Extraction of Rapeseed, Linseed, Safflowerseed and Tobaccoseed With a New Laboratory Extractor

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In this study, extractions of rapeseed, linseed, safflowerseed and tobaccoseed have been conducted with a new extractor which has been developed for the extraction of vegetable tannins and which was later applied to sugar beets and sugar cane.

Using the optimal conditions of the new extractor for oilseeds, that is, 315μ particle size and 62 C temperature, technical hexane has been employed, and the variations of the rate and distribution of oil extraction from solid materials have been determined.

Optimal extraction periods with the new extractor were found to be 88 min for rapeseed, 90 min for linseed, 118 min for safflowerseed and 90 min for tobaccoseed. The same seeds have been extracted with the Soxhlet extractor for a period of 10 hr. Soxhlet extractor yields compared to Gülbaran yields with the new extractor were found to be only 98.18% for rapeseed, 98.15% for linseed, 97.79% for safflowerseed and 97.39% for tobaccoseed.

The shortage of basic food materials including alimentary oils has been increasing in proportion to the ever-growing world population. Therefore, obtaining alimentary oils from various seeds has increased in importance.

The Soxhlet extractor is used internationally for research and in quality control studies for the determination of oil yields in seeds and meal. Although easy to use, the Soxhlet extractor has some important disadvantages. The drawbacks are the length of the extraction period and the deterioration of quality of the oil which remains in the boiling solvent during the operation. In order to eliminate these disadvantages, the Gülbaran extractor has been chosen for the extraction of vegetable tannin, sugar beet, sugar cane and some oilseeds because it performs extractions in a much shorter time and maintains the extract at lower temperatures (1-4).

The purpose of this research was to study extraction mechanisms by carrying out systematic experiments with various oilseeds in the Gülbaran extractor, to determine optimal extraction times, and to compare the results with results from the Soxhlet extractor, thus revealing the advantages and disadvantages of the two extractors.

EXPERIMENTAL PROCEDURES

Equipment. The new laboratory extractor used for the extraction of solid materials is shown in Figure 1. It is made of two glass cylinders (E,E_1), one inside the other and both 40 cm high. 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).

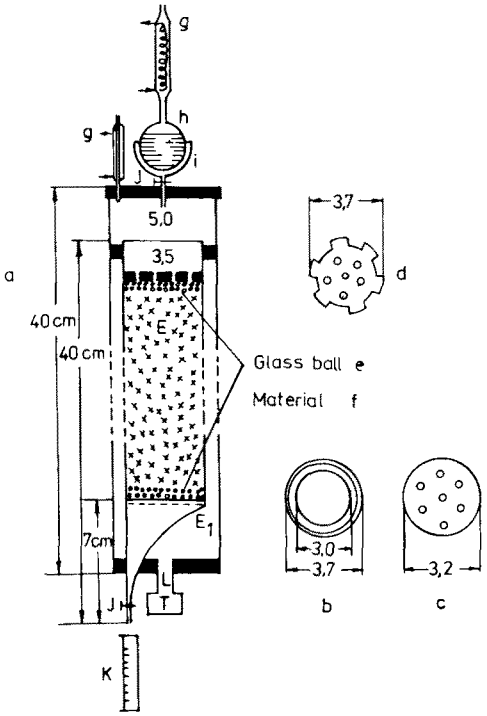

FIG. 1. Extractor for solid materials (Gülbaran, E., German Patent 1,024,269).

This ring is filled with glass ball bearings (e) 2-3 mm in diameter. 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) 2-3 mm in diameter 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 (L). Taps are shown in the figure with the symbol (J). Miscella is collected through the lower tap (J) into a graduated cylinder (K).

Materials. Rapeseed, linseed, safflowerseed and tobaccoseed which are grown in Turkey were used in the experiments. One kg of each material was ground in the MLW (Veb Kombinat Medizin und Labor-Technik, Leipzig) mill and sifted through a JEL (J. Engelsmann) sieve machine.

The solvent used for extraction both in Gülbaran and Soxhlet extractors was produced by Istanbul Petroleum Refinery Association Corp. under the name of Iprahex,

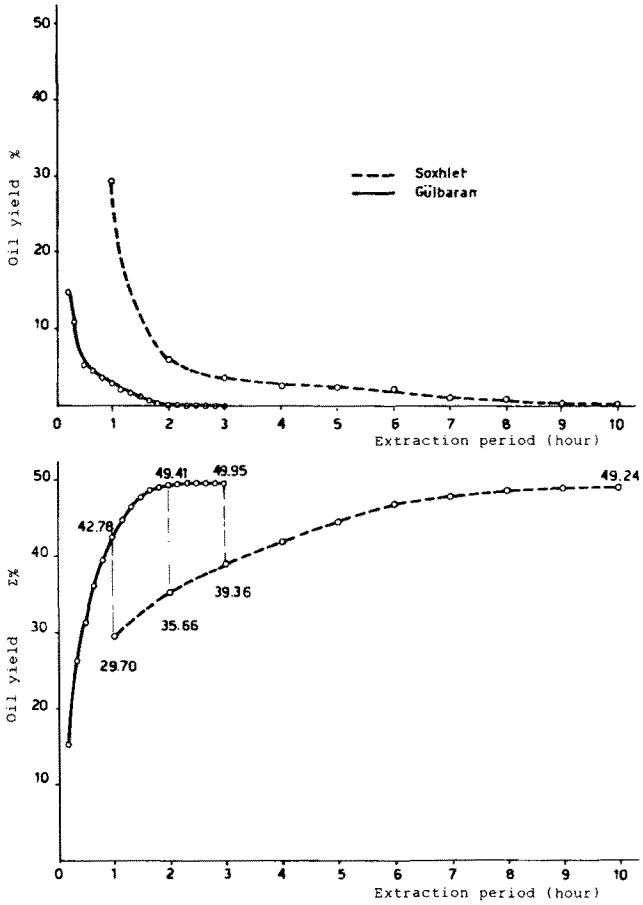

FIG. 2. The comparison of the variation of the oil yield to the extraction period for rapeseed with Soxhlet and Gülbaran extractors.

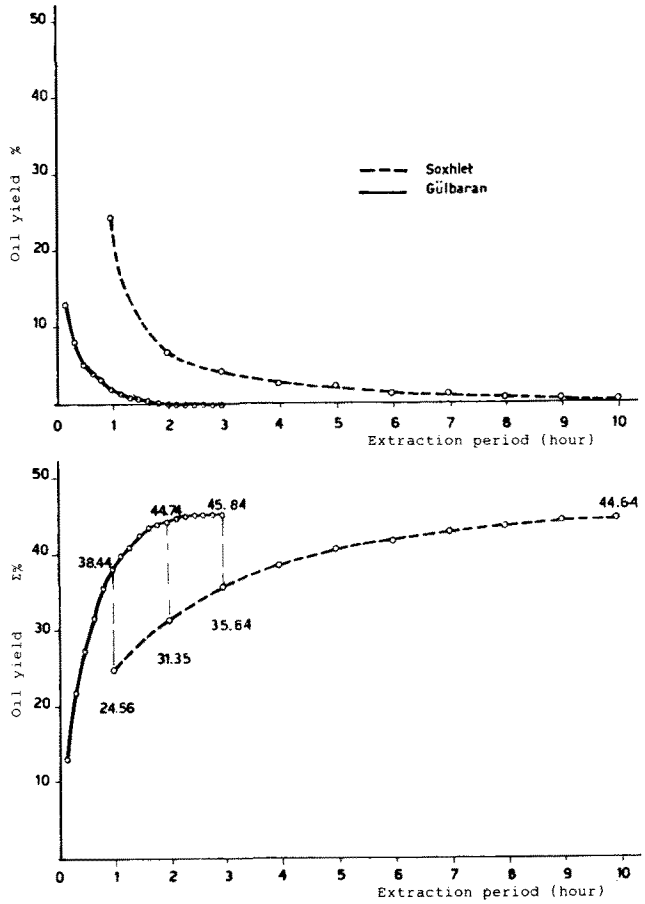

FIG. 3. The comparison of the variation of the oil yield to the extraction period for linseed with Soxhlet and Gülbaran extractors.

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

Experiments. The study has been done with the products of the sieve no. 0.315 (315μ) of the German DIN, which earlier was determined to be the optimal oilseed size for the extractor, and at the optimal temperature for the extraction, 62 C (4,5).

Optimal height of bed for the extractor is 20 cm (4,5). 75 g of material has been mixed with a glass tube with 7 cc of Iprahex at 62 C, and has been placed in the extractor. The average height of material in the extractor is 20 cm. While the external water jacket (E_1) has been kept at 62 C by a thermostat (T), the tap (J) of solvent tank (h) on the upper part of the apparatus has been opened and Iprahex which is heated to 62 C and placed below the condenser (g) has dripped on the material at regular intervals. 200 cc of miscella has been taken during the extraction periods, varying from 60 to 120 min (4,5). No pools of hexane can be allowed to form on top of the material during extraction with the Gülbaran extractor, and the dripping rate must be adjusted in such a way that hexane dripping onto the material is absorbed by the material immediately. Hexane residues on top of the material are contrary to the principles of this extraction system and elution theory (6).

In order to compare the extraction efficiency of the Gülbaran type extractor, a standard Soxhlet extractor of type DIN 12602-100 was used with extraction periods of 10 hr.

In order to determine the variation and distribution of the rate of oil extraction from the seeds used, and to compare the results from the Gülbaran and Soxhlet extractors, miscella was taken at the end of every hour from the Soxhlet extractor and at the end of every 10 min from the Gülbaran extractor, and oil yields were determined (Figs. 2-5).

All the extraction experiments in this study were repeated twice. The average values obtained in these experiments are indicated in the study.

The chemical and physical characteristics of the raw oils obtained either by the Gülbaran or Soxhlet extractors have been determined by standard methods and have been compared with the values given for refined oils in the literature.

RESULTS AND DISCUSSION

With the Gülbaran extractor, 10 pairs of experiments were carried out on rapeseed of an average ash content of 3.83% and an average moisture content of 6.59%; extraction periods ranged from 62 to 121 min. An average oil yield of 49.94% based on dry material was found. Hexane/material ratio was 1.534 kg/kg. The oil yield obtained with the Soxhlet extractor after 10 hr averaged 49.58% based on dry material. Hence, the efficiency of the Soxhlet extractor is only 99.28% compared to the performance of the Gülbaran extractor. At the end of the op-

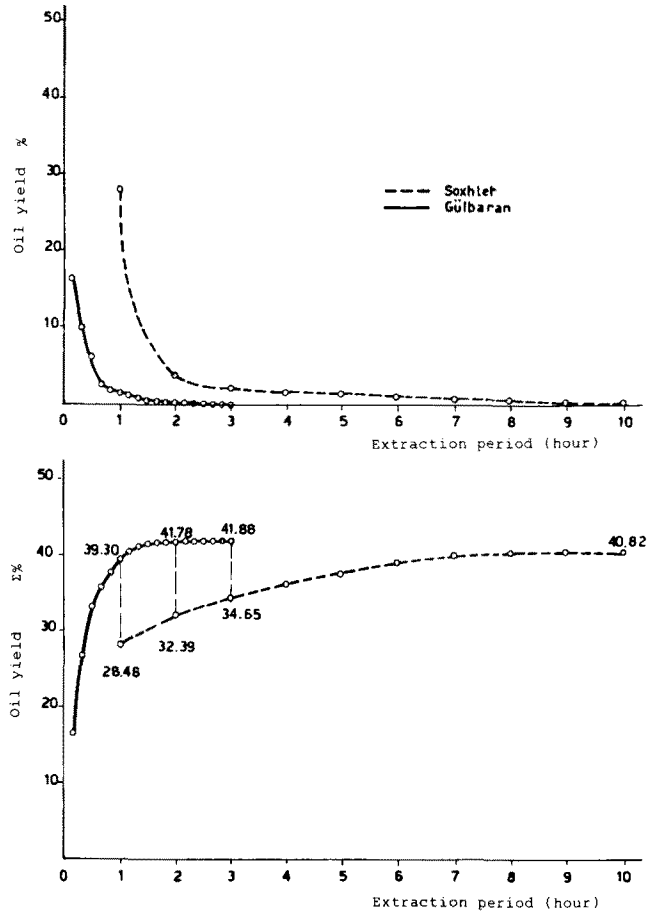

FIG. 4. The comparison of the variation of the oil yield to the extraction period for safflowerseed with Soxhlet and Gülbaran extractors.

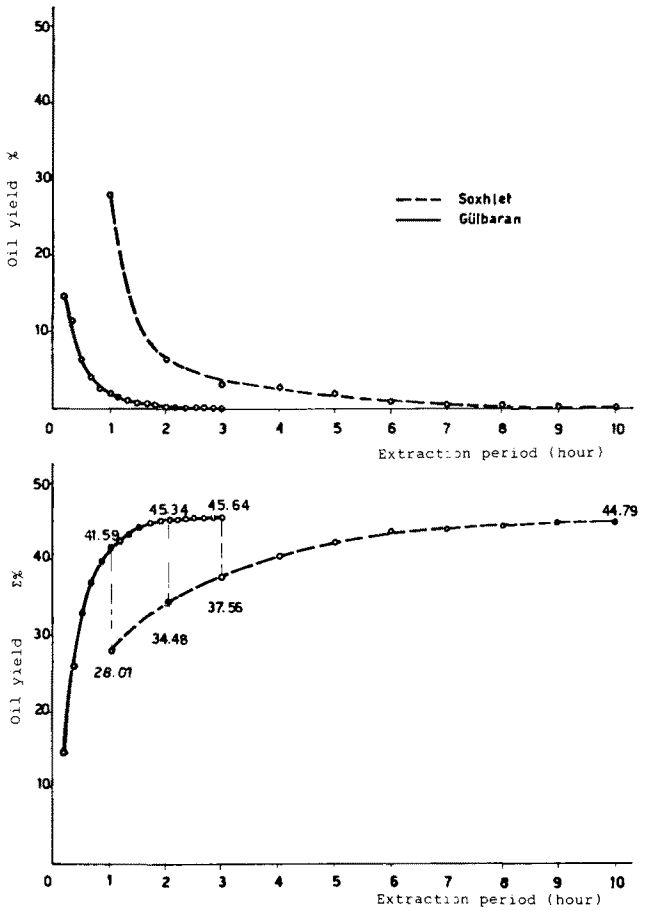

FIG. 5. The comparison of the variation of the oil yield to the extraction period for tobaccoseed with Soxhlet and Gülbaran extractors.

timal extraction period of 88 min, 50.50% oil yield was obtained, whereas the Soxhlet extractor reached 98.18% of this amount.

Seven pairs of experiments were carried out with the Gülbaran extractor on linseed containing an average ash content of 4.48% and an average moisture content of 5.07%. For the extraction periods ranging from 82 to 120 min, an average oil yield of 45.44% was obtained, based on the dry material. During these experiments the hexane/material ratio was 1.874 kg/kg. An average oil yield of 45.02% was obtained with the Soxhlet extractor after 10 hr based on the dry material. This value corresponded to 99.08% of the value achieved with the Gülbaran extractor. The optimal extraction period for linseed was determined to be 90 min together with an average oil yield of 45.87% based on the dry material. In Soxhlet extraction only 98.15% of this value was achieved.

At the end of six pairs of extraction experiments with the Gülbaran extractor on safflowerseed, for extraction periods ranging from 64 to 118 min, an average of 41.64% of oil yield was found based on dry material. The average ash and moisture contents have been determined as 2.20% and 7.34%, respectively. Hexane/material ratio was 1.943 kg/kg. At the end of 10 hr of the Soxhlet analysis, an average oil yield of 41.10% was found based on the dry material. Soxhlet can extract only 98.70% of the oil obtained by the Gülbaran extractor. The optimal extraction period with the Gülbaran extractor for the safflowerseed and the corresponding oil yield on dry material basis were determined to be 118 min and 42.03%, respectively. 97.79% of this value was obtained by the Soxhlet extractor at the end of 10 hr.

Nine pairs of experiments were done with the Gülbaran extractor on tobaccoseed. Extraction periods of these experiments ranged from 62 to 120 min, and average values of the ash and the moisture were 4.07% and 5.23%, respectively. At the end of these experiments an average oil yield of 45.66% was obtained based on the dry material. During these experiments the hexane/material ratio was 1.857 kg/kg. At the end of 10 hr of the Soxhlet extraction an average oil yield of 45.19% was obtained based on dry material, which was 98.97% of the oil extracted by the Gülbaran extractor. The optimal extraction period for tobaccoseed was determined to be 90 min. The oil yield obtained at the end of this time was 46.40%, and the Soxhlet extraction can obtain 97.39% of this value.

The general results of the Soxhlet and the Gülbaran extractors for rapeseed, linseed, safflowerseed and tobaccoseed are summarized in Table 1.

The remaining meals from the 10-hr Soxhlet extraction were subjected to extraction in the Gülbaran extractor for 120 min. For these experiments average oil yields of 0.16%, 0.67%, 1.22% and 0.65% were obtained for rape-

TABLE 1

Comparison of Experimental Results of Soxhlet and Gülbaran Extractors

Material	% Ash[a]	% Moisture[a]	Soxhlet extraction		Gülbaran extraction			
			Oil yield[b]	Extraction period[c]	Oil yield[b]	Range of extraction period[d]	Optimal extraction period[d]	Oil yield[e]
Rapeseed	3.83	6.59	49.58	10	49.94	62-121	88	50.50
Linseed	4.48	5.07	45.02	10	45.44	82-120	90	45.87
Safflowerseed	2.20	7.34	41.10	10	41.64	64-118	118	42.03
Tobaccoseed	4.07	5.23	45.19	10	45.66	62-120	90	46.40

[a]Average.
[b]% based on dry material.
[c]In hours.
[d]In minutes.
[e]% based on dry material at optimal extraction period.

TABLE 2

Physical and Chemical Characteristics of Raw Oils

Type of oil	Free oleic acid (%)	Iodine number	Saponification number	Refractive index at 20 C	Specific gravity at 20 C	Color (Lovibond Tintometer)
Rapeseed (7)	—	97-108	170-180	1.470-1.474 (25 C)	0.906-0.910 (25/25 C)	—
Gülbaran extraction	1.4	102.5	172	1.471	0.907	10
Soxhlet extraction	1.5	100.8	172	1.471	0.907	11
Linseed (7)	—	165-204	189-195	1.477-1.482 (25 C)	0.931-0.936 (15.5/15.5 C)	—
Gülbaran extraction	1.83	178	187	1.481	0.927	10-11
Soxhlet extraction	1.96	176	185	1.481	0.927	10-11
Safflowerseed (7)	—	140-150	188-194	1.472-1.475 (25 C)	0.919-0.924 (25/25 C)	—
Gülbaran extraction	1.0	143	191	1.473	0.914	7-8
Soxhlet extraction	1.25	140.5	191	1.473	0.914	8
Tobaccoseed (7)	—	129-142	186-197	1.474-1.483 (25 C)	0.923-0.925 (15/15 C)	—
Gülbaran extraction	2.28	138	190	1.476	0.919	6
Soxhlet extraction	2.46	133	190	1.476	0.919	6-7

seed, linseed, safflowerseed and tobaccoseed meals, respectively. On the other hand, the meals which previously had undergone 120 min of extraction in the Gülbaran extractor were subjected to extraction in the Soxhlet extractor for 10 hr; no oil was obtained.

As will be seen by the examination of Figures 2-5, the oil yields obtained by the Soxhlet extractor compared to those of the Gülbaran extractor at the end of first, second and third hours, are, respectively, 69.43%, 72.17% and 78.80% in rapeseed, 63.89%, 70.07% and 77.75% in linseed, 72.47%, 77.53% and 82.74% in safflowerseed and 67.35%, 76.05% and 82.30% in tobaccoseed. Under these operating conditions, the oil which has been extracted by Soxhlet extractor at the end of 10 hr has been obtained by Gülbaran extractor in 110-120 min for rapeseed and linseed, 70-80 min for safflowerseed and 90-100 min for tobaccoseed.

These results indicate that the Gülbaran extractor has a much shorter extraction time and higher oil yields than the Soxhlet extractor. This is because the new extractor operates under a system very different from 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 difference in concentration between the material and the solvent is kept very large throughout the extraction period, compared to the difference in the Soxhlet. Thus, extraction is done in a shorter period. The elution theory of Gülbaran explained that the new extractor would give such results (6).

It has been observed that miscella obtained by extraction with the Gülbaran extractor is clean and clear and has no solid particles in it. Because of the shortness of extraction periods, and because miscella leave the system continuously and accumulate at room temperature, the raw oil has been observed to be within the quality limits given for refined oils in the literature. The results of the physical and chemical experiments are shown in Table 2.

One of the most important characteristics of the

Gülbaran extractor is that the environment and solvent temperatures can be adjusted as desired. Owing to this property, it is advantageous in extraction procedures which should be done at low temperatures in research laboratories. In addition to giving more accurate results, the Gülbaran extractor can reduce personnel expenditures in research laboratories and lower cost in technology, product and quality control laboratories of oil factories. The latter effects are attributable to shortened extraction times.

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The Flash Devolatilization of Cocoa Butter

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A water-induced flash devolatilization process has been developed for the deodorization of cocoa butter. Per flash devolatilization efficiencies of 65-75% are high enough to give commercial quality products in a single flash.

Cocoa butter is an important ingredient in the production of chocolate. It is obtained from the cocoa bean and gives chocolate its characteristic taste, odor and consistency. Unfortunately, cocoa butter is of little value in its raw form because it contains many impurities which adversely affect its quality. A variety of refining processes have been developed to remove these impurities and improve uniformity.

An important step in the refining of cocoa butter is the deodorization process. Deodorization removes low molecular weight fatty acids and other volatile impurities from the butter. It is performed after the butter has been washed and filtered to remove bulkier impurities. Presently, the method most commonly used for deodorizing cocoa butter is steam distillation. In this process, hot, liquid cocoa butter is passed through a distillation column countercurrently to steam. The volatile compounds diffuse out of the cocoa butter into the water vapor and are removed with this water at the top of the column. Steam distillation may be performed in batch, semi-continuous or continuous modes and produces a highly deodorized butter (1). However, steam requirements are high because the interfacial contact with the cocoa butter is quite poor.

Other deodorization processes, such as chemical treatment, have been used in cocoa butter refining, but have been shown less efficient than steam distillation.

Flash devolatilization is a recently developed method for cocoa butter deodorization. It is a more efficient and economical process than steam distillation because it promotes a very large interfacial contact area between the cocoa butter and the devolatilizing medium. Flash devolatilization is a single stage process. Therefore, it requires relatively little monitoring. In addition, it requires a fairly low residence time to produce a highly devolatilized butter.

EXPERIMENTAL

Process. Flash devolatilization is a rather simple physical separation process, conceptually consisting of a single equilibrium stage. The necessary raw materials are prewashed butter and deionized, deaerated water.

The devolatilization process begins with the formation of a dispersion of about 2 wt.% water in cocoa butter. A relatively stable dispersion is achieved, without the use of emulsifying agents, when the mean particle size is less than 1 μm.

After the dispersion is formed, the mixture is quickly heated to 100-150 C under sufficient pressure to prevent boiling, e.g., 50 psi. The dispersion then undergoes a flash in which the pressure is dropped suddenly to approximately 5 torr. This pressure drop causes the water droplets to become gaseous. Because of this

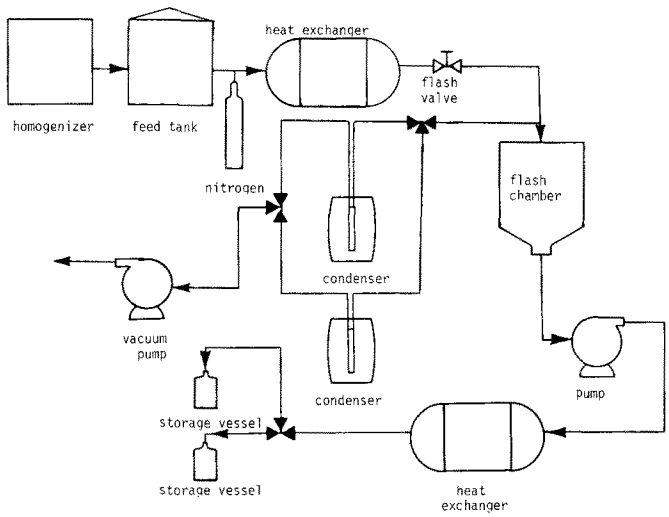

FIG. 1. Flash devolatilization process.

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