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Design and Operation of a Commercial Soybean-Oil Refining Plant, Using Acetic Anhydride as a Degumming Reagent

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THE CONVENTIONAL REFINING PROCESS for soybean oil has been the combination of degumming, caustic refining, water-washing, bleaching, and deodorizing steps to produce a deodorized, liquid soybean oil. The degumming step is used by those refiners who produce lecithin. For some inherent physical reason it has never been possible to remove more than about 90% of the lecithin in the water-degumming step because water will not completely hydrate the lecithin. Hence a caustic refining has always been required to remove the residual lecithin or "break" material and the free fatty acid. It has long been a challenge to inventive chemists and process engineers to find a way in which 100% of the lecithin or "break" material could be removed in the degumming step. This would eliminate the need for caustic refining because the free fatty acids could be removed in the subsequent deodorization steps.

Such a process, discovered by Hayes and Wolff (2), consists of adding a small amount (about .1%) of acetic anhydride to the crude oil before the degumming step. The subsequent addition of degumming water causes all the lecithin to hydrate and to be removed in one step. The oil is then water-washed, bleached, and deodorized into a high-grade deodorized product without being contacted with caustic. The process is known as the Staley 50 oil process because the fundamental research work was begun in 1950.

The Process Operations

After some five years of continued research and development work the process is today being used commercially in the Staley oil refinery. The flow diagram is shown in Figure 1. The process in this particular case was designed to refine 10 tank cars per day of crude soybean oil.

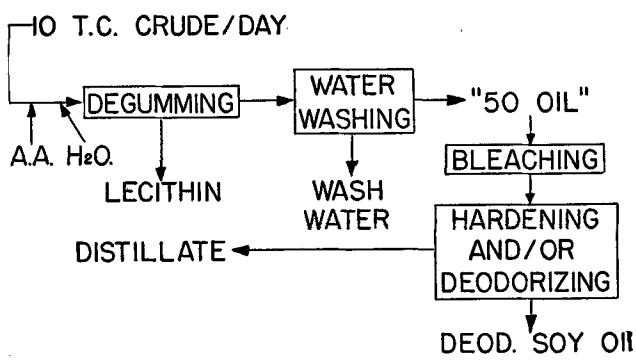

FIG. 1. Flow diagram of the Staley 50 oil process.

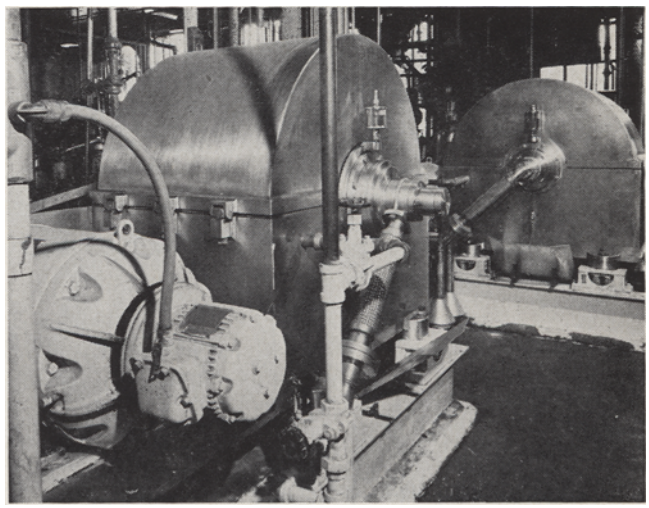

FIG. 2. Staley 50 oil plant, Podbielniak extractors.

The degumming step removes essentially 100% of the "break" material as lecithin at 66–68% acetone-insoluble concentration. The oil is then water-washed to remove traces of acetic acid and final traces of "break" material. The oil at this point is called 50 oil. It is completely acid heat-break free, has good heat-bleach characteristics, and is suitable at this point for finishing into any type of edible product. The 50 oil can also be used for industrial purposes. It is entirely comparable to the conventional once-refined or caustic-processed oil except that the fatty acid content is some .25% instead of about .05%. The hydrogenation step is shown because the 50 oil process is adaptable to the production of either hardened or liquid oil. The data in the paper are concerned chiefly with the production of liquid soybean oil.

The Physical Plant Layout

After some five years of research and development the Staley Company constructed a full-scale, 50 oil plant in the Decatur oil refinery. This unit was placed on stream on February 27, 1956.

The 50 oil physical plant consists of two centrifuging steps. One centrifuging step removes the lecithin, and the second centrifuging step removes the wash water from the oil. The centrifugal separations are carried out in Podbielniak extractors. The degumming operation is performed on two No. 9710 Podbielniak Duozon extractors in parallel while two No. 9715 Podbielniak Hydrozon extractors operate in series as single-stage washers. The oil leaving

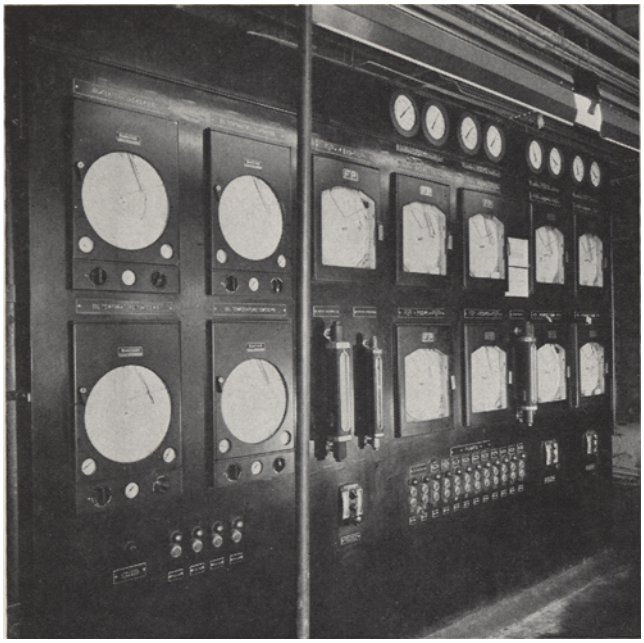

FIG. 3. Staley 50 oil plant, panel board

the last Hydrozon extractor is dried and sent directly to the bleaching operation and thence to the final deodorizing step. Two views of the Staley 50 oil plant are shown in Figures 2 and 3.

Collection of Plant Data

The degumming yield was determined by the amount of lecithin packed during the month. The acetone-insoluble in the finished lecithin averaged about 63%. The washing loss was analyzed on a D. S. basis by oven drying of the wash water. The bleaching loss was determined by analyzing the oil in the cake and by knowing the weight of bleaching earth used.

The deodorization loss needs some explanation because it is a critical factor in this paper. The deodorization losses were determined on a standard three-tank-car per day, semi-continuous Girdler deodorizer. Three streams of distillate-effluent from the deodorizer were measured over the six-month period. These streams were a) the condensate that was collected from the shell of the deodorizer, b) the distillate collected in the entrainment separator in the vacuum system, and c) the organic loss in the down-leg water from the main barometric condenser. The first two streams were collected and measured over monthly intervals. The latter stream was measured daily.

The measurement of the organic loss in the down-leg water bears special mention. The down-legs were sampled four times daily, and the sample was composited. This 32-oz. composite sample was extracted with CCl_4, dried, weighed, and reported as p.p.m. of CCl_4 extract or organic material. The raw water ran about 4 p.p.m. CCl_4 extractable. This was about 5% of the total extractable, and correction was made for it in the final results. The water flow on the down-leg was measured continuously by an orifice meter. Thus the water flow (lbs./day) multiplied by p.p.m. organic gave the organic loss to the sewer in lbs./day.

Operational Results

The operational results for the six-month period of March through August are shown in Table I.

TABLE I
Operational Results of the Staley 50 Oil Plant

Period	% De-gumming Yield	% Washing Loss	% Bleaching Loss	% Deodorization Loss
1956				
March	3.46	.127	.301	.54
April	3.38	.117	.302	.54
May	2.99	.063	.32	.57
June	3.52	.125	.303	.51
July	3.52	.125	.297	.61
August	3.08	.114	.297	.50

Since February 27, 50 oil has been used continuously as the feedstock for the deodorizers of both the batch and the semi-continuous type. The quality level of the deodorized soybean oil showed no difference within normal variance before or after the 50 oil plant start-up (Table II). This point had been illustrated repeatedly to management's satisfaction in the laboratory and on pilot-plant scale before the commercial scale plant was authorized for construction. Hence this quality factor merely substantiated numerous previous observations and conclusions; namely, that the quality of the deodorized, liquid

TABLE II
Flavor Evaluation by Staley Control Laboratory of Daily Production of Deodorized Liquid Soy Oil

Period	Process	% Production [a]	No. Samples Tested
January through December, 1954	NaOH	92	905
January through September, 1955	NaOH	94	745
October, 1955, through January, 1956	NaOH	95	346
March through August, 1956	50 Oil	93	480

[a] Flavor score of 7 or above.

or hardened soybean oil from the Staley 50 oil process is exactly comparable to the caustic-refined product with respect to taste and stability.

Crude Oil Quality

The quality of the crude soybean oil fed the 50 oil process over the six-month period of time is outlined in Table III. At least 15 individual determinations are included in the figures presented for each month.

Predicted Plant Losses by the Caustic Process

In order to compare the over-all loss achieved by the 50 oil process it is necessary to predict the plant losses which would have been experienced if the same crude oil had been refined by the conventional methods of degumming, caustic refining, and water washing, bleaching, and deodorizing.

TABLE III
Quality of Extracted, Crude Soybean Oil to Staley 50 Oil Plant

Period	% FFA	% M&V	% Gardner Break	% Cup Loss
1956				
March	.64	.15	.50	5.1
April	.62	.17	.49	4.8
May	.51	.13	.38	3.8
June	.66	.20	.49	4.4
July	.65	.16	.50	4.5
August	.68	.18	.46	4.4

For several years the following formula has been used at Staley's for predicting plant losses. It was developed by Freyer and Shelburne (1):

% theoretical loss = % FFA + % M&V + 4.37 (% Gardner break).

Over a period of several years the production control department evolved a factor of 1.50, by which to multiply this theoretical value to obtain a predicted degumming, caustic refining, and water washing loss that the refinery can attain a reasonable part of the time. It is important to notice that this predicted loss includes a degumming step, the product of which is sold at 63% acetone-insoluble or conversely 37% neutral oil.

Thus from the data in Table III, the predicted degumming, refining, and water-washing loss is calculated. This is presented in the second column of Table IV. The third column contains the same bleaching loss as obtained on the 50 oil, *i. e.*, the actual and predicted bleaching losses are considered identical. The deodorizing loss is our standard Girdler deodorizing loss on caustic-refined, bleached stocks established over a long period of time at 0.47%.

TABLE IV
Predicted Conventional Caustic Plant Loss, Based on Crude Oil Quality to Staley 50 Oil Process

Period	% Loss D/G-Ref.-WW	% Bleaching Loss	% Deodorization Loss
1956			
March	4.45	.301	.47
April	4.39	.302	.47
May	3.45	.32	.47
June	4.50	.303	.47
July	4.48	.297	.47
August	4.30	.297	.47

Table V shows in greater detail on a monthly basis the performance of the 50 oil plant compared with the predicted losses from the conventional caustic process. All the figures in the fourth column are within the same area of magnitude except May where the saving is only 0.08 lb. It is believed that something unorthodox occurred in the sampling of the crude oil for that month because the cup loss is lower than expected, when taking into account the cup loss of crude oil produced in the Staley extraction plant. In August the degumming yield of 3.08% compares quite well with the 2.99% in May. However the per cent Gardner and the cup loss are considerably lower in May than in August. This indicates that the sampling of the crude in May may have been in error. It is believed that the May result could be stricken since there is reasonable doubt of its accuracy. However, to be on the conservative side, it is included in the figures that make up the average of .89 lb. crude oil saved per cwt. of refined oil.

Discussion of Results

A comparison of the deodorization losses of the 50 oil process compared to the conventional process (Tables I and IV) shows a gain of only about 0.08% loss on an average even though the fatty acid of the feed stock was increased from about 0.06% FFA for conventional oil to about 0.29% on the average for 50 oil (Table VI).

An observer might comment that the deodorization losses of 50 oil might be expected to be increased in proportion to the increase of the fatty acid in the feed stock. This does not seem to be the case from the data collected so far. This has been a point of much deliberation.

TABLE V
Crude Oil Savings of the Staley 50 Oil Process Over the Conventional Caustic Process

Period	50 Oil Process [a]	Conventional Process [b]	Oil Savings [c]
1956			
March	104.72	105.77	1.05
April	104.63	105.63	1.00
May	104.45	104.53	.08
June	104.77	105.76	.99
July	104.87	105.83	.96
August	104.23	105.51	1.28
			Av. .89

[a] Lbs. crude oil required per cwt. of deodorized product.
[b] Lbs. crude oil required per cwt. of deodorized product.
[c] Lbs./cwt. of deodorized product.

A fatty acid balance across the deodorizer was made to see if the total amount of fatty acid distilled was accounted for. This survey showed that 90–100% of the fatty acid distilled could be accounted for each month. The distillate did increase in FFA content from 26% to 50–60%. The feed stock does not contain any measurable acetic acid. Several explanations could be presented to account for this non-relationship in deodorization loss:

The molecular weight of the acidic materials in the oil may be less than the standard 282 assumed for oleic acid.

The total distillables in the plant deodorization may be nearly a constant for both conventional oils and 50 oil, *i.e.*, the fatty acid may displace other less volatile distillables.

These explanations bear further investigation.

The 50 oil process produces more lecithin than the conventional degumming process. The average degumming yield of Staley's crude production for 13 months prior to February, 1956, was 3.15%. The degumming yield of the 50 oil process, excluding May and August, averaged 3.47%. (May and August were excluded for comparison purpose because the Staley extraction plants were down part of each of those months, a fact which voids rigorous yield comparisons). Thus the 50 oil process yields some 0.32 lb. more of lecithin per 100 lbs. finished product. This figure of 0.32 lb. is not obtained by merely subtracting the percentages but rather by an involved mathematical calculation, in which the crude oil requirements are calculated in a reverse fashion, beginning with the deodorization step and working back across the process steps.

The net savings of 0.89 lb. of crude per cwt. of deodorized product deserves further comment. There are three contributing reasons:

The deodorization loss is 0.15% lower than expected.

TABLE VI
Comparison of the Deodorizing Losses in 50 Oil to the Free Fatty Acid Content of the Feedstock

Period	% FFA in Feedstock	% FFA in Deodorized Oil	% Deodorization Loss	% of Fatty Acid Accounted for in Distillates
1956				
March	.22	.010	.54	
April	.29	.009	.54	
May	.21	.009	.57	100
June	.33	.011	.51	90
July	.29	.008	.61	95
August	.32	.012	.50	94

A considerable portion of the savings can be attributed to omission of caustic. The caustic must saponify more oil than formerly suspected.

Part of the 0.89 lb. of crude oil saved results from the close operational control the 50 oil process provides. The dried lecithin is collected, measured, and sampled on a daily basis. The per cent acetone-insoluble is analyzed, and if this property is in the desired range, the refiner knows he is obtaining minimum losses. Also an excess of anhydride, unlike an excess of caustic, does not attack neutral oil and increase losses.

Advantages and Disadvantages

Based on the successful commercial experience of the 50 oil process in the Staley oil refinery the process can be evaluated as follows for a refiner and lecithin producer.

Advantages

0.89 lb. less of crude oil is required for 100 lbs. of finished deodorized product.

0.32 lb. additional lecithin is made.

The deodorization loss is lower than expected.

The flavor and stability of the deodorized oil are unchanged.

No acidulation plant is required with its attendant waste disposal problem.

Saponification of any neutral oil is eliminated.

The net savings of the process amount to some 10¢/cwt.

Disadvantages

The process is corrosive and requires 316 stainless-steel equipment and piping.

The process requires somewhat more care in deodorization.

Some highly colored vegetable oils present special problems for adaption to this 50 oil process.

Economic Significance of the 50 Oil Process

The 50 oil process has economic considerations for two classes of soybean oil refiners:

Class I: Oil producers and/or refiners who degum, refine, and sell finished oils and lecithin

For this category of refiner already established in the lecithin business the monetary advantage of the 50 oil process amounts to 10.6¢/cwt. of deodorized product. The following values were used: crude oil 11.5¢/lb., acid soapstock @ 5¼¢/lb., and lecithin @ 10.5¢/lb.

Class II: Oil refiners who refine only crude into finished oil.

For this category of refiners the lecithin would be acidulated at 72% AI into soapstock. The net savings per cwt. of refined oil are calculated to be 5.2¢/cwt. of deodorized product.

Summary

The 50 oil process has been demonstrated on a commercial scale to be physically possible and economically attractive to refiners of soybean oil. The flavor and stability characteristics of the finished oil are identical to the conventional caustic-refined product.

Acknowledgment

Special acknowledgment is made to the personnel of Podbielniak Inc., whose efforts aided materially in the success of this project.

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The Antioxidant Properties of Garden Cress (Lipidium Sativium) and Wild Mustard (Sinapsis Arvensis) Oils[1]

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LINSEED OIL is obtained from the seeds of the flax plant, *Linum usitatissimum*. Its production dates back to 259 B.C. when sesame, linseed, and castor oils were first recovered by pressing in Egypt (1). Although it contains a high percentage of unsaturated fatty acids, it is used as an edible oil on a rather large scale in Egypt. There is a general belief among its producers that the presence of foreign oils, particularly those of wild mustard *(Sinapsis arvensis)* and garden cress *(Lipidium sativium)*, increases its keeping quality and retards its rancidity. The presence of these foreign oils is attributed to the contamination of flax seeds with those of wild mustard and garden cress. The present work was undertaken to test this belief and to isolate and identify the stabilizers in these foreign oils.

Resistance of fats to oxidation derives from the presence of minute amounts of natural antioxidants, which inhibit oxidative processes (2, 3, 4, 5, 6). Vegetable oils are generally rich in such substances (*e.g.*, lecithin, tocopherol), but animal fats contain only insignificant quantities. Addition of antioxidants provides an important means of increasing stability to oxidative rancidity, but it has not proved successful in delaying flavor reversion in highly unsaturated fats.

Recently Lundberg *et al.* (7) examined the antioxidant properties of samples of 32 spices. Ground mustard exhibited an antioxidant effect on lard when tested by the active oxygen method at 98.6°C.

Materials and Methods

Flax seeds used in this work were obtained from the Ministry of Agriculture farm at Borg El-Arab. Wild mustard and garden cress seeds were procured from the University farm at Giza. Great care was taken to collect the seeds for each plant in as pure a manner as possible. Two methods were used for obtaining the oil from each seed: namely, solvent extraction and pressing.

Measurement of Keeping Quality. The onset and progress of rancidity in the different oils were determined and followed by the determination of the per-

[1] From a dissertation submitted by Moustafa Lotfy to Cairo University in partial fulfillment of the requirements for the Ph.D. degree, May 1954.
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