PROCEDURE

The operation is outlined in Figure 2. The converter is loaded with oil from a scale or a measuring device. The valve for the vacuum system is opened. The vacuum deaerates the oil and prevents it from oxidation. Steam for heating is turned on and the catalyst, previously suspended in oil, is added any time during the heating period. When the oil reaches the specified temperature, the vacuum is closed and hydrogen is added until the specified pressure is attained. Pressure is kept unchanged during hydrogenation. When the exothermic reaction raises the oil temperature close to the maximum temperature specified, water is introduced into the coils. Samples are withdrawn from time to time to determine the refractive index (related to the iodine value) for measuring hydrogenation progress. When the operator judges that the endpoint is very close, the agitation and or hydrogen circulation are suspended until laboratory results are ready. With some practice, operators may determine the endpoint proximity by the pressure drop in hydrogen storage tanks. Each plant has its preference for a method to determine endpoint. Iodine value, refractive index, congeal point, cloud point and C number are some methods used. If this analysis shows that the hydrogenation is over, hydrogen is vented to the atmosphere through the vacuum system, the agitator is run again and the oil is cooled or transferred to a cooling tank or heat exchanger. The cooling continues until a temperature of 65 C is attained. The oil is filtered through the "black press" that is precoated with oil, and then filtered to separate catalyst from oil. Generally, it is necessary to recirculate the oil several minutes to obtain a clear filtrate. Recently developed catalysts appear to have excellent filterability so that filter aid is not required. The oil is poured into a tank supplied with coils and agitator. This tank is used frequently to rebleach the oil or for the addition of other stocks. Citric or phosphoric acid is added to the oil before filtering it again. This acid inhibits any adverse effects that traces of nickel impart to the oil.

After the oil is filtered through this press, called the "white press," it is ready to be deodorized. It is not advisable to invert the order because hydrogenation gives the oil a peculiar and undesirable odor.

De Smet Process for Physical Refining of Soybean Oil

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INTRODUCTION

Physical refining–also called steam-refining–is the combined neutralization and deodorization by steam distillation under high vacuum. The process will give, with the right equipment, a first class edible oil from a good quality crude.

However, to obtain a good final product, it is essential to make sure the crude oil entering the neutralizer/deodorizer is free of impurities. Considering the process of water-degummed soybean oil with 0.2–0.5% phosphatides content, the three steps of the physical oil refining process are discussed.

CENTRIFUGAL ACID DEGUMMING

Water-degummed oil contains remaining phospholipids and other gum-like impurities. The object is to eliminate them as much as possible. To accomplish this, centrifugal degumming using a small quantity of phosphoric acid is most recommended; the acid attacks the hydratable and nonhydratable phospholipids and eliminates trace metals. Centrifugal separation of the formed compounds reduces the phosphorous content of the oil to 10–20 ppm.

This process can be replaced by the degumming stage of an alkali centrifuge refinery, if this is available in the factory.

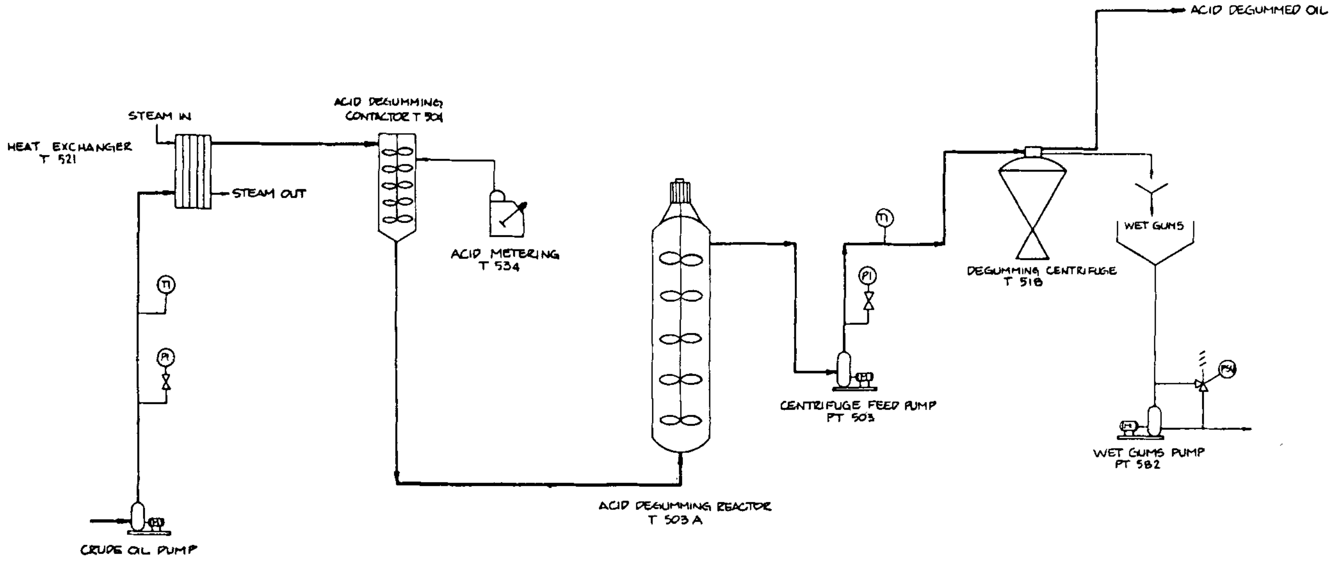

FIG. 1. Acid-degumming.

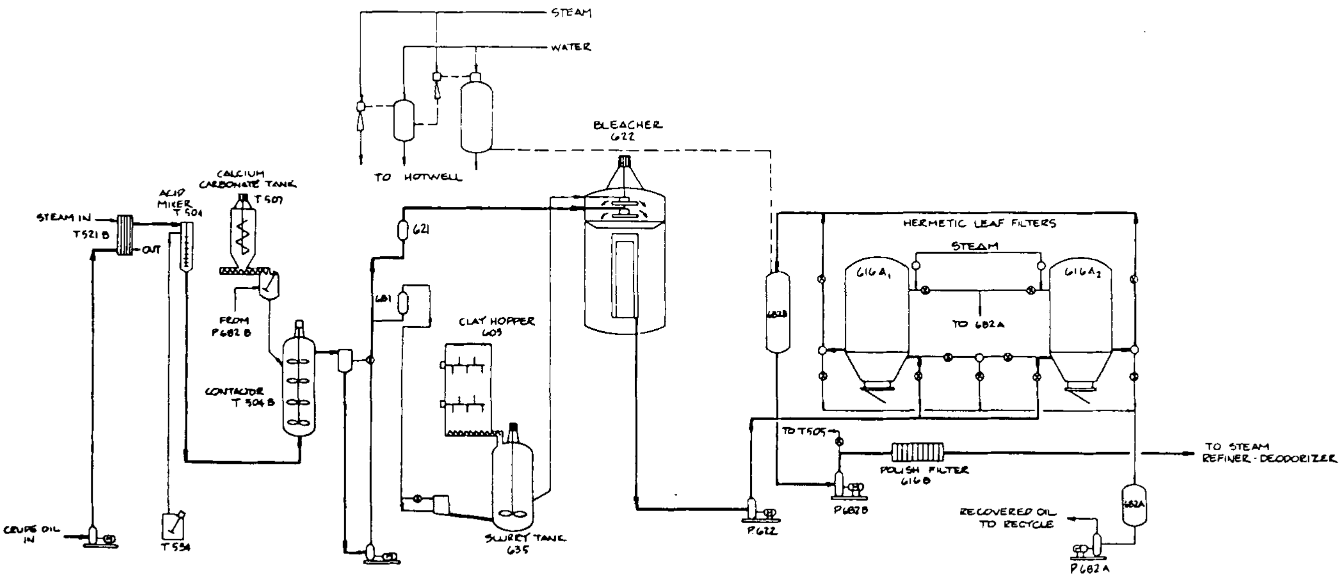

FIG. 2. Pretreatment, bleaching and filtration.

COMBINED DRY PRETREATMENT AND BLEACHING

After the oil is centrifuged, a subsequent dry pretreatment combined with bleaching is used for eliminating the remaining phosphatides and for decoloring the oil before steam neutralizing and deodorizing. Filtration is used in this step for removal of the small amount of solids. Filtration is combined with bleaching.

The successive phases in pretreatment bleaching are: (a) preheating of predegummed oil and mixing with concentrated phosphoric acid; (b) neutralizing excess acid by a weak alkali to form an insoluble salt precipitate; (c) continuous metering of activated clay in an oil-clay mixture, and then operation of vacuum continuous bleaching.

At the end of the dry pretreatment, the oil recovery from spent clay can be accomplished by steam stripping so that oil content of spent clay does not exceed 25%. Solvent extraction can be used to recover the oil from clay to avoid having to dump this hazardous material in restricted areas as required by new federal regulations.

STEAM REFINING-DEODORIZING

Deodorizing, the ultimate stage in refining, is combined with steam refining as the last step of the process. The succession of the different steps with emphasis on energy saving are: (a) the incoming oil is first preheated in a plated heat-exchanger using the finished oil; (b) a second heat exchange is effected during continuous heating and deaeration. The incoming oil is gradually deaerated and heated under high vacuum for ca. 2 min. This occurs in a countercurrent heat exchanger where the oil to be heated flows as a falling film on the heating coils, in which an intermediate exchange fluid circulates. This fluid is either glycerol or thermal oil and circulates in an exchange tube coil installed in the compartment of the buffer vessel. The heat balance of this system shows that 70% of the total heating energy required to raise the oil temperature from 50 to 250 C is recovered.

Finally, continuous heating of the oil at the desired temperature, 250 C, is accomplished under the same high vacuum as just described and with live steam injection in the bottom of the heat exchanger-deaerator.

During this phase, rapid acid removal is accomplished. The semicontinuous deodorization and final acid removal occurs within the deodorizer. The deodorizer/neutralizer consists of a vertical, cylindrical body composed of one central and six peripheral compartments, each permitting intimate contact of oil with sparge steam. The central buffer compartment has about twice the capacity of a peripheral one. It is equipped with a coil heated by high pressure steam, Dowtherm or thermal fluid. The heating coil is always submerged in oil. From the central compartment, steam eductors controlled by a programmer feed oil sequentially and at a high transfer rate to each of the peripheral compartments. Each separate charge is then treated separately in its compartment with sparge steam under high vacuum. In other words, five charges are always being deodorized while one is being filled or discharged.

Major energy-saving is achieved in the deodorizing plant by reducing the steam consumption for vacuum production. Our newest deodorizers divide into two layers each of the six peripheral deodorizing compartments to expose 12 oil surface areas to vacuum, and thus double the number of points for live steam contact. This optimizes the efficiency of the live steam and reduces the quantity of steam injection into the deodorizer, reducing the quantity of steam necessary for producing vacuum while still maintaining the optimal deodorization of the treated product.

After deodorizing time has been reached, the programmer sequentially opens each of the discharge valves located

TABLE I

Steam, Water and Heating Energy Consumption per Metric Ton of Processed Oil: Semicontinuous Deodorizing Plant

	Without heat recovery	With full heat recovery
Live steam for vacuum production (water at 28 C)	275 lb	275 lb
Live steam	37 lb	---
Heating energy (oil at 250 C)	474,000 BTU	142,200 BTU
Cooling water for vacuum production (water at 28 C)	57 gpm	57 gpm
Water for oil cooling (water at 28 C)	110 gpm	12 gpm

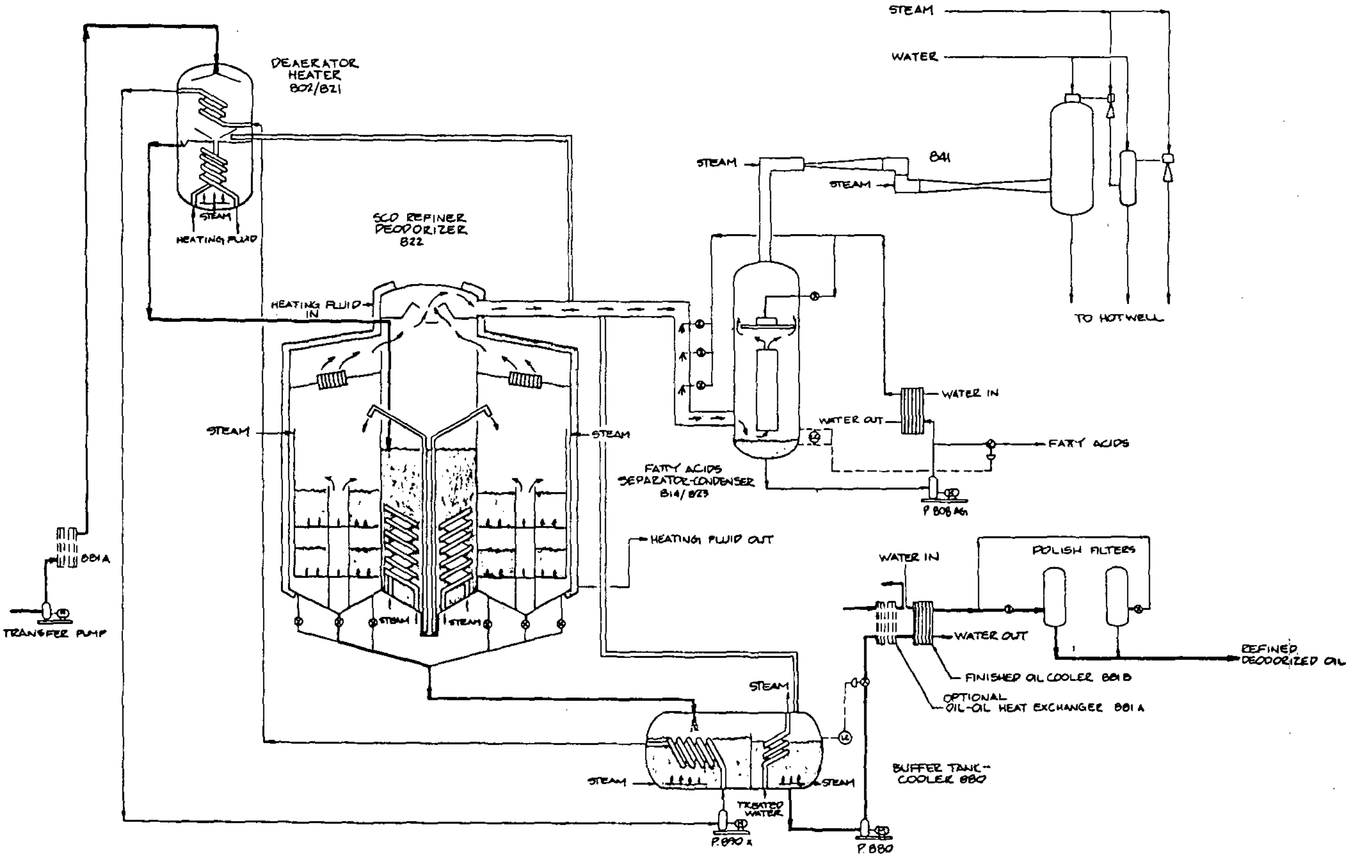

FIG. 3. Steam-refining and deodorizing.

at the bottom of each peripheral compartment. The oil leaving the compartments is collected in a two-section buffer tank cooler maintained under the same vacuum as the neutralizer/deodorizer. In the first section, the discontinuous oil flow is transformed into a continuous one and oil is precooled by heat exchange with incoming oil. In the second section, the oil is further cooled by contacting with a coil in which cooling water is circulating. Sparge steam is injected during cooling and, if necessary, citric acid or antioxidants can be added to the oil with a metering pump.

On the other hand, an enormous cooling economy is obtained because no water is used to cool the oil from 250 down to 70 C. Cooling water is only required when shutting down the plant.

Table I shows the savings obtained when incorporating heat recovery for semicontinuously deodorizing 150 MT/day.

Physical neutralizing of a degummed soybean oil produces a finished oil with 0.04–0.06% FFA, 2–8 pPM phosphorus, 1–2 R color and 13–17 hr AOM.

Substantial advantages are offered with the "physical refining," compared to conventional alkali refining.

For a normal quality of degummed soybean oil, refining losses by the conventional system range from 1.7 to 1.8 times the free fatty acids present. With steam acid removal, the same coefficient will not exceed 1.1 in the neutralizing section itself, plus normal oil losses in the bleaching clay and removal of compounds of phosphoric acid treatment. On the other hand, in physical neutralizing, fatty acids distilled together with the other distilled components have a purity of ca. 85%. In the conventional system, the soapstock produced must be disposed of, or has to be further treated with sulfuric acid for producing acid oils.

The conventional refinery and soapstock treatment require a complicated water treatment system to process the effluents. The steam refinery process recycles all water in a cooling tower and, therefore, requires very little pollution control.