (Iprahex) heated at a temperature of 62 C with a glass tube in a beaker. The height of the material loaded in the extractor is ca. 20 cm. The solvent heated at 62 C is dripped on the material with regular intervals by slightly opening the solvent tap (J) of the tank (h) heated at a boiling point and placed below the condenser (g) which is found on the top part of the extractor. Miscella is collected in the three graduated cylinders below (k), first in an amount of 100 cc, then of 50 cc, followed by another at 50 cc.

No pools of hexane must ever be allowed to form on top of the material during extraction and the dripping rate must be adjusted in such a way that hexane dripping onto the material is immediately absorbed by the material. Hexane residues on top of the material is against the principles of our extraction system and elution theory (2); such an operation is excluded in this system.

All the extraction experiments in this study have been carried out under these conditions and have always been repeated twice. The average values obtained in these experiments are indicated in the study.

MATERIALS

Corn germ and corn germ press products used in our research were commercial products supplied by the corn starch plant at Vanikoy in Istanbul. Corn germ press product is obtained when some amount of oil is taken off while the corn germ is pressed. One kg of each material was ground in the MLW (Veb Kombinat Medizin und Labor-Technik, Leipzig) mill.

The solvent used for extraction is produced by Istanbul Petroleum Refinery Association Corporation under the

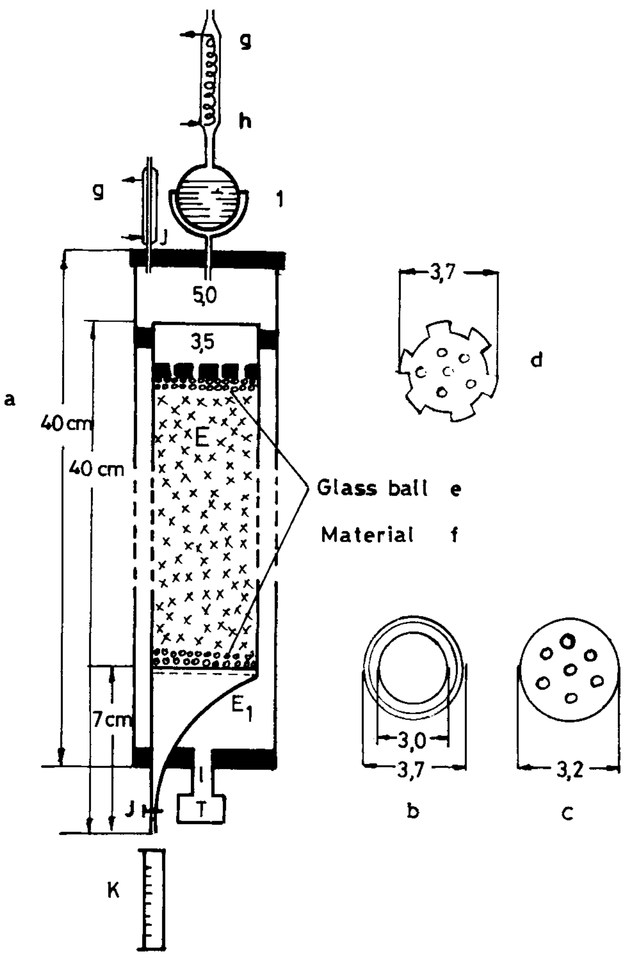

FIG. 1. Extractor for solid materials (E. Gülbaran, German Patent 1,024,269).

TABLE I

Determination of Optimal Extraction Temperature

Expt. no.	Operation temp. (C)	Oil yield (% based on dry material)
1	18	24.47
2	30	28.21
3	40	35.96
4	50	41.05
5	62	47.43

name of Iprahex, a technical hexane, for which the boiling point is between 60 and 70 C.

DETERMINATION OF THE OPTIMAL OPERATION CONDITIONS FOR THE EXTRACTOR

Samples of ground corn germ (75 g) obtained from sieve no. 1.00 (1,000 μ) of the German DIN were extracted for a period of 50 min at various temperatures. The extractor was held at constant temperature throughout the experiments by means of the thermostat (T) and the water jacket connected to it. Oil yield obtained in these experiments is shown in Table I. Experiment 5 shows that the optimal extraction temperature is 62 C, which is around the boiling point temperature of the solvent. In comparison, an average of 54.06% of oil was found using the Soxhlet.

In order to determine the optimal size of the particles, experiments were done on the extraction of particles of various sizes 1,000, 630, and 315 μ at 62 C, and the respective oil yields determined.

Oil yield was 93.9% on average compared to Soxhlet in 8 pairs of experiments done with the product obtained from sieve no. 1.00 (1,000 μ) in an extraction period of 80-160 min. Average percentages of ash and moisture in the corn germ were 0.71 and 6.86, respectively. Hexane/material ratio has been 1.325 kg/1 kg.

Eleven pairs of experiments were made on corn germ obtained from the product of the sieve no. 0.63 (630 μ) in 83-138 min. In all the samples, the average ash was 0.70% and the average moisture was 9.23%. Oil yield compared to Soxhlet averaged 92.04%. The average of the hexane/material ratio of the experiments was 1.336 kg/1 kg.

Twelve pairs of extraction experiments were made on the products of sieve no. 0.315 (315 μ) in 65-120 min. Optimal extraction period was 90 min, and oil yield was 102.85% compared to Soxhlet. The average ash and moisture were, respectively, 0.7 and 7.76%. Oil yield based on dry material in all these experiments compared to Soxhlet averaged 100.93%. The average of the hexane/corn germ ratio of the experiments was 1.299 kg/1 kg.

All the experiments have demonstrated the fact that a greater oil yield compared to the yield by Soxhlet was obtained from the corn germ derived from the products of the 315 μ. This is the optimal size for particles in our extraction system, and we have found experimentally in this new system that oil yield does not always increase when the particle sizes diminish, and that sometimes, it even decreases.

It should not be surprising that the yield is slightly higher than the yield obtained by the Soxhlet extractor, which is used internationally. This difference is due to the fact that this new extractor operates with a highly different system than that of the Soxhlet. In this system, each particle of the material is in continuous contact with pure solvent in every stage of the extraction and the concentration difference between the material and the solvent is kept very wide throughout the extraction period compared to the difference in the Soxhlet. Thus, extraction is done in a shorter period. It had already been explained in the elution theory of Gulbaran (2) that this new extractor would give such results.

EXTRACTION EXPERIMENTS WITH PRESSED CORN GERM CAKE

Extraction experiments on corn germ press product were done under exactly the same conditions as the ones applied in the previous experiments.

Experiments were made on the first press cake of corn germ obtained from sieve no. 1.00 (1,000 μ) in various different extraction periods. Average percentages of ash and moisture were, respectively, 1.11 and 8.59 in the samples. An oil yield of 24.85% based on dry material was obtained using the Soxhlet extractor. The average moisture was 6.27%.

Total oil based on dry material was 23.28% as an average of the eight pairs of experiments. The ratio of the solvent consumed for these experiments to the amount of raw material is 1.569 kg/1 kg; the average oil yield of the eight experiments is 93.69% compared to Soxhlet.

Extraction experiments made on the corn germ first press cake obtained from no. 0.63 (630 μ) were completed in an extraction period of 69-140 min. The samples had 6.27% moisture and 1.14% ash on average. Oil yield from Soxhlet extraction averaged 24.06% based on dry material.

Oil based on dry material was 22.63% in 8 pairs of experiments. Oil yield compared to Soxhlet was 94.05%. The ratio of hexane to material was 1.575 kg/1 kg.

Eleven pairs of extraction experiments made on the first

TABLE II

Physical and Chemical Characteristics of Raw Oil

Type of oil	Free oleic acid (%)	Peroxide number	Iodine number	Saponification number	Reflux index abbe (20 C)	Spec weig (15 C)	Color (x)[a]
Reference (4)			117-123	188-193	1.473-1.476	0.920-0.928	
Corn germ by extractor	0.58	1.18	120.77	192.18	1.4742	0.9250	4
1st press cake, by extractor	0.69	1.47	118.36	191.76	1.4746	0.9261	14
Corn germ by Soxhlet	0.62	1.21	119.99	192.01	1.4754	0.9258	4
1st press cake, by Soxhlet	0.71	1.56	118.04	192.07	1.4757	0.9265	14

[a]x = Colorimetric color determination, according to the potassium dichromate scale (5).

press cake obtained from sieve no. 0.315 (315 μ) were completed in 76-159 min. Oil (20, 43%) based on dry material has been obtained on average from this material in Soxhlet analysis. Average moisture of the material was 9.26%, and average ash was 1.10%. Average oil yield of the first press cake obtained from sieve no. 0.315 (315 μ) was 100.18% compared to Soxhlet.

The ratio of the solvent to the material was higher in the experiment made on corn germ press cake compared to the ones made on corn germ. It was 1.594 kg/1 kg on average. These results were due to the fact that while the corn germ press cake was being pressed, its particles were subject to high pressure and were squeezed together. In order to have a satisfactory extraction, it was necessary to use more hexane compared to the amount used for the corn germ.

The results we obtained in the physical and chemical experiments made on the raw and unrefined oils were found to be within the range of the characteristics of the refined corn oil given in the literature (4). The results of these experiments are shown in Table II.

A Gulbaran Extractor & Diffuser of the pilot plant type has been designed and constructed. Experiments made with this extractor in both batch and continuous systems have given excellent results technologically. These studies were done on various materials, such as flaxseed, rapeseed, moonflower seed and soybean, will soon be published with the methods of applying them to technology.

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[Received June 6, 1980]

The Occurrence of Dihydromalvalic Acid in Some Seed Oils

Sir:

Bohannon and Kleiman (1) found small amounts of dihydromalvalic acid, 9-10 methylenehexadecanoic acid (DHM), in eight of 19 seed oils containing cyclopropene fatty acids (CPFA).

In a study of the occurrence of CPFA in 40 seed oils (Vickery [2]), the gas liquid chromatographic techniques used were unable to separate very small amounts of DHM from oleic esters, but they detected 4.0-4.9% of DHM in *Litchi chinensis* seed oil.

In our studies, 39 seed oils were reanalyzed and two additional oils were assayed to detect the presence of DHM, using a 40-m glass capillary column coated with All-Tech CS-10 (Alltech Associates, Sydney), a cyano-silicone similar to Silar 10C, in a Packard chromatograph with an oven temperature of 175 C. All other experimental methods were similar to those outlined in the previous paper (2).

DHM was prepared by hydrogenating methyl malvalate dissolved in dry methanol using the catalyst Pd on activated carbon (Fluka A.G.). A mixture containing 8% 16:0, 32% 18:0, 22% DHM and 38% 18:1 was used for reference. The

TABLE I

Concentrations of Dihydromalvalic Acid (DHM) in Halphen-Positive and Halphen-Negative Oils

Halphen-positive			Halphen-negative		
Family	Species	Mass %	Family	Species	Mass %
Thymelaeaceae	*Pimelea decora*	Tr[a]	*Anacardiaceae*	*Harpephyllum caffrum*	0.1
	P. linifolia	0.2		*Mangifera indica*	0.2
Eleaocarpaceae	*Elaeocarpus reticulatus*	0.5		*Melanorrhoea pubescens*	0
Malvaceae	*Abutilon auritum*	0.2		*Pistacia chinensis*	0.2
	Gossypium sturtianum	0.4		*Schinus molle*	0.1
	Hibiscus diversifolius	0.1	*Celastraceae*	*Elaeodendron melanocarpum*	0.3
	H. trionum	0.2	*Ebenaceae*	*Diospyros australis*	0
	Lagunaria patersonia	tr	*Elaeocarpaceae*	*Elaeocarpus alaternoides*	0.3
	Lavatera plebeia	1.0		*E. persicifolius*	0.2
	Pavonia hastata	0		*E. rotundifolius*	0.1
	Radyera farragei	tr	*Rhamnaceae*	*Emmenosperma pancheranum*	0
Sterculiaceae	*Brachychiton gregorii*	1.2	*Sapindaceae*	*Dodonaea boroniifolia*	0.6
	Heritiera actinophylla	0.3		*D. petiolaris*	0
	Lasiopetalum macrophyllum	0		*D. triangularis*	0
	Rulingia corylifolia	0.8		*D. truncatiales*	0
Sapotaceae	*Pouteria wakere*	0.1		*D. viscosa*	0
Celastraceae	*Elaeodendron australe*	0		*Jagera pseudorhus*	0.2
Sapindaceae	*Dodonaea triquetra*	0.1	*Sapotaceae*	*Mimusops commersonii*	0.4
	Koelreuteria elegans	0.2		*Planchonella australis*	0.1
Gnetaceae	*Gnetum gnemon*	0.5		*P. myrsinoides*	0.4
				Pyriluma sphaerocarpum	0

[a]Tr = trace (<0.1%).