quinoline, and employs an alkaline interesterification catalyst such as sodium methoxide.

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[Received August 16, 1951]

The Flavor Problem of Soybean Oil. X. Effects of Processing on Metallic Content of Soybean Oil

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IN a previous publication (1) data were presented, showing the serious effect which trace metal contamination has upon the stability of soybean oil. Spectrographic analysis showed that practically all soybean oils contained iron and copper in amounts which could be definitely detrimental to the stability of the oil. Since this work was reported we have extended our studies and have also included the examination of a number of crude oils. The spectrographic method (2) described previously was employed for the iron and copper analysis, and the organoleptic evaluations were made by our regular published procedure (3).

Through the cooperation of several soybean oil processors we have been able to examine a great number of oil samples taken from each of the unit processes employed in the extraction and refining of soybean oil.

Figure 1 summarizes the data obtained for commercial oils and shows the concentrations of iron and copper that were found in refined, deodorized, and crude edible oils. The oils examined included, besides soybean oil, cottonseed, corn, safflower, sunflower, and sorghum oils. As judged from the limited number of samples examined, there appears to be no difference in the metal contents of these different types of oils. This conclusion is further substantiated by the data presented by O'Connor *et al.* (4) for the crude oils of cottonseed, peanut, rice, okra, and calabash seeds.

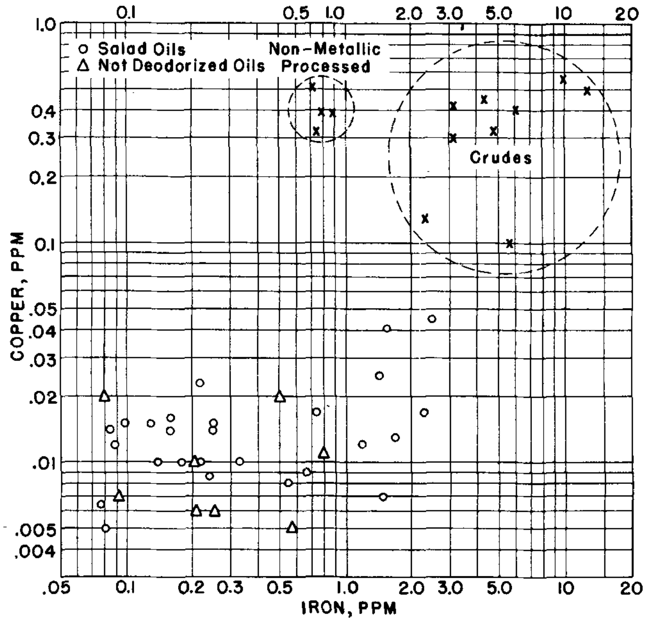

FIG. 1. Iron and copper content of commercially processed edible oils.

TABLE I

Metal Contents and Stability Evaluations of Soybean Oil Sampled From Units of Two Commercial Extractors

Sample Description	Metal Content p.p.m.				Stability		
	Iron		Copper		A.O.M.[a] 8 Hours	Flavor Score	
	Crude	Refined	Crude	Refined	100°C.	0	4 Days
Company A							
Miscella	2.70	.088	.16	.002	5.5	8.9	7.2
Falling film	3.80	.12	.26	.012	1.8	8.5	7.7
Stripper	5.60	.13	.10	.008	1.9	7.8	6.9
Control 176E	......	.015	......	.006	19.4	8.3	6.1
Company B							
Miscella	3.55	.077	.21	.011	17.8	8.9	7.0
Horizontal	4.10	.10	.50	.006	11.4	8.9	6.9
Falling film	4.70	.11	.33	.012	16.0	8.5	7.0
Stripper	2.10	.11	.28	.009	14.7	8.7	6.9

[a] Peroxide value milliequivalents of peroxide per kg. of fat.

The average iron content for laboratory-refined soybean oil has been reported at 0.12 p.p.m.; for commercial undeodorized oils, 0.19 p.p.m.; and for deodorized oils, 0.91 p.p.m. (1). The iron content of crude soybean oils will vary from 2 to 10 p.p.m. while samples of badly contaminated crudes will run from 10 to 100 p.p.m. or more.

The copper content has shown less than a 10-fold variation, and most crude oils will have between 0.2 and 0.5 p.p.m., and the refined oils less than 0.02 p.p.m. Because of the high copper content found when beans are processed in all-glass equipment, it is believed that most of the copper is "native" to the oil. The native copper is more than likely tightly complexed, or at least bound in such a manner that it is not free to act as an oxidation catalyst. "Native" iron and possibly some of the contaminating iron may be complexed with the phosphatides in the crude oil. Our analyses however show consistent trends in the contamination with iron, and it is this free or freshly added iron that is especially detrimental. The stability of both crude and refined soybean oil would be greatly lowered if most of the iron and copper present in the oil were not complexed.

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Contamination in Commercial Extraction Plants

The results obtained from the examination of the products of two commercial extractors are shown in Table I. The iron content of the oil was found to increase constantly as the oil progressed through separate units of the extraction plant. Copper may have increased some, but the values do not show a consistent trend throughout all operations of the extraction process. Laboratory refining has removed 95-98% of the iron and 92-99% of the copper present in these oils. Laboratory refining performed with careful centrifugation has consistently given oils with low metal contents, and, almost without exception, these oils have exhibited excellent stability. No evidence was obtained in this examination that the levels of contamination shown here are detrimental to the quality of the soybean oil when the crude oils are refined within a short time after extraction. The high oxidative stability values (i.e., low peroxide values under A.O.M. conditions) and the high organoleptic scores showed that these oils were all essentially equal and of excellent quality and stability.

The apparent discrepancy in the iron content of the crude oil obtained from the stripper of company B is explained as a sampling error. This plant had been shut down for a short time just prior to the taking of the sample, and it is quite likely that a separation of the phosphatides occurred and a representative sample was not obtained. In drawing any conclusions, one should remember that a large sampling error can occur in the sampling of oil from commercial processing equipment. Large sampling errors are introduced when the samples are collected from the various units in a matter of a few hours, while the actual flow of material through these units may require 24 hours or more.

Iron and Copper in "Native" Soybean Oil

To establish the level of what is termed the "native" concentration of iron and copper in crude soybean oil, three samples of soybeans were processed by hand and the oil removed in an all-glass Soxhlet extractor. The beans were carefully cleaned before being smashed with a wooden mallet, and, after the first few extractions with hexane, the crushed beans were removed from the Soxhlet thimble and ground in a porcelain mortar. Sample 4 was ground in a relatively new burr mill, but otherwise it was treated like the other samples. Over 96% of the oil was removed, and a crude oil with an acid number of 0.38 was obtained. Table II shows the iron and copper content of these nonmetallic-processed crude oils. Samples 1 and 2 were obtained from the same lot of beans which was being processed at the time the commercial extractors were sampled. Samples 3 and 4 were from a laboratory lot of Lincoln beans.

The analyses show an iron content of about 0.80 p.p.m., which was only 20% of that found in the commercial samples. The copper analyses show fair agreement, but with a slightly higher value for sample No. 2 of the glass-extracted series. This difference may be the result of a sampling error. The accuracy of the spectrochemical method is too great to account for this difference.

TABLE II

Iron and Copper Content of Crude Soybean Oil (All-Glass Extractor)

Sample	Iron p.p.m.	Copper p.p.m.
1	.88	.39
2	.71	.51
3	.74	.32
4	.78	.40

A laboratory refining of a composite sample, made from the 4 glass-extracted crudes, removed 93% of the iron and 97% of the copper from the oil.

A check on the physical analysis of the beans in samples 1 and 2 showed that they were good quality beans. Foreign material other than dockage was given as 0.4% and 0.7%, respectively. No samples of beans were taken during the commercial cleaning or flaking, but it seems logical to assume that dirt and foreign material present may also add iron to the crude oil.

Contamination in Refining Operations

Through the cooperation of three commercial refiners, oil samples were obtained at each of the unit operations in the refinery. Sampling errors are probably higher in the refinery investigation than in any of the others. In the case of company C an attempt was made to follow the same batch of oil through the entire refinery, and samples were collected over a period of 30 hours. In the other two refineries all the samples were collected in the course of a few hours.

TABLE III

Metal Content of Oils Sampled at the Various Stages of Commercial Refining

Sample	Company A		Company C		Company D	
	Fe p.p.m.	Cu p.p.m.	Fe p.p.m.	Cu p.p.m.	Fe p.p.m.	Cu p.p.m.
Crude	4.5	0.40	4.9	0.24	2.6	.14
Degummed	2.5	.10	....	....	....	....
Refined	1.05	.011	.56	.045	.28	.011
Washed	.15	.002	.18	.021	.079	.016
Washed	.17	.016	....	....	....	....
Dried	.09	.013	.26	.017	.050	.005
Bleached	.29	.023	.11	.010	.057	.025
Deodorized	.06	.014	.13	.027	1.50	.026

Table III shows the trend in the metal contents as the crude oil passes through the various refinery units. Most of the iron and copper are removed in the degumming and refining steps. The bleached oil might be expected to have the lowest metal content because of the nature of the bleach and the degree of filtration employed. Actually little change was found in the metal content after washing was completed; however in one case a very great rise in the iron content of the oil occurred during deodorization. Assuming that the average value for the washed, dried, and bleached oils will give a more correct indication of the metal content of the refined oil, we found that 96 to 98% of the iron in the crude oil was removed by these refineries. The removal of copper, when calculated on the same basis, showed a removal of 96.8, 80.0, and 89.3% for the three refineries.

No metal contamination resulted from the deodorization in two of the refineries, but the third showed a 24-fold increase in the iron content. No significant increase in the copper content was found in any of the deodorized samples. This agrees with previous findings.

Refining was completed in the laboratory for the samples collected after each of the unit operations in the commercial refineries. The oxidative stability and

TABLE IV

Evaluation of Oils Sampled at the Various Stages of Commercial Refining

(Refining Completed in the Laboratory)

Sample	Company A			Company C			Company D		
	A.O.M.	Score		A.O.M.	Score		A.O.M.	Score	
	8 Hours 100°C.	0-4 Days		8 Hours 100°C.	0-4 Days		8 Hours 100°C.	0-4 Days	
Crude	15	8.3	6.3	19	8.7	6.5	10	8.6	7.4
Degummed	9	7.1	6.6	....	....	....	....	....	....
Refined	9	7.9	6.8	13	8.8	5.7	11	8.2	7.0
Washed	12	8.6	6.3	8	8.8	6.4	12	8.6	6.4
Washed	9	8.7	6.5	....	....	....	....	....	....
Dried	11	8.1	6.2	12	8.8	6.8	17	8.3	6.3
Bleached	39	7.2	4.8	42	8.7	6.1	50	7.9	4.7
Re-deodorized	75	4.8	4.5	53	8.8	5.3	80	6.5	3.4
Deodorized[a]	68	4.9	4.1	2	8.0	6.9	69	5.0	3.5

[a] Evaluated as received.

TABLE V

Comparison of Commercial and Laboratory Deodorizations of Soybean Oil

(Oil Obtained at Beginning of Crushing Season)

Sample	Metal Content		A.O.M.	Flavor Score	
	Fe p.p.m.	Cu p.p.m.	8 Hours 100°C.	0	4 Days
Commercial A	1.50	.007	70	5.4	4.1
Laboratory	.25	.006	16	8.0	5.5
Commercial C	.33	.010	7	4.5	4.1
Laboratory	.21	.006	18	8.8	6.5
Commercial D	—	—	Lost	—	—
Laboratory	.09	.012	25	6.7	3.8
Laboratory refined	.35	.014	5	8.1	7.3
Commercial E	.22	.010	4	8.1	6.4
Laboratory	.21	.010	26	8.4	6.1

the organoleptic evaluation of these oils are shown in Table IV. The oxidative stability as measured by the A.O.M. peroxide values was good through the drying stage. After bleaching however, the oxidative stability showed a definite decrease in each case, as is shown by the greatly increased peroxide values. Through the use of a metal scavenger, refinery C markedly improved the stability of the deodorized oil while oil from the other refineries showed a distinct decrease in stability. Company A used no metal scavenger; company D used citric acid, but its finished oil received a low score because of the very high iron content. Citric acid is very beneficial, but it is not able to prevent entirely the effects produced by a high level of iron contamination.

Organoleptic evaluations made with an 18-member taste panel agree remarkably well with the oxidative data. Flavor scores shown are the average of three separate evaluations. All oils through the bleaching stage were equal and showed high initial flavor scores and storage scores. Initial flavor scores for the bleached samples were not significantly different from the scores of the washed sample, but the storage scores for the bleached samples of companies A and D were inferior and significantly lower than the score of the preceding stored samples.

For comparisons of oils processed under the same conditions, part of the commercially deodorized oils were redeodorized in the laboratory before they were submitted for taste panel evaluation. The commercial oils were also evaluated as received, by comparing each against the other two commercial oils. A comparison of the data in the last two lines of Table IV will show that the laboratory deodorization improved the zero time scores slightly for two oils but did not greatly change the storage flavor scores.

An examination of the stability and metal contents of soybean oil before and after commercial deodorization has given valuable information concerning the effect of metals on the stability of edible oils. Laboratory deodorization has shown that soybean oil cannot be deodorized in the presence of 0.1 p.p.m. of added iron without affecting the initial flavor score. The Northern Regional Research Laboratory taste panel will score a sample containing 0.3 p.p.m. of iron significantly lower than the control. At levels of 0.1 p.p.m. iron the scores will be lower, but they may not be significantly lower.

Contamination During Deodorization

The results of an investigation of four commercial deodorizers are shown in Table V. These oil samples, obtained before and after deodorization, were produced early in the crushing season of 1949. The samples shown in Table VI were obtained from the same four processors but at the end of the 1949 crushing season. The metal contents of these oils agree with the data reported previously. Data (on another deodorizer) are also presented and show a 6-fold increase in the iron content of the freshly deodorized oil. The copper content of these oils was low, and no difference was found between commercial and laboratory deodorized oils. All the samples were analyzed by code numbers, and the agreement achieved in the metal analysis was remarkably good. Each pair of samples were essentially duplicates, and excellent agreement in analysis of copper was shown when copper was present in concentrations of only 10 parts in a billion.

TABLE VI

Comparison of Commercial and Laboratory Deodorizations of Soybean Oil

(Oil Obtained at End of Crushing Season)

Sample Description	Metal Content		A.O.M.	Flavor score	
	Fe p.p.m.	Cu p.p.m.	8 Hours 100°C.	0	4 Days
Commercial A	1.20	0.012	65.0	5.0	4.8
Laboratory	0.093	0.007	35.0	7.1	5.1
Commercial C	0.67	0.009	80.0	5.7	4.2
Laboratory	0.57	0.005	141.0	5.8	3.5
Commercial D	0.18	0.010	61.0	6.5	5.3
Laboratory	0.079	0.020	47.0	6.3	5.3
Commercial E	0.13	0.015	2.4	7.1	6.0
Laboratory	0.08	0.011	58.0	6.7	4.6

The oxidative stability values agree well with the metal analysis, and the effect of using a metal scavenger is shown by the low A.O.M. peroxide values obtained in commercial samples E and C. No metal scavengers were employed by company A, and the low flavor score and high peroxide values correlated with the high iron content. The A.O.M. peroxide values from the laboratory all-glass deodorized samples are low and approximately equivalent, but they are not as low as the metal-scavenger treated oils.

In making organoleptic comparisons laboratory samples were compared against laboratory and commercial against commercial. These scores are the average of three separate evaluations, made by comparing each oil against the other three oils in that series. The organoleptic scores of the four laboratory deodorized oils showed that all oils are equal and of good quality and stability. The commercially deodorized oils however varied in score from 4.5 to 8.1. Yet only with oils from company A can the low scores be attributed to the presence of iron. Many reasons can be advanced for the cause of low flavor scores of which the presence of iron is only one. It is known that high scores are never obtained in the presence of dissolved and uncomplexed iron.

Results of the second sampling of the same four commercial deodorizers are shown in Table VI. The copper and iron analysis is somewhat comparable with the previous examination, and company A again showed a high iron contamination. The A.O.M. peroxide values were all high except in the single case of the salad oil from company E. This low value again indicates the importance of a metal scavenger even though the iron and copper contents are low and contamination is nil. Although flavor scores of sample E were superior for both the initial and storage-time evaluations, the other flavor scores showed little difference. These laboratory samples taken at the end of the crushing season were scored much lower than those taken at the beginning of the crushing season.

Quality of Oil from New vs. Old Beans

The difference in the flavor scores of the two samplings might indicate a difference between the oil obtained from fresh beans or beans carried over to the end of the crushing season. Many processors hold to the opinion that soybean oil obtained early in the season is of better quality and of greater stability than the oil obtained at the end of the season. In Table VII are shown results obtained from evaluating oils processed from both new and old beans and deodorized by either commercial or laboratory methods. For oils processed commercially, no difference could be established in the quality of the oil obtained from new or old beans. For three of the four laboratory deodorized samples, a significant difference was shown, and in the fourth the score was higher but not significantly in favor of the new beans. The variability in commercial processing has a greater effect on the quality of the oil than could be demonstrated between oils from new and old beans. The variability in quality as evidenced by the flavor scores shown in this and previous tables is a result of the variable and different deodorization conditions.

TABLE VII

Comparison of Quality of Oil Obtained From New Beans vs. That Obtained at the End of the Crushing Season

Sample	Flavor Scores					
	0-Time		Sig. Diff.	4-Day Storage		Sig. Diff.
	New	Old		New	Old	
	Commercial Deodorization					
Company A	5.4	5.1	+	4.8	5.2	+
Company C	4.5	6.1	**	3.9	4.8	+
Company D	5.5	5.8	+	4.2	4.8	+
Company E	8.0	7.2	+	6.3	5.8	+
	Laboratory Deodorization					
Company A	6.9	5.7	*	6.0	4.7	**
Company C	8.1	5.2	**	6.1	2.4	**
Company D	6.7	5.8	+	5.2	5.1	+
Company E	8.2	6.3	**	6.5	4.3	*

A direct evaluation of the eight pairs of samples was made by the taste panel to determine if there were any statistical difference between the commercial and the laboratory deodorized oils. As shown in Table VIII, two of the three evaluations made of oils obtained from early crushings and deodorized under laboratory conditions gave highly significant results in favor of the laboratory deodorization. For oils obtained late in the crushing season no difference could be established between the two deodorization processes.

TABLE VIII

A Comparison of Commercial vs. Laboratory Deodorization on Soybean Oils Obtained Early and at the End of the 1949 Crushing Season

Sample Description	Flavor Score[a]		Flavor Score[a]		Oxidative Stability A.O.M. 8 Hours 100°C.
	0	Sig. dif.	4-Day	Sig. dif.	
Oil From Early Crushings in 1949					
Company A	4.6	**	3.5	**	70
Laboratory deod.	7.1		5.8		28
Company C	4.1	**	4.1	+	7
Laboratory deod.	8.1		5.1		40
Company D	—		Lost		—
Laboratory deod.	6.4	**	4.5	**	22
Lab. ref. and deod.	8.1		7.3		5
Company E	7.5	+	6.1	+	3.8
Laboratory deod.	7.6		6.0		39
Oil From Late Crushings in 1949					
Company A	6.0	+	4.4	+	65
Laboratory deod.	7.5		4.5		31
Company C	5.4	+	4.6	+	80
Laboratory deod.	6.5		3.8		140
Company D	6.9	+	4.5	+	62
Laboratory deod.	5.4		3.9		44
Company E	7.4	+	6.2	**	2.4
Laboratory deod.	6.3		4.2		52

[a] Flavor scores are from single paired tastings.

From the data presented in the last two tables it might be concluded that there is a difference in the quality of the oil crushed from new and old beans. This difference can be demonstrated by organoleptic scores or oxidative stability measurements. The conditions of commercial deodorization were sufficiently variable to destroy the quality difference found in the oil processed from fresh beans.

Summary

1. "Native" content of iron in crude soybean oil was approximately 0.8 p.p.m., and the copper content was about 0.4 p.p.m.

2. Commercial extraction increased the iron content of crude oil 4- to 8-fold; passage through each of the unit operations increased the iron content.

3. No significant and consistent increase was noted in the copper content at any stage of processing for either the beans or the oil.

4. The contaminating iron accumulated during extraction was not deleterious to the quality or stability of the oil when the crude oils were refined promptly after extraction.

5. Refining removed over 95-98% of the iron present. The removal of copper was in the same order of magnitude.

6. The lowest metal content was observed in the washed oils. The iron content of a refined oil can be lowered to less than 0.1 p.p.m.

7. Commercial bleaching lowered the oxidative stability but not the initial organoleptic flavor score.

8. Deodorization is the most critical and sometimes a very damaging process to the stability and quality of soybean oil. In many cases the deleterious effect

can be attributed to the iron contamination. Observed increases in the iron content have been as high as 40-fold for this one processing operation.

9. Laboratory experiments have shown that the presence of 0.3 p.p.m. of iron during deodorization for 3 hours at 210°C. is very detrimental to the quality and stability of soybean oil. Iron at levels of 0.1 p.p.m. also lowers the initial flavor score but not significantly.

10. Oil from new beans was shown to be significantly higher in quality than oil obtained from beans crushed at the end of the season.

11. Variability in commercial deodorization of oil was sufficiently great to prevent recognition of any flavor superiority resulting from use of fresh beans.

12. Deodorization is the processing step in which improvements most logically could be made on both the quality and stability of soybean oil. A reduction of the metal contamination and the use of a metal scavenger will improve both the quality and stability of soybean oil.

Acknowledgment

The authors are grateful to Robert Beal of the Engineering and Development Division of the Northern Regional Research Laboratory for the collection of many of the commercial oils; to members of the research staffs of Allied Mills Inc., Clinton Foods Inc., The Capital City Products Company, and to the other refiners and processors who cooperated in this investigation but prefer to remain anonymous. Tribute must also be paid to the 18 members of the Northern Laboratory's Taste Panel for their continued support and services in the organoleptic evaluation of edible oils.

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[Received July 16, 1951]

Re-Refining Cottonseed Oil at High Rates of Shear[1]

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RE-REFINING, which is essentially a process for removing color bodies from an oil, is an important operation in the production of cottonseed oils intended for use in the manufacture of shortening and sometimes for other uses as well. Recent records indicate that 20-40% of the crude cottonseed oil produced in some areas is off-grade with respect to color under the trading rules of the National Cottonseed Products Association (5).

Re-refining consists of mixing a small amount of an aqueous solution of sodium hydroxide with a once-refined oil and separating the soapstock and oil. Basically, refining and re-refining are very similar processes except for variations in the strength of the sodium hydroxide solution used and several other details (2, 3), according to the ideas of the operator and the equipment available.

Cavanagh (4) has reported that crude cottonseed oil which has been degummed by the continuous soda ash process will yield a lighter-colored oil if it is subsequently treated batchwise with a solution of relatively concentrated caustic soda while being agitated at very high rates. Although Cavanagh reported data with respect to the effect of a number of variables involved in the process, he did not mention other equally essential data. For example, he reported data for the bleach colors of the re-refined oils, but not for the corresponding oils prior to bleaching, and, more important, he reported no data for the refining losses.

The following report is concerned with the application of high rates of agitation and shear in re-refining caustic soda-refined cottonseed oils, and particularly with the effects produced by the systematic variation of the factors involved in the process as applied to oils of widely divergent origins. These factors are a) rate of agitation and shear of the oil-caustic soda mixture, b) duration of agitation and shear, c) temperature of the mixture, d) amount of caustic soda used, and e) concentration of the caustic soda. The rate of agitation and shear is itself a complex variable, but at present this variable cannot be resolved into simpler components.

The relative effectiveness of the color removed by conventional and high-shear re-refining is compared, and data are reported for the comparative refining losses by the two methods.

Apparatus and Test Procedures

All re-refinings at high rates of agitation and shear were made in a one-liter stainless steel beaker, 12.4 cm. inside diameter. Three baffles, consisting of strips of stainless steel (1.5 x 15.0 cm.), were attached equi-

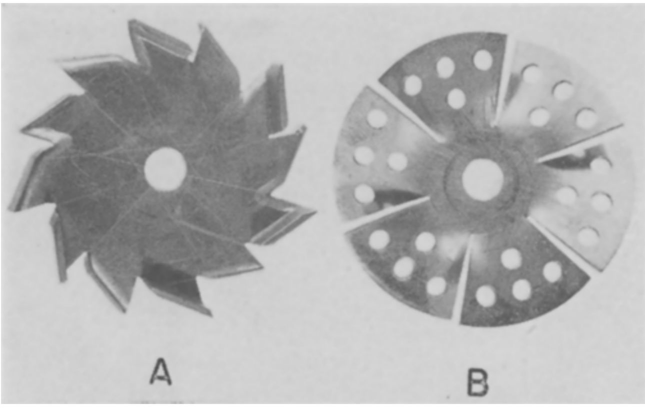

FIG. 1. Impellers employed in high-shear re-refining.

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[1] Presented at the 42nd Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 1-3, 1951.

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