TABLE I

Composition of Hulls and Components of Hulling Operations (Dry Basis)

No.	Location of huller	Moisture in fruit[a]	Oil in fruit	Oil in nuts	Kernel in nuts	Extract in coarse hulls	Extract in fine hulls	Fine hulls in total	Oil lost in hulls (on basis of fruit)	Ratio of fruit to nuts	Oil lost in hulls,% of total
		%	%	%	%	%	%	%	%	%	
1	Field	16.9	23.4	49.7	81.4	1.02	3.51	38.3	0.51	2.17	2.2
2	Mill	18.8	22.2	43.4	70.4	1.13	3.28	50.8	0.55	2.00	2.5
3	Mill	15.5	22.1	47.7	73.3	0.98	2.55	36.9	0.31	2.19	1.4
4	Mill	33.9	22.0	45.7	73.2	1.05	4.15	47.4	0.79	2.15	3.6
5	Field	21.7	22.6	53.5	86.8	0.86	5.97	53.5	1.66	2.54	7.3
6	Field	19.2	19.6	43.9	78.2	0.57	2.23	44.8	0.42	2.29	2.1
7	Field	15.2	24.1	50.4	85.3	1.27	4.25	52.8	0.85	2.16	3.5
8	Mill	16.4	21.1	48.6	75.7	1.06	2.20	41.2	0.27	2.33	1.3
9	Mill	23.0	22.7	45.1	72.6	0.82	2.87	37.2	0.39	2.02	1.7
10	Mill	14.6	24.8	51.6	79.2	1.25	2.38	72.2	0.43	2.12	1.7
11	Mill	20.2	24.4	48.2	74.4	0.83	2.34	43.1	0.33	2.00	1.4
12	Mill	17.2	23.1	46.0	72.2	0.62	3.28	32.4	0.44	2.03	1.9
13	Mill	18.1	26.9	56.8	83.0	1.50	4.26	55.3	1.41	2.19	3.2
14	Field	25.7	23.8	41.9	64.0	0.66	1.52	36.6	0.15	1.76	0.6
15	Field	30.9	20.4	55.8	83.7	1.23	2.96	42.8	0.56	2.82	2.8
16	Field	15.4	22.6	45.2	71.3	1.05	2.59	53.9	0.44	2.04	1.9
17	Field	13.8	25.2	47.3	71.6	1.11	2.67	58.1	0.44	1.91	1.7
18	Mill	15.6	24.4	54.3	81.6	1.56	5.99	61.9	1.62	2.38	6.6
19	Mill	14.7	25.7	51.5	76.1	1.24	4.72	46.5	0.87	2.08	3.4
20	Mill	23.8	24.7	50.4	78.6	1.08	4.57	59.4	1.30	2.16	5.3
21	Mill	14.0	21.7	50.1	75.4	1.69	2.36	44.8	0.19	2.43	0.9
Av		19.2	23.2	48.9	76.6	1.08	3.36	48.1	0.66	2.18	2.7

[a] On an as is basis.

higher in the hulls than in the nuts except under very unusual conditions.

Summary

Methods for analyzing commercial tung hulls for oil have been developed. Samples of tung hulls from mill and field hulling operations have been collected and analyzed. The loss of oil when the fruit are hulled was found to vary from 0.6% to 7.3%, with an average loss of 2.7% based on the total amount of oil in the fruit. The difference in the loss of oil between grove and mill hulling was not significant. With a loss of 2.7% of the oil in hulling, a recovery of 87.9% oil on the hulled nuts would be equivalent to a recovery of 85.5% oil on the whole fruit.

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An Inexpensive Soap Stock Conversion Plant[1]

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ENGINEERS in the United States have excelled in carrying out large scale chemical plant operations both efficiently and profitably. Very often however the careful planning for maximum economy which goes into large scale plant design is not carried over to small operations. The result is generally discouraging from the point of view of the magnitude of the investment required and the anticipated return on this investment. This paper presents an illustration of the successful application of sound engineering design to the small scale conversion of vegetable oil foots to a crude fatty acid product. This plant (shown in Figures 1 and 2) was designed for a vegetable oil refiner in the Southwest in 1950. Constructed the same year, it has since given over two years of satisfactory operation.

Product Economics

The factors imposing rigid economy on the plant design are to be found in the product economics. At best, the conversion of soap stock (raw foots) to crude fatty acid is a marginal operation. This is seen by a comparison of the recent average delivered value of 1.5c/lb. of 50 wt. % raw cottonseed foots and 3.5c/lb. of the 95 wt. % acidulated foots (i.e., crude fatty acid). On the basis of an average freight cost of 2c/ton-mile and a processing cost of 0.75c/lb. of 95% product, the maximum economical shipping distance for soap stock is seen to be 120 miles.

Thus with the nearest possible consumer 300 miles away, faced with a negligible or non-existent demand for his crude foots and prevented by antipollution regulations from discharging his soap stock into the municipal sewerage system, the refiner had no alternative but to upgrade his crude foots to a marketable product in order to dispose of this waste material. The low profit margin available made it mandatory that the capital investment required to accomplish this processing be held to a minimum without any corresponding sacrifice in process efficiency or increased labor or maintenance costs.

Process Requirements

Processing of the raw foots (soap stock) discharged from the vegetable oil refining plant consists of three basic steps:

a) acidification or acidulation (with 66°Bé sulfuric acid) of the highly basic, diluted soap stock feed to convert the soap into free fatty acids;

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FIG. 1. Side view of soap stock conversion plant.

FIG. 2. Front view of soap stock conversion plant.

b) breaking the emulsion of fatty acid and foreign material in water by means of heat and agitation;
c) phase separation (of which there are three):
   1. Fatty acid product (top layer). Recovered and transferred to storage;
   2. Recycle (middle or interphase layer). This material is reprocessed;
   3. Acidified waste water (bottom layer). Removal, neutralization with caustic solution, and subsequent disposal into sewer.

Prior to the actual plant design, sufficient laboratory work was carried out on a representative sample of the soap stock to determine optimum conditions of operation. More specifically, the following were investigated:

a) pH requirements to break rapidly the emulsion and obtain high free fatty acid recovery;
b) requirements for minimization of foaming during agitation;
c) caustic requirements for neutralization of the waste water phase prior to discharge into the public sewer system.

It is recognized that the varying composition of soap stocks, especially the nonfatty acid constituents, will vary both the pH and the agitation required to break the emulsion for satisfactory separation of the fatty acid and nonfatty acid aqueous phases.

With the basic laboratory data obtained, the daily operational cycle was decided upon. This was based on an eight-hour operating day and is shown in the following table:

TABLE I

Daily Operational Cycle of Soap Stock Plant

Time required, hours	*Description of operation* (in sequence)
2	Discharge and separation of settled tank contents from previous day's run
2	Soap stock charged to treating tank
1	Soap stock preheated to temperature of 200-212°F. (93-100°C.)
1	Acidulation of soap stock with 66 Bé sulfuric acid, followed by air agitation to obtain intimate mixing of both phases
2	Steam cleaning of various lines carrying soap stock, recovered fatty acid, and recycle material
8 (total)	

In addition, it was planned to allow the treating tank contents to settle overnight before separation of the waste water and fatty acid phases.

Process Design Features

Factors receiving careful attention during the design, regarding both economy and simplicity of operation, were materials of construction, transfer equipment, and metering and proportioning devices. All transfer lines were black iron pipe (i.e., soap stock feed, fatty acid product, waste water, concentrated sulfuric acid, etc.). Sulfuric acid and neutralizing caustic metering drums and storage tanks were also of iron construction. The only corrosion-resistant material specified was for heating coils and other lines within the treating tanks and the tank bottom discharge fittings.

The major items of equipment were two 12-ft. diameter by 14-ft. high (12,000-gallon) wooden treating tanks, one 30-gal./min. gear type transfer pump (steel construction), one 10,000-gallon sulfuric acid storage tank, one 4,000-gallon soap stock hold-up tank, two 25,000-gallon fatty acid product storage tanks, a caustic solution metering drum, and a sulfuric acid weigh drum (acid egg). By specifying wooden treating tanks, corrosion problems were avoided. In addition, the equivalent of conical bottoms was obtained by tilting these treating tanks 2.5 degrees.

Only one pump was specified. This was a gear pump of steel construction designated for handling soap stock feed and fatty acid product only. Transfer of corrosive streams (i.e., sulfuric acid, acidified recycle foots, etc.) was achieved by air pressure or by means of simply constructed yet highly effective steam ejectors.

Process control is accomplished manually. There are two basic control points. One is at the overhead platform between the two treating tanks (position A in Figure 2); here the operator controls the treating portion of the process. The other point is at the bottom of the two tanks and at the transfer pump (position B in Figure 2). At this point the transfer of the materials, cleaning of lines, and metering of treating chemicals are controlled.

Simple metering devices were designed for both low cost and trouble-free operation. The sulfuric acid metering system is a good example of how economy and simplicity of operation was achieved at no sacrifice in efficiency or excessive labor requirements. A

small acid egg was fabricated from 12-inch diameter black iron pipe. This egg was attached to one end of a balanced, steel beam loaded at the other end with a 100-lb. spring scale. The egg was designed to hold the anticipated acid requirement for a 4,000-gallon charge of representative soap stock. In this way the operator need only fill the egg to the desired weight and then, by pressurizing the acid egg, transfer the acid up to a control valve located at the overhead platform. From here acid may be directed into either of the two treating tanks.

Sulfuric acid is introduced at the top of each treating vessel in a mixing tee where it is diluted with water. This is done to avoid locally concentrated reactions which would otherwise result in sulfation of the fatty acids and excessive foaming. The dilution water also aids in phase separation. The mixing tee is very simple, being composed of standard cast iron fittings. The operator controls the proportions of water and acid from the overhead control platform at the top of the tanks. He is also in a position to observe the acid weigh tank below so that he can determine when acid addition has been completed and can thus stop the flow of dilution water.

Neutralization of the waste mineral acid bottom layer is also accomplished very simply. The caustic metering tank discharges into the drain at the same point as a common, external swinging joint, adjustable back pressure head discharge line from both treating tanks. Operational experience will indicate to the operator just about the appropriate level at which to set this adjustable discharge line (for example, perhaps 1.5 or 2 feet above the bottom of the tank). This will allow the waste mineral acid layer to be completely discharged without loss of either the turbid interphase layer (which is recycled) or the free fatty acid layer (which is sent to product storage). By means of a manually adjusted, calibrated valve at the side of the caustic metering tank, a regulated flow of neutralizing caustic is fed to the discharging waste water from the treating tank.

Because the recycle emulsion layer of material contains dilute sulfuric acid (among other things), it was decided not to use a pump to transfer it. Instead a simple steam jet is used to send the recycle liquid overhead into the next treating tank to be charged. These ejectors are made up simply from pipe fittings. Half-inch sample cocks, adjacent to them in the recycle line, are opened when the recycle is being transferred, and they indicate visually when to stop recycling and to begin moving the recovered free fatty acid to storage. This differentiation is possible because the recycle possesses a milky brown color whereas the free fatty acid is dark brown.

Process Operation

The operator charges soap stock into one of the two treating tanks. He does this from a point on the ground between the two tanks adjacent to the transfer pump. While the treating tank is being charged, steam is admitted to a two-inch monel heating coil in the bottom of the treating tank to begin warming up the soap stock. At the same time live steam is admitted directly into the charge through a one-half inch monel line to begin raising its temperature also. While this is taking place, he weighs out his sulfuric acid charge into the acid weigh tank and then pressurizes the sulfuric acid system up to the control valve at the overhead platform. By this time the soap stock charging has been completed, and he shuts off the charging pump. Still at the bottom control position he flushes the soap stock charging lines to prevent caking and solidification of this material in these lines.

He then goes up to the overhead platform and begins adding the sulfuric acid with a diluting stream of fresh water, meanwhile continuing to bring the soap stock charge up to the desired temperature range of 200-212°F. (93-100°C.). By the end of the sulfuric acid addition the vessels' contents are generally up to the desired temperature range, and the live steam agitation of the charge is replaced by air. He proceeds to agitate the treating tank contents vigorously for from one-half to one hour, depending on the soap stock feed and the requirements for breaking the emulsion. At the end of the agitation period he turns off this air and continues to circulate steam in the two-inch circular monel heating coil in the bottom of the vessel and allows the acidulated charge to begin settling.

Two points might be noted. An indication that acidification is complete occurs when the materials become uniformly milky. The other point is that less foaming is encountered with a caustic soap stock when the diluted sulfuric acid solution is added to the soap stock; however just the reverse is true when soda ash soap stock is being treated. The reason appears to be that, in the case of the caustic soap stock, the foaming action is chiefly caused by soaps. In the case of the soda ash soap stock the problem appears to be one of liberation of carbon dioxide as the soap stock material is being neutralized. Consequently, in treating soda ash soap stock, the diluted sulfuric acid (acidulating solution) is added to the treating tank first and then the soap stock is charged in on top of this.

In either case, after the acidulated, blown charge has settled overnight, the waste layer is discharged (as described earlier) in conjunction with a neutralizing flow from the caustic metering drum. Both streams enter an open drain leading to the sewerage system. Complete neutralization of the waste water layer does not require attention by the operator. By means of sample cocks at the bottom of the treating tank he can determine when he is approaching the interphase layer. This layer is recycled into the next treating tank by means of a steam jet ejector as has been described previously. By means of a sample valve in this recycle line the operator can tell when to stop transferring recycle and begin sending his product over to the product storage tank. This transfer of product he accomplishes by means of the transfer pump. It will be noted that before the transfer of product takes place, the recycle lines in the common tank discharge manifold which may have trapped recycle material and waste water are flushed with steam. After the fatty acid product layer has been transferred to storage, the entire transfer line is flushed with steam also. At this point the processing cycle is ready to begin over again with a charge of soap stock going to the next treating tank, which contains the recycle from the previously treated batch just described.

Summary

The design of an inexpensive, small scale plant for converting soap stock to crude fatty acid has been described. It has a capacity for producing 6 tons of product (90-95 wt. % fatty acid) per day from 4,000 gallons of soap stock feed (35-40 wt. % fatty acid). Installed at a cost of $11,000, the unit investment has

proved to range between $7 and $8 per annual ton of product. Operating continuously for the past two years since construction (at varying percentages of capacity), total processing costs have averaged 0.75c per pound of fatty acid product.

This plant design is presented to demonstrate that a small scale and marginal operation, such as soap stock conversion, can be carried out both economically and competitively when preceded by sound engineering analysis.

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Note on the Use of Calcium Hydroxide in the Preparation of Peanut Protein

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STUDIES at the Southern Regional Research Laboratory (1, 2, 5, 6) on the preparation of peanut protein have stressed the use of sodium hydroxide as a preferable means of peptizing protein in hexane-extracted peanut meal although the effects of other materials have been reported (3). In the course of investigating the mechanical dewatering of meal residue, calcium hydroxide, a lower-priced material, was used to replace sodium hydroxide. The information obtained on the yields of protein with use of calcium hydroxide is noted here for its scientific interest. Protein so prepared is reported to be suitable for spinning (4).

Preliminary Peptization Studies

The range of protein solubilities with calcium hydroxide was studied in laboratory peptizations to provide information needed for pilot-plant production. Hexane-extracted peanut meal, previously described (6), was ground to pass a 60-mesh screen and was peptized at various pH's at a solution-to-meal ratio of 40 to 1. Distilled water at room temperature (approximately 80°F.) was used. As shown by analytical data in Table I, the nitrogen solubility was practically a constant at about 88% between the pH range of 7.2 and 9.5. This solubility compares with 88.1% for sodium hydroxide solutions at pH 7.5.

TABLE I

Meal Analyses

Moisture,[1] %	3.58
Lipids,[1] %	1.85
Total nitrogen[1]	
As is, %	9.44
Dry, %	9.79
Nitrogen solubility	
NaOH dispersion[1,2]	
As is, %	82.2
Ground, %	88.1
$Ca(OH)_2$ dispersion[2]	
As is, %	84.0
Ground, %	87.8

[1] Previously reported (5).
[2] Determined at pH 7.5, 40:1 solution-to-meal ratio for 3 hours at room temperature (approx. 80°F.).

Pilot-Plant Preparation of Protein

Experimental. In the pilot plant work 100 lb. of meal at approximately 85°F. was peptized at pH 7.5 with calcium hydroxide in solution with tap water, with a solution-to-meal ratio of 15 to 1. Average analysis of the tap water (6) by the New Orleans Sewerage and Water Board over the period of experimentation were in parts per million: Na_2CO_3 as $CaCO_3$, 34.2; chlorides as Cl, 14.5; sulphates as SO_4, 44.2; dissolved solids on evaporation, 147.8; calcium as Ca, 17.8; magnesium as Mg, 5.2; and total hardness as $CaCO_3$, 65.5. The undissolved solids were separated by means of a continuous, horizontal solid-bowl centrifuge (6).

To investigate the possible contamination from formation of calcium salts, the clarified liquor was divided into two equal portions, and the protein was precipitated from one portion by use of sulfur dioxide and from the other by the use of hydrochloric acid, adjusting the pH to 4.5 (2). The precipitated protein was recovered in a solid-bowl, vertical centrifuge and dried at 125°F. (6).

TABLE II

Data on Protein Recovery and Dewatering of Residual Meal

	Run No. 1[1]	Run No. 2	
Meal, lb.	241	100	
Solution-to-meal ratio	15:1	15:1	
Peptizing agent	NaOH	$Ca(OH)_2$	
Spent meal recovery			
Feed rate to centrifuge, g.p.m.	7.2	7.6	
Moisture in spent meal, %	82.5	84.5	
Protein recovery			
Precipitant	SO_2	HCl	SO_2
Protein,[2] basis of original meal, %	35.8	34.4	36.6
Nitrogen in protein, m.f.b., %	16.68	16.76	16.55
Nitrogen, % of original nitrogen	61.0	58.9	61.9
Calcium in protein, m.f.b., %	.01	.025	0.05

[1] Protein produced from portion of same meal and using identical method described previously (5).
[2] Yield = Protein, m.f.b./Meal, m.f.b.

Results. Table II shows the data on protein recovery and on the dewatering of the residual meal. Yields of protein obtained from calcium hydroxide-peptized protein were equal to those obtained from sodium hydroxide-peptized protein (5). While obviously there

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