Degumming and Refining Practices in the U.S.

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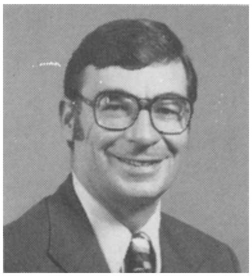

ABSTRACT

The "state of the art" of degumming and refining crude vegetable oils in the U.S. is discussed with reference to (a) production volumes and significant quality characteristics of oils processed; (b) processes and equipment for degumming, refining, and water-washing the oils, illustrated by diagrams and flow charts; and (c) key process controls and analytical testing requirements for each unit operation.

INTRODUCTION

The objective of this presentation is to provide an overview of present degumming and refining practices in the U.S. Available raw materials will be reviewed for utilization and quality features important to these practices. The primary processing system for purifying these raw materials, and the conditions required, will be described using unit operation flow charts and illustrations of key equipment. Finally, control systems for achieving refined oil quality and yield standards will be discussed.

RAW MATERIALS

Utilization

Of the 29.67 million metric tons of vegetable oils produced worldwide in 1974, 55.9% came from three sources: soybean, sunflower, and peanut. Soybean oil dominated with a 30.6% share of the total. A projection of world volumes to the year 1985, calculated from 1965 to 1973 production trends, predicts that soybean oil's share will increase to 33.7% of total production (Fig. 1). The top three oils should then be soybean, sunflower, and rapeseed, comprising 57.2% of the world production of vegetable oils.

On a U.S. basis, the importance of soybean oil is even more significant in view of its 71.5% share of the 4.6 million metric tons of vegetable oils utilized during 1974 (Fig. 2). Other oils of significance to U.S. refiners, by order of importance, are cottonseed, lauric (coconut and palmkernel), corn, palm, peanut, and safflower oils.

Since 1940, U.S. consumption of fats and oils has increased by 40%. During this same period, margarine consumption rose 620%, primarily at the expense of butter. The combined effect increased soybean oil consumption by an astounding 1,300%! As a result, soybean oil is now the primary raw material factor affecting degumming and refining practices in the U.S., as well as the world.

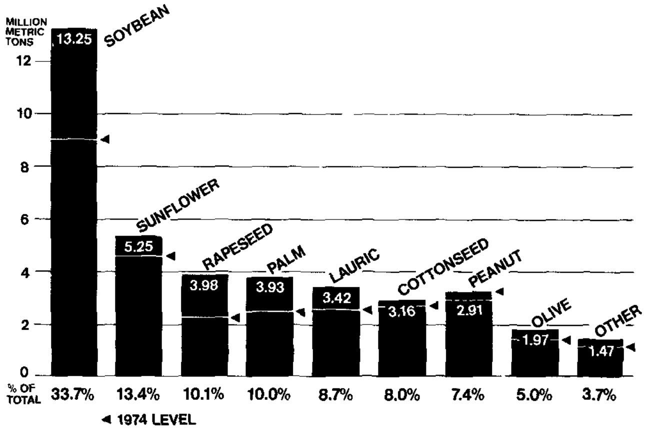

FIG. 1. Estimated world production of vegetable oils in 1985 from 1965-1973 trends.

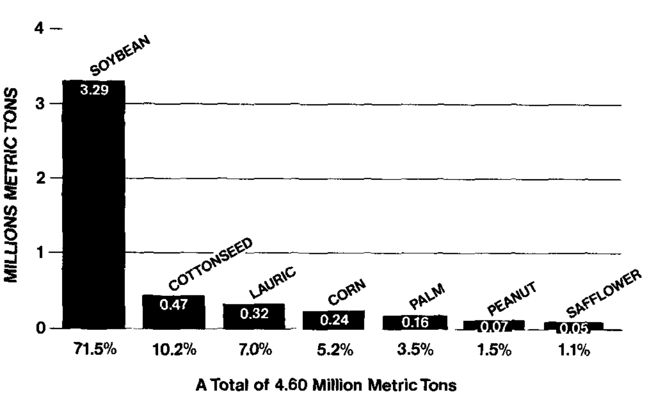

FIG. 2. U.S. utilization of vegetable oils in 1974.

Quality

Crude vegetable oils contain variable amounts of nonglyceride impurities, such as free fatty acids (FFA), nonfatty materials generally classified as "gums" or phosphatides, color pigments, moisture, and dirt. Most of these impurities are detrimental to finished product color, flavor, foaming, and smoking stability, and must be removed by a

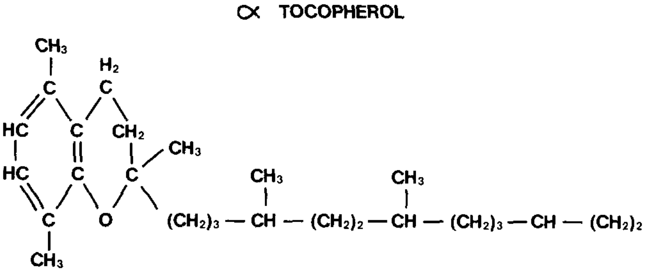

FIG. 3. Natural antioxidants.

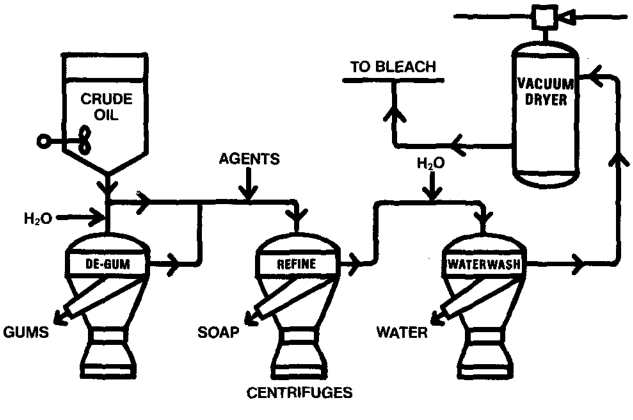

FIG. 4. Primary crude oil processing system (U.S.).

TABLE I

Phosphatide Content in Crude Oils

Crude oil	Phosphatides (%)
Soybean	1-3
Corn	1-2
Cottonseed	1-2
Rapeseed	0.2-2
Peanut	Relatively free
Safflower	Relatively free

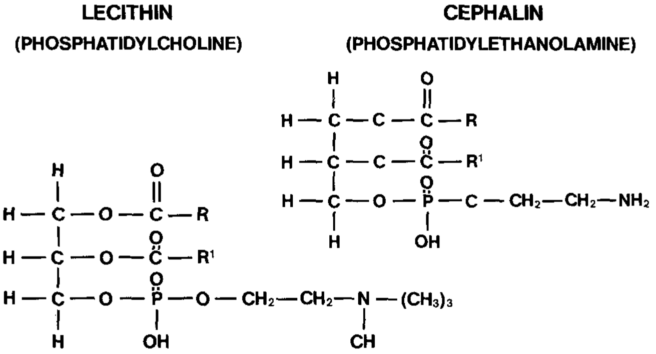

FIG. 5. Phosphatides.

purification step. Tocopherols, however, perform the important function of being natural antioxidants, protectors of oil keeping quality (Fig. 3). Thus, the object of the purification step is to remove the objectionable impurities, with the least possible damage to the neutral oil and tocopherols, and minimum loss of oil during processing.

PRIMARY PURIFICATION PROCESS

The primary crude oil processing system used in the U.S. to achieve this objective is a combination of degumming and caustic soda centrifugal refining (Fig. 4). As an option, crude oil may be degummed before refining by a water treatment, followed by centrifugation to remove the hydrated gums. Crude or degummed oil is treated with caustic soda to saponify impurities, which are subsequently removed as soapstock by a primary centrifuge. The refined oil is washed to remove the last traces of soap in a secondary centrifuge. Refined, water-washed oil is finally dried under vacuum and is ready for additional processing, usually bleaching.

DEGUMMING

Some crude oils, such as soybean oil, contain significant quantities of phosphatides (Table I). Degumming exploits the affinity of phosphatides for water by converting them to hydrated gums, which are insoluble in oil and readily separated by centrifugal action. Because of a strong demand in the U.S. for the lecithin emulsifying agent derived from the hydrated gums, crude soybean oil is frequently degummed prior to refining.

The principal phosphatides are lecithin and cephalin, which are triglyceride compounds with one fatty acid radical replaced by phosphoric acid (Fig. 5). The position of this phosphoric acid radical is important. In the position shown in the illustration, it is termed an alpha-lipoid. If found in the center position, the compound is called a beta-lipoid. Alpha-lipoids are hydratable and may be removed by water degumming prior to caustic refining. The beta-lipoids are not hydratable and must be removed by caustic refining.

Batch water treatment followed by centrifugation is the principal degumming process used in the U.S. (Fig. 6). Water, at ca. 2% of the oil volume, is contacted with crude soybean oil by mechanical agitation in a mix tank. Agitation is continued for 30 min to 1 hr at 70 C to complete gum hydration, being careful to prevent air entrainment during mixing.

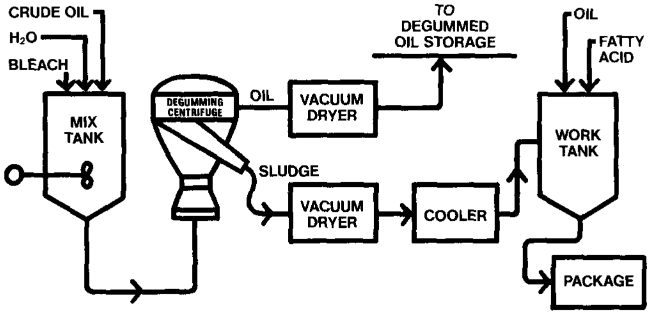

FIG. 6. Degumming crude soybean oil.

TABLE II

Degumming in SRPX-317

Oil discharge (psig)	Gums (dry basis)		Degummed oil heat break	
	AI[a]	Oil	(C)	(F)
45	72	28	252	485
50	69	31	265+	510+
54	65	35	265+	510+

[a] AI = acetone insolubles or phosphatides.

Hydrogen peroxide may be added during hydration if single-bleached lecithin is the desired by-product. If double-bleached lecithin is required, then benzoyl peroxide powder is also added to the mix. For commercial food grade lecithin, the crude oil should be filtered prior to the degumming operation.

Following hydration, the contents of the mix tank are pumped to degumming centrifuges, such as the De Laval SRPX-317. The mixture of oil and gums is continuously separated into sludge and degummed oil phases. Initially, the centrifuge oil discharge pressure control valve is adjusted to obtain a 40-50% moisture content in the sludge phase for satisfactory separation. Back pressure is then fine tuned to achieve the desired characteristics in the sludge and degummed oil phases. De Laval data illustrate that the highest phosphatide content of 72% in the sludge phase was obtained by the lowest oil discharge pressure of 45 psig (Table II). Increasing the oil discharge pressure to 54 psig improved the degummed oil quality, as measured by heat break, but the sludge phase now contained higher oil content and lower phosphatides.

The sludge phase from the centrifuges is vacuum dried at 115 C to 0.5% moisture and cooled to 50 C, prior to pumping to the lecithin work tank. Ca. 7% soybean oil and 3% vegetable fatty acids are blended with the lecithin in the work tank to obtain fluidity at room temperature. This final blend is filled into drums and sold as lecithin.

Degummed oil from the centrifuge is passed continuously through a vacuum dryer and on to degummed oil storage. It may then be sold as degummed crude oil or transferred to the refining process.

REFINING

Of all the unit operations to which vegetable oils are subjected during conversion to finished products, the refining process has the most impact on quality and economic performance. If oils are not adequately refined, subsequent operations such as bleaching, hydrogenation, winterizing, deodorization, etc., will be troublesome, and finished products will fail quality standards for fresh and aged performance. Inefficient refining will also reduce the

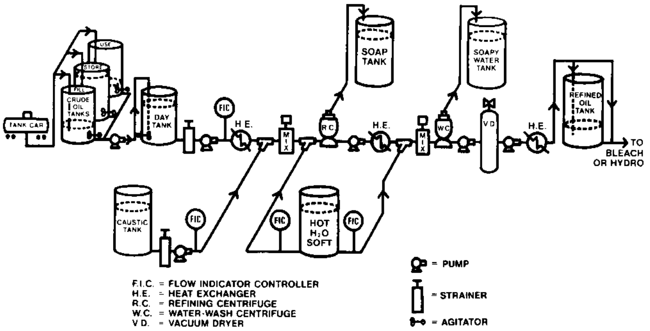

FIG. 7. Primary crude oil refining system (U.S.).

yield of finished product and adversely affect manufacturing profits.

Present refining practices in the U.S. are the result of a gradual application of science to the basic art of batch refining in open kettles, as practiced during the first third of this century. The key technological breakthrough was the development of a "conventional caustic soda refining" process in which a solution of sodium hydroxide was mixed with crude oil and separated by centrifugal action in a continuous process. This new continuous refining system became a commercial reality in 1932.

Despite the development of other techniques, such as the "soda ash," "modified soda ash," "caustic soda-soda ash," "ammonia," "miscella," and "steam refining" systems, the vast majority of refiners in the U.S. are currently using the conventional caustic soda continuous refining system. Science has improved the refining efficiency of this basic system by (a) upgrading the average quality of crude oils by selective breeding and milling advances, (b) evaluating the effects of variables in each refining step, and (c) providing improved equipment for flow control, pumping, heat exchange, mixing, separation, and instrumentation. As a result, today's commercial caustic soda refining system is a relatively simple operation, with the necessary flexibility to efficiently refine all the crude oils presently utilized in the U.S.

Caustic Soda Process

The system may be summarized as follows: Crude oils are received in tank cars, tank trucks, barges, or from the degumming operation (Fig. 7). Receipts are sampled, graded, and then transferred to appropriate storage tanks. As needed, they are pumped by oil type to a day tank in preparation for refining. Crude oil from the day tank is continuously mixed with a proportioned stream of dilute caustic soda solution and heated to obtain a break in the emulsion. Soapstock is continually separated from the neutral oil by centrifugal action. The resultant refined oil is mixed with hot, soft water and again centrifugally separated to remove small amounts of residual soap. This water-washed refined oil, containing traces of moisture, is then passed through a continuous vacuum-drying stage and on to the refined oil storage tank.

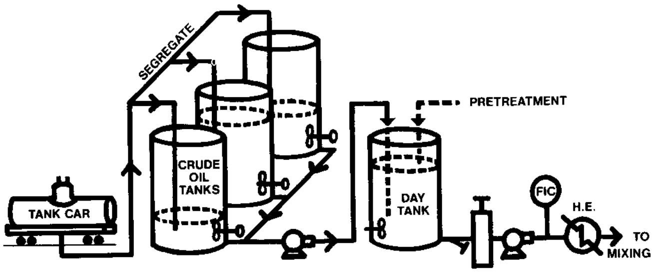

FIG. 8. Crude oils storage and preparation. HE = heat exchanger, FIC = flow indicator controller.

Crude Oil Storage and Preparation

Crude oil receipts, following the sampling and analytical steps, should be segregated by oil type, color, FFA, or refining loss, and, as necessary, by the previous history of the supplier (Fig. 8). Storage tanks for phosphatide-containing crude oils, such as nondegummed soybean oil, should be equipped with slow speed, side entering agitators.

Feedstock uniformity is essential to a steady-state refining operation, hence agitated day tanks are utilized to provide homogenous batches of crude oil sufficient for 24-hr minimum continuous refining runs. Several day tanks may be necessary to provide sufficient time to prepare the crude oil batch, test the oil, and select the appropriate refining conditions.

Crude oils with significant levels of phosphatides, such as soybean oil, are usually treated with 300-1,000 ppm of food grade, 75% phosphoric acid at least 4 hr prior to the refining step (Table III). For optimum refining performance, the acetone insoluble or phosphatide content of degummed soybean oil should be below 0.3%. When this level is exceeded, degummed oil should be blended with nondegummed soybean oil to a 1.0% minimum acetone insoluble level. Crude oils such as palm, palmkernel, coconut, and corn should be rapidly heated to 82-88 C immediately before they are pumped to the caustic-oil mixing step.

Caustic Storage and Preparation

Refiners usually receive concentrated caustic at 50°Bé in tank cars or tank wagons and unload the contents to the caustic storage tank (Fig. 9). Prior to the refining run, concentrated caustic is transferred to the caustic dilution tank and blended with water to obtain the desired concentration. Some installations utilize a caustic-water proportionating system, such as the Bran & Lubbe metering unit, to replace the caustic dilution tank. Diluted caustic of the desired concentration is pumped through a strainer, heat exchanger as necessary, and flow indicator/controller, to the caustic-oil mixing "T".

Key control points for caustic preparation are concen-

TABLE III

Crude Oil Preparation Conditions

Crude oil	Conditioning (ppm H_3PO_4)	Preheating (HE)[a]	(C)	(F)
Soybean nondegummed (NDG)	300-1,000	No	33	90
Degummed (DG)	0	No	33	90
Mixture (NDG+DG)	300-1,000	No	33	90
Cottonseed	0	No	33	90
Lauric (palmkernel and coconut)	0	Yes	88	190
Corn	0	Yes	82	180
Palm	0	Yes	88	190
Peanut	0	No	33	90
Safflower	0	No	33	90

[a] HE = heat exchanger.

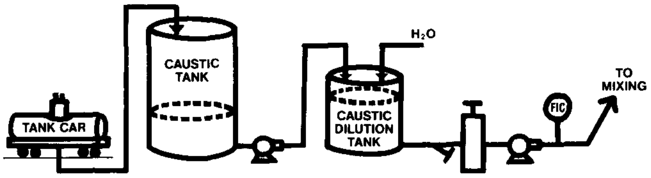

FIG. 9. Caustic storage and preparation.

tration, flow rate or "treat," and temperature (Table IV). Selection of control levels is determined by the type of crude oil to be refined, laboratory tests, past refining experience with similar oils, and refining equipment available. In general, the minimum amount of the weakest strength caustic necessary to achieve the desired endpoint control is used to minimize saponification of neutral oil and prevent "three-phasing," or emulsions during separation. Caustic strengths of 17-18°Bé are usually prescribed for oils other than palm, palmkernel, and coconut. The latter require weaker caustic of ca. 12°Bé to optimize centrifugal separation, reduce saponification of neutral oil, and minimize emulsions.

The treat selected for the crude oil to be refined will vary with the FFA content of the oil and the level of caustic "excess" over theoretical, determined for each oil type from previous experience. Most oils are refined with 0.10-0.13% excess, but there are important exceptions. Lauric and palm oils require a minimal excess of ca. 0.02% because they are refined for FFA reduction purposes. Cottonseed oil is primarily refined for color reduction and requires a larger excess, ca. 0.16%. Diluted caustic, for use with lauric and palm oils, is usually preheated to 65 C to minimize emulsion formation in the separators.

A smooth, reproducible flow of caustic to the mixing T is of prime importance to efficient refining. Pulsating delivery will carry through the mixers and produce varying mixture densities in the separators. Refiners now utilize ratio units with proportional band, reset, and rate action to achieve a nonpulsating reagent delivery to the crude oil. In addition, rotameters are installed in the reagent line for visual observation and emergency manual control.

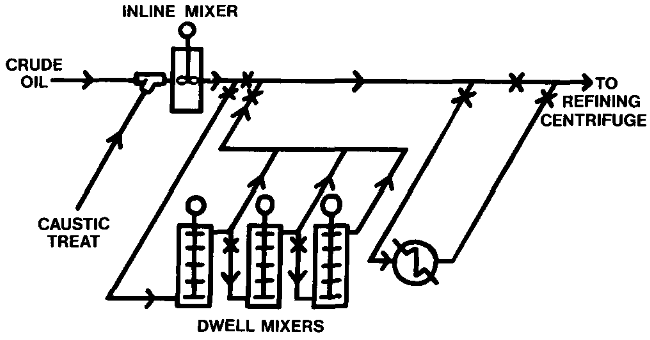

FIG. 10. Caustic-oil mixture treatment.

Caustic-Oil Mixing

After the caustic reagent has been proportioned into the crude oil at the mixing T, it must be sufficiently blended to ensure adequate contact with the FFA, phosphatides, and color pigments (Fig. 10). Caustic reacts with FFA to form soapstock, while hydrolyzing phosphatides and removing unsaponifiable matter from the crude oil. After mixing, the soap-oil blend is heated as necessary to a temperature selected for optimum separation in the refining centrifuges.

The usual mixing system provides a high speed, inline mixer for all oils, plus an optional bank of dwell mixers for soft oils, such as soybean. Suitable piping connections are supplied to allow the inclusion of zero to four dwell mixers in the mixing system. Lauric and palm oils require low speed mixing with only the inline mixer. This mixture should be immediately delivered to the separator to

TABLE IV

Caustic Preparation Conditions

Crude oil	Concentration (° Bé)	Excess[a] (%)	Preheating (HE)[b]	(C)	(F)
Soybean nondegummed (NDG)	17	0.12	No	-	-
Degummed (DG)	17	0.10	No	-	-
Mixture (NDG + DG)	17	0.10	No	-	-
Cottonseed	18	0.16	No	-	-
Lauric (palmkernel and coconut)	12	0.02	Yes	65	150
Corn	18	0.13	No	-	-
Palm	12	0.02	Yes	65	150
Peanut	17	0.12	No	-	-
Safflower	17	0.12	No	-	-

[a]% Treat = $\frac{(\%\ \text{FFA crude} \times 0.142 + \%\ \text{excess}) \times 100}{\%\ \text{NaOH in caustic}}$. FFA = free fatty acids.

[b]HE = heat exchanger.

TABLE V

Caustic-Oil Mixing Conditions

Crude oil	Inline mixer (rpm)[a]	Dwell mixers (Number)[b]	Heating (C)	(F)
Soybean nondegummed (NDG)	1,750	3	74	165
Degummed (DG)	1,750	3	74	165
Mixture (NDG + DG)	1,750	3	74	165
Cottonseed	1,750	4	74	165
Lauric (palmkernel and coconut)	500	0	88	190
Corn	1,750	1	82	180
Palm	400	0	88	190
Peanut	1,750	1	74	165
Safflower	1,750	1	74	165

[a]Nettco mixer equivalent.

[b]De Laval No. 2 equivalent.

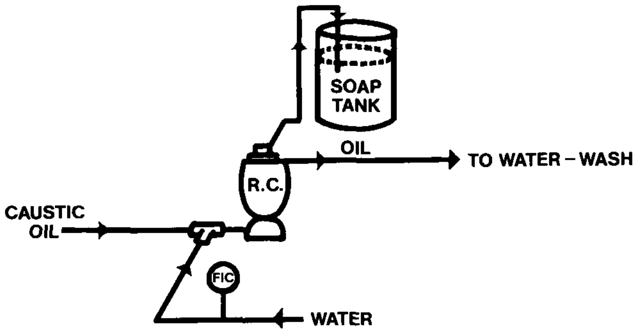

FIG. 11. Refining centrifuge (RC). FIC = flow indicator controller.

minimize emulsification and saponification of neutral oil. Soft oils receive high speed mixing by the inline mixer to obtain intimate contact between the caustic and oil. This is followed by a delay period in a number of dwell mixers, varied to achieve the refined oil endpoint control (Table V). Soybean oil usually requires three dwell mixers for adequate phosphatide removal. One additional mixer is generally used for cottonseed oil to maximize color removal.

Once the mixing operation has been completed, the soap-oil mixture should be delivered to the separators at a temperature suitable for optimum separation. Most soft oil mixtures are heated to 74 C to obtain the "break" necessary for efficient separation, while hard oils bypass the caustic-oil heater.

Soap-Oil Separation

All of the previous operations are in preparation for the primary separation step, the key to refinery yield efficiency (Fig. 11). From the caustic-oil mixer, the resultant soap-in-oil suspension is fed to high-speed centrifuges for separation into light and heavy density phases. The light phase discharge is comprised of refined oil containing traces of moisture and soap, while the heavy phase is primarily soap, meal, free caustic, phosphatides, and small quantities of neutral oil.

In *either a top-feed or bottom-feed centrifuge*, the composition of the phases can be adjusted by changing the position of the neutral zone in the centrifuge. In the old atmospheric centrifuges, zone changes were obtained by modifying the diameter of the heavy phase discharge port and using various ring dam sizes. Most centrifuges in the U.S. are now of the pressure or hermetic type in which zone changes can be readily achieved by adjusting the back pressure applied to the light phase discharge. Regardless of the system employed, complete separation of the two phases can never be achieved.

Increasing the refined oil discharge back pressure reduces the soap content in the oil phase but increases the neutral oil lost in the soapstock. Conversely, reducing the back pressure decreases the neutral oil loss in the soap phase but increases soap in refined oil to a level which is beyond the capacity of the subsequent water-washing step.

For centrifuge startup conditions, most refiners use refined oil back pressure guidelines selected from previous refining experience. Once steady-state is achieved, refined oil back pressure is carefully adjusted until the refined oil, as viewed through a lighted sight glass, becomes slightly turbid from included soap particles. A high speed test tube centrifuge may then be used to determine the quantity of heavy phase left in the refined oil. Back pressure adjustment and test tube centrifuge evaluations are continued until the soap content in the refined oil is compatible with water-washing capability, usually 300 ppm maximum. Refining centrifuges are often equipped with breakover warning systems or automatic sealing devices to minimize high loss of oil in the soap phase during periodic disruptions in the neutral zone balance.

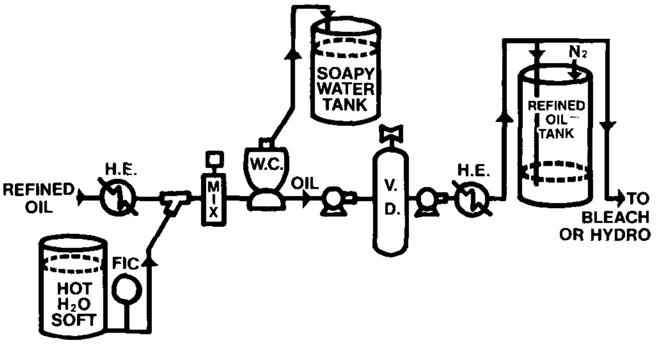

FIG. 12. Water-washing, drying, storage. HE = heat exchanger, WC = water-wash centrifuge, VD = vacuum dryer, FIC = flow indicator controller.

Separation efficiency for palm and palmkernel oils is usually improved by metering water or brine into the soap-in-oil suspension as the mixture enters the refining centrifuge. Water is generally used for crude oils containing < 6% FFA. For higher FFA content crude oils, a 10% sodium chloride brine solution may be used at flow rates not exceeding 5% of the crude oil throughput.

Thick, sticky soapstock from the refining centrifuge can reduce separation efficiency by increasing the soap phase back pressure or blocking the soapstock discharge line. Hot water may be added to the soap discharge port to lower soapstock viscosity to the desired level for satisfactory centrifuge operation.

The refined oil phase, containing minute quantities of soap, is pumped continuously from this primary refining step to the secondary refining stage, namely, water-washing. Soapstock from the refining centrifuge is transferred to the soap tank prior to acidulation or sale.

Water-Washing, Vacuum Drying, and Storage

Refined oil from the primary centrifuge is reheated as necessary to 88 C (Fig. 12). Softened water or recovered steam condensate, at ca. 93 C, is proportioned into the refined oil at a rate of 10-20% by wt of the oil flow. This *water-oil combination passes* through a high speed inline mixer to obtain intimate contact for maximum soap transfer from the oil to the water phase. The soapy water-oil mixture continues through secondary separators, such as a De Laval B-214-C centrifuge. Similar to the action of the refining centrifuge, water-washed oil is discharged as the light phase, and the soapy water solution is the heavy phase. The water-washing operation removes ca. 90% of the soap content in the refined oil, and a single washing pass is usually sufficient. Problems, such as emulsions or high soap content in the washed oil, are typically caused by improper separation at the refining centrifuge.

Washed oil is passed through nozzles into the evacuated section of a continuous vacuum dryer to reduce the moisture content of the washed oil below 0.1%. The typical dryer operates at 70 cm Hg and is equipped with high level alarm and automatic shutdown capability. Before entering the refined oil storage tank, the dried oil is continuously cooled to ca. 49 C. If extended storage is necessary, a nitrogen blanket may be applied to the surface of the oil to minimize oxidation.

CONTROL

Control systems in U.S. refineries are designed to evaluate the quality level of the refined oil, monitor the refining efficiency over the system, and trigger adjustment of processing conditions for out-of-control incidents.

Refined oil quality standards are established for each oil type, compatible with the finished product quality objectives of the individual company. In most cases, FFA

TABLE VI

Refined Oil Quality Control

Oil	Key endpoint
Soybean	Acetone turbidity
Cottonseed	Lovibond red color
Lauric	Free fatty acids
Corn	Free fatty acids
Palm	Free fatty acids
Peanut	Free fatty acids
Safflower	Free fatty acids

content of 0.05% maximum is the primary endpoint control for refined oil (Table VI). Phosphatide-containing crude oils, such as soybean, are normally controlled by the residual gum level in the refined oil. These gums precipitate when the oil sample is treated with acetone. The quantity of precipitate can then be measured by visual comparison with a standard tube or by nephelometric equipment to determine whether or not the oil is fully refined. In the case of cottonseed oil, completeness of refining is controlled by comparing laboratory bleach test colors of plant refined oil vs. similar tests on the laboratory refined crude oil samples.

Refining efficiency is generally considered to be the yield of dry neutral refined oil as a percentage of the available neutral triglyceride content of the crude oil. The former is either measured volumetrically and adjusted as necessary for temperature by specific gravity tables or weighed by scale tanks. Crude oil quantity is determined in a similar manner and then adjusted to available neutral triglyceride by a laboratory loss evaluation.

The laboratory loss method, and hence the efficiency terminology, varies with the oil type (Table VII). Gum-containing crude oils, such as soybean, are usually evaluated by the chromatographic method for lab loss, and the refining efficiency is expressed as the ratio of neutral oil produced over the calculated neutral oil in the crude oil. The lab loss basis for cottonseed and corn oils is the AOCS Cup Loss method, and refining efficiency is expressed as "savings over cup." Palm and lauric oils' refining efficiency is controlled by the refining factor, a ratio of plant loss to FFA content of the crude oil.

Some refiners reduce feedback lag by utilizing the Sodium Balance Method for estimating loss at the primary centrifuges. Metering equipment, such as the Sullivan system, is used to compare flow rates of crude oil input with refined oil output for instantaneous estimates of refining loss performance at any time throughout the run.

TABLE VII

Refining Efficiency

Lab basis	Term	Formula
Chromatographic	Refining efficiency	$\frac{(100 - \%\ \text{plant loss})}{(100 - \%\ \text{lab loss})}$
AOCS cup	Savings over cup	$\frac{(\%\ \text{cup loss} - \%\ \text{plant loss})}{(\%\ \text{cup loss} \times 100)}$
Free fatty acids	Refining factor	$\frac{\%\ \text{plant loss}}{\%\ \text{Free fatty acids}}$

Despite all the improvements in equipment and instrumentation, the prime control factor is the attentiveness of the refinery personnel. Various motivation techniques have been tried, but the best approach is to increase job satisfaction and knowledge. Yield control can be improved by involving operators in refining efficiency goal setting, providing trend charts for tracking yield and quality performance, and clearly defining corrective actions for out-of-control incidents.

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