

Refining and Degumming Systems for Edible Fats and Oils

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ABSTRACT

Crude edible fats and oils contain variable amounts of nonglyceride impurities, such as free fatty acids, non-fatty materials generally classified as "gums," and color pigments. Most of these impurities are detrimental to end product fresh and aged quality characteristics, hence must be eliminated by a purification process before the finished fats and oils are suitable for human consumption. The object of this process is to remove these objectionable impurities with the least possible loss of neutral oil and tocopherols. Key theoretical and practical factors for degumming and refining crude edible oils are discussed with particular reference to processes, flow charts, control systems and analytical testing requirements. In addition to typical large volume oils, such as soya and cotton, techniques are also reviewed for smaller volume oils, including palm, lauric and corn.

INTRODUCTION

The primary system used to purify crude edible source oils is the conventional caustic soda refining process, in which a solution of sodium hydroxide is continuously mixed with crude oil and separated as soapstock from the neutral oil by centrifugal action. As an option, crude soybean oil may be degummed before refining by a water treatment, followed by centrifugation to remove the hydrated gums.

If oils are not adequately refined, subsequent operations such as bleaching, hydrogenation, winterization and deodorization will be troublesome, and finished products will fail to meet quality objectives. Inefficient refining will also reduce the yield of neutral oil and adversely affect manufacturing profits. Hence, the refining process has the greatest impact on the quality and economic performance of today's edible oils manufacturing facilities.

Factors important to an efficient refining operation include the source oils supply, composition, receiving and storage techniques; type of crude oil purification system selected; processing conditions and equipment used in the selected system; and control methods used for yield and quality performance during processing. Each of these factors will be reviewed for their effect on refinery performance.

SOURCE OILS

Supply

Of all edible fats and oils available to the world, and more specifically the United States, soybean oil is by far the most important factor in the supply of source oils to refiners. At the present time, consumption of soybean oil in North America is higher than all other sources of fats and oils combined. Cottonseed, lauric (coconut and palm kernel), corn, palm, peanut and safflower oils are also used by U.S. refiners. On a worldwide basis, soybean oil contributes approximately 31% of the total production of vegetable oils. By 1985, projections of USDA data indicate that soybean oil's share may increase to over 34% of the total. Sunflower and rapeseed oils, combined with soybean oil, will probably supply more than half of the world's production of edible oils. Other oils of international importance are palm, lauric, cottonseed, peanut and olive oils.

Composition

Crude edible oils consist primarily of triglycerides, or triacyl glycerols, which are esters resulting from the union of one unit of glycerine with three units of fatty acids. This is the portion of the crude oil, approximately 95%, that we wish to recover and use as neutral oil in the manufacture of finished products. The remaining nontriglyceride portion contains variable amounts of impurities, such as free fatty acids (FFA), nonfatty materials generally classified as "gums," phospholipids (phosphatides), tocopherols, color pigments, sterols, meal, oxidized materials, waxes, moisture and dirt. Most of these impurities are detrimental to finished product color, flavor, foaming and smoking stability; hence, must be separated from the neutral oil by a purification step. Of these impurities, our primary concern is with the adequate removal of the FFA and phosphatides.

As the name suggests, FFA is the amount of fatty acid, occurring naturally or produced during storage or processing, which exists in edible oils as a distinct chemical unit in an uncombined state. The quantity of FFA present is a good measure of the quality of the crude oil, as well as the purified oil. Some crude oils, such as palm oil, may contain as much as 5% FFA by the time they are readied for processing. FFA content can be lowered by alkali treatment or by other methods to be described later.

Phosphatides consist of polyhydric alcohols combined with fatty acids, phosphoric acid, and a nitrogen-containing compound. Their primary classifications are phosphoglycerides, phosphoinositides, and phytosphingosines. Lecithin and cephalin are common phosphatides found in edible oils. In lecithin, the nitrogen base is choline, while hydroxyethylamine is the nitrogen base for cephalin. Soybean, corn, cottonseed and rapeseed are the major oils which contain significant quantities of phosphatides. Alkali treatment used for FFA reduction is also capable of removing most of the phosphatides from these crude oils.

On the other hand, tocopherols are important minor constituents of vegetable oils. They perform the important function of serving as natural antioxidant protectors of oil keeping quality by retarding the development of rancidity. One of the tocopherols is referred to as Vitamin E. Proper processing allows most of the tocopherols to remain in the finished oils. Animal fats, however, lack natural tocopherols and require the addition of antioxidant agents. 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.

Receiving

Domestic crude oils are generally purchased and graded for quality according to industry-accepted rules, such as the National Soybean Processors Association Trading Rules Book, or the Trading Rules by the National Cottonseed Products Association. Prior to the unloading of crude oils from tank cars, trucks or barges, a representative sample is taken for comparison with the official loading sample of the lot submitted by the mill. Soybean oil, as one example, is usually evaluated by the American Oil Chemists' Society (AOCS) methods for percent FFA, neutral oil loss, bleached color, odor and green color. Hard fats receipts, such as tallow and palm oil, can be damaged by extended heating periods in colder climates, prior to unloading. An unloading shed with heated agitators can reduce the heating/unloading period from two days to ca. five hours.

Following these sampling and analytical steps, the weighed receipts should be segregated by oil type, color, FFA or refining loss, and, as necessary, by the previous history of the supplier. Some companies assign their quality control (Q.C.) department the responsibility of authorizing the unloading of all receipts. If poor quality receipts are significant, a supplier/quality monitoring system will highlight the suppliers causing the problem. An ongoing segregation and blending program will minimize the complications of the purification steps due to variability in the raw materials.

Storage

Storage of crude oils is necessary to build up sufficient inventories to minimize oil losses associated with frequent start ups and shutdowns during short refining runs. The storage objective is to eventually deliver oil to the refining operation as close as possible to the quantity and quality of the crude oils received.

Problems can start from the moment crude oils are unloaded. Physical loss of oil can be caused by faulty piping design, leaking valves and inadequate volumetric or weighing equipment. Cross-contamination of oils can occur from pumping errors due to operator mistakes, poor equipment, or faulty system design. Improper storage conditions will result in significant quality degradation from FFA rise, color reversion or fixation, and damage to the oils' natural keeping quality or shelf life. Quality degradation during storage is generally caused by moisture, temperature, metal soaps, oxygen and equipment condition or design.

Moisture is to be avoided for several reasons, the most important being that it causes fat hydrolysis with a resulting rise in FFA. This deterioration will produce higher refining losses, hence higher processing costs for crude oils. Contamination by moisture can also degum soybean oil in the storage tanks. In order to keep moisture content low, the moisture of the crude oil should be carefully checked when received. Any crude with a high moisture (e.g., above 0.1%) should be rejected or used as soon as possible after receipt. Heating coils must be kept in good repair to prevent moisture leakage into storage tanks, and the storage tanks should be rubbed out regularly to prevent a buildup of moisture and sludge.

Oils should be stored at as low a temperature as possible. When stored in proper tanks which will not be used for some time, they should be allowed to cool down to ambient temperature. Those oils in tanks from which pumpings are being made regularly should be kept liquid at a temperature just high enough to prevent graining. This is usually on the order of 5 C above the complete melting point of the stock. The rate of oxidation of an oil increases rapidly as temperature increases. A general rule is that for each 10 C increase in storage temperature, the oxidation rate triples. Additionally, with elevated temperatures, water solubility increases in the oil, and the rate of hydrolysis increases, causing an increase in the rate of FFA rise and metal soap formation.

During processing, oils contact metal surfaces of storage and handling equipment in the presence of FFA and moisture. All metals, with the exception of aluminum and nickel, exert varying degrees of catalytic prooxidant effect. This detrimental effect is accentuated by increases in temperature and the formation of metal soaps by the interaction of metals with FFA.

Storage or handling activities which increase the chances of oxygen coming into intimate contact with the oil should be eliminated. A typical oil can absorb 2% by volume of oxygen when stored under air. If any significant portion of this absorbed air reacts with the oil, flavor deterioration will result. When storing oil under air, tanks should be filled completely so that the oil surface-to-volume ratio is mini-

mized and the loss in quality is reduced. Oil storage under nitrogen is desirable because of the stability protection. It is good practice to periodically monitor oils in extended storage for FFA, bleached color, peroxide value and moisture content. Lots showing deterioration trends should be refined promptly.

Storage tanks should be empty, free of foreign matter, with coils tested for leaks, before filling with new oil. Tank design should include side-entering agitators, a cleanout manhole at ground level, bottom-filling capability, adequate inventory and temperature measuring equipment. Steam pressure to the heating coils should be 10 psig maximum and the flow controlled by a temperature indicator controller (TIC). Where possible, provide separate piping and tank systems for expensive oils with critical physical properties. Provide adequate insulation and heating to piping and tanks used to handle oils in colder climates. Tank farm design should provide the capability of blending degummed and nondegummed oils prior to refining.

CRUDE OIL PURIFICATION

Crude oils which have been carefully graded, segregated and stored are now ready for the first processing steps towards their conversion to finished products. The objective of this step is to refine or cleanse the crude oils of undesirable impurities, with minimum loss of the neutral oils and natural tocopherols. We use the term "refining" for any purification treatment designed to remove free fatty acids, phosphatides, gums or other gross impurities in the oil.

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. Crude oil may be degummed before refining by a water treatment followed by centrifugation to remove the phosphatides as 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.

Processors have the option of approaching this purification step in two ways. Soybean oil phosphatides can either be recovered as valuable by-products through degumming, or considered as bothersome impurities which must be removed from the oil to produce finished products with satisfactory quality attributes.

DEGUMMING

Degumming is the treatment of crude oils with water, salt solutions or dilute acids such as phosphoric to remove phosphatides, waxes and other impurities. Some crude oils, such as soybean, contain significant quantities of phosphatides. 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 (ca. 100,000 tons per year) for the lecithin emulsifying agent derived from the hydrated gums, crude soybean oil is frequently degummed prior to refining. This soybean oil by-product is the only current commercial source of lecithin used as emulsifiers in margarine, salad dressings, shortening, peanut butter and many other products.

The principal phosphatides, lecithin and cephalin, are triglyceride compounds with one fatty acid radical replaced by phosphoric acid. The position of this phosphoric acid radical is important. When it is attached to an outer carbon link with the glycerol molecule, it is termed an Alpha-

Lipoid. If found in the center position, the compound is called a Beta-Lipoid. Alpha-Lipoids are hydrateable and may be removed by water degumming prior to caustic refining. The Beta-Lipoids are not hydrateable and cannot be removed during degumming.

Degumming by batch water treatment followed by centrifugation is commonly practiced in the U.S. 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 ca. 30 minutes at 60-70 C to complete gum hydration, being careful to prevent air entrainment during mixing. In a continuous hydration system, the preheated oil is treated with water and mixed in a hold tank for ca. 15 min. The hydrated gums are a complex mixture consisting mainly of phosphatides, neutral oil and water, while the degummed oils are unaltered chemically from the neutral oil in the source crude oils. It is important to add only the amount of water necessary to precipitate the gums, as any water excess causes unnecessary oil losses through hydrolysis. Phosphatide removal efficiency can range from 80 to 95% of the crude oil content.

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 continuously separates into sludge and degummed oil phases. Initially, the centrifuge oil discharge pressure control valve is adjusted to obtain a 40% to 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. Hermetic centrifuges can deliver 65% to 80% Acetone Insoluble (A.I.) gums on a dry basis without sacrificing quality in the degummed oil. Residual unhydratable phospholipids remaining in degummed oil must be subsequently removed by an Alkali refining process.

The sludge phase from the centrifuges is vacuum dried at ca. 90 C for 1-2 min. by semibatch and continuous drying film evaporators to a 0.5% moisture level, then cooled to 50 C prior to pumping to the lecithin work tank. Residence time and temperature control are critical to the production of light colored products. Approximately 7% soybean oil and 3% vegetable fatty acids are blended with the lecithin in the work tank to obtain fluidity at room temperature. As an option, this final blend can be bleached with peroxides prior to filling for sale as lecithin. For commercial food-grade lecithin, the crude oil should be filtered prior to the degumming operation.

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. Removal of gums prior to alkali refining often improves the overall yield, because phosphatides act as emulsifiers in a caustic solution and increase the quantity of neutral oil entrained in the soapstock.

REFINING SYSTEMS

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 yield of finished products and adversely affect manufacturing profits. Commercial worldwide purification systems include physical refining, miscella refining, the Zenith process and alkali refining.

Physical Refining

When dealing with high FFA and low phospholipid content crude oils, physical refining, or steam refining, can provide an economic advantage over caustic refining. In Europe, physical refining of high-FFA oils has been utilized for many years to deacidify source oils down to lower FFA levels of 0.2-0.5%, prior to the traditional caustic refining process. Domestic interest in this system increased coincidental to the rise in palm oil's share of the world edible oils market. A fullscale plant was constructed in 1973 at Portland, Oregon, to steam refine crude palm oil.

The process, similar to deodorization, is a steam distillation of water-degummed and bleached oil. Source oil is pumped through a deaerator to a deodorizer-deacidifier with a dwell time of ca. one hour at 260 C and 1 mm mercury vacuum. Injection of steam volatilizes most of the free fatty acids, which are condensed and collected in a tank separate from the neutral oil.

When used with high-acidity crude oils, physical refining can reduce the loss of neutral oil in by-products, reduce the number of unit operations in the purification process and eliminate the acidulation problems associated with the soapstock by-product produced by alkali refining. On the other hand, this process is not as successful as alkali refining in readily achieving low FFA contents in the refined oil and satisfactory oil colors during the subsequent bleaching step. Thorough removal of prooxidants and phosphatides is crucial to preparing quality oils by steam refining techniques.

Miscella Refining

Facilities with an existing oilseed solvent extraction system may find miscella refining to be an advantage by using one solvent recovery unit for both purposes. It can be performed by either a continuous or a batch process for most U.S.-utilized fats and oils such as soybean, cottonseed, palm, safflower, sunflower, tallow and coconut.

The process usually starts with the blending of one to two parts of an inert solvent with one part of source oil for refining in a mix tank. Two solvents are presently being used commercially in miscella refining. Hexane is used in the De Laval, Ranchers and De Smet systems and acetone in the Vaccarino Process. The blend may then be treated with chemicals to condition gums and pigments prior to the addition of caustic soda. Properly conditioned miscella is homogenized, heated to 65 C, cooled to 45 C and then passed through a centrifuge to separate the two phases into refined miscella and soapstock. The former, or light phase, is filtered and either stripped of hexane to produce the refined oil or passed on to the next miscella-based process such as winterization.

Miscella refining, when compared to conventional methods, can result in lower refining losses, lighter colored oil and elimination of the need to water wash and vacuum dry refined oil. Today, more than a dozen plants are miscella refining vegetable oils around the world.

Zenith Process

Another refining method, used by a number of refineries in Europe, is the Zenith Process. After a pretreatment with concentrated phosphoric acid, followed by sludge removal if necessary, the oil is neutralized as droplets, rising by gravity through a lye column. The alkali lye is weak and keeps the formed soap in solution. This virtually eliminates the formation of emulsions and any saponification of neutral oil. It is claimed that water washing of the neutralized oil is not necessary, that the yield is very much improved and that the quality of the refined oil is excellent.

Alkali Refining

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 refining method. The present system is 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. The primary advantage of the continuous process over the batch process is a markedly reduced contact time between oil and caustic. This, in turn, reduces the saponification of neutral oil and results in a higher yield. Plants with daily capacities of 20 tons or more will generally use the continuous system in preference to the batch refining method.

Through the years, refining efficiency of this basic system has been improved 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 (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.

CONVENTIONAL CAUSTIC SODA REFINING SYSTEM

Basic Process

The following is a summary of the system. Crude oils are received in tank cars, tank trucks, barges, or from the degumming operation. 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.

Five key factors determine the success of any edible fats and oils refining operations (1) uniform feedstock; (2) proper quantity of refining reagent; (3) proper mixing of reagent and oil; (4) proper residual contact time and temperature control; and (5) efficient centrifugation. Each of the unit operations within the caustic soda process will now be reviewed for features important to the overall refining system.

Crude Oil Preparation

Crude oils are pumped from segregated storage to temporary holding tanks (day tanks) by oil type, quality and quantity, as necessary for production planning requirements. Because feedstock uniformity is essential to steady-state refining operation, these day tanks should be equipped with slow speed, side-entering agitators. This is particularly important for phosphatide-containing source oils, such as nondegummed soybean oil. Tanks should be sized to provide homogeneous batches of crude oil, sufficient for 24 hr minimum continuous refining runs. Smaller batches increase refining losses from frequent start ups and shutdowns. 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. Prior to the refining start up, the crude oil in the day tank should be evaluated for FFA, Neutral Oil or Cup Loss, and Bleach Test on cup refined oil.

Crude oils with significant levels of phosphatides, such as soybean oil, are usually treated with 300 to 1000 ppm of food-grade, 75% phosphoric acid, at least four hr prior to the refining step to increase the efficiency of phosphatide removal during caustic refining. The Phosphoric acid also acts as a sequestering agent by combining with metal ions in the oil, retarding their catalytic potential for oxidizing the neutral oil and facilitating their removal during subsequent processing.

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 1.0% minimum acetone insoluble level. Crude oils such as palm, palm kernel, coconut and corn should be rapidly heated by an in-line heater to 82-88 C immediately before they are pumped through a Cuno type crude oil filter 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. Prior to the refining run, concentrated caustic is transferred to the caustic dilution tank and blended with water to obtain the desired concentration. After thorough mixing, the strength is checked with a hydrometer by the operator. When on target, a sample is submitted to the laboratory for percent NaOH and degree Bé, prior to startup. 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 concentration, flowrate or "treat," and temperature. 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 to 18° Bé are usually prescribed for oils other than palm, palm kernel and coconut. The latter require weaker caustic of ca. 12° Bé to optimize centrifugal separation, reduce saponification of neutral oil and minimize emulsions.

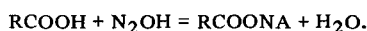
The treat selected for the crude oil to be refined will vary with the FFA content of the oil and the level of caustic "excess" over "theoretical," determined for each oil type from previous experience. The theoretical quantity of caustic is based on the ratio of molecular weights of sodium hydroxide to oleic acid. Most oils are refined with 0.10% to 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 only. 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. This ratio controller permits only the specified quantity of caustic to enter the crude oil stream, even if there are fluctuations in the crude oil flow. In addition, rotometers are installed in the reagent line for visual observation and emergency manual control. Prompt, accurate readings of oil and caustic flow rates are essential for the refining operator to control his refining efficiency.

Caustic-Oil Mixing

After the caustic reagent has been proportioned into the crude oil at the mixing T, it must be sufficiently blended to insure adequate contact with the free fatty acids, phosphatides and color pigments. Caustic reacts as follows with free fatty acids 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 to zero to four dwell mixers in the mixing system. Lauric and palm oils are refined by the "Short-Mix" process which requires low speed, short time mixing with only the inline mixer. This mixture should be immediately delivered to the separator, to 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. The gums are hydrolyzed by water in the caustic solution and become insoluble in the oil. Inline mixing is followed by a delay period in a number of dwell mixers, varied to achieve the refined oil endpoint control. 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 provide the thermal shock necessary to "break" the emulsion of the oil-caustic-soap mass. Too high an oil temperature during the addition of caustic can increase the saponification rate of neutral oil and reduce the yield of refined oil. The degree of refining is controlled by the caustic treat, plus the caustic-oil mixing temperature and time. Hard oils, such as palm and lauric oils, should bypass the caustic-oil heater.

Some refiners use pH to ensure that the reaction mixture contains the proper caustic treat before it is sent to the refining centrifuge. pH targets will range from 9.8 for lauric oils to 10.8 for cottonseed oil. Periodic monitoring, either manually by the operator or by a continuous pH unit, minimizes under refining and excess losses from neutral oil emulsification, saponification and entrainment.

Soap-Oil Separation

All of the previous operations are in preparation for the primary separation step, the key to refinery yield efficiency. 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. These separators are designed to divide suspensions of insoluble liquids and solids in suspension with different specific gravities. The light phase discharge is comprised of refined oil containing traces of moisture and soap, while the heavy phase is pri-

marily insoluble soap, meal, free caustic, phosphatides and small quantities of neutral oil.

Centrifugal separation is several thousands times faster than gravity separation used in batch refining, and the separation efficiency is controlled primarily by the temperature of the oil-caustic feed. The basic principle is that an object in flight will travel in a straight line unless prevented by an outside force. For example, a weight twirling on a string will exert a force away from the center of rotation. The string pulls back on the weight, causing it to take a circular path. The faster the spin, the greater the force generated, until the force becomes great enough to break the string.

A centrifuge contains a bowl or hollow cylinder turning on its axis. As the flow of material to be treated by centrifugal force enters this rotating bowl, it is forced outwards to the disc stack. The flow then separates and the soap, having a heavier specific gravity, is thrown out to the bowl periphery. The lighter specific gravity phase, which is the desired neutral oil, is displaced to the center of the bowl and eventually discharges from the neck of the top disc. In the meantime, the soap phase flows over the top disc and out the soapstock discharge port.

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, or interface, 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.

For good operation, the interface should be near the midpoint between the center and periphery of the bowl. Its position depends upon the refined oil discharge back pressure. Increasing this 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. Key factors improving the completeness of separation are: (a) greater difference in specific gravity of the phases; (b) lower viscosities, (c) higher temperature; (d) shorter travel distance for the heavy particles; (e) increased centrifugal force, and (f) longer centrifuge dwell time.

For centrifuge start up conditions, most refiners use refined oil back pressure guidelines selected from previous refining experience. Once the process starts to line out, 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. The spin test provides a rapid guide to separation efficiency and back pressure adjustment. Spin test evaluations and back pressure adjustments are continued until the soap content in the refined oil is compatible with water-washing capability, usually 300 ppm maximum.

Low loss oils, such as degummed soybean oil, should be separated by centrifuges equipped with bowl flush units to dilute the viscous soap phase. 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 also be added to the soap discharge port to lower soapstock viscosity to the desired level for satisfactory centrifuge operation.

Separation efficiency for palm and palm kernel 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 less than 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. The "Ultra-Short-Mix" method can be used for high acidity palm and lauric oils to shorten contact time further by introducing caustic directly into the hollow spindle of the centrifuge.

Once steady state is achieved, key control features such as flow rates, temperatures and pressures should be frequently monitored and adjusted as necessary. Permanent log sheets for processing data and equipment maintenance are very important for long term satisfactory performance. 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 or "foots" formed during the alkali neutralization of FFA and removed during this primary centrifugation can be sold to soapmakers for further processing, or can be split by sulfuric acid to form a mixture of crude FFA, phospholipids, proteins and other impurities. This mixture can be sold to animal feed manufacturers as "acidulated soapstock" or used as a raw material for the production of purified products, such as fatty acids.

Water-Washing, Vacuum Drying and Storage

Refined oil from the primary centrifuge is reheated as necessary to 88 C. Hot softened water, or recovered steam condensate, is proportioned into the refined oil at a rate of 10 to 20% by weight 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. Wash water temperature is important to efficient separation in the centrifuge, while the water-wash flow rate controls soap removal and oil losses in the wash water.

The control of the water-washing separator is easier than the refining centrifuge and trouble is seldom serious. Breakover will occur only because of carelessness, such as the use of an oversize discharge ring, poor refining conditions, low oil or water temperatures, emulsion in refined oil or an excessively dirty bowl. The only critical factors are the selection of the correct discharge ring and the relationship of the temperatures of the oil and the water. The wash water must be as hot as, or preferably 5 to 8 C hotter than, the oil to prevent emulsions. Problems, such as emulsion or very high soap content in the washed oil, are typically caused by improper separation at the refining centrifuge.

Washed oil at ca. 82 C is passed through nozzles into the evacuated section of a continuous vacuum dryer, which controls the moisture content of the washed oil below 0.1%. A 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.

EQUIPMENT

The overall refining equipment package should be designed and selected for its capability of producing a high yield of quality product, with operating flexibility to handle all types of edible fats and oils. For most refining installations, hermetic centrifuges are the cornerstone of

the equipment presently utilized to achieve these requirements. In addition to their efficiency and flexibility, hermetic centrifuges provide a closed, air free system which eliminates the risk of oxidizing the oil during refining. For example, peroxide values of all-hermetic refined oils are ca. 0.2 m.e. vs. 2 to 4 m.e. values for batch refined oils.

Centrifuges

Typical hermetic centrifuges are the De Laval VO, SRG and PX series. VO hermetic machines, such as the VO 194, are widely used by vegetable oil processors for lower volume applications. The interface between the light and heavy phases is controlled by varying the back pressure of the light phase discharge. Capacity of the VO 194 is ca. 5000 lbs/hr. SRG-214 hermetic machines, with ca. four times the capacity of the VO 194, can be used in vegetable oil refining wherever the VO 194 is used, except for miscella refining. The B-214 is used for water-washing in larger refineries, due to its large capacity of ca. 40,000 lbs/hr. It uses the same principle of balanced columns as the SRG-214, but since it is not hermetic, it differs in the method of controlling the interface location. To vary the interface position in the bowl, the diameter of the discharge ring must be changed. The ring traps the heavier layer of water in the bowl, thereby confining the lighter layer of oil under the top disc for discharge from the center of the bowl. PX hermetic machines are used to separate liquids with high solids content and are designed for self cleaning operation. When solids build up in the bowl, a bottom sliding section is hydraulically opened to discharge the solids from the bowl. Within a few seconds, the bowl bottom section returns to its original position and normal operation is resumed. Throughput capacity, depending upon the type of feed product and centrifuge selected, varies from the range of 25,000 to 50,000 lbs/hr.

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. A breakover occurs when the interface suddenly moves toward the bowl periphery and no longer contains the column of oil under the top disc. Large amounts of oil are discharged through the soap outlet in a partial breakover. When a total breakover occurs, the large oil flow quickly forces all the soap from the bowl and all the oil follows through the soap outlet port. Breakovers can happen when the centrifuge bowl is clogged, the refined oil outlet back pressure is too high, or the refined oil temperature or caustic concentration drops considerably. Breakovers can usually be corrected by quickly opening the back pressure valve and readjusting until the soapstock and refined oil are discharging properly. The bowl may have to be reprimed or back flushed to correct large breakovers.

PERFORMANCE

Refinery performance control systems should be designed to monitor the refining yield efficiency over the system, evaluate the quality level of the refined oil and initiate prompt adjustment of processing conditions for out of control incidents.

Yield

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. 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.

Key features requiring close supervisory control are properly calibrated tanks, accurate daily inventories of stocks in all refinery vessels, stock transfer control systems to eliminate overfilling tanks or mixing stocks, and a responsive maintenance program to prevent stock leaks from valves and piping connections. Improper refining conditions can also lose neutral oil through excess saponification, emulsions and process upsets. During refining, the diluted caustic supply and the caustic-oil feed to the primary centrifuge should be checked for sodium content at least once per shift. Soapstock should be monitored for neutral oil content at least every four hours. Wash water from the secondary centrifuge should be composited over a shift and analysed for neutral oil content.

Quality

Refined oil quality standards are established for each oil type, compatible with the finished product quality objectives of the individual company. Final refined oil control samples are generally taken downstream from the vacuum dryer. At this point in the process, moisture content of the oil should not exceed 0.1%. In most cases, 0.05% FFA and 50 ppm soap content maximums are the primary endpoint limits for all refined oils. Phosphatide containing crude oils, such as soybean, are also controlled by the residual gum level. These gums precipitate when the refined 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 base of cottonseed oil, completeness of refining is also controlled by comparing laboratory bleach test colors of plant refined oil vs. a similar test on the laboratory refined crude oil samples.

Feedback

In order for refinery personnel to adequately control yield and quality performance, process instrumentation and analytical information should be promptly returned to their

control station. Any response delay will increase the amount of time that the process operates out of control. Some refiners reduce this feedback lag by utilizing the Sodium Balance Method for estimating loss at the primary centrifuges. Metering equipment, such as the Elliott or Sullivan system, can be used to compare flowrates of crude oil input with refined oil output for instantaneous estimates of refining loss performance at any time throughout the run.

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 provide management feedback for operating personnel to increase job satisfaction and knowledge. Yield and quality control can be improved by involving operators in refining efficiency goal setting, providing trend charts for tracking performance and clearly defining corrective actions for out of control incidents. The same approach can be utilized to upgrade the responsiveness of the plant maintenance program to plant equipment problems.

Troubleshooting

Periodic process control problems will arise, and operating personnel should be trained to quickly identify causes of these problems from the symptoms observed.

Consistently poor yields can be caused by frequent start ups and shut downs from short refining runs, discontinuous refining due to plugging filters or centrifuge bowls exceeding the centrifuges design capacity and improper processing conditions. Excess neutral oil loss to soapstock may be due to high centrifuge back pressure, improper feed temperatures or caustic flow and plugging of centrifuge bowls. High neutral oil loss in the wash water can be caused by the use of an improper ring dam or low water-oil feed temperature.

High refined oil FFA or bleach color indicates under-refining, which can be due to low caustic strength or flow, insufficient caustic-oil mixing, or a rise in crude oil FFA or temperature. Conversely, a refined oil bleach color significantly lower than the crude oil refined and bleach color indicates unnecessary overrefining. If high soap content is observed in the vacuum dried oil, check primary centrifuge refined oil solids by a spin test and adjust back pressure as necessary. Also investigate for low wash water rate and the oil-water mixing temperature. High moisture content in the dried oil is usually due to low vacuum in the vacuum drier, poor separation at the water wash centrifuge or improper oil-water mixing temperature.