

Some Further Data on Pressure System Centrifugal Refining¹

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REFINING is one of the most important steps in processing a crude oil. The quality of refining will determine the quality of the finished product, and the efficiency of refining will greatly affect the manufacturer's profits. Increased efficiency should be the keynote of any refining system. In order to evaluate more fully methods of processing, several new refining systems will be described.

Soybean Oil Degumming

Commercial lecithins, for the most part, are produced by water-degumming of crude soybean oil. Essentially a predetermined amount of water is added to the crude oil and mixed thoroughly. By using centrifugal separators, the oil is separated from the gums. This process may be either batch or continuous. The centrifugal separation, until recently, has taken place in an open type of separators which, due to their construction, are limited to a certain viscosity of the discharged material.

By referring to the numerous articles published on this subject and to our own recorded plant information, the average lecithin content of various commercial products may readily be ascertained.

Bailey (1) refers to a typical soybean oil, yielding 3.5% of a material that consists of 25% water and 75% oil-soluble. The oil-soluble will contain about one-third oil and two-thirds acetone insolubles (phosphatides, etc.). This statement from Bailey would show a commercial product about 66% acetone-insoluble on a dry basis. A recent article gave acetone insoluble contents of many commercial lecithins, and again the average was 65% on a dry basis (2).

The pilot plant tests of degumming under pressure, using the Hermetic Centrifuge, gave much higher yields of lecithin. It was possible to discharge from the centrifuge a very high acetone-insoluble gum which was low in oil content. This material was extremely viscous and required from 40 to 60 lbs. p.s.i. pressure to discharge. A product could be continuously discharged containing more than 80% acetone insoluble on a dry basis. The moisture content of the discharged gums before vacuum drying was generally less than 20%.

The pilot plant tests were generally run on lots of 800 to 1,200 lbs. of crude oil at a processing rate of 500 lbs. per hr. through the system. After two or three test runs, of 1,000 lbs. each, were completed, the data were evaluated for the most optimum conditions. A longer test run would then be made which consisted of 3,500 to 4,000 lbs. of crude oil to confirm the initial trials. In this manner, after many long series of test runs, the data could then be safely interpreted for full-size, commercial plant operations.

The pilot plant data were then confirmed by actual commercial installations, using the Hermetic pressure degumming system.

On a commercial installation located at one of the largest soybean oil producers, the dried gums analyzed between 79% to 81% acetone-insoluble on a run of 3½ million lbs.

It is also possible to produce dried gums of lower acetone-insoluble content, if desired, for special purposes. The lower acetone-insoluble content gums may be produced simply by increasing the back pressure on the control valve located on the oil discharge outlet. The increase in back pressure will move the neutral zone or zone of separation out towards the periphery of the bowl. The average acetone-insoluble content of the dried gums was over 77% on more than four months of production analysis from this same commercial installation.

The increased separating efficiency and gain in oil is apparent from the analysis and more particularly from an actual example.

A typical, crude-extracted soybean oil contains 0.5% free fatty acid and 2.0% acetone-insoluble and 2.5% Wesson Loss. When this oil is water-degummed with the conventional open type of centrifugal separator (Case 1), the results would be, on a 100-lb. basis, as follows. Total gums discharged, at 65% acetone-insoluble on a dry basis, would be 3.08 lbs. ($2.0 \div 0.65$). The total oil content of the gums is then 1.08 lbs. (3.08×0.35).

When we degum the same crude soybean oil, using the Hermetic centrifugal separator under pressure (Case 2), the results would be, on a 100-lb. basis, as follows: Total gums discharged, at 80% acetone-insoluble on a dry basis, would be 2.50 lbs. ($2.0 \div 0.80$). The total oil content of the gums is then 0.50 lbs. (2.50×0.20).

This would result in a net gain of 0.58 lbs. of oil per 100 lbs. of crude oil degummed or 350 lbs. per tank car of crude oil treated.

These calculations are based on the same amount of acetone-insoluble material removed from 100 lbs. of crude oil, employing each type of separator. The gain in efficiency lies in the higher acetone-insoluble content of the gums from the Hermetic compared with that from the open type of centrifugal separator. There is less oil in the gums from the Hermetic than from the open type of separator.

The pressure-degumming flow sheet is shown in Figure 1.

These data illustrate the increased efficiency of the Hermetic Separator. The higher efficiency, which results in a 50% savings of neutral oil in the gums, now makes it economical separately to degum crude soybean oil before caustic refining. This permits a reclaiming of the lecithin, followed by an easy refin-

¹ Presented at the spring meeting, American Oil Chemists' Society, San Antonio, Tex., April 12, 1954.

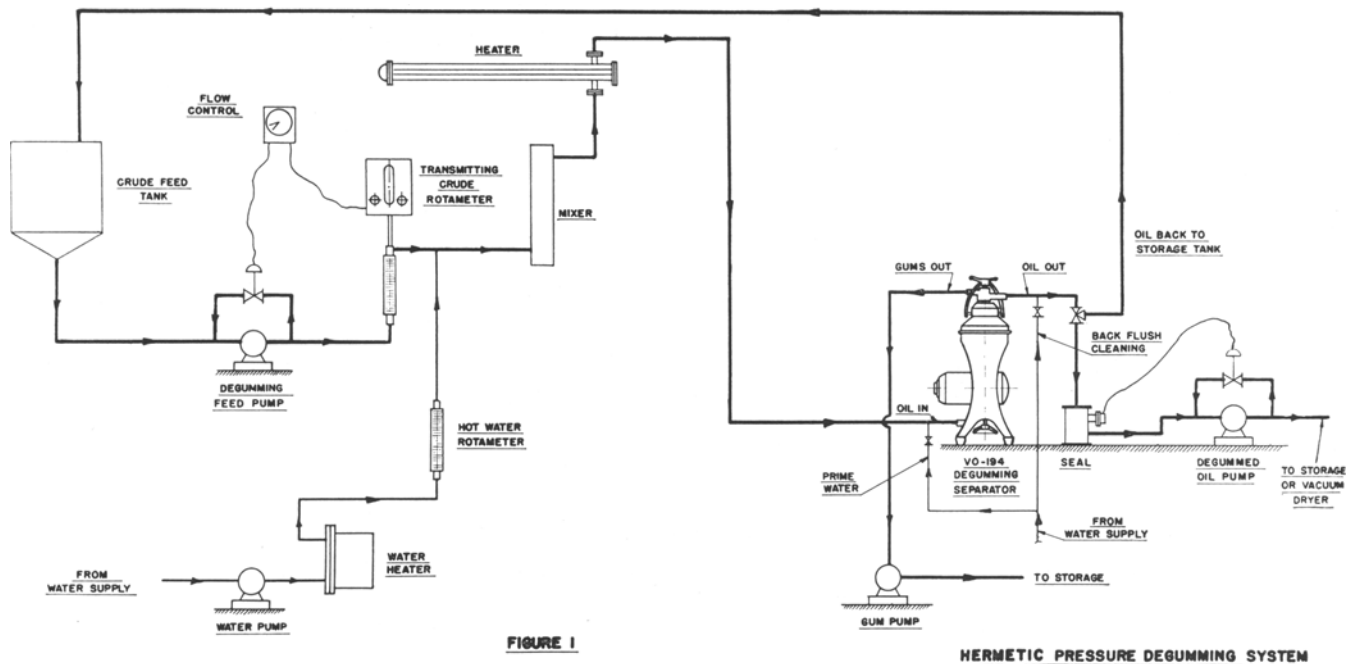


FIGURE 1

HERMETIC PRESSURE DEGUMMING SYSTEM

ing of the degummed oil. Several typical examples of soybean oil treated first by degumming, followed by caustic refining, are shown in Table I.

Pressure Soda Ash Refining

Many refinings have been made in our semi-commercial size pilot plant on crude cottonseed and soybean

TABLE I
Degumming Followed by Refining

Type Oil	Crude Analysis			Total Refining Loss	Savings Compared to Cup
	Free Fatty Acid	Wesson Loss	Cup Loss		
Crude Soybean Extracted.....	0.62	2.30	4.5	2.98	33%
Expeller.....	0.57	2.36	4.5	3.05	32%

All analyses on dry basis.

oil, using the pressure soda ash system according to the teaching of a Clayton patent (3). In this system the crude oil is reacted with the soda ash solution and, after sufficient mixing, is heated and separated while under pressure. The reaction between the free fatty acids and the soda ash proceeds smoothly without foaming as the entire system is under pressure, including the centrifugal separator bowl. In the treatment of some oils we have found superior results to be obtained where the crude oil is preheated to 160°F.-180°F. (71°C.-85°C.) and then separated at 180°F.-200°F. (85°C.-97°C.), all under operating pressure. The separated oil is then treated with caustic for a re-refining color removal step. The total refining losses have been very low, and usually the savings compared to the Official Cup Loss have been between

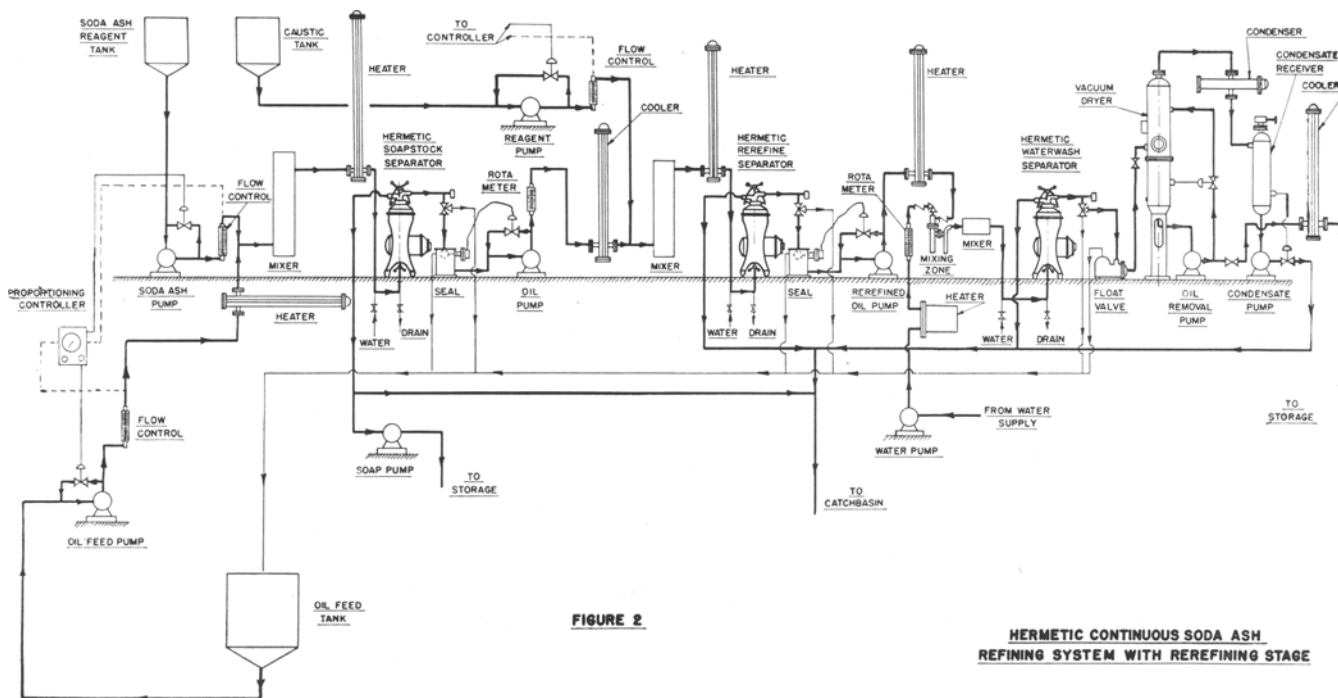


FIGURE 2

HERMETIC CONTINUOUS SODA ASH REFINING SYSTEM WITH REREFINING STAGE

35% to 45%. Several typical examples are shown in Table II.

This system offers several advantages over the conventional soda ash process employing the open type of centrifugal separators. The pressure system elimi-

TABLE II
Pressure Soda Ash Refining Data

Type of Oil	Crude Analysis			Total Refining Loss	Savings Compared to Cup
	Free Fatty Acid	Wesson Loss	Cup Loss		
Crude Cottonseed					
Extracted.....	0.8	2.4	6.2	3.5	43%
Expeller.....	2.0	3.4	9.7	5.8	40%
Expeller.....	6.1	8.6	22.6	13.5	40%
Crude Soybean					
Extracted.....	0.6	2.3	4.5	3.0	34%
Expeller.....	0.6	2.3	4.5	2.9	36%

nates the dehydration step in which the water from the neutralizing soda ash must be evaporated. The pressure system also eliminates the rehydration step which always followed the dehydration in the conventional system in which additional soda ash was added to wet the dried soap-oil mixture. With the low amounts of soda ash solution used in this pressure system the Hermetic centrifugal separator will discharge a soapstock which is high in total fat, generally over 60%, and low in neutral oil. The flow sheet for the pressure soda ash system is shown in Figure 2.

Ammonia Refining

This method of refining utilizes ammonium hydroxide as the primary neutralizing reagent. The refined oil after separation of the ammonia soapstock, or ammonia lecithin as sometimes termed, is caustic re-refined for color removal as in the soda ash process.

The ammonia soap may be heated to near dryness and the ammonia reclaimed. This material then consists of fatty acids, neutral oil, and phosphatides and offers possibilities as a nutritional additive to solvent extracted meal.

The neutral oil loss over the Wesson Loss has been very low, and the savings compared to cup loss have been high, usually more than 40%. A few examples of the results which may be expected from this system are shown in Table 3.

Here again the color of the re-refined oil was comparable to the cup refining and was identical to soda ash refined oil.

In this system, as in the soda ash, there is a tendency for foaming from the gaseous evolution of ammonia unless the system is pressurized. With this system, operating under positive pressure (10-100 lbs. p.s.i.) including the centrifugal separator, the reac-

TABLE III
Ammonia Refining Data

Type Oil	Crude Analysis			Total Refining Loss ¹	Savings Compared to Cup
	Free Fatty Acid	Wesson Loss	Cup Loss		
Crude Cottonseed					
Expeller.....	2.5	3.9	9.9	6.3	36%
Extracted.....	0.8	2.4	6.2	3.4	45%
Expeller.....	6.0	8.6	21.4	12.8	40%
Crude Soybean					
Extracted.....	0.8	2.2	4.1	2.9	29%
Expeller.....	0.6	2.3	4.5	3.0	34%

¹ Includes ammonia refining, caustic re-refining and water washing.

tion proceeds without gasification. Separation will therefore be efficient.

The process flow sheet for the ammonia process is identical to that shown for the Hermetic pressure soda ash system in Figure 2. Ammonium hydroxide reagent, usually 14% as NH₃, is substituted for the soda ash reagent in the former process.

The ammonia soap which is discharged from the primary Hermetic separator may be heated in a jacketed screw type of unit for removal of ammonia and water and then added to the meal. The ammonia from the soapstock may be recovered and reused or may be removed as ammonium sulphate, or a like product.

Evaluation of a Refining System

Along with any discussion of refining systems, the term, "Neutral Oil in the Soapstock," always predominates. When refining with the conventional caustic soda system, the amount of neutral oil analyzed in the soapstock is usually a function of the length of time in the centrifugal bowl and the length of time before analyzing the sample. With the high excess amounts of sodium hydroxide used for refining, any neutral oil that may have been present at the moment of separation is generally saponified by the time the analyst obtains the sample. It is therefore important clearly to define the difference between the soapstock from a saponifying reagent, such as caustic soda, and that from a nonsaponifying reagent, such as ammonia or soda ash. An average crude cottonseed oil was selected, which had the following crude analysis of 0.8% free fatty acid, 2.4% Wesson Loss, and 6.2% A.O.C.S. Cup Loss.

The soapstock from the caustic refining analyzed 43% moisture and only 5.4% neutral oil, or 9.5% on a dry basis, which was apparently very low. The actual refining loss was 4.6%, which represented a savings compared to the official cup loss of 26%.

The true neutral oil loss, before saponification by the excess of caustic used in refining, can only be shown by calculation. This calculated, or true, neutral oil loss, on a dry soapstock basis, would then be 43.5% instead of the misleading 9.5% analyzed in the laboratory.

This same oil was refined with a non-saponifying reagent, ammonia, and had a soapstock which analyzed 34.4% moisture and 9.7% neutral oil. This neutral oil, on a dry basis, would be 14.8%. The difference in analysis now is that the ammonia soapstock represents all the neutral oil lost while the caustic soapstock only shows small portions of the neutral oil lost during refining. The ammonia refining gave a loss of only 2.8%, which was a savings compared to a cup loss of 55%. This oil was caustic re-refined for color reduction, which resulted in a small additional loss of 0.5%, to give an over-all refining loss of 3.3%, or a savings compared to the cup loss of 47%.

It therefore becomes obvious that the neutral oil analysis of the soapstock may be misleading when used to interpret a method of refining. The over-all criterion should be the actual yield of refined oil, the cost of obtaining that yield, and the quality of the final product.

REFERENCES

1. Bailey, A. E., "Industrial Oil and Fat Products," 2nd ed., p. 624, New York, Interscience Publishers Inc., 1951.
2. Stopper, H. R., Saffer, B. A., and Bauer, S. T., J. Am. Oil Chemists' Soc., 30, 401-410 (1953).
3. Clayton, Benjamin, U. S. Patent 2,607,788 (1952).

[Received April 23, 1954]