

Refining Methods for Drying Oils

MORRIS MATTIKOW, Refining Uninc., New York, New York

CRUDE VEGETABLE OILS are mixtures of triglycerides, which for all practical purposes are neutral oil and a relatively small per cent, 0.5 to 2.5% for most common crude oils, of minor constituents, which in addition to the free fatty acids, must be removed substantially completely to produce the refined oils of commerce. Upwards of 75% of the minor constituents but not the free fatty acids are taken out in the degumming of crude vegetable oils. This paper will be devoted to methods of degumming and refining, particularly to the latter, of linseed and soya oils, the two most widely employed in the drying oil industry.

The gums or minor constituents are complex mixtures of chemical compounds, some of which have only recently been identified. The phosphatides (0.5 to 2.5%) are present in the greatest quantity. The unsaponifiables (0.5 to 1.4%) come next in order of magnitude. They include the sterols (0.25%), sterol glycosides (0.03%), tocopherols (0.1–0.2%), and hydrocarbons (0.2%). The coloring matters are carotinoid pigments and chlorophyll (less than 0.01%).

The gums influence greatly the course of refining and determine to a large extent the losses of emulsified or occluded oil incurred. The gums may be removed in a separate operation, called degumming, or may be taken out along with the F.F.A., usually referred to as complete refining, or just refining, in which caustic alkali is the most common reagent.

Extracted crude soybean oils in this country normally contain 1.5 to 3.0% gums and 0.4 to 0.8% F.F.A. These gums have been characterized as precipitable or nonprecipitable with water. Jamieson reported in 1926 that some phosphorus-containing compounds remained in crude cottonseed oil after repeated washings with water (1). More recently a procedure was developed to determine the hydrophilic or hydratable gums (Lipoid A) and the non-hydrophilic or nonhydratable gums (Lipoid B) in crude vegetable oils. The proportion of Lipoid B in crude soya and linseed oil is small. From figures on crude soya oil made available to the writer, the estimate is approximately 5.0 to 10%. It is however the major gum component of a crude soya oil that has been well degummed with water. Lipoid B appears to be resistant to attack by weak caustic soda solutions. Strong caustic soda solutions, 20° Bé. and higher, are employed by some refiners for the effective removal of the residual phosphorus, Lipoid B, from degummed soya oils.

Degumming of crude soya and linseed oil is practiced extensively. The amount of water to use is dependent on the percentage of gums in the crude oil. The hydrated gums occlude oil. In practice this occluded oil is approximately 30 to 35%, dry basis, in the gums, separated centrifugally, when the water added to the crude oil is 1.5 to 3.0% for maximum removal of hydratable gums. It is possible to reduce the occluded oil in the gums by employing less water to leave behind a significant amount of hydratable gums in the degummed oil. The specific gravity of a

crude linseed oil has been made a measure of the water to add for degumming (2). The greater the gum content of the crude oil, the higher the specific gravity. It is recommended that the water to add is equal to the amount of gums.

COMMERCIAL INSTALLATIONS for degumming are most frequently placed in the extraction plant where the crude oil is produced. One type of continuous degumming has two kettles, each fitted with an agitator. The required amount of water is added to the crude oil in one kettle. The mixture is agitated for 30–45 min. at a temperature of 130°–160°F. As stated above, the water will usually vary from 1.5 to 3.0%. The mixture of oil and hydrated gums is sent to a centrifuge. While the mixture from the first kettle is being centrifuged, the second kettle is filled with crude oil, water is added, and the mixture therein is treated in the same manner as the first kettle. There is thus a continuous cycle, with one kettle on stream while the other is being readied. The discharged gums, dried under vacuum in a kettle, are commercial lecithin. For a bleached grade of lecithin a small amount of hydrogen peroxide is incorporated with the gums before or during drying. Other bleaching agents have been proposed, but hydrogen peroxide is the one generally employed.

The other type of continuous degumming is a stream flow process (3). Proportioned streams of crude oil and water, pumped from the respective supply tanks, meet in a mixing zone or in a tee before a mechanical mixer. The mixture goes through a heat exchanger in which the temperature is maintained or raised to 130°–160°F. before entering a centrifuge or battery of centrifuges. Degumming with steam instead of water is also used. The steam, in the desired amount as water when condensed, is injected into the stream of crude oil. This affords an efficient initial dispersion of the reagent (water) in the crude oil.

The gums or minor constituents of crude soya and linseed oil, predominantly phosphatidic in nature, have such nutrient components as choline bound in the lecithins, inositol in the phosphoinositides, glycerophosphoric acid in most of the phosphatides. It is interesting to note that linseed phosphatides have the highest yield of phytoglycolipids, a constituent that Carter and his associates have isolated and largely established its composition (4). Phytoglycolipids contain fatty acids, phytosphingosine, inositol, glycosamine, galactose, arabinose, mannose, phosphate, and cerebronic acid.

Many aids to degumming have been proposed. The number of compounds or substances that have been urged for improving the degree of removal of the gums, reducing the free oil in the gums and producing a better quality degummed oil, is legion: inorganic acids, organic acids, acid salts, salts, to name a few classifications (5). An interesting development has been the acetic anhydride process for degumming crude oils, soya oil in particular (6). The anhydride (about 0.1%) is mixed with the crude oil for a period of time, after which water is added in amounts equivalent to those used in conventional de-

gumming. The acetic anhydride is converted to acetic acid and water. The gums are separated from the oil in a centrifuge. The degummed oil is water-washed to extract the residual acetic acid. The resulting degummed oil, flash-dried *in vacuo*, is claimed to be HCl-break-free.

Degumming crude linseed oil with (0.25 to 2.0%) sulfuric acid, 50 to 85% concentrations, was practiced in the past, but this method of degumming is being resorted to less and less. The action of the acid is one of charring and denaturing the gums that are converted into insoluble, tarry masses that precipitate. Not only must the equipment be acid-resistant, which is expensive, but sulfonation of the oil must be avoided through careful control of mixing the acid with the oil so as to minimize local over-concentration of the acid. The temperature is also regulated to compensate for any rise because the reaction is exothermic. The oil is washed with water to extract the residual inorganic acidity. Sometimes the oil is given a lime treatment to neutralize the free sulfuric acid remaining.

THE GUMS are powerful emulsifiers that influence the loss of neutral oil in refining, which in the fatty oil industry is normally taken to mean the removal of the minor constituents, *i.e.*, the gums and the fatty acids, most commonly by neutralization with aqueous solutions of caustic alkalis, 12° to 20° Bé. caustic soda solutions. Despite the complex nature of the minor constituents, strong alkalis remove them fairly completely whether by adsorption, chemical attack, or decomposition, or a combination of these.

Caustic soda solutions are effective for refining crude fatty oils. An excess of alkali over that required to neutralize the acidity, measured as the free fatty acids and calculated as oleic acid, must be added to obtain the desired quality of refined oil in respect to low F.F.A., less than 0.05%, and low residual impurities, particularly phosphorus compounds and coloring matter. This excess results in saponification of some neutral oil. Refining was carried out with caustic soda solution in excess in large kettles, fitted with an agitator. The soapstock settled by gravity was high in occluded oil.

The first continuous processes of refining crude fatty oils were with caustic soda solution. A stream of crude oil, pumped from a supply tank through a meter or other measuring device, was mixed with a proportioned stream of caustic soda solution (in excess of 0.25 to 0.5%, dry caustic) pumped from a supply tank. The streams went into a mechanical mixer directly or met in a tee or mixing zone before going into the mixer. From the area in which the mixing took place, the emulsion of caustic soda solution and oil passed through an agglomerating coil or heat exchanger in which the temperature was raised to 130°–160°F. The water-in-oil type of emulsion was broken during passage through the coil. The small soapstock particles that first separated from the oil coalesced to form large agglomerates. The mixture of oil and coalesced soapstock particles, kept in suspension by the correct velocity of flow through the coil, was separated in a centrifuge. The centrifuged oil was neutral, and the soapstock was a viscous mass. The refined oil was washed with 10 to 20% of water in equipment similar to the refining step. A centrifuge separated the wash water from the oil, temperature 170°–180°F. A second water-wash step, the

same set-up as the first water-wash, was not uncommon in order to reduce the soap content of the washed oil to below 60 p.p.m. The washed oil was flash-dried in a vacuum drier. The operation was continuous from the pumping of the crude oil and caustic solution to the withdrawal of refined oil from the vacuum drier.

The continuous process of refining crude fatty oils with caustic soda solution was superior to kettle refining in respect to higher yields of refined oil, a more positive control of the physical operation, and the better quality of the refined oil produced. Changes in caustic soda solution and temperatures could be made rapidly as corrective measures for or improvements in the refining without shutting down. If a shut-down was required, the amount of oil in process was very small compared with the 60,000 to 120,000 lbs. in the refining kettle. The losses of neutral oil from saponification with the excess caustic soda and occluded oil in the separated soapstock were still significant.

The next phase in the development of continuous fatty oil refining processes was the substitution of sodium carbonate, soda ash, a nonsaponifying alkali in respect to appreciable attack on the neutral oil, for the caustic soda, with the obvious primary intent of eliminating the loss of oil through saponification (7). The use of soda ash meant coping with the problem of either avoiding the evolution of carbon dioxide or providing means or techniques for effectively removing the gas which interferes with the efficient separation of the soapstock from the oil. Soda ash acts as a weighting agent for the soapstock and is an electrolyte that assists in breaking the emulsion of soapstock. The dehydration-redishydration soda ash process, the Clayton process, of continuously refining crude fatty oils, offered the advantages of a higher yield of refined oils and a simplification of the variables in the technique of the refining operation over straight caustic refining.

SODA ASH SOLUTION (20° Bé.), in the amount of approximately 1.5 times to neutralize the F.F.A. of the crude oil, was mixed with a stream of the crude oil pumped from a supply tank through a heat exchanger, temperature 140°–160°F., in a mixing zone. The emulsion went through another heat exchanger, 200°–210°F., to a dehydrator, maintained under vacuum. The carbon dioxide and most of the water vapor, flashed off, are removed in the dehydrator. The dehydrated soapstock-oil mixture, moisture less than 0.3%, was pumped from the dehydrator and rehydrated with a soda ash solution. The mixture went through a heat exchanger before being centrifuged at a temperature of 195°–210°F. The amount of rehydration soda ash solution (2 to 7%) is enough to make the soapstock sufficiently fluid for continuous discharge. This is one of the most efficient and facile separations of soapstock from oil in the refining of oil. The soapstock is very low in free oil, 3.0 to 10.0%, dry basis, for many crude oils. The separation is so positive that priming of oil into the soapstock or *vice versa*, was practically eliminated. The centrifuge could be operated without change of ring dam in processing different crude oils of varying F.F.A. and gum content. Priming of oil in the centrifuge is not an uncommon occurrence in the continuous refining of fatty oils with caustic soda solution. Changing ring dams in the centrifuge to accommodate to a

change in the strength of caustic treatment is a normal procedure in continuous refining of fatty oils. The dehydration-rehydration soda ash process narrowed the operating variables to the amounts of soda ash for the neutralization and for the rehydration, both of which were adjustments of the valves that controlled the flow of the soda ash solution. The physical appearance of the discharging soapstock was used by the operator to determine the correct amount of rehydration solution. Excesses of reagent in either step means a waste of chemical but no loss of oil by saponification or significant increase in occluded oil in the soapstock because the soda ash is an electrolyte that promotes separation of the soapstock without emulsification of oil. It is important to note that the oil from the primary centrifuge is low in F.F.A., 0.03 to 0.05%.

Soda ash does not attack and remove the color and the phosphorus compounds so effectively as caustic soda. For further color reduction and removal of residual phosphorus compounds from the soda ash-refined oil, the latter is treated, or re-refined as it is commonly termed, with caustic soda solution.

Strong caustic soda solutions, approximately 20° Bé. and higher, are preferred to lower concentrations because the former act more thoroughly on the residual phosphorus compounds and color of soda ash-neutralized oil. The use of strong caustic soda solution in re-refining however resulted in the salting out of the soap by the caustic soda to produce a three-phase system in the centrifuge that can make a clean separation of two phases only. Here the gums play a negative role by their absence. They act as emulsifiers that tend to bind the soap with the caustic in one phase in the separation of caustic soapstock from nondegummed crude soya or linseed oils in which the gums predominate.

The soda ash-neutralized oil is very low in gums. The strong caustic salts out the small amount of soap, which forms an intermediate layer between the oil and the niger. The oil discharges with some intermediate layer and niger, and after a period the oil may begin to prime over into the soapstock discharge. The centrifugal separation is unsatisfactory.

The problem was solved by introducing water into the centrifuge to dilute the caustic soda sufficiently to prevent or overcome the salting out or stratification of the soap in the soapstock (8). In other words, instead of a three-phase system in the centrifuge, a two-phase soapstock, and an oil phase, there is a one-phase soapstock. The flush water dissolves the soap and dilutes the excess strong caustic soda sufficiently to prevent the salting out of the soap that occurs in the absence of the added water. The important practical advantage of this process of re-refining with strong caustic is the very low losses incurred, 0.12 to 0.25% for soya, linseed, and other oils. The re-refined oil is water-washed and vacuum-dried as in conventional continuous caustic soda processes. Refining degummed crude soya oil with caustic soda solution, 20°–30° Bé., with a flush of water in the centrifuge results in an efficient separation of clear oil from the soapstock, which is liquid. A neutral, well-refined oil as to low residual phosphorus and low color is produced.

THE SODA ASH-REFINING PROCESSES are, generally speaking, two-stage refining processes. The gums and free fatty acids are taken out in the first stage

with the soda ash and the residual phosphorus and some color in the second or re-refine stage. Two-stage processes require more equipment and are therefore more expensive to install than single-stage neutralization, straight caustic refining, for eliminating the gums and free fatty acids. The former offer many practical advantages not only in greater yields of refined oil but in simplifying the over-all refining. A first-stage soda ash-refined oil is for all practical purposes "uniform" as to the very small amount of F.F.A. and residual gums it contains, irrespective of significant variations in the amounts of F.F.A. and gums of the crude oil processed. The second-stage treatment with strong caustic soda with a water flush in the centrifuge is a routine, now standardized, procedure. This re-refining may be practiced on straight caustic refined oil to improve the quality of the oil. One refiner, with a dehydration-rehydration soda ash-refining unit has standardized on 2.0% of 20° Bé. caustic soda solution for cottonseed oil and 1.3% for soya oil with a water flush for the re-refine step.

The Short-Mix is a two-step process, the first of which is degumming with water and the second is refining with caustic soda solution. The neutral oil loss incurred in water degumming of crude soya and linseed oil is higher than in soda ash refining. The gums have 30–35% free oil, dry basis, while the soda ash soapstock from a dehydration-rehydration refining has approximately 10.0 free oil, dry basis.

Simplifications of the dehydration-rehydration soda ash-refining process have been proposed. They are all characterized by the elimination of the dehydration step and, as a consequence, of the rehydration too. One is the Modified Soda Ash Process in which 2.5 times (22° Bé.) soda ash solution is mixed with preheated crude oil (9). The mixture goes through a fin-tube heater, 190°–200°F., to a small, closed, pressure de-aeration tank, fitted with a relief valve that is designed to allow carbon dioxide to escape but shuts off automatically for oil. The mixture, under pressure of the system, is led from the bottom of the de-aeration tank to the centrifuge. The neutralized oil is then re-refined as in other soda ash processes. Several refiners with dehydration-rehydration soda ash process units changed to the Modified Soda Ash Process. Nondegummed crude soya oils often require an amount greater than 2.5 times the 22° Bé. soda ash solution to neutralize the F.F.A. because the resulting soapstock is not fluid enough to be discharged at a sufficient rate and begins to accumulate in the centrifuge with consequent dirtying of the oil effluent with soapstock. In those instances, at least four to five times the soda ash solution to neutralize the F.F.A. are needed for a soapstock that is discharged at a normal rate from the centrifuge.

The Modified Soda Ash Process begins to lose its effectiveness when the F.F.A. of the crude oil is greater than about 1.8 to 2.0%. The F.F.A. of the oil from the primary centrifuge then is higher than 0.1%. As a result, the losses in the re-refining step rise.

THE C.S.A. PROCESS is mentioned to illustrate some of the latest techniques of refining fatty oils (10). Although designed to overcome the limitations of some soda ash processes in respect to the F.F.A. of crude oils that can be effectively refined and to reduce the consumption of chemicals, we see no reason

why its principles cannot be advantageously applied to the refining of crude soya and linseed oils. In the C.S.A. Process caustic soda solution, 20 to 40° Bé., usually 24–32° Bé., in an amount approximately enough just to neutralize the F.F.A., is mixed with the crude oil. A small amount of soda ash solution (20° Bé.) is admixed with the emulsion of caustic soda-oil to precipitate the caustic soda soapstock and provide an electrolyte that weights the soapstock while furthering the separation of a low free oil soapstock. It is apparent that accurate, reliable proportioning devices and suitable mixers are essential for optimum yields of oil. The oil from the primary centrifuge of the C.S.A. Process is neutral, F.F.A. 0.03%. It is re-refined with caustic soda solution and a water flush as in the other soda ash processes. An 8-tank-car-a-day C.S.A. refinery unit has been in operation for more than one and one-half years. It was adopted after comprehensive tests in which it was compared with the Modified Soda Ash Process. The operating conditions of the C.S.A. Process are the same as in the Soda Ash processes except that the caustic soda solution is added to the crude oil at 100°–110°F. rather than preheat the crude oil to 140°F. The temperature of the mixture to the primary centrifuge is 195°–210°F.

The purpose of solvent extraction for the production of crude vegetable oils, now the common practice in this country for crude soya oil and being extended rapidly to other vegetable oils, is the maximum yield of crude oil, which means a concomitant meal, very low in oil. Extracted meal lacks the nutritive and caloric value of the 3 to 5% oil that was left in pressed meal and does not pellet as well as the latter. The addition of tallow and other fats, as a rule less expensive than vegetable oils, was recommended for incorporation with extracted meal.

The soapstock product from low excess soda ash-refining of nondegummed crude soya and linseed oil has phosphatides that have not been appreciably decomposed by the alkali. The phosphatides in the product are soluble in petroleum ether. Low excess-soda ash soapstocks from nondegummed crude oils are thus sources of fat and phosphatides. This soda ash product has also been referred to as carbonated lecithin and carbonated phospholipids. The phosphatides in caustic soapstock from nondegummed crude oils have been attacked and decomposed. The addition of low excess-soda ash soapstocks to meal and feed concentrates improves pelleting. Feeding tests on steers (11) and chicks given low excess-soda ash soapstocks at levels of 0.75 to 3.0% of the ration have demonstrated that these products increase growth.

REFINING CRUDE OILS with ammonium hydroxide offers the possibility of producing a neutralized oil and a soapstock that when dried, is a phosphatide product, because the ammonia is removed as a gas under the conditions of drying. This is realized on a commercial scale in the ammonium-hydroxide-refining process recently introduced (12). A 4-tank-car-a-day capacity unit has been in operation in a Midwest soybean oil-extraction plant. Nondegummed crude soya oil is treated with ammonium hydroxide solution. The ammonia soapstock is separated from the oil, which in a second step is re-refined with 20° Bé. caustic soda solution. The separation of the soapstock from the oil and the washing of the oil with

water are accomplished in one centrifuge, a Modified Podbielniak Contactor. The process of combining caustic refining and wash-washing steps in one operation has shown promise. Degummed crude soya oil has been successfully caustic-refined and water-washed in one step with approximately 80 p.p.m. soap in the washed oil. The ammonia soapstock is mixed with the meal in the desolventizer, where the small amount of ammonia is readily volatilized. Ammonia soapstock dried is a lecithin product. The ammonium soaps lose ammonia to revert to fatty acids and the ammoniated phosphatides to phosphatides.

Refining the crude oil in the miscella at the extraction plant has many attractive features, such as minimum loss of occluded oil in the soapstock and reduced saponification loss with large excesses of caustic soda solution. The refining unit is expensive because it must be vapor-tight and explosion-proof. The Thurman continuous miscella refining process has been practiced for the past decade in commercial installations (13).

It is the practice of the refiner to convert the caustic soda and soda ash soapstock to acidulated soapstock or acid oil by adding sulfuric acid in considerable excess. The mass of soapstock is boiled and agitated with live steam. Enough acid is added, and boiling is continued to decompose and degrade the phosphatides and other gum constituents. When acidulation is completed, the mass is allowed to settle. A dark, oily layer that assays upwards of 90.0% T.F.A., to meet trading rules requirements, separates. At best, acid oil is a low-grade fat.

Soapstock from an average nondegummed crude soya and linseed oil has gum or gum decomposition products as the major portion, dry basis. The nonfat components are destroyed on acidulation in order to liberate and salvage the combined fatty matter. The T.F.A. of the acetone-insolubles of the gums of crude soya oil is 55.0 to 60.0%, but only a portion of this is recovered in acidulation. The sterol glycosides and the inositides are not completely split. They collect in the interface and act as emulsifiers that help stabilize the emulsion or heel that often forms between the acid oil layer and the acid waste water.

To speed up the subsequent acidulation and to obtain a maximum T.F.A. in the acid oil, the soapstock is boiled with added caustic soda solution to effect a complete saponification of the occluded oil and hydrolysis of the phosphatides before the sulfuric acid is admixed. This, of course, increases the cost of the operation. A continuous process of caustic saponification of soapstock and salting out of the soaps has been offered (14).

The efficiency of refining the crude oils of commerce has been improved to such an extent for specific processes that the gap between plant refining-loss and the theoretical loss or the maximum neutral, refined oil obtainable has become so small that the attractive features of a new process may depend on factors other than refining loss, such as greater value of the by-products or better or unique qualities of refined oil.

THIS BRINGS US to the important consideration of refining efficiency, which is related to a sound, reliable method for the determination of the maximum or total triglycerides or neutral oil in a crude oil (15). The loss by difference is the Absolute or Theoretical Loss.

The Cup Loss (A.O.C.S. Official Cup Method), which served its purpose both as a basis for trading and as a yardstick for measuring the refining efficiency when refining was done in the kettle, is now recognized by many in the industry to be inadequate, in effect obsolete, in an era of continuous refining. In any event the cup loss does not reflect quantitatively the nature and percentage of minor constituents of the crude oil. The Cup Loss was useful to compare with the loss from the continuous refining process when the latter was introduced about 25 years ago. It was a practical means of demonstrating the greater efficiency of continuous overkettle refining in quantitative terms because the cup loss was considered the lowest loss obtainable with the kettle.

In Europe the refiners express the efficiency of refining as the refining factor which is the plant refining-loss divided by the F.F.A. In this country some have proposed refining efficiency, the percentage of plant yield of refined oil times 100 divided by the percentage of neutral oil in the crude oil. The latter is determined from the Wesson Loss or Chromatographic Loss, most commonly, or the Acetone-Insolubles plus F.F.A. and moisture, of the crude oil. Another method of defining the plant loss is to refer to the Absolute Loss as Unavoidable Loss and the difference between the Absolute Loss and the Plant Loss as the Avoidable Loss. When the Clayton Soda Ash Process was introduced, its efficiency, that is, the plant loss, was expressed as per cent above the Wesson Loss.

Some refiners use the F.F.A., or the acid number, in the acid oil from the acidulation of the soapstock produced as a measure of the efficiency of the refining. The higher the neutral oil, the lower the acid number and therefore the lower the efficiency. The gums and any saponification of oil from excess caustic soda solution are not taken into account in this estimation of efficiency. It is however a practical indication of the occluded neutral oil loss.

The method for the determination of the Wesson Loss, modified by Jamieson, with improvements in the technique and manipulation, has given reliable results. The Chromatographic Loss however is a simpler procedure that is more adaptable to routine analysis. An analyst can set up several columns and make determinations of a corresponding number of crude oils simultaneously (16). This is a practical advantage that would favor the adoption of the Chromatographic Absolute Loss over the Wesson Absolute Loss. The prospects are bright that the Chromatographic Absolute Loss determination (A.O.C.S. Tentative Method Ca 9f57 for Neutral Oil) will be actively used by the industry not only for comparison with the plant loss but as a basis for the value of crude oils for trading purposes.

Chromatographic Losses of nondegummed crude soya oils are higher than the Wesson Losses while the

values for the two agree within experimental error for degummed soya oils. The difference, it is logical to assume, is attributed to the gums. One hypothesis is that in the Wesson Loss Determination the potassium hydroxide solution, in very large excess, hydrolyzes the phosphatides to di- or monoglycerides that are soluble in the petroleum ether solution of the oil. It is to be noted that, in the Wesson Loss determination, the soapstock layer is extracted several times with petroleum ether. Any nonsaponifiables in the soapstock go into the petroleum ether extracts along with the oil.

Results of analysis of several "synthetic" oils in our laboratory support the assumption that the gums are responsible for the higher values of the Chromatographic Loss than the Wesson Loss. An oil made by mixing 97.96% refined deodorized soya oil and 2.04% of a distilled oleic acid gave a Wesson Loss of 2.05%, almost a 100% recovery of the fatty acid added. Another oil composed of 97.65% of a nondegummed crude soya oil, Wesson Loss 2.23%, and 2.35% oleic acid, had a Wesson Loss of 4.60%. The Chromatographic Loss of the crude oil was 3.37%, and 5.80% after the addition of the oleic acid. The fatty acids were recovered quantitatively in both instances. In our opinion the value of the theoretical neutral oil probably lies between the Chromatographic Loss and the Wesson Loss.

In present-day continuous-refining installations that are provided with accurate, reliable proportioning devices, mixers and automatic controls to regulate rates of flow, temperatures, and pressures, and with efficient centrifugal separation, the oil is refined in a matter of minutes. Air can be excluded during the entire refining operation. The oil can be heated or cooled rapidly to the desired temperature in heat exchangers. These features of continuous refining offer advantages in the refining of soya, linseed, safflower, and fish oils, the most commonly used drying oils.

REFERENCES

1. Jamieson, G. S., *J. Oil & Fat Ind.*, **3**, 153 (1926).
2. Kantor, Max, *J. Am. Oil Chemists' Soc.*, **27**, 455-462 (1950).
3. U. S. Pat. No. 2,150,732.
4. Carter, H. E., *et al.*, *J. Am. Oil Chemists' Soc.*, **35**, 335-343 (1958).
5. Andersen, A. J. C., "Refining of Oils and Fats," Pergamon Press, London, 1953.
6. Hayes, L. P., and Wolff, Hans, *J. Am. Oil Chemists' Soc.*, **33**, 440-442 (1956).
7. U. S. Pat. No. 2,190,594.
8. U. S. Pat. No. 2,412,251.
9. Smith, F. H., and Ayres, A. U., *J. Am. Oil Chemists' Soc.*, **33**, 93-95 (1956).
10. U. S. Pat. No. 2,641,603.
11. Virginia Agricultural Experiment Station, Research Report, 1953-1957, p. 137 and 143.
12. U. S. Pat. No. 2,686,794.
13. U. S. Pat. No. 2,260,731.
14. Kieth, F. W., Jr., Blachley, F. E., and Sadler, F. S., *J. Am. Oil Chemists' Soc.*, **31**, 298-303 (1954).
15. James, E. M., *J. Am. Oil Chemists' Soc.*, **32**, 581-587 (1955).
16. Sipos, Endre, *J. Am. Oil Chemists' Soc.*, **35**, 233-236 (1958).