Relation of the Quality of Solvent-Extracted Soybean Oil and Residual Oil Content of Meal

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Flaked soybeans were extracted by commercial hexane in a continuous countercurrent pilot-plant and in glass extraction-rate apparatus. The quality of the oil extracted at various stages was evaluated in terms of iodine value, saponification value, refractive index, color, refining loss, free fatty acids, phospholipids, and neutral oil. As the residual oil content of the meal decreased, the refining loss, phospholipid content, and color of the extracted oil increased. There was no significant change in other properties. The maximum amount of refined oil was obtained with a meal containing about 1% residual oil.

IN THE SOLVENT EXTRACTION of soybean oil it is commonly assumed that the maximum gross return is obtained with the highest yield of oil from a given batch of beans since the oil sells for a higher price per pound than the meal. The highest net return would have to take into account other factors, such as the amount of solvent recovery (evaporation and condensation) required, solvent loss, and extraction time. Another consideration would be the quality of the products, particularly the oil. The selling price of the oil is dependent partly upon the refining loss and the refined oil color.

The advantage of reducing the oil content of seeds, such as soybeans, to low levels, such as 1%, has been questioned (7). It has been suggested that the lower selling-price of the oil resulting from the lower quality might more than balance the increase resulting from a greater amount. Bull and Hopper (9) have reported high phosphatide content in the last 1.1% of oil extracted. Karnofsky (10) has reported a high refining loss in a similar fraction.

In the current studies an attempt has been made to determine the significant changes in oil quality that take place with an increasing degree of extraction of the oil. As a check on the countercurrent studies in the pilot plant, glassware extraction studies were also carried out.

Pilot-Plant Studies

The equipment used for the countercurrent extraction was a modified form of that used for various other extraction studies in this laboratory (4,5). The glass extraction section, forming the bottom run of the equipment, was replaced by a steel section. Copper tubing was wound spirally around this for heating by steam or hot water. The meal-desolventizing sections were equipped with individual steam-pressure controls.

The operating procedure for each run was as follows. The proper drive sprockets with connecting chain to produce the desired conveyor chain speed with its corresponding extraction time were installed and the conveyor chain was put into operation. Flaked soybeans were fed continuously into the extractor. Solvent feed was adjusted to the desired solvent-feed ratio. This combination of extraction time and solvent-feed ratio determined the amount of residual oil remaining in the meal. About two hours were required to bring the unit to a steady rate of operation for each run, as evidenced by the constant temperature and concentration of the miscella flowing out. During this time the flake feed-rate was checked by timing weighed amounts of flakes going into the extractor. A sample of miscella sufficient for analysis, usually from one to two gallons, was collected after a steady state of operation was attained. A sample of the meal was taken at such a time as to correspond to the miscella sample. Extraction times were varied from 19 to 66 min. with corresponding solvent-feed ratios of 0.36 to 2.28. Residual extractables varied from 4.24 to 0.68% (Table I).

TABLE I

Pilot-Plant Operational Data

Run No.	Extraction time	Solvent-feed ratio	Extraction temp. (av.)	Flake moisture	Residual extractables	Miscella concentration
	min.	*lb./lb.*	*°F.*	*%*	*%*	*% oil*
1	19	0.36	155	12.9	4.24	45.2
2	19	0.50	155	10.7	3.21	42.5
9	33	0.48	155	9.3	2.34	34.0
10	33	0.94	140	9.3	1.82	28.2
3	19	0.70	153	14.2	1.52	33.5
8	48	1.95	133	9.5	1.40	14.0
4	19	1.10	150	13.2	1.10	27.0
7	48	1.23	150	9.5	0.94	17.5
5	66	1.83	151	12.8	0.73	11.9
6	66	2.28	150	12.8	0.68	10.0

The soybeans used in the series of extraction runs were from a common batch. However, to avoid oxidative changes in the flaked beans each batch was flaked as needed after being stored over-night with sufficient added water to adjust the moisture content to approximately 10%. Actual moisture contents ranged from 9.3 to 14.2%. Average flake thickness was 0.011 in. The solvent was "high-purity normal hexane"[1] with an 88% hexane content.

The solvent was removed from the miscella sample by distilling under reduced pressure, followed by vacuum evaporation. The following were determined on the crude oil by official A.O.C.S. Methods (11): iodine value, saponification value, free fatty acids, and refractive index. Refining loss was determined by a modification of the official method developed in this laboratory (1) for use on 50-g. samples. This method uses a special refining cup, making it possible to obtain the arbitrary evaporation losses necessary to check the official method. Weighings are made on an analytical balance. The following were determined on refined oil from the refining loss determination, using official methods: iodine value, saponification value, refractive index, and color (spectrophotometric method). The oil content of the flaked soybeans and the extracted meal was obtained by extraction with the solvent in a Soxhlet extractor for three hours, followed by grinding and extracting for another hour. Moisture content was determined by the official method. Miscella concentration was deter-

[1] Phillips Petroleum Company.

mined from specific gravity data that were obtained by weighing in a specific gravity bottle.

The properties of the crude oils from the various runs are given in Table II. The relation between the refining losses and residual extractables in the meals is shown in Figure 1. Data on the refined oil showed the following ranges: iodine value, 129.1 to 130.0; saponification value, 193.0 to 193.9; refractive index, 1.4731 to 1.4738; and color 6.0 to 8.2.

Glassware Studies

As a check on the pilot-plant work two series of studies were made by extracting the oil in glassware rate-extraction apparatus similar to but somewhat larger than that used in previous studies in this laboratory (6). The extraction chamber was 12 in. high by 2 in. in diameter, designed to use 100-g. samples. The extraction chamber and the incoming solvent were heated to 131°F. The solvent, the same as that used in the pilot-plant work, was passed through the sample continuously at 10 ml./min.

TABLE II

Crude Oil Results

Run No.	Residual extractables in meal	Free fatty acid	Refining loss	Iodine value (Wijs)	Saponification value	Non-saponifiable matter	Refractive index
	%	%	%			%	
1	4.24	0.54	5.12	127.3	192.4	0.40	1.4739
2	3.21	0.49	5.10	126.9	192.3	0.71	1.4736
9	2.34	0.57	5.28	126.7	192.0	0.60	1.4731
10	1.82	0.54	5.45	126.4	192.3	0.41	1.4729
3	1.52	0.54	5.45	125.7	192.2	0.44	1.4728
8	1.40	0.59	5.54	125.7	192.2	0.44	1.4730
4	1.10	0.54	5.66	125.5	192.1	0.73	1.4729
7	0.94	0.59	5.92	125.1	191.5	0.56	1.4730
5	0.73	0.54	7.03	125.0	191.3	0.54	1.4723
6	0.68	0.54	7.46	124.5	190.3	0.67	1.4719

In the first series, extractions were made on separate lots of flakes from a common batch for times from 50 to 120 min. All of the miscella from each extraction was desolventized in the same manner as the pilot plant samples. The oil content of the residual meal was also determined. The following determinations were made on the oil using official A.O.C.S. Methods (11): free fatty acids, iodine value, and color (spectrophotometric). The neutral oil and phospholipids were determined by the methods of Arnold and Choudhury (2) (Table III). The relations between the residual extractables in the meal and the phospholipids and the neutral oil content are shown in Figure 2.

In the second series, samples of flakes were extracted for 120 min. Samples of miscella were taken at the end of 50, 60, 70, 80, 100, and 120 min. Analytical results are given in Table III.

Discussion

The pilot-plant data show an increase in refining loss with a decrease in the residual extractables. Since the highest refining loss is well within the 12% maximum for commercial crude soybean oil, it might be assumed that from the practical standpoint there is little need to be concerned about oil quality. The increase in refining loss of 5.12 to 7.46% does not appear great.

If we check on the increase in the refining loss in relation to the increased oil extracted, we arrive at somewhat different figures. For example, when the residual extractables drop from 1.40 to 1.10%, the apparent refining loss on the additional increment of oil is 13.3%. Between 1.10 and 0.94 the refining loss

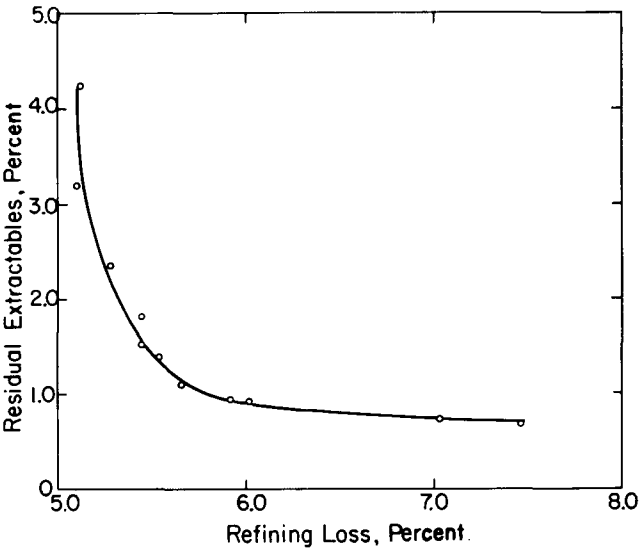

FIG. 1. Refining loss of crude soybean oil.

jumps to 37.5%. From 0.94 to 0.73 the figures show 114% with an increase to 180% for the 0.73 to 0.68 increment.

Obviously all of the refining loss attributed to the last two increments does not occur here, nor could it all result from experimental error. It was noted that with low residual oils the soap stocks tended to be soft, thus making separation difficult with a tendency toward high results. It should be considered that, since each run was separate and made with an individual combination of solvent-feed ratio and extraction time designed to produce a certain residual extractable content, the increments as calculated above are not necessarily directly comparable. The general trends of the phospholipid and neutral oil percentages in the first glassware series (Table III) are similar to the refining loss trend.

The phospholipid values appear high, compared with those commonly reported for commercial oils that are obtained with corresponding residuals in the meals. This may, in part, be the result of the analytical method used. This was a modification by the authors (2) of the chromatographic method (8) widely used in biochemical work, which basically determines the material soluble in methanol after the neutral oil has been removed by ether or chloroform. On some oils this gives a higher result than that obtained by multiplying the phosphorous content by an average conversion factor. The amount

TABLE III

Soybean Oils Extracted in Glass Rate-Extraction Apparatus

Extraction time	Residual extractables	Color	Free fatty acids	Iodine value	Neutral oil	Phospholipids
Series 1. Oils from Individual Samples						
min.	%		%		%	%
50	3.64	9.24	1.15	125.5	95.5	3.5
60	2.63	9.24	1.10	125.4	95.0	4.2
70	2.02	9.51	1.10	125.8	94.2	5.0
80	1.01	9.29	1.12	127.5	93.0	5.9
100	0.40	9.90	1.16	127.5	92.6	6.4
120	0.20	9.95	1.15	127.1	91.4	7.5
Series 2. Successive Increments from One Sample						
0–50	3.64	9.1	1.03	125.0	95.5	3.4
50–60	2.63	9.5	1.61	126.0	94.0	4.3
60–70	2.02	11.2	1.67	126.0	93.3	5.1
70–80	1.01	17.8	1.69	125.0	92.2	6.2
80–100	0.40	18.1	1.87	127.1	91.2	7.0
100–120	0.20	18.5	2.30	127.1	87.0	11.0

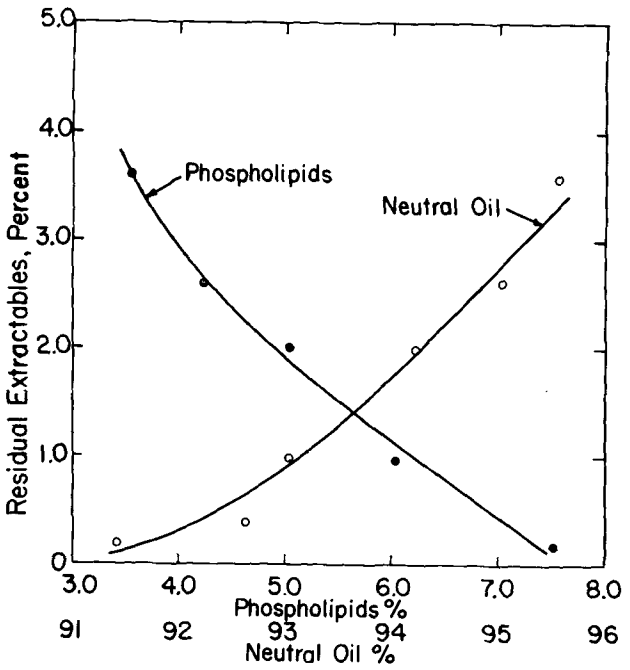

FIG. 2. Relation between residual oil content of meal and phospholipid and neutral content of corresponding oil.

of phosphatides in the oil is also affected by the amount of heating to which the seeds are subjected prior to or during extraction. Coagulation of the protein by the heat is said to bind the phosphatides and reduce their solubility. Bull and Hopper (9) extracting to a 0.43% residual with Skellysolve-B obtained 18.62% phosphatides in the last fraction compared with our value of 7.0%. These results were based on phosphorous data. Their extraction temperature was 104°F. compared with our temperature of 131°F. This comparison is valid only in a very general way since the variations in such factors as soybeans, flaking conditions, solvent, and detailed methods of operation between the two studies are not known.

Neither free fatty acid percentages nor color values show any measurable differences between the different fractions from the pilot plant and the first glassware studies. However definite differences do appear in the second glassware series (Table III). The color values and free fatty acid percentages in the last 1% of oil extracted are twice those in the first 82%. However if we calculate the average free fatty acid content of the composite sample extracted in the second series in 120 min., it is 1.15%, or the same as the free fatty acid content of the oil extracted over a 120-min. interval in the first series. In the pilot plant and first glassware series the highly-colored fractions which appear in the second series are mixed with a much larger amount of oil, which tends to make the color appear less.

The relative amounts of neutral oil, free fatty acids, and phospholipids in the complete oil sample extracted in the second glassware series are shown in Figure 3. The composition of the neutral oil was not determined. Since the iodine values, saponification values, and refractive indices show little more variation than probable experimental errors, it is believed that there is no significant difference in the glyceride composition of the various fractions. This agrees with the results of previous work (3) on cottonseed extraction, in which it was shown that there was practically no difference in the fatty acid compositions of the glyceride portions of the oils extracted at different residual levels.

We can calculate the amount of refined oil produced in each pilot-plant run from the total oil extracted minus the refining loss. The corresponding figures for meal residues of 1.52, 1.40, 1.10, 0.94, 0.73, and 0.68% are 18.31, 18.39, 18.60, 18.67, 18.61, and 18.56 lbs. per 100 lbs. of moisture-free soybeans. This indicates the maximum amount of refined oil was obtained with a meal residue of 0.94%. Since extraction and refining costs and prices of oil and refining foots vary, no attempt has been made to determine the oil residual in the meal corresponding to the greatest net return. It seems reasonable to assume that it will be approximately 1.0%.

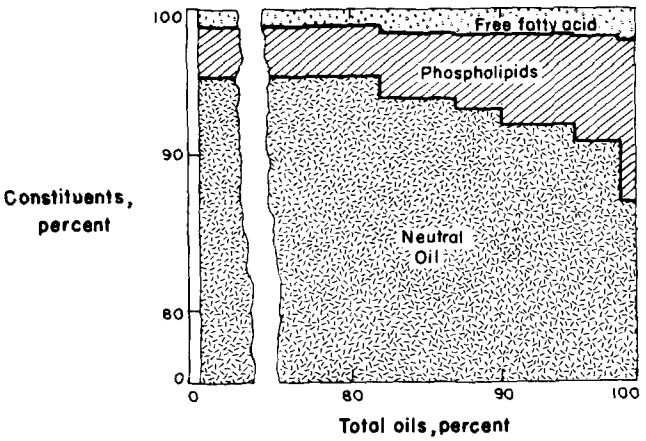

FIG. 3. Relative amounts of various constituents.

Conclusions

The authors did not expect to obtain close checks between the results of the pilot-plant studies involving countercurrent extraction and the glassware studies involving continuous extraction with fresh solvent. The results do show good general agreement and would appear to have some practical value.

While in the pilot-plant operation the increase in refining loss and color with decreased residual extractables was not sufficient, even down to 0.68% residual, to produce an oil below the usual commercial standard, the increase in refining loss was significant below about 1%. There is no evidence of any change in neutral oil composition with increased oil extraction. The point of greatest net return based on refined oil yield is about 1%.

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