Direct Extraction of Sunflower Seed by Filtration-Extraction

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THE OBJECT of this paper is to report on the industrial-scale operation of our continuous, direct solvent-extraction plant, using dehulled sunflower seed as the feed material, and to discuss the influences of certain process variables upon plant operation.

The high initial oil content of dehulled sunflower seed and the production of fines as the oil content is reduced during extraction are factors which eliminate the use of conventional solvent-extraction systems for this material. For these reasons sunflower seed is normally pressed or prepressed, followed by solvent extraction for oil recovery.

Runs conducted in our plant were made to determine the processing conditions required for an efficient extraction with relatively low solvent/feed ratios for maximum oil content in the full miscella. The information obtained permitted smooth, full-scale operation of our direct, continuous solvent-extraction installation, using dehulled sunflower seed as feed material, a feat which is believed to be a significant advance in oil seed technology.

Description of the Process

Filtration-extraction (Filtrex) has been described previously in considerable detail (1, 2, 3), hence the following will be limited to a brief description of the processing steps to which sunflower seed is subjected.

Preparation Section. The seeds were first cleaned, then dehulled. The commercially dehulled meats (Table I) were then dried and rolled through a single-pass roll stand. The rolled meats were cooked in a five-high stacked-kettle cooker. Under the influence of moisture, heat, and a rapid evaporative cooling step termed crisping, the meats assume a porous structure which facilitates oil release during extraction.

TABLE I

Dehulled Sunflower Meats Composition

	%
Oil content	53.00
Free fatty acid	1.26
Moisture	5.60
Meats	90.90
Hulls	9.10

Cooking and crisping are processing steps not common to other extraction systems. These two unit operations however are responsible to a large extent for Filtrex's ability to process high oil-content materials with or without fine meat particles present.

Extraction Section. The solvent-extraction operation in filtration-extraction is divided into two distinct phases. One is the extracting or the putting of the oil into solution with the extracting solvent, and the second is the separation of the oil-solvent mixture from the spent meal by filtration.

Figure 1 is a view of the horizontal filter, showing pan drive, view ports, and piping.

Figure 2 shows the compartmented miscella tank which receives, keeps separate, and supplies countercurrent wash miscellas of various concentrations to appropriate pumps.

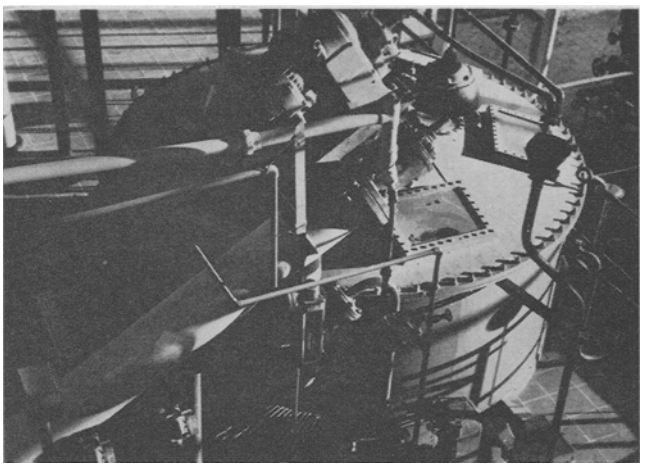

FIG. 1. Horizontal rotary vacuum filter.

Figure 3 is a view of the solvent area of the plant including extraction, oil and solvent separation and recovery, and marc (solvent damp meal) desolventization. Capacity of the plant is 50 tons of dehulled sunflower seed meats per 24 hrs.

The prepared material is fed continuously to the extractor, where it is slurried for 30–35 min. under gentle agitation with a miscella of medium concentration. The slurry formed is continuously deposited on the rotating pan of the horizontal vacuum filter, where most of the concentrated miscella is removed. The solids which remain on the pan form a cake of approximately 2 in. in thickness. The concentrated miscella previously removed is passed through this cake bed for clarification. The cake receives three washings before being discharged for solvent removal. Miscellas of decreasing concentrations are used for the first two washes, and oil-free solvent is used for the final wash. The miscella resulting from the first wash goes to the extractor to form the slurry.

Preparation Conditions

In all of the following runs, water was added to the second ring of the cooker; the first ring was used as a preheater. Previous experiments have shown that water added to the relatively cool material in the first ring of the cooker caused oil release, which adversely affected the cooking operation. Those same experiments also showed that preheating prior to moisture addition permitted the production of meats which extracted to lower residual lipids contents. Similar results have been obtained with peanut kernels (4).

Rolling, drying, cooking, and crisping were done on an industrial scale. The material resulting from these various preparations was extracted on a bench scale (5).

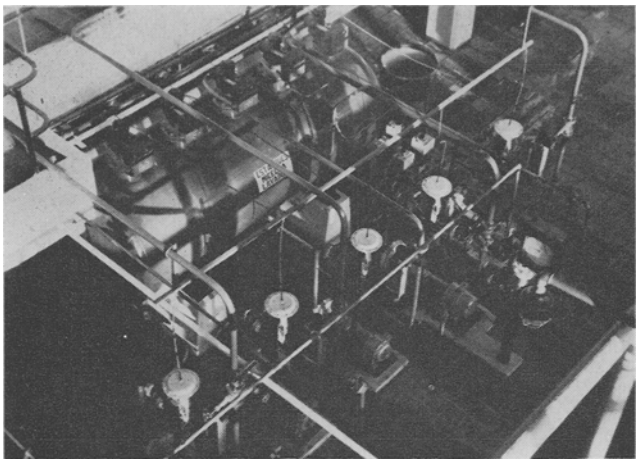

FIG. 2. Miscella tank with liquid-level control pilots, valves, and pumps.

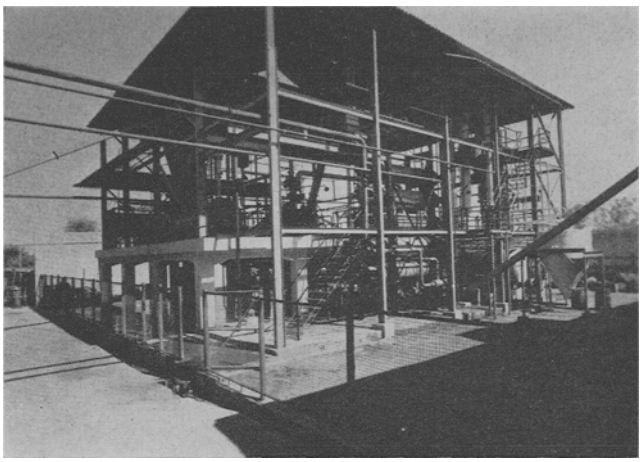

FIG. 3. Over-all view of solvent area, including extraction, oil and solvent recovery, and marc desolventization.

Experimental Results

The studies included the effect of rolling, drying, cooking, solvent/meats ratio and mass velocity upon residual lipids content. Solvent/meats ratio is the weight ratio of oil-free solvent to prepared meats introduced into the plant. The solvent used for these studies was a commercial grade of n-hexane. Mass velocity is the rate at which miscella passes through the cake bed and filter medium, measured in pounds of filtrate per hour per square foot of filter area.

The results of these runs are tabulated in Table II. From these results it can be seen that a single rolling before cooking is not as effective as a double rolling before cooking. It is also shown that drying before a single rolling is more effective than a double rolling. This results from the higher rolling efficiency possible with dried material. The best results were obtained with material which had been rolled, cooked, and re-rolled after cooking. Re-rolling after oil cells have been weakened by heat and moisture during cooking seems to promote more complete oil-release in the extraction step.

As previously mentioned, mass velocity is a measure of the filterability of miscella (or solvent) through the cake bed. For a high value of this variable (6,000–7,000), the wash liquids percolate rapidly through the cake bed without covering the bed completely. This uneven washing results in a meal containing an unnecessarily high residual lipids content. Conversely a low mass velocity (1,000 to 2,000) allows the wash liquids to accumulate on the surface of the cake bed; and, in addition to the undesired intermingling of the wash fractions, high residual lipids also result.

An efficient washing is obtained with a mass velocity of 3,000 to 4,000. Under these conditions a favorable distribution of wash liquids was obtained with no intermingling of the various wash fractions.

That the removal of oil from the oil-bearing material takes place in the extractor is an important concept. Practically no extraction takes place on the filter. If insufficiently extracted material is conveyed to and washed on the filter, even ideal washing conditions will not produce an extracted meal with an acceptably low residual lipids content. A second concept is that the mass velocity of a particular preparation is not only a measure of probable washing efficiency but also an indication of the degree of extraction to be expected in the extractor itself.

Actually a particular preparation will give two distinct mass velocity values. The first, and higher, value measures the ability of the material after cooking and crisping to pass solvent. The second value measures the ability of the material to pass solvent after the concentrated miscella has been refiltered through the cake bed. The fines removed from the concentrated miscella, the original cake bed, and the filter medium add up to a greater resistance, hence a lower resultant mass velocity value. These two mass velocities will be identified in the following discussion as the mass velocity before refiltration (mass velocity during extraction), the higher value, and mass velocity after refiltration (the wash liquid mass velocity).

Figure 4 shows the relationship between these two mass velocities. An initial mass velocity of 6,000 to 7,000 lbs. per hour (sq. ft.) is reduced to about 3,000 lbs. per hour (sq. ft.) after deposition of the fines upon the cake bed. Preparation of the sunflower meats used for this evaluation are shown in Table III.

TABLE II

Data on Extracted Meals

Type of rolling	Run No.	Lbs. processed	Rate lbs./hr.	Moisture content	Rolls separation	Flake thickness	Mass velocity	Solvent ratio	Vacuum	Slurry temperature	Residual lipids
				%	mm.[b]	mm.	Lbs./hr./sq. ft.[c]	lb./lb.	Inches Mercury	°F.	%
Rolled[a]	1	5,000	4,409	5.6	0.1	0.15–0.18	4,000	1.7	4.0	140	2.94
Re-Rolled before cooking[a]	2	4,500	4,409	5.6	0.1	0.15–0.18	4,000	1.7	4.0	143	1.15
Re-Rolled after cooking[a]	3	4,800	4,409	5.6	0.1	0.15–0.18	4,000	1.7	4.0	145	0.67
Dried before rolling	4	4,600	4,409	1.8	0.1	0.15–0.18	4,000	1.7	4.0	141	1.05

[a] Processed without drying.
[b] Flaker one pair rolls 800 mm. in diameter.
[c] Determined after crisping.

TABLE III

Sunflower Meats Preparation Data
(for bench-scale extraction)

Run number	1	2	3	4	5
Flaking conditions	Dry before rolling	Dry before rolling	Dry before rolling	Dry before rolling	Dry before rolling
Moisture after drying, %	1.8	1.8	1.8	1.8	1.8
Rolls separation, mm	0.1	0.1	0.1	0.1	0.1
Flake thickness, mm	0.15–0.18	0.15–0.18	0.15–0.18	0.15–0.18	0.15–0.18
Lbs. processed	8,850	8,000	7,900	8,300	8,100
Flake feed rate to cooker, lbs./hr	4,409	4,409	4,409	4,409	4,409
Cooking conditions					
Total time, min	55	55	55	55	55
Temperature, °F.:					
Ring: 1st	190	190	190	210	215
2nd	200	200	200	210	220
3rd	200	200	206	212	220
4th	206	210	210	214	225
5th	210	222	225	225	230
Ring, 2nd, moisture, %	7.1	7.6	8.1	8.3	11.6
After crisping, moisture, %	1.21	1.3	1.31	1.36	1.32
Mass velocity, lb./hr. (sq. ft.)	1,800	3,000	4,000	5,200	7,000
Cake thickness, in	2	2	2	2	2

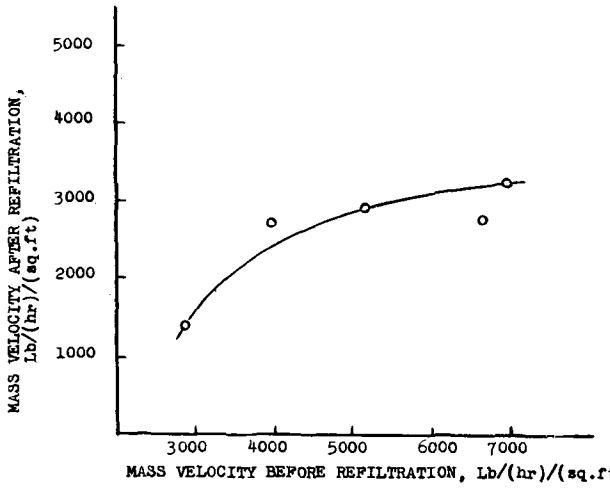

FIG. 4. Relationship between mass velocity before and after refiltration.

Figure 5 shows the relationship between mass velocity before refiltration, solvent/meats ratio and residual lipids content. It is apparent that a) residual lipids content decreases with an increase in solvent/meats ratio, b) residual lipids content decreases with an increase in mass velocity, and c) mass velocity has a more pronounced influence upon residual lipids content at lower solvent/meats ratios. Meats prepared as shown in Table III were also used for this evaluation.

In view of the foregoing results, it is evident that a high mass velocity material is required during the extraction step for optimum oil release and that a somewhat lower mass velocity is required during countercurrent washing on the filter for optimum cake-washing efficiency. A good preparation meets both of these requirements since the initially high mass velocity value in the extractor is reduced to the proper range for washing by refiltration of the concentrated miscella through the cake bed.

Tables IV and V show the preparation and extraction conditions of a full-scale industrial run conducted to verify the experimental findings. Table V also includes the results of that run.

The physical layout of the preparation section of the plant does not readily lend itself to re-rolling after cooking. The second best procedure, that is, drying before rolling followed by cooking and crisp-

TABLE IV

Sunflower Meats Preparation Data
(for industrial run)

Flaking condition	Drying before rolling
Moisture after drying, %	1.8
Rolls separation, mm	0.1
Flake thickness, mm	0.15–0.18
Wt. of sunflower meats processed, tons	58
Cooking conditions	
Temperature, °F.:	
Ring: 1st	215–218
2nd	220
3rd	220–222
4th	224–228
5th	228–232
Moisture, %	
Ring—2nd	11.8
After crisping	1.41
Mass velocity, lb./hr./(sq. ft.)	7,000

TABLE V

Sunflower Meats Extraction Data
(for industrial run)

Feed rate, lb./hr	4,409
Wt. of sunflower meats processed, tons	53
Solvent ratio, lb./lb	1.7
Extractor temperature, °F	115
Cake thickness on filter, in	2
Vacuum required, in. of mercury	3.8–4.5
Lipids in filtrate, %	
1st	30.1
2nd	29.8
3rd	10.0
4th	2.8
5th	0.4
Solvent in marc, %	39.0
Residual lipids in desolventized meal, %	1.68
Moisture in desolventized meal, %	3.8

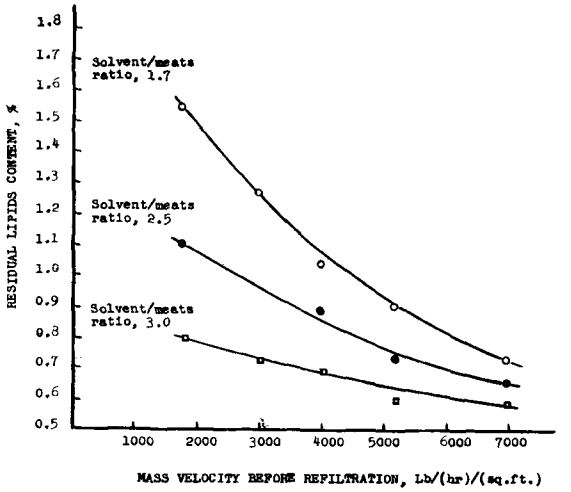

FIG. 5. Relationship between mass velocity, residual lipids content, and solvent/meats ratio.

ing, was employed. Nevertheless residual lipids content was reduced to 1.68% oil at 3.8% moisture, or *1.58% oil calculated to 10% moisture.*

Conclusions

The influence of certain variables upon the direct, continuous, solvent extraction of sunflower seed meats has been discussed. It has been shown that the ideal preparation consisted of first reducing the hull content to about 10% by weight. Then the dehulled meats were rolled, cooked, crisped, and re-rolled prior to solvent extraction. Drying before rolling, followed by cooking and crisping, was also helpful in the reduction of residual lipids content but to a lesser degree than was re-rolling after cooking.

It has been shown that the use of relatively low solvent/meats ratios required initially high mass velocities and that mass velocity during extraction was higher than that realized during cake washing.

The full industrial-scale run confirmed the experimental findings; thus the direct, continuous, solvent extraction of sunflower seed meats is a commercial *reality. An oil-content reduction from 53% to 1.68%* in one operation is a technological advance of great magnitude.

Except for periods when rice bran is processed, the plant is in continuous operation on sunflower seed meats and consistently produces meals containing between 1.5% and 1.7% oil content.

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Ozonolysis as a Method for Establishing the Position of Olefinic Linkages

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OZONOLYSIS combined with chromatography has not been an entirely satisfactory method in our laboratory for locating the positions of double bonds in unsaturated fatty acids. Literature reports (1, 2, 3, 4, 5, 6), including some which appeared after this work was begun, had indicated a reasonable degree of success with the method. We therefore examined the procedures in greater detail, using oleic acid as the representative compound.

The methyl oleate employed in our investigation satisfied the requirements for high purity on the basis of melting and freezing curves and infrared spectra. The presence of methyl linoleate was not detected in its ultraviolet spectra, and both iodine and saponification values lay well within the accepted limits of purity. Because the methyl oleate was converted to oleic acid under saponification conditions which previously had been shown to effect no conjugation of the double bond system of methyl linoleate, it was assumed that no isomeric octadecenoic acids were formed during saponification. When oleic acid was ozonized however and the products of ozonide decomposition were chromatographically resolved, relatively large fractions corresponding to seven-, eight-, and ten-carbon mono- and dicarboxylic acids were obtained in addition to the expected pelargonic and azelaic acids.

This report is concerned with our attempts to determine, in a semi-quantitative manner, the origin of these unexpected cleavage products. These acids might have been formed during either ozonization or ozonide decomposition or might have resulted from positional isomers contained in the methyl oleate which were not detected by the methods employed to establish its purity. Our results are summarized in Table I. Observations and conclusions regarding fractions other than the C_9 follow.

C_7 Dicarboxylic Acid Fraction. Runs 1, 2, 3, and 4 are representative of ozonizations carried out in solvents commonly used for this purpose; a relatively large C_7 dicarboxylic fraction was obtained in each case. It has been shown however that this fraction results from lower-molecular-weight monocarboxylic acids, such as acetic acid or other acidic substances. Thus, when acetone was treated with ozone, then heated with alkaline peroxide, and the product was chromatographically resolved, a broad peak in the region of the C_7–C_8 fraction was obtained. Similar results were also obtained with methanol and tetrachloromethane. n-Hexane was not attacked by ozone under the conditions employed, and its use as a solvent eliminated the C_7 fraction completely (Runs 6, 7, 8, and 11). Thus the C_7 fraction was an artifact and not pimelic acid.

C_{10} Dicarboxylic Acid Fraction. The C_{10} fraction, isolated by the same chromatographic procedure used in the analytical procedure, contained some azelaic acid and an unidentified amorphous substance but not sebacic acid. When the ozonide was subjected to vigorous decomposition (heated under reflux for 3 hrs. with alkaline peroxide, Run 11) or when ozonization was effected in acetic acid at 90–95° (at which temperature the ozonide probably decomposed as rapidly as it formed, Run 12), the C_{10} fraction was