in the 2-position of TAG were determined by pancreatic lipase hydrolysis procedure (Table II). Normally, TAG of mature seed oils contain predominantly 16:0 and 18:0 in the 1,3-positions of TAG. But, considerable proportions of 16:0 and 18:0 were present in the 2-position of TAG in HE in the initial stages of maturation until the 13th day after flowering. As maturation proceeded and TAG synthesis became active, these proportions were drastically reduced and the distribution pattern became normal with 18:2 as the major acid in the 2-position. A gradual reduction occurred from a high value (80%) in the percentage proportion of 18:1 in the 2-position. This pattern was not observed in the TAG of HC because the samples were analyzed only after the TAG synthesis was complete. Once TAG synthesis was more or less complete, no change occurred in the positional distribution pattern in both species.

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Physical Refining of Edible Oil[1]

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ABSTRACT

Physical refining of edible oils has received renewed interest since the early 1970s when the process was reintroduced on a large scale to refine palm oil in Malaysia. Subsequent laboratory and field tests have also shown that physical refining can be used as a substitute for caustic or chemical refining, not only for high free fatty acid (FFA) oils such as palm, but also on low FFA oils such as soybean oil. In either case, the physical refining system results in lower oil loss than chemical refining and also eliminates pollution problems associated with soapstock acidulation. In physical refining, however, the oil pretreatment and efficiency of the distillation are two very important factors that must be considered to guarantee continuous production of high quality products. This paper reviews the physical refining system as it is today and how it can be used on two different edible oils. An actual case study showing the effects of the pretreatment in a commercial operation is also presented.

INTRODUCTION

Physical refining of edible oils has been a principal topic of discussion in the fats and oils industry for the past few years. Until that time, edible oil refining was almost always thought of as a four-step process: degumming, deacidification, or neutralization, bleaching and deodorizing. Since the alkalai used in neutralization most often was caustic, the process was widely known as caustic or chemical refining. One problem of the chemical refining process, however, is the production of soapstock, a mixture of sodium salts of fatty acids, neutral oil, water, unused caustic and other compounds resulting from the reactions of the caustic with the various impurities in the oil. The disposal of this soapstock, or the waste streams from soapstock processing systems, has become very expensive as governmental regulations on plant waste water discharge quality tighten. Another problem associated with chemical treatment is that it also causes a loss in neutral oil, thereby reducing the overall yield of refined product. As long as the primary oils being refined, however, were those with naturally low free fatty acid contents (FFA), such as soybean, there were few attempts to introduce physical refining on a large scale. In the early 1970s, however, action by the Malaysian government forced the development of a large refining industry for their palm oil which had grown rapidly in the world market. A typical palm oil contained 3-5% FFA and, therefore, chemical treatment resulted in excessive losses in neutral oil and production of large quantities of soapstock. For this oil, physical refining offered an attractive alternative.

Block flow diagrams for both the chemical and physical refining processes are shown in Figure 1. The chemical method contains a water degumming step, which is used to remove impurities generally classified as gums; this is followed by caustic treatment for removal of additional impurities and most of the FFA; then bleaching to improve color, and finally deodorization or steam distillation to

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[1] Presented at the 73rd AOCS annual meeting, Toronto, 1982.

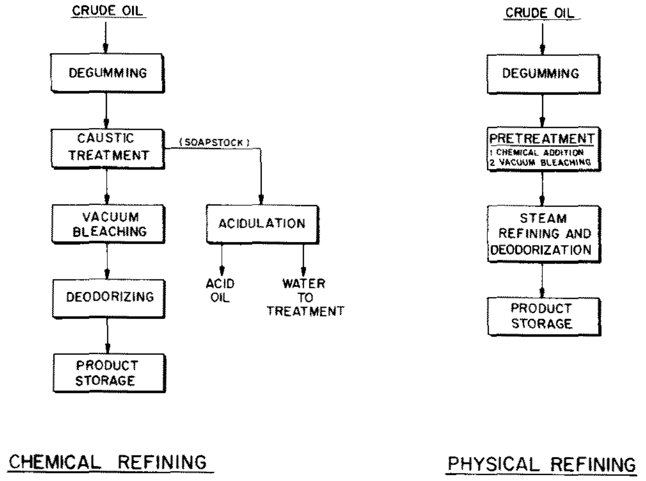


FIG. 1. Block diagrams for chemical and physical refining.

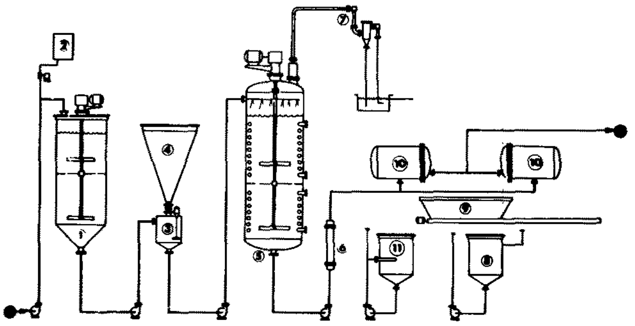


FIG. 2. Pretreatment process.

remove the unsaponifiables and any traces of fatty acids and impurities remaining. The soapstock, which also contains neutral oil from the chemical treatment, can be reacted with sulfuric acid in a process called acidulation to form acid oil and highly acidic water which must be treated before discharging into a waste treatment system.

Physical refining will remove all the FFA, as well as the unsaponifiable and other impurities by steam stripping, thereby eliminating the production of soapstock and keeping neutral oil loss to a minimum. A degumming and pretreatment of the oil is still required, however, to remove those impurities which will darken or otherwise cause a poor quality product when heated to the temperature required in the steam refining deodorizing unit. The pretreatment is normally a two-step operation; the addition of a chemical to remove any trace quantities of gums remaining after water degumming and bleaching for color reduction. Following pretreatment, all the FFA and any remaining trace impurities are removed by steam distillation in a single unit. Acidulation is eliminated and the FFA from the oil is recovered directly without major pollution problems. The block diagram in Figure 1 is for an oil containing a large quantity of gums, generally greater than 1%, such as soybean; for other oils such as palm or coconut which contain only small quantities of these gums, the water degumming step may be omitted, and all the gums removed in the pretreatment.

PROCESS DESCRIPTION

A complete review of the various types of physical refining systems available has been presented recently (1). The flow diagrams for one of the most commonly used systems which was described in a previous article (2) are shown in Figures 2 and 3. In the pretreatment process (Fig. 2) the crude oil is pumped into the treat tank [1] along with a regulated amount of the pretreat chemical from a storage tank [2]. After thorough mixing the solution is pumped into a slurry tank [3]. Clay and filter aid are metered into the slurry tank from the storage hopper [4] and after thoroughly mixing the slurry is pumped into a vacuum bleacher [5]. The slurry is sprayed into the head space of the bleacher to obtain complete deaeration and then heated to the desired temperature by internal steam coils. After sufficient time with thorough mixing, the mixture is then cooled in heat exchanger [6] and filtered. The filters [10] are usually precoated with material from precoat tank [11], and two filter units are normally supplied, one on stream, while the cake in the other is being steamed and air blown to provide maximum recovery of oil. Oil from the steaming operation is collected in the steamings tank [8] and recycled into the system. Spent filter cake collects in the hopper [9] and is discarded. The bleaching process not only produces a lighter colored oil but the clay also provides a surface to absorb the precipitated or coagulated gums from the pretreatment reactions in the treat tank.

The pretreated oil is then steam refined and deodorized. The system shown in Figure 3 uses a continuous single shell deodorizer [3] which contains individual sections for oil heating, stripping, holding, final stripping and cooling, all within a common shell under the same vacuum. The oil is first pumped continuously through a filter [1] and sprayed into the vacuum deaerator [2] to remove entrained and dissolved air. It is then pumped through the heat recovery section into the heating section in the top of the steam refining deodorizer [3]. In this section the oil is heated to the required processing temperature with coils, which are heated by a thermal fluid, typically Dowtherm vapor from a vaporizer [10]. The oil is then "refined" by flowing over the top series of trays countercurrent to the flow of stripping steam which is injected below the bottom tray. This "refined" oil then flows down to a holding section which provides the retention time required for heat bleaching of some oils; then to a second series of trays for additional steam stripping to deodorize the oil completely; then to the heat recovery section where the deodorized oil transfers some of its heat into the incoming oil; and finally to the cooling section. The cooled oil is pumped through polishing filters [7] to storage. To complete the system there is a distillate tower [4] where the entire vapor stream from the steam refining deodorizer is cooled to condense the fatty acids. This allows for recovery of a pure acid stream and reduces the pollution in the cooling water from the multistage vacuum system [5] which maintains the vacuum on the entire system.

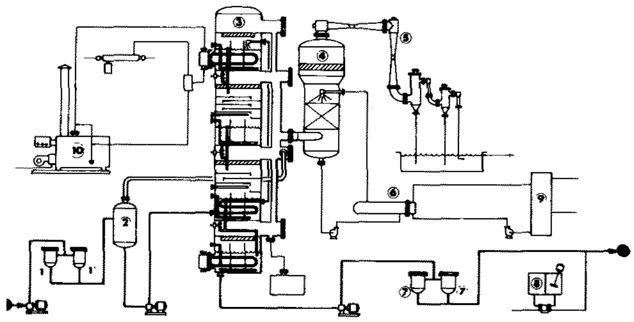


FIG. 3. Steam refining-deodorizing process.

In the steam refining deodorizer of Figure 3, steam stripping of the oil is done on high efficiency distillation trays; other types of steam refining deodorizers use various other internals, but their purpose is the same, to provide sufficient contact between the steam and oil so all the FFA and other impurities are stripped from the oil. In a chemical refining system, the unit is called a deodorizer since it removes only trace quantities of these components, but in physical refining the unit is called a steam refining deodorizer since it removes all the FFA and impurities. A deodorizer and steam refining deodorizer must be constructed differently because of the different functions they perform. For the unit in Figure 3 the following design modifications must be made to convert from a normal deodorizing to a steam refining and deodorizing operation. (a) The number and design of the trays has to be changed since the FFA of the feed is higher for steam refining than for deodorizing. (b) For the deodorizer, the shell and the internals of the tower are 304 SS and the vapor pipe shown on the right is carbon steel, since hot oil never enters this pipe. The steam refining deodorizer is constructed completely of 316 SS, however, to prevent excessive corrosion, because of the larger quantities of FFA present during this operation.

MATERIAL BALANCES

One of the advantages of physical refining over chemical treatment is an increase in product yield. The chemical treatment and separation of the soapstock results in additional loss beside that which would be calculated for the FFA reduction. Inefficiences in the centrifugal separation and reactions of the caustic with triglyercides in the oil cause this "neutral oil loss." It will not happen to as great an extent in a physical refining system since all the FFA is removed by steam stripping. This advantage can be seen by comparing material balances.

A comparison of material balances for low FFA oil such as soybean is shown in Figure 4. In chemical refining, the amount of caustic used and the overall loss during neutralization are based on standard calculations for soybean oil. The gums, amounting to 2.0% of the feed, are removed in the degumming step. The amount of clay and filter aid used in bleaching are typical for commercial operations as is the percent of oil lost in the filter cake and in the deodorizer distillate. The individual process losses result in a yield of ca. 954 lb for each 1,000 lb of oil that is treated, or a total loss of 4.6%; this value is within the range reported in the literature (3). In the case of physical refining, oil is degummed in the same manner; the amount of pretreat chemical, bleaching clay, distillate and the amount of oil left in the filter cake are again based on typical commercial operations. The yield is ca. 962 lb, or a total loss of only 3.8%. Thus, physical refining will result in an improved yield of ca. 0.8%.

The more significant advantage of physical refining from a material balance point of view is with a higher FFA oil such as coconut. In the material balance shown in Figure 5 the neutralization loss is based on values reported in the literature (4) for oil with similar FFA contents; since normal equations used to calculate this loss are usually not applicable on high FFA oils. Other parameters are based on actual operations with coconut oil. The individual process losses in chemical refining result in ca. 910 lb of product for each 1,000 lb of feed, or a 9.0% overall loss. For physical refining, however, this overall loss is only ca. 5.9%. Besides the 3.1% increase in yield, the FFA in the physical refining system is also recovered in a pure form and, therefore, can be sold as a byproduct without additional processing such as acidulation.

PRETREATMENT OF OILS FOR PHYSICAL REFINING

The purpose of pretreatment in a physical refining system is to remove those impurities in the oil that will darken and cause a poor quality product when the oil is heated to the temperatures required in the steam refining deodorizer. A considerable amount of work has been reported (5,6) and is currently being done on numerous oils such as soybean, rapeseed, peanut, corn, sunflower, palm, coconut, palm kernel, and others to determine the proper conditions for this pretreatment. The major process variables in pretreatment are as follows (Fig. 2): (a) the pretreat chemical, its concentration and the amount added to the oil in treat tank [1], (b) the type of bleaching clay and the amount added to the oil from storage hopper [4], and (c) the operating temperatures and pressures.

On a single crude oil of fairly consistent quality, the chemical and bleaching clays used, the amount of each, and the optimum operating conditions can be expected to remain fairly constant, but in the more typical commercial operation, where more than one crude oil, each of varying qualities, is encountered, various chemicals, clays and operating conditions may have to be considered.

Phosphoric acid, citric acid and acetic anhydride offer

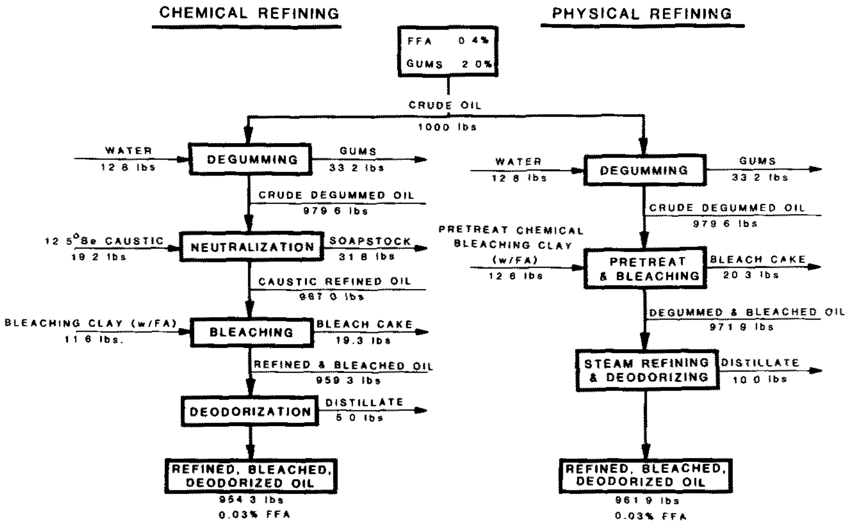


FIG. 4. Material balance–chemical and physical refining of crude soybean oil.

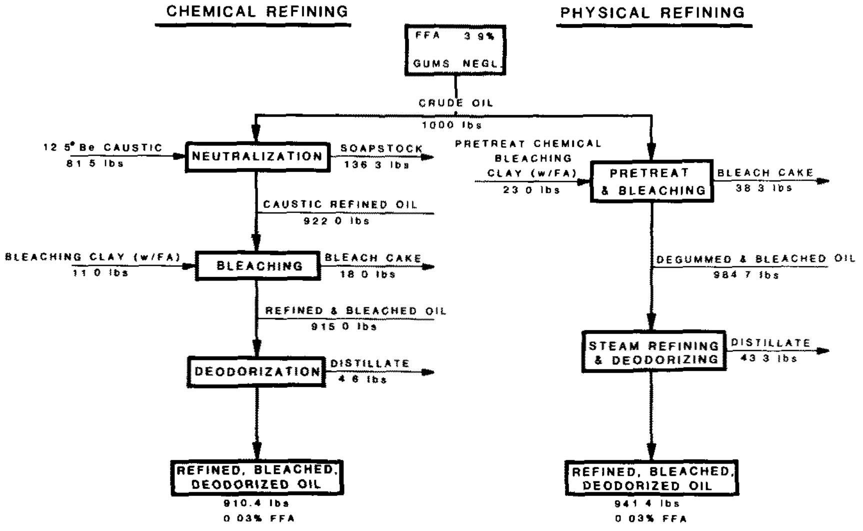


FIG. 5. Material balance—chemical and physical refining of crude coconut oil.

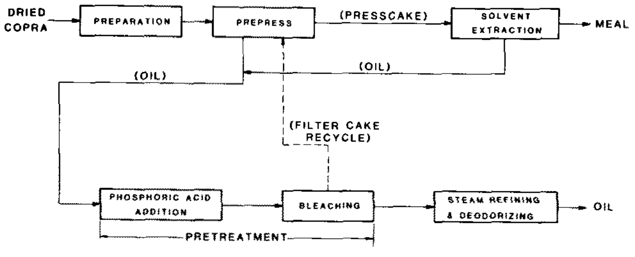
good potential as chemicals in pretreating since they have been used successfully in oil processing in the past; also, the characteristics of the various clays as they pertain to edible oil treatment are well known and this information can be helpful in selecting the proper clay to use.

As an example, a case study will now be presented showing how changes in pretreatment procedures may be necessary once experience with a particular oil is obtained.

Case Study: Physical Refining of Coconut Oil

This case study on the effect of pretreatment in a physical refining system involves a coconut oil refinery. The initial operation of the plant was satisfactory producing an oil with 0.03-0.05% FFA and with odor and flavor equivalent to that from an existing chemical refinery at the same location. Phosphoric acid and a commonly used bleaching clay were used in the pretreatment section. However, a few months after start-up a noticeable rise in the FFA content in two of the shipments was reported by a customer. At the same time the refiner noticed similar increases in his storage tanks. When the obvious causes of this instability such as water contamination of the oil or poor deodorizer operation were ruled out, an investigation of the effect of pretreatment was begun.

The process used to recover and refine the coconut oil was basically a standard prepress/solvent extraction system followed by physical refining. A block diagram is shown in Figure 6. Shortly after the physical refining system was put in operation, however, a process change was made to recover oil from the bleach filter cake by recycling the cake back to the expellers as shown by the broken line. This not only increased oil recovery, but also improved the color of the oil to the physical refining system feed as evidenced by the lower red color value. The amount of bleaching clay required was also reduced. In effect, this recycled clay was partially bleaching the oil in the screw presses. Since the instability of the finished oil was first noticed after this process change had been made, however, the effect of clay recycle became the first target to the investigation.



	BEFORE RECYCLE	AFTER RECYCLE
COLOR	11-14R	8-9R
BLEACHING CLAY REQ'D, % OF OIL FEED	1 8	1 4

FIG. 6. Block diagram—copra extraction and coconut oil refining process.

The instability of the oil from various batches was erratic, sometimes showing up in storage immediately after processing and sometimes not appearing until after shipment. However the first problem that remained was to develop a quick laboratory test which could predict the stability of any oil produced. The test developed over the period of this investigation consisted of heating a sample of the oil with 10% water added at 90 C for 16 hr. Experience with this rapid stability test indicated that if the FFA of the oil after the test was below 0.10%, the oil would remain stable indefinitely, and that the higher the FFA obtained in the test, above the 0.10% limit, the more unstable the oil tended to be.

From an analysis of the crude and deodorized oils the following was determined. (a) The crude analysis was typical for a coconut oil, containing ca. 3.7% FFA, except for the metal analysis, which showed high levels of iron, 76 ppm compared to a typical oil which is less than 10, and high levels of other trace metals. (b) The deodorized oil contained relatively high levels of phosphorous and the level of phosphorous was related to the rapid stability value as shown in Figure 7. Although the data was somewhat inconsistent a clear trend was developed. It is important to note that the phosphorous measured was organic in nature, or part of a compound that was oil soluble. The apparent limit of 5 ppm is the same as that which has been reported for soybean oil (6).

It appeared, therefore, that the first thrust of the investigation should be to examine the effect of the processing on the level of organic phosphorus in the finished oil and the effect of the metal content on the oil quality. The sources of the metal are that naturally occurring in the coconut oil and that due to recycling of the bleach filter cake.

The source of the phosphorus is threefold; that naturally occurring in the coconut oil; that due to recycling of the bleach filter cake; and that from addition of phosphoric

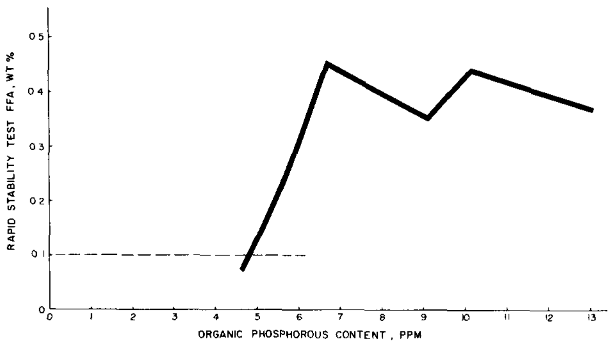


FIG. 7. Rapid stability FFA vs organic phosphorous content.

acid during the pretreatment step. Obviously, the first thing to be done was to eliminate the recycle of bleach clay to prevent both the phosphorus and iron compounds from recycling back into the crude oil, thus, increasing their concentration. The reduction or elimination of the phosphoric acid was also considered.

Table I is a summary of some of the initial test runs made on the commercial unit. Run no. 1 is the standard system, without bleach clay recycle, consisting of phosphoric acid pretreatment and bleaching with a commonly used clay, here identified as "A." Phosphoric acid and bleaching clay were used at 0.2% and 1.5% of the oil feed, respectively. Although the filter cake recycle had been stopped, the rapid stability value of 0.46% clearly indicates this alone will not produce a stable oil. In run 2, the crude oil was water washed and centrifuged prior to physical refining to reduce the iron, other trace metals and the naturally occurring phosphorus concentrations. The stability is greatly improved but still is at the upper limit of 0.10% as required by the rapid stability test. This result led to run 3, in which the phosphoric acid pretreatment was eliminated. Since previous operations had shown that the oil would darken in the steam refining-deodorizer when clay "A" was used without sufficient phosphoric acid, a very highly acidic clay, here referred to as "B," was substituted. This clay was chosen since it had been used successfully in the past in bleaching of poor quality vegetable oils. Clay "B" is basically identical to "A" in particle size, apparent bulk density and moisture content; it has a surface area slightly greater than "A" but an acidity 8 times that of "A." As shown the quality of oil was improved as to pass the rapid stability test. The color, however, was slightly higher than desired. Economics dictated that to reduce this color activated carbon be used instead of increasing the level of clay, and as shown in run 4, the color greatly improves when carbon was added. A small benefit in stability as evidenced by the improved rapid stability value also resulted.

TABLE I

Summary of Test Runs on Coconut Oil Physical Refining System

	TEST			
	1	2	3	4
WATER WASH (DEGUMMING)	NO	YES	YES	YES
CITRIC ACID TREATMENT	NO	NO	NO	NO
PHOSPHORIC ACID TREATMENT	YES	YES	NO	NO
PREHEAT TEMP °C	90	90	90	90
BLEACHING TEMP. °C	100	100	100	100
DEODORIZING TEMP °C	250	250	250	250
BLEACHING CLAY	A	A	B	B
ACTIVATED CARBON	NO	NO	NO	YES
INITIAL FFA, WT%	0.028	0.028	0 028	0 028
RAPID STABILITY FFA, WT%	0 460	0.107	0.062	0 045
COLOR	-	-	1.7 R	1 1 R

Under the conditions of run 4 it was now possible to consistently produce a stable oil. However, work now began on reducing the overall treatment cost for the system. First, the water washing step was eliminated and it was found that a stable oil could still be produced with clay "B." A third clay, "C" lower in cost than "B" was also tried, but with the water washing step eliminated, a small amount of phosphoric acid, 0.05%, was required in the pretreatment process to produce a good quality, stable oil. The combination of clay "C" and phosphoric acid was lower in cost than just using clay "B," and it was possible to produce a stable oil as long as the amount of phosphoric acid added was monitored carefully.

Since the tests were made on a commercial unit in actual operation, it was not possible to run all the tests that would be necessary to positively identify all the factors. Production schedules, mechanical breakdowns, shipping and customer requirements all reduced the amount of experimentation that could be done, but the data in Table I does show the importance and effect of pretreatment, on a commercial scale, in a physical refining system.

PHYSICAL REFINING OF SOYBEAN OIL

Since the advent of the new physical refining systems for the Malaysian palm oil, researchers have begun to take another close look at the process for soybean oil, which had been tried many years ago but abandoned (7). Numerous papers have appeared in the literature on this subject and recently a symposium was held devoted to just this one topic (8). The speakers at this symposium generally agreed that physical refining of soybean oil will produce an oil of comparable quality to that which is chemical refined although they had some concern whether physical refining could be used with poor quality oils. There was also some concern expressed that the process was more expensive than chemical refining because of the reported high usage of bleach clay.

The quality of physical refined soybean oil was also studied recently by the United States Department of Agriculture (9). In this work the flavor and oxidative stability of physically refined and chemically refined oils were compared by a taste panel. Although the results were somewhat variable, it appeared that physically refined oils, that is oils that were treated with phosphoric acid, degummed steam refined and deodorized, were equal in flavor to chemically refined not only on fresh samples but also after storage.

Several refiners, both inside and outside the U.S.A. have experimented with physical refining of soybean oil on a commercial scale and many continue to do so. There have also been reports of successful physical refining systems in operation on soybean oil and recently a commercial run was made on a physical refining unit similar to that shown in Figure 3. In this run 100,000 lb of a typical soybean crude, not preselected, were first treated with phosphoric acid and then water washed in a centrifuge. Thus, the water degumming and pretreat operations were combined. No additional phosphoric acid was added after degumming. After a bleaching operation with a standard bleaching clay, the oil was continuously steam refined in the steam refining deodorizer. An analysis of the bleached and steam refined oil is shown in Table II. The FFA and peroxide limits of the deodorized oil were equal to or better than those from oil produced by chemical refining. In addition the color was

TABLE II

Analysis of Physically Refined Soybean Oil

	DEGUMMED & BLEACHED	STEAM REFINED
FFA, WT %	0 4	0015
PEROXIDE, ME/KG	---	0.0
COLOR	3 6R/35Y	0 3R/5Y
FLAVOR	---	8 5

excellent. A flavor score of 8.5 (scale 1-10) was reported by a taste panel. They described the flavor as excellent as was flavor stability as measured by standard AOCS procedures.

The amount of bleaching clay as well as the steam usage and operating temperature of the steam refining deodorizer were at the same levels commonly used for deodorization in a chemical refining system. The high levels of clay reported in the recent symposium (8), which were a cause for the concern about economics of physical refining, were not required to make a satisfactory product. In addition it appeared the yield of product was somewhat greater than would be expected from chemical refining, but the run was too short to quantify this observation.

Whether physical refining can be used for soybean oil, regardless of quality, at this time is unknown. But now that extensive research is being done, commercial operations are beginning, and a considerable amount of interest in physical refining is being generated, it can be assumed that processing techniques will be developed to handle crude soybean oil regardless of its quality.

The physical refining system has an inherent lower installed cost when compared to chemical refining, lower steam, water and power requirements, and eliminates soapstock and its associated waste treatment problems. These facts will most certainly provide the impetus to bring physical refining of soybean oil to the forefront in the very near future.

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Evaluation of Extractive Contact Units for Oil Extraction from Dehulled Sunflower Seed

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ABSTRACT

The efficiency of an extractive system generally depends on kinetic, thermodynamic and technological parameters. Each of these factors affects the extractive effectiveness, although the overall result depends on their interaction. In the present work this interaction is analyzed for oil extraction from dehulled sunflower seed. Experiments on laboratory scale were made to calculate the kinetic and thermodynamic parameters of the extraction. The behavior of the system in 2 typical contact units (mixer-settler and semicontinuous extractor) was studied and models assuming equilibrium conditions were formulated. The soundness of the models was checked by pilot-plant tests and good agreement was obtained as long as the residual oil concentration in the solid was higher than ca. 0.01 c_{so}. At lower oil concentrations the contact time becomes the main factor of the process, and the extraction is much less affected by the extraction ratio and the composition of the liquid.

INTRODUCTION

This work is part of a more extensive research program that aims at developing a technology for producing edible proteins from sunflower seeds. The characteristics of this technology are: the near-total dehulling of the seed and the direct oil extraction (without prepressing) at a relatively low temperature. The apparatus normally used for the extraction of oil from seed (percolation) can be hardly used with this type of material for rheological reasons (1-3). Because of this, immersion systems must be taken into account. At the moment they are not very widespread owing to their bulkiness and low efficiency (4-5).

In this work the possibility of improving the traditional immersion systems by affecting the rate limiting factors has been investigated.

EXPERIMENTAL PROCEDURES

Three groups of tests were performed.

Batch Laboratory Tests

Dehulled and flaked sunflower seeds were placed in contact with n-hexane in a 5 L stirred thermostatic vessel. The seeds had 3% residual hull (evaluated by direct weighing) and 60% lipids (petroleum ether extraction, according to AOCS method Ac 3-44). The kinetic of extraction was followed by measuring, as a function of time, the amount of oil in the liquid (spectrometric analysis at 232 nm) and in the solid (6-8). The tests were repeated by varying: seed/hexane extraction ratio (R') in the range of 0.1-0.0125 kg/L; extraction temperature (T) in the range 22-50 C; seed characteristics (humidity in the range 5-12%, flaking in the range of 0.1-0.5 mm).

Semicontinuous Pilot-Plant Tests

This system consisted of a number of extraction units (N) (600 L volume each) connected in series cyclically and fed with solvent for a time (t). Specifically, the connection allowed the isolation of the loading and discharging unit