A New Continuous Process for The Refining of Vegetable Oil

By E. M. JAMES

A continuous process for the refining of cottonseed and other vegetable oils has for many years been desired by refiners. From time to time such processes, some of them embodying centrifugal machines for the separation of foots and caustic from neutral oil have been proposed and some even attempted on the commercial scale. All without exception failed in practice, on account of the combination of faulty mechanical design and a failure to appreciate or understand the necessity for a proper sequence of mixing, heating and separating the oil and caustic soda solution.

The continuous process for the refining of vegetable oils to be described below is in daily use in many of the largest refineries in this country and has proven eminently successful in daily operation. The process is simple in its essentials and for its mechanical operation depends upon four component parts. These consist in—

1. A proportionometer for accurately proportioning two continuously flowing liquids one to the other,
2. A mechanical mixer,
3. A suitable heater for rapidly raising the temperature of the mixture of oil and lye,
4. High speed centrifugal separators for continuously separating neutral oil and soap stock.

The proportionometer developed for our process is so designed that the rate of caustic feed is definitely regulated by the flow of oil through a meter. The percentage of caustic solution to oil is adjustable through a fine pitch calibrated screw and such adjustment can be made at any time without discontinuing the operation of the unit. For instance, if a refining has been started with let us say 4% of 14° Baume caustic soda, and a sample of the refined oil should not bleach properly when treated with standard earth, according to laboratory procedure, the percentage of caustic may be increased to 4.5 or 5% in a few seconds, merely by adjusting the screw. Conversely, should the refiner feel that he is over-refining the oil with his original lye prescription, the amount can immediately be reduced through the same agency.

Crude oil, drawn from a holding tank, is fed through the proportionometer by means of a centrifugal pump. Oil and lye solutions are discharged through independent lines into the mixer, in which the crude and caustic first come together.

Refining takes place in the mixing unit, consisting in a small chamber providing very intimate mixing for a period of approximately fifty seconds. From the mixer the emulsion is forced through a heater, where the temperature is raised to 140° F. The source of heat is hot water, and circulation of water through the heater is maintained by means of a small centrifugal pump.

On leaving the heater, the mixture of oil, foots and lye enter the feed line to a battery of centrifugal separators connected in parallel where, under centrifugal force, the foots are separated from the oil. Neutral oil is discharged from one spout of the machine, and soap stock from the hopper. The elapsed time from the first contact of oil and lye in the mixer to the discharge of refined oil and foots from the centrifugal is approximately two minutes.

It was found necessary to develop a special centrifugal separator for the continuous refining process, on account of the problem presented by the continuous discharge of heavy soap stock. The centrifugal is of the suspended tubular type, and the rotor or bowl is driven at a speed of 15,000 r.p.m. Driving power is furnished by 3,600 r.p.m. motor, belted to a pulley connected to a spindle which in turn is connected to the bowl through a flexible coupling. A separating force of 13,200 times gravity is developed. This high centrifugal force, acting upon the mixture of soap stock and oil, drives the foots particles (the heavy phase) to the periphery of the bowl, where the mass is discharged through several ports into a hopper integral with the frame and flows away from the machine through a four inch line leading to the foots pan. The refined oil (light phase) free from foots, discharges through another series of ports at the center of the bowl, is collected in the oil cover, and led away to further processing through suitable pipe lines. The machine is completely enclosed, thus preventing the escape of oil and caustic vapors, minimizing the aeration of the oil.

Centrifugal force has two functions in the continuous separation of soap stock and oil. In the first place, it causes the particles of soap stock to settle in a fraction of the time required under gravity, and secondly, under the pressure, the individual foots particles are pressed into a compact mass to form a relatively homogeneous liquid, substantially free of oil, thus greatly reducing the entrainment of oil with the soap stock.

The completeness of subsidence under centrifugal force of one liquid suspended in another depends upon the fineness of division, the viscosity of the continuous *liquid, and the time during which the* mixture remains in the centrifugal bowl, which latter varies inversely with the rate of flow. In the continuous refining operation the rate of flow or capacity per centrifugal for optimum results is *2.5 g.p.m. of crude oil, no allowance* being made for increased percentages of caustic soda solution. Hence, a con*tinuous unit rated at a capacity of one* tank car per day will handle one tank car of crude oil per 24 hours whether the F.F.A. of the crude be 0.6 or 6.0%.

The clarity of the refined oil discharged varies with the individual crude, but the neutral oil is free from suspended soap stock as such. Some oils are bright at the temperature of separation, others are somewhat cloudy, but all contain slight moisture in the form of small amounts of suspended caustic soda solution. Immediately upon discharge the refined oil shows alkaline to alcoholic phenolthalein, but after standing for an hour or so this alkalinity disappears, the caustic having acted upon the oil to form traces of soap. This is true even when an oil is bright as discharged, for no centrifugal can remove dissolved or colloidal moisture from an oil.

The centrifugals used in the continuous process should be cleaned once every twenty-four hours. With many oils longer operation is possible, but as a plant routine this limit has been established. Cleaning is accomplished by first reducing the flow of crude oil through the unit by 2.5 gallons per minute and then shutting down one machine at a time for cleaning. By this method operation is uninterrupted. The solid matter retained in the bowl usually consists in a small quantity of dirt and fibre particles which are too coarse and heavy to be discharged with the soap stock. Most of the meal and settlings pass through with the foots, and the solids removed in each twenty-four hour cleaning seldom exceed three to four pounds per centrifugal bowl.

Many refiners are now washing all of their oils after refining before further processing, such washing being accomplished by adding hot water to the oil, heating, agitating, and settling. In some plants washing is carried out in vacuum kettles, the oil being agitated and dried under vacuum and heat, after the wash water has been drawn off.

As an adjunct to the continuous refining process, at the request of those refiners who wash their oils, a continuous water washing process has been developed. The oil from the refining centrifugals is received in a small tank equipped with an agitator, steam coils, and a temperature regulator. Hot water is continuously added, in amount about 10% by weight, and is thoroughly mixed with the neutral oil. This mixture is heated to 165° F.

From the wash tank the wet oil flows to a battery of centrifugal separators. The wash water is continuously separated and run to the sewer, while the oil is accumulated in a suitable receiving tank, from whence it either goes direct to the bleach kettle for bleaching or to the driers for winterizing or storage.

An analysis of the wash water from a number of runs on cotton and corn oils showed an oil and soap content combined of about 35 pounds per 60,000 pounds of crude oil refined and washed.

The advantages of the continuous washing unit lie in the fact that all lye and traces of soap are removed immediately, and the washing and separation are complete in approximately five minutes' time. There is no loss of oil in emulsion such as occurs in drawing off the wash water from a gravity wash, and the amount of moisture to be removed in the driers is considerably less than in the case of gravity washed oil, unless the latter is allowed to settle for a long period of time.

The continuous refining process described has been operated commercially only on the soft oils; cotton, corn and peanut. Successful experimental runs in semi plant equipment have been undertaken with soya bean oil both domestic and imported and linseed oil.

The continuous process will refine cotton oil with a reduction in loss of approximately 30% below the loss suffered in refining by the conventional batch method. This figure is based upon commercial test runs made with oil originating in almost every part of the continental United States in which cotton is grown.

The average bleach color of the continuously refined oil is approximately equal to the bleach obtained by batch refining.

With cotton oil, a rough idea of the saving in loss to be expected from any given tank of crude may be gained by comparing the ratio of loss to F.F.A. as shown by the settlement sample. When this ratio is low we should expect a somewhat lower saving than when it is high. For instance, to cite a concrete example:

Car	F.F.A.	Settlement Loss	Continuous Loss	% Saving	% Absolute Saving
1	1.4	6.6	4.00	44.4	2.6
2	1.4	5.8	4.10	29.3	1.7

The F.F.A. loss ratio of the first car is nearly normal while that of the second car is low. The continuous process gave almost identical yields of oil with each tank, but the saving in the case of the second was 15% less than with the first.

We have also noted that the relative percentage saving decreases as the F.F.A. of the crude increases, but the absolute saving in refined oil is greater. As an example of this phenomenon:

Car	F.F.A.	Settlement Loss	Continuous Loss	% Saving	% Absolute Saving
1	3.3	11.8	8.8	25.4	3.0
2	8.5	21.9	16.7	24.9	5.2

For an average season the saving in neutral oil throughout the year will amount to approximately 2.5% of the crude refined.

The soap stock produced by the continuous method shows an average F.F.A. of approximately 40.0%.

When corn oil is continuously refined, the saving over conventional batch methods will amount to approximately 25% of the loss normally suffered, and with peanut oil the saving in a comparatively small number of runs on which we have data will amount to approximately 25%.

Work has been done on the possibility of using the Continuous Refining Process for hard oils such as cocoanut, palm and palm kernel. The normal losses by the conventional method on such oils as cocoanut and palm kernel are so low as to minimize the savings possible through loss reduction, so that factors other than reduction in loss must be found to justify the installation of the continuous process on an economic basis. Research along these lines is being continued. However, some refiners are using their washing equipment to wash cocoanut oil with marked success, and have been able to produce as good a washed oil with one wash through the continuous plant as with two or more washes through regular batch equipment.

INTERPRETATION OF COTTONSEED OIL MILL PRODUCTS ANALYSIS

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Hundreds of thousands of chemical tests are made each year on cottonseed and its products, some for settlement purposes, others for control, a few for experimental purposes; a fair proportion, we suspect, are made from habit. What do these tests convey to you? Probably very few of us see in a laboratory certificate all the meaning it carries. The significance of analytical reports varies in considerable degree among the different individuals who have occasion to examine them. To your laboratory-bound and laboratory-minded chemists who make them they often represent hardly more than percentage compositions, although high and low protein values generally spell "loss" to anyone close to an oil mill. To the average mill executive high or low values of certain determinations imply that circumstances or his superintendent is losing or saving him money. The mill superintendent regards laboratory reports as the yardstick of his performance; and well may he! This individual, more closely than anyone, watches the percentages and their daily variations, and considers perhaps their implication regarding the quality of his work rather more than the actual losses or savings represented to his employer. But thanks to this employer, he is not unaware of these.

We have intended to indicate that interpretation of oil mill analyses is generally qualitative. Let us bring the chemical engineering attitude to bear on this matter. Engineering is, above all, quantitative whenever possible. With a slide rule constantly within reach, and accustomed to calculating losses or saving as might be due, for example, to variations in the amount of smoke issuing from his stack, or perhaps to changes in the average daily humidity, we should expect the engineer to examine very inquiringly all the oil mill products analyses for the possibility of quantitatively assigning the effect of variations of the percentages of each of the components or values or properties determined. Unfortunately, the contact between chemical engineers, as a class, and oil mills has been very slight. Although a few superintendents might possess the curiosity and insight to penetrate into the deeper interpretation of their laboratory reports, the majority of them appear to develop their talents rather more on mechanical lines. Admittedly, the problems of an oil mill are very largely mechanical, and it is not surprising that advancement has proceeded principally in this direction.

In this paper, a system of laboratory reporting will be described, which was developed with the purpose of deriving the maximum benefits from chemical control of oil mill operations; or, expressed in plain words, of saving the most money. This is no new aim in the industry, and some of the practices here related also are not new. Our aim has rather been to breathe new life into an old striving by a clear definition of all factors involved, no matter how slight they may appear to be. There is no need to dwell on the necessity of this in these latter days of shrinking spread between manufacturing costs and products' price.

Cottonseed

Our practice in reporting seed results differs in no important respect from the universal one. Under yields, in addition to the oil and cake yield, we report loss in terms of pounds per ton. This figure is determined by the moisture and foreign matter contents and is taken from a table calculated by assuming certain cake, hull, oil, and lint yields and certain moisture contents of these products. The assumption should of course be based on the experience of the locality in which a mill or group of mills occurs. Subtracting the sum of loss and cake and oil yields from 2,000 gives the yield in hulls and lint, which products have been grouped together. A system of lint-cut control based on a direct lint-on-seed determination is in the experimental stage; and should a method of rapid lint estimation, which is being tried, prove to be successful, hull yields and lint yields may be reported separately.

Hulls

The losses due to the hulling and separating operations are probably the most easily evaluated of any of the chemical losses in an oil mill. The meats or meat fragments which go out with the hulls represent just so much cake loss and loss of the oil contained in the meats. The following constituents of hulls are reported: whole seed, meats in whole seed (assumed to be ½ of seed), whole meats, meat dust, and total meats. The calculation of the cake loss from the percentage of total meats is very simple assuming a hull yield of 600 lbs. per ton of seed. It is usual practice to assume meats to be ⅓ oil. As it is so common, however, that meat dust is the only form of meats present, and as varying and uncertain quantities of hull bran may go through the screen with the meat dust in separating it, quantities of these fine screened particles were accumulated and the oil content determined. The average of several determinations was very close to 20%, only ⅗ or 0.6 the oil content assumed for meats. We therefore use a factor of .60 for converting the percent of hull bran-meat dust mixture into percentage of meat dust. The percentage of total meats is divided by three to give the percentage of oil in the meats. Cake loss is given below the analysis expressed in pounds per ton of seed and in cents per ton of seed.

As a basis for calculating oil loss in hulls, one may set an arbitrary value of "total oil in hulls" as representing average good work, and figure a loss or gain in pounds of oil and cents per ton of seed, according to whether the reported value is above or below this standard. Or the minimum amount of oil in clean hulls might be determined by occasionally extracting the hand-separated hulls from carefully hand cut seed, and consider this value as an ideal of practically perfect work toward which the mills should strive. The authors favor and practice this method of evaluation, especially since certain mills under their observation have been able for many days at a time to attain the ideal. Repeated determinations made on seed from various localities in Texas yielded values of the minimum oil content of hulls ranging, in general, from