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## Data on edible vegetable oil processes

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"DATA ON EDIBLE VEGETABLE OIL PROCESSES"

A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS

FOR THE DEGREE OF CHEMICAL ENGINEER

AT THE

SCHOOL OF MINES AND METALLURGY

OF THE

UNIVERSITY OF MISSOURI

BY

JOHN CHARAVELLE INGRAM.

\*\*\*\*\*

*approved by*  
*A. W. Turner*

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## A) INTRODUCTION

### 1. USES OF EDIBLE VEGETABLE OILS

The designation Edible Vegetable Oils when regarded in its broadest sense will include most liquid oleaginous substances of vegetable origin. The only noticeable exceptions are those having cathartic or other medicinal properties. Crude Palm Oil is an Edible Oil in the Congo and Linseed Oil is used for culinary purposes in Siberia. The only criterion of edibility of a fat or oil seems to be the taste of the users and that taste demands fatty materials that range from the distinctly rancid crude oils to the perfectly neutral salad oils produced by modern refining methods.

The term American Edible Vegetable Oils includes only four distinct oils and under certain economic conditions perhaps a fifth. All these oils are to a certain extent interchangeable in their uses, their employment in specific commodities depending largely on their relative price. Listing them in the order of their economic importance, their principal usages are given in the following paragraphs.

#### COTTON SEED OIL

a) By far the largest use of this oil is in the manufacture of Lard Substitute called Compound Lard or simply Compound. The oil is stiffened to the consistency of Pure Lard either by the mixing in of vegetable or animal "stearine" or by the addition of hydrogen to the unsaturated bodies in the oil thus producing stearine "in situ".

b) As a cooking and salad oil it is used largely in place of Olive Oil. Salad dressings were formerly all made with this product as a base.

c) In the production of true Oleo-margarin Cotton Oil forms 50-60% of the fat base used in the churn charge.

d) Large quantities of off-color refined oil finds its way to the soap makers to be utilized as one ingredient in laundry soaps.

e) Petroleum derivatives have almost totally displaced this oil for burning purposes although a small amount of de-stearinized Cotton Oil is still sold for burning at religious ceremonies.

#### COCOANUT OIL

a) The Nut-Margarin manufacturers absorb about three-fourths of all cocoanut oil imported into this country. The fat base of this product contains ninety to ninety-five percent of this oil after refining.

b) When cocoanut oil is cheaper than cotton oil the Compound makers substitute from five to ten percent of it for the latter in their formulae. There are also a few Compounds on the market made of one hundred percent coco oil, but its use in these products has not become general because of foaming phenomena which are met with in the application of this Compound to deep frying.

c) Considerable Virgin Cocoanut Stearine is sold to the candy manufacturers and is used by them in the preparation of a dip for chocolates. This product is made by winter-pressing cocoanut oil and is the solid portion remaining in the cloths after the liquid glycerides have been pressed out.

#### CORN or MAIZE OIL

a) In the last ten years Corn Oil has come rapidly to the front as a salad and cooking oil till today it probably enjoys greater popularity than Cotton Oil.

b) In the Manufacture of Compounds Corn Oil is used to a limited extent as a substitute for a portion of the Cotton Oil. It is a specially desirable ingredient in Compounds containing Edible Tallow and is used there to mask the odor of the Tallow.

PEANUT or GROUND NUT OIL

a) Virgin Peanut Oil, made by cold pressing sound decorticated nuts, is put on the market as a high class salad oil.

b) Five to ten percent of the refined and deodorized oil is found in the Nut Margarin makers formula. It is used here to prevent the formation of a visible crystalline structure in the product.

c) When the price will permit this oil finds a ready market among Compound makers and is an ideal oil for high class shortenings.

SOYA BEAN OIL

During the war large quantities of soya bean oil were imported from the Orient for use as an edible oil. At that time it was made into both compounds and salad oils. Since 1920 the importations have fallen off; that which is being brought in at present being used in paints as a substitute for linseed oil. While this oil is classed with the edible oils there is practically none being refined for edible use.

2. GEOGRAPHIC DISTRIBUTION OF OIL BEARING MATERIAL

Although, in this discussion, we are interested mainly in the domestic industry, it seems advisable to at least mention the sources of supply of crude oils and oil seeds of foreign origin. This is best done in tabular form.

<u>OIL or OIL SEED</u>	<u>VARIETY</u>	<u>COUNTRY</u>	<u>REMARKS</u>
Cotton Oil	Foreign	South China	Pacific Coast Only.
Cocoanut Oil	Cochin	Indo-China ( ) India ) E. Indies ( )	2¢ lb. duty
Cocoanut Oil	Ceylon	Philippines E. Indies	No duty. 2¢ lb. duty.
Cocoanut Oil	Manilla	Philippines	
Peanut Oil	European Virgin	France	4¢ lb. duty.

*Area of Cotton Oil Production  
Principal Refineries ©*





Peanut Oil	Oriental	S. China Japan	4¢ lb. duty.
Soya Bean Oil	Green	Manchuria	2¢ lb. duty.
Soya Bean Oil	Brown	Manchuria Japan	

It is interesting to note here that the American Tropics furnish no oil bearing materials, although numerous attempts have been made to establish the oil cocconut and oil palm industry in Brazil and the Central Americas. The main condition that has arrested the development of plantations of oil bearing seeds in Tropical America is the greater profits that can be realized by raising other crops on the land. Tropical fruits, edible cocoanuts or sugar will yield a greater profit than the oil palm or oil cocconut.

The distribution of domestic oil bearing material is shown on the accompanying map. The more important oil refining centers are indicated by means of circles. The numerals placed on each southern state designates its relative standing in amount of cotton seed and oil produced during the season August 1, 1922 to July 31, 1923.

### 3. STATISTICS OF PRODUCTION.

Because of the fact that the cotton growing industry is distinctly an American development rather complete statistics are available on the production of cotton by-products. In round numbers the cotton crop has shown, in the last decade, a minimum of seven and a maximum of thirteen million bales. Under these crop conditions the amount of seed brought to the mills would vary from two and one-quarter to four million tons and the amount of crude oil produced would range from three-quarters of a billion to one and one-quarter billion pounds.

All cocconut oil used in this country is originally of foreign origin although considerable is now being produced on the Pacific

Coast from imported copra or cocoanut meats. This production amounts to about two-hundred million pounds annually while an equal amount is imported as oil from the Orient.

The production of crude corn oil is confined to those companies milling corn for the manufacture of starch and glucose. Since this oil is distinctly a by-product accurate statistics of production are not available. About one hundred million pounds of crude oil were expressed during 1923.

Only a small portion of the total edible peanut crop finds its way to the oil mills. By far the larger portion of the crop goes to the roasters and candy manufacturers. At the present time only nuts unsuitable for confections are being crushed, the total production amounting to about twenty million pounds annually. The large importations of Oriental crude oil were practically stopped several years ago by a prohibitive duty of 4¢ per pound. Since that time peanut oil has been refined and processed for the margarin makers only.

Soya bean oil made its appearance in the edible oil field during the fat shortage occasioned by the war when the refineries handled several hundred million pounds yearly. About twenty million pounds were imported in 1923, practically all of it being taken up by the paint trade.

The latest Department of Commerce statistics on the production of cotton seed and oil are given in Tables 1 and 2.

TABLE 1.

COTTON SEED

	Rec'd. at Mills Aug. 1st to July 31st Tons		Crushed Aug. 1st to July 31st. Tons	
	1922	1923	1922	1923
UNITED STATES	2,923,658	3,244,960	3,007,717	3,241,464
Texas	731,954	973,676	764,892	970,260
Mississippi	329,750	386,269	331,868	386,063
Tennessee	241,029	289,491	241,947	289,556
N. Carolina	299,500	288,583	299,677	288,973
Arkansas	228,917	263,053	229,718	260,979
Georgia	315,065	255,923	326,740	356,429
Alabama	161,864	202,120	163,819	203,720
Oklahoma	154,295	187,120	157,162	187,554
S. Carolina	253,160	152,058	262,415	152,566
Louisiana	102,984	102,949	102,619	102,420
All Other	105,140	143,691	126,884	142,945

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TABLE 2.COTTON SEED PRODUCTS

Produced Aug. 1st to July 31st.

	<u>Season 1921-22</u>	<u>Season 1922-23</u>
Crude Oil, pounds	930,474,738	1,003,025,212
Refined Oil, pounds	839,898,417	910,539,168
Cake and Meal, tons	1,354,604	1,486,861
Hulls, tons	936,990	943,595
Linters, 500# bales	400,371	608,708
Hull fiber, 500# bales	42,069	76,689
Grabbots, notes 500# bales	11,481	18,317

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#### 4. ESTABLISHED TRADING STANDARDS

The various vegetable oils are bought and sold on different bases which take into account the various characteristics of the oils. Certain rules have been established by the different interested parties and practically ninety percent of the trading is done in accordance with these rules, of which those of the Interstate Cottonseed Crushers Association are the most universally used. A summary of these rules is given in Table 3.

It will be noted in the table that the trading bases for Cotton and Peanut Oils have been worked out in greater detail than have those for Soya Bean, Corn or Cocoanut. The specifications for the latter include no provisions for quality from the standpoint of flavor, so that all off flavored oils must be accepted as prime. The specification "Fair Average Quality" means absolutely nothing from the refiner's standpoint and makes a good tender out of any corn oil that tests under 3.0% Free Fatty Acid.

TABLE 3.

TRADING BASES

BASIS	REFINING LOSS	F.F.A.	COLOR REFINED	FLAVOR
COTTONSEED OIL	:	:	:	:
Choice Crude	6.0%	:	:	:Prime
Prime Crude	9.0%	:	35y - 7.6 red	:Prime
Basis Prime Crude	:	:	35y -16.0 red	:Prime
Off Crude	Within 5% of sample	:	:	:Off
Prime Summer Yellow	:	0.25%	35y - 7.6 red	:Prime
Off Summer Yellow	:	0.50%	35y -12.0 red	:Off
Red Off Summer Yellow	:	0.75%	35y -20.0 red	:Off
Bleachable P.S.Y.	:	0.25%	Bleach 20y - 2.5 red	:Prime
PEANUT OIL				
Choice Crude	3.0%	:	:	:Prime
Prime Crude	5.0%	:	35y - 5.0 red	:Prime
Off Crude	Within 3% of sample	:	:	:Off
Prime Yellow	:	0.25%	35y - 5.0	:Prime
Good Off	:	0.25%	35y - 5.0	:Off
SOYA BEAN OIL				
Prime Crude	5.0%	:	35y - 9.0 red	:No spec- ifications
Crude	7.0%	:	35y - 11.0	:"
COCOANUT OIL				
			Color Crude	
Choice Crude- Cochin	:	3.0%	12y - 2.0 red	:No speci- fications
Prime Crude - Ceylon	:	5.0-6.0%	30y - 5.0 red	:"
Basis Prime Crude	:	6.0%	30y - 6.0 red	:"
Manilla	:	5.0-7.0%	No specifications	:"
CORN OIL				
Fair Average Quality	:	3.0%	No specifications	:No speci- fications
OFF	:	SOLD ON SAMPLE ONLY	:	:

## B) CRUSHING or MILLING

Although the present thesis deals primarily with the refining and processing of oils after production from the oil bearing materials, a short chapter is included here on the extraction of the crude oils from the primary source. Oils occur in plant life principally in the seed where, with starch, it serves as an important source of food for the young plant. Its occurrence is invariably in tiny sacs, the liquid or semi-solid oleaginous material being surrounded by a thin membrane of vegetable protein and fibre. All expression processes aim at the complete rupturing of these sacs and the subsequent draining out of the oil. Solvent extraction processes depend on the ability of the solvent to either penetrate or partially dissolve the membrane surrounding the oil. The former are strictly mechanical in their operation, the latter mainly chemical. The physical character of the material to be handled is the deciding factor in the selection of the type of equipment best adapted.

### 1. PREPARATION OF RAW MATERIALS

For reasons readily appreciated few materials are sent directly to the presses or extractors without some preliminary treatment. As the seeds arrive at the mill the "meats" or oil bearing portions are separated from the hulls or shells. If this is not done the yield of oil is very low due to the absorption of the oil by the dry, fibrous hulls. In the extraction process decortication of seed is essential because of the action of the solvent in removing coloring matter from the hulls.

The preliminary mechanical operations undergone by the several raw materials are listed below.

COTTONSEED

- GINNING - to separate cotton fibre from seed. Cotton bolls contain by weight about 2/3 seed and 1/3 cotton fibre.
- DELINTING - to remove short fibre called linters.
- HULLING - to loosen hulls from "meats".
- SCREENING - to separate hulls from "meats".
- CRUSHING - to flatten "meats".
- COOKING - to soften sac membranes and distend sacs.

PEANUTS

- SHELLING - to remove shells.
- POLISHING - to remove skins.
- PULPING - to expose oil sacs.
- COOKING - to soften sac membranes and distend oil sacs.

COCOANUTS

- SPLITTING - to remove milk and meat.
- DRYING - to preserve meat from fermentation insect ravages.
- PULPING - to expose sacs.
- COOKING - to soften sac membranes and distend sacs.

CORN

- STEEPING - in H<sub>2</sub>SO<sub>3</sub> solution - to soften hulls.
- CRACKING - to loosen hulls from starch and "germ".
- FLOATING - to separate starch from hull and "germ".
- DRYING - to remove moisture.
- WINNOWERING - to separate "germ" from hulls.
- CRUSHING - to flatten "germ".
- COOKING - to soften sac membranes and distend sacs.

SOYA BEANS

- PULPING - to expose oil sacs.
- COOKING - to soften sac membranes and distend oil sacs.



## 2. EXPRESSION PLANTS

At the present writing practically all edible vegetable oils produced in this country are being made by the expression process. Solvent extraction has made considerable progress in the last decade in England and on the Continent but American producers have not yet accepted this process or erected anything but experimental units.

American oil mills, especially those engaged in crushing cotton seed, have been built in smaller units and distributed widely thru the cotton growing section. The tendency has been toward a large number of cheaply constructed plants with capacities ranging from fifty to two hundred tons of seed a day during the crushing season which extends from September 1st to about May 1st. Several large mills have been constructed with a view to more economical production and use of power but they have not yet proven successful.

The average one hundred ton mill contains the following equipment in addition to boilers and engines.

1. Three triplex hydraulic pumps capable of developing 4500 pounds pressure - using cottonseed oil as the compression medium.
2. Four Accumulators - two high and two low pressure for equalizing the pressures.
3. Fourteen (14) Box Presses with fifteen boxes each.
4. Six (6) Single compartment, steam jacketed cookers or two (2) fire-compartment cookers.
5. Two (2) cake strippers - Two (2) cake formers.
6. About one thousand yards of camels hair press cloth 18" wide.
7. Necessary oil pumps and storage tanks.
8. Cake grinding machinery for making cotton seed meal.

The yield of oil from cotton seed varies widely in different localities and in the same locality during different seasons.

The limits of variation are from about thirty (30) to about forty-six (46) gallons per ton of seed with the average over a ten year period about forty gallons. A mill with the above equipment would average about 4,000 gallons per day or about 50,000,000 pounds per season. Owing to the uncertainty of the cotton crop in certain sections of the South, especially the Southeast, oil milling has become a rather unprofitable business. During the season of 1920 - 1921 many mills operated only four to six weeks and then were shut down for lack of seed.

The box type of hydraulic press has been standard equipment in the oil milling industry since its beginning. This piece of apparatus is simply a vertical hydraulic press equipped with from twelve to twenty-eight rectangular boxes or plates. When the ram, which is fastened to the lower box, is down there is about a three inch space between the boxes. The bottom of each box is a perforated steel plate which acts as a drainage member. As the hydraulic pressure is applied from below the boxes are moved upward and together squeezing the oil from the cakes of meats which have been placed in the boxes. The oil runs from the sides and ends of the cake and from the bottom thru the perforations in the plates. This oil is caught in a trough attached to the lower box and is piped to the conveying gutter set in the floor.

In operating the hydraulic press in oil milling the ram is lowered and the cakes inserted in boxes by hand. A cake usually weighs fifteen pounds and is made in a cake former which is fed directly by the cookers. In making the cake a cloth usually 18" wide and 6' long, is placed in the cake former, cooked meats run in and the ends of the cloth lapped over. When the boxes are filled the low pressure valve is opened and 500 pounds pressure allowed to build up on the ram. Since

the area of the box is usually twice that of the ram the pressure on the cake is 250 pounds. After about five minutes at this pressure the high pressure is applied by means of a quick change valve and in about ten minutes the maximum of 4,000 pounds per square inch is attained on the ram. Fifteen minutes is usually allowed for draining and then the pressure is released, the ram allowed to descend and the cakes removed and stripped of their cloths. The cloths are returned to the cake former for refilling and the operation repeated.

Further development and improvement of the expression process centers around the adoption of mechanical changes in the construction of the hydraulic press which will allow the use of higher pressures. The limit of pressure practical with the box press is the pressure the cloth will stand at the doubled edges, i.e. the tensile strength of the camels hair cloth, which is about 2,000 pounds per square inch. Progress has been made in this direction thru the development of the cage or cylinder press in which the cloth is subjected to compression only.

Cylinder presses are being made at present in sizes from a capacity of 200 to 2,000 pounds per press per charge. The press is essentially a heavy hollow cylinder mounted vertically on a concrete base. The cylinder is accurately bored and fitted with a ram which operates from the bottom. A drainage duct is provided in the side of the cylinder and runs the entire length. Drainage plates which exactly fit the cylinder are made of a corrugated steel plate fastened to a plane plate. The corrugated plate is drilled to allow drainage of oil from the bottom of the cake. In filling the press the ram is raised to the top of its stroke, a plate placed on it, a filter cloth placed on the plate and the meal run in on top of the cloth. The ram is then lowered about 3 inches, another plate placed on the meal and the press

built up by repeating this operation. Since the cake has the same area as the ram, total ram pressure can be applied; pressures up to 6,000 pounds per square inch can be used.

Table 4 is a summary of an extended series of tests using the box and cylinder press on identical materials.

TABLE 4.

HYDRAULIC PRESS vs. CYLINDER PRESS

	<u>% Oil in Meats</u>	<u>% Oil in Cake</u>		<u>% Yield</u>	
	:	:	:	:	:
	:	:	:Box Press- Cylinder Press	:	:Box Press-Cylinder Press
	:	:	:	:	:
	:	:	:	:	:
Cotton Seed (decorticated)	: 28.7	: 7.8	4.5	: 23.1	25.9
Peanuts (Shelled)	: 44.1	: 9.4	5.2	: 37.4	41.7
Copra	: 69.3	: 8.7	5.6	: 61.9	65.8
Soya Beans	: 18.0	: 9.2	5.4	: 11.6	15.4

Continuous presses have been tried in oil milling but have not yet succeeded in establishing themselves as the important crushing units. One type of continuous press, the Anderson Expeller, is used quite successfully in the preliminary operation of crushing oil bearing materials high in oil, i.e. corn germ, copra and peanuts. The meal from the Anderson is always repressed in an hydraulic type press because of the inability of the former to make a cake of sufficiently low oil content.

### 3. EXTRACTION PLANTS

Theoretically the extraction of edible oils by means of solvents offers many advantages over the expression method. The most obvious advantage is the greater yield of oil obtained, it being possible

to extract the cake down to less than 1% of oil. Practically, the difficulties encountered in the mechanical operation of large units result in costs which more than balance the saving due to increased yield.

The two types of extraction plants, the Stationary and the Rotary, are illustrated in Figures 1 and 2. The principles behind both types are the same and involve the following operations.

1. Charging the Extractor with ground meats which have been dried to not over 8% moisture.
2. Extraction by percolation thru the mass.
3. Evaporation to free oil from solvent.
4. Condensation to recover solvent.
5. Steaming of meal in Extractor to free it from solvent.

A commercial solution of the following problems would do much to advance the cause of solvent extraction of edible oil seeds in this country.

a) Finding A Suitable Solvent and Reducing Solvent Losses.

The most suitable solvents involve fire hazard. Those which are non-inflammable are either too expensive or have a corroding effect on iron and copper. Those which have been tried commercially are listed below, together with their principal advantages and disadvantages.

	<u>Advantage</u>	<u>Disadvantage</u>
Gasoline (Gas Machine)	Price and Supply	Fire Hazard, Flavor of Oil
Benzol	Price and Low B.P.	Fire Hazard, Extraction of Non Glyceride Materials
Carbon Tetrachloride	No Fire Hazard	Hydrolyzes to liberate HCl
Di-chlor-ethylene	No Fire Hazard, Constant B.P.	Price and Supply
Tetra-chor-ethane	No Fire Hazard, Constant B.P.	Price and Supply

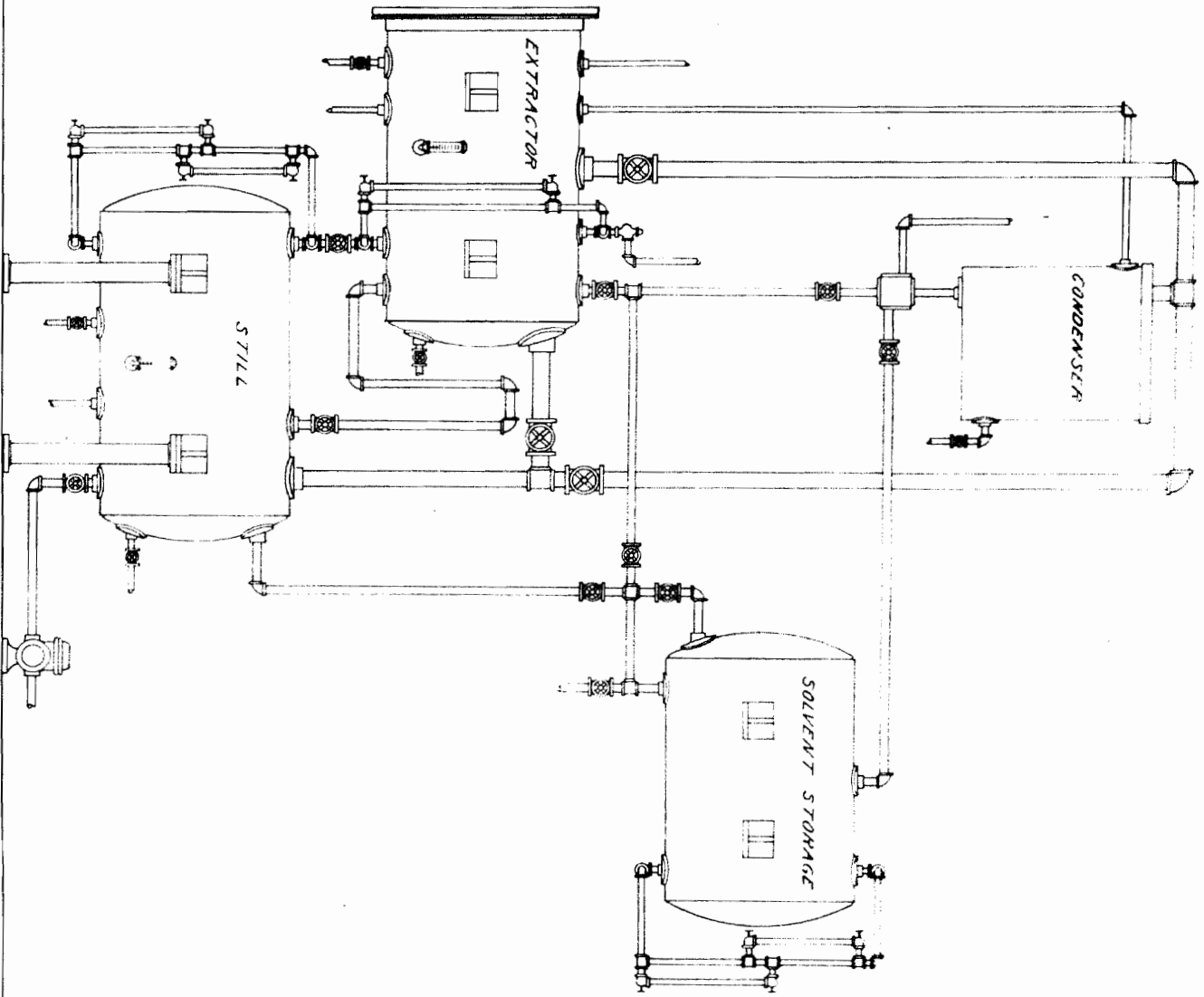


Figure 1  
 Stationary  
 Solvent Extraction  
 Plant.

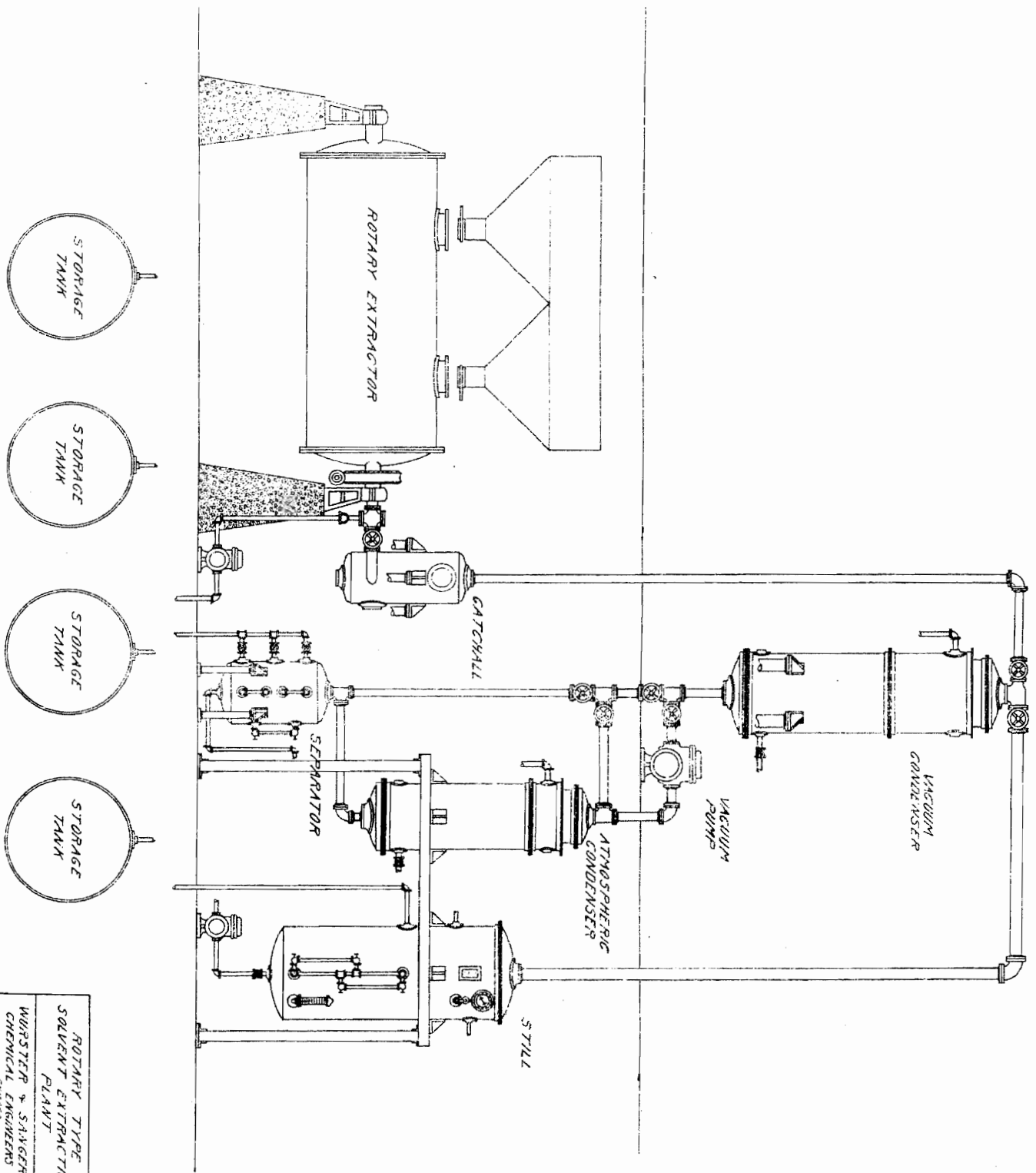
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STATIONARY  
 SOLVENT EXTRACTION  
 PLANT

WURSTER & SANGER  
 CHEMICAL ENGINEERS  
 CHICAGO

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Figure 2  
Rotary Solvent Extraction  
Plant



ROTARY TYPE  
SOLVENT EXTRACTION  
PLANT  
WURSTER & SANGER  
CHEMICAL ENGINEERS  
INCORPORATED  
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The loss of solvent is usually a large item in the cost of operating these plants. Many designers of extraction apparatus claim that this loss can be kept down to about 1/2 to 1% of total solvent used per cycle. Most plants show between 2 and 5% loss during continuous operation.

b) Freeing Oil and Meal of Last Traces of Solvent.

All extracted oil can be readily identified by the odor. The elimination of solvent odor from both oil and meal has not yet been accomplished commercially without injuring the color of the oil.

c) Refining Extracted Oil.

Extracted oil presents some interesting problems for the refiner. In the refining kettle its reaction to caustic makes necessary radical changes in the mechanical handling of the kettle. The refining losses are on the average considerably higher than those of expressed oils.

C) REFINING

1. SAMPLING AND UNLOADING

Both crude and yellow oils are invariably transported in tank cars of approximately sixty thousand pound capacity. Immediately on receipt of a tank of oil a twenty pound sample is drawn by means of the gun sampler. This sampler is simply a  $2\frac{1}{2}$  inch brass tube about ten feet long and fitted at the bottom with a valve operated by means of a cord. In operating this sampler it is thrust thru the tank dome into the oil, the valve opened and the tube allowed to fill. Since all oils are accepted or rejected on the tests made on samples taken in this manner, this sampling becomes a rather important item in refinery operation.

This method of sampling, the best that has yet been devised, is not entirely satisfactory from the standpoint of the refiner.



During the last half of the cotton oil season the oils always contain more or less meal which during transit is concentrated at the ends of the tank. This meal is not reached by the sampler and in consequence the sample is invariably better than the true average oil in the tank. For this reason the above method of sampling favors the seller and operates against the buyer.

Oil is usually unloaded from tanks by pumping from the bottom thru a special connection which is equipped with a cock, inside opening plug valve and cap. Sometimes this connection is in bad shape, which necessitates unloading the tank by pumping over the top. Of the two methods the former is much to be preferred for the following reasons.

- a) It is much easier to thoroughly squeegee the tank.
- b) Less air is incorporated in the oil if it runs to the pump than when it requires a suction lift.

In this connection it would be well to mention the fact here that the amount of air in the oil has an important bearing on the refining loss. All suction lines should be tight to prevent air leaking at this point. Since no refining should be attempted till all air has left the oil the incorporation of air in pumping up causes a delay which may result in the loss of a days time in refining.

## 2. TESTING

Before any oil is unloaded, tests must be made in the refinery laboratory to determine whether the oil comes up to the specifications under which it was purchased. These tests before unloading are quickly made in the case of Corn and Coconut Oils and consist only of a determination of Free Fatty Acid content. In the case of the other oils a test must be made to determine the refining loss.

A detailed description of the standard method for

determining refining loss is given in the Rules of the Interstate Cottonseed Crushers Association. This method is, briefly, a miniature imitation of the procedure carried<sup>out</sup> in refining on a large scale. Three tests are usually made on 500 gram samples using three different strengths of lye solution, all of which contain the same actual weight of caustic. The following example will illustrate the calculations necessary to determine the amount of each solution to use.

Cottonseed Oil containing 1.4% Free Fatty Acid (as oleic).

7 / 1.4	
<u>0.2 %</u>	Pure Caustic necessary to neutralize F.F.A.
0.35%	" " " " secure color.
<u>0.55%</u>	Total pure caustic to refine.

12 degree Be Lye contains 8.0 % Pure Caustic

14 " " " " 9.42% " "

16 " " " " 10.97% " "

$\frac{0.08}{0.55}$	$\frac{0.0942}{0.55}$	$\frac{0.1097}{0.55}$
6.87% 12° Lye	5.84% -14° Lye	5.01% -16° Lye

If any of the refining tests produce an oil with a color and loss equal to or lower than the specifications call for, the oil must be accepted as the grade tendered. Should all of the tests indicate higher loss or darker oil than that allowed for the particular grade, then a new series of tests must be made to determine the minimum loss that must be sustained in order to secure the desired color. Should this loss exceed that allowed in the specifications the tank is held on track and a new fifty pound sample taken by the Board of Trade Inspector. The seller must be notified by wire of the result of the tests. Three one-gallon samples are then prepared and disposed of as follows: one is sent to the seller, one to a referee chemist and one is held to be used in case of further dispute. The results of the referee's tests are used as bases for settlement of conflicting claims.

After the oil is unloaded into the refining kettle it

is well mixed and another sample drawn. This sample is more representative than the tank car sample and should be used in making another series of tests to determine the amount and strength of lye to be used in the actual refining. That lye is selected for commercial refining which gives the lowest loss or the best color or a compromise between the two.

### c) REFINING IN GENERAL

By "refining" we mean caustic refining or neutralization of the acid and coagulation of the non-glyceride bodies in the oil. All crude vegetable oils contain the following impurities in varying proportions:

1. Moisture in solution and suspension.
2. Meal in suspension.
3. Resins and gums in solution.
4. Albuminoids and phosphatides in solution.
5. Ferments in solution and suspension.
6. Coloring matter in solution (mainly gossypol)
7. Fatty acids in solution.

It can be readily understood that with the varying proportions of the above constituents in the oils the caustic refining cannot be done by any set rules and can be controlled only to a certain degree by laboratory tests. The proper handling of a refining kettle is largely an art and must be learned thru actual experience on a wide range of oils. The biggest factor in refining is the judgment of the refiner who controls the process mainly by the appearance of the oil and soap stock during the varying stages of the operation. In a general way the various steps in the caustic refining process are as follows:

1. Determination of the amount and strength of caustic to be used. The refiner's judgment is the main factor

in making the decision, although the laboratory tests are usually the basis upon which this judgement rests.

2. Determination of the necessity of using agents other than caustic soda, i.e. sodium silicate, salt, water, etc.
3. Temperature at Start and Speed of Agitation.

The lye is introduced into the oil at a rate which will complete the introduction in about fifteen minutes for a tank car of oil. Too rapid introduction of the lye puts too great a concentration of caustic into the oil and increases the saponification of neutral oil. When lye is added slowly a large portion of it is used up in neutralizing the free Fatty Acids about as fast as added.

The agitation used while adding the lye and while the mixture is being stirred to get color should be about 20 to 30 R.P.M. with the horizontal sweep paddle. This speed of agitation is usually continued until a visible "break" occurs in the oil. This "break" is the separation of minute particles of soap stock or flocs from the oil and indicates that the flocs are beginning to coagulate or break from the emulsion. The saponified fatty acids, the majority of the coloring matter and impurities and the excess lye are gathered together in these flocs. At this stage a sample should be drawn from the kettle, filtered and read for color in order that the refiner may know how the operation is progressing. If the color is darker than desired, longer agitation or more lye may be necessary.

As to temperatures at the start of refining, best results are obtained on cotton, peanut, corn and soya oils at 80 to 90° F. Coconut and animal oils can be refined successfully by starting at 140 - 150° F.

4. Coagulation of the Soap Stock.

After the "break" occurs the oil is refined and the

remainder of the operation is devoted to getting a good separation of soap stock from oil. If separation is to be accomplished by settling, the foots must be put into a condition in which they will pack and run together. The handling of the kettle to attain this condition requires considerable experience and directions for procedure cannot be set down. Different types of the same oil act differently at this stage in the process.

In general, the operation is about as follows:

The oil is heated slowly with the paddles turning 10-12 R.P.M. The grain size of the particles of soap stock gradually increases and the particles seem to be getting heavier. The temperature to which the oil is heated is governed by the appearance of the foots. So far as the quality and color of the oil is concerned, it should be the object of every refiner to finish each kettle at as low a temperature as will give compact soap stock. If the finishing temperature is too high some of the color will be redissolved from the foots. With the one exception of cocconut oil the finishing temperatures seldom exceed 135° F.

#### 5. Settling

After the coagulation of the soap is complete the agitation is discontinued and the steam turned off. The kettle should be settled at least twelve hours. In cases of emergency six hours settling has been tried but invariably resulted in increasing the loss 3/4 to 1%. The oil is drawn from the kettle thru a swinging suction, leaving the foots packed in the bottom. Some oil is recovered from the foots by remelting in the kettle after the oil has been drawn off. This oil is seldom as good as the original oil and in most cases should be re-refined before using in edible products.

The refining loss can be determined in two ways. This

is usually done by weighing the oil drawn off although it is possible to arrive at the percent lost by weighing the foots. In well regulated refineries both methods are used; the latter acting as a check on the former. In order to obtain good check results it is necessary to weigh the refined oil, the refining solutions used and the soap stock produced, the difference between the weight of soap stock and the weight of the refining solutions being the oil loss. The losses figured by the two methods should check within 1/2% refining loss.

#### D) OIL REFINING EQUIPMENT

Figure 3 is a show drawing of simple refining and bleaching equipment giving ideal arrangement of apparatus and piping.

#### E) REFINING OF TYPICAL OILS

Although each individual tank of oil is a problem in itself there are certain general facts in regard to refining which are helpful in handling the various types of crudes. It must be thoroughly understood that there are exceptions to all the following generalities and that they are included as guides to refining and not rules for refining.

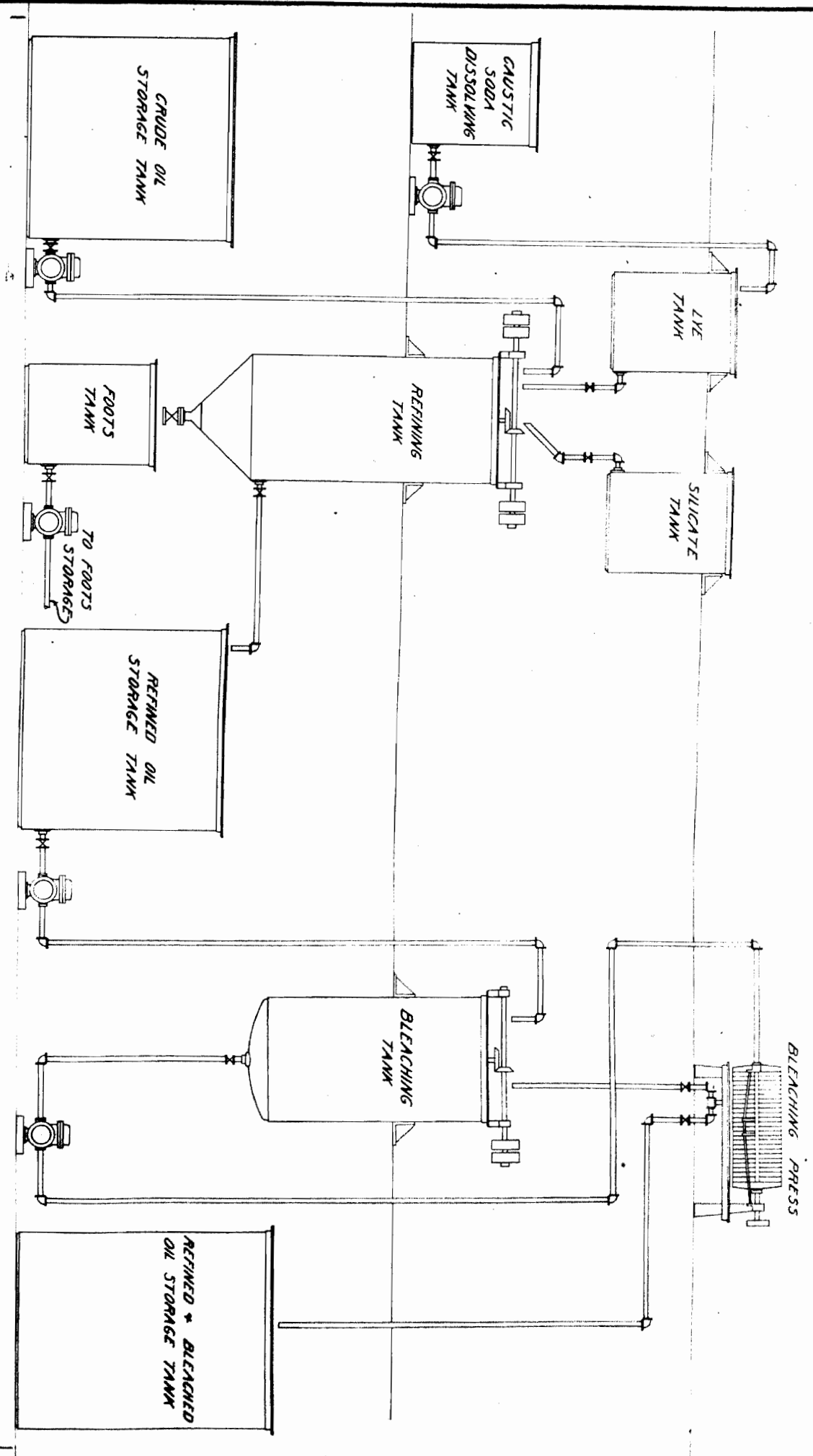
### THE REFINING OF VEGETABLE OILS

#### COTTON SEED OIL

The many different grades of crude cotton seed oil that are available each season as well as the varying quality each season, practically eliminates any set basis for refining this oil.

For Prime crude oil, clean and with a F.F.A. content of less than 2.5%, the best results are usually obtained with 12 to 14 degree lye. Which to use is shown by the usual laboratory tests. Enough lye should be used to furnish plenty of solution, so that the foots produced will melt and run together without exceeding a temperature

Figure 3  
Oil Refining Plant



OIL REFINING  
PLANT

WURSTER & SAASEN  
CHEMICAL ENGINEERS  
CHICAGO

Sheet No. **1055**  
Drawing No. 214

Fig. 3

of 130 degrees F. For example, an oil of 1% F.F.A., clean and free from settlings can be refined with 5% of 12 degree caustic. If this same oil should contain quite a percentage of meal and extractive matter it is often advisable to use as high as 6% of 16 degree caustic and 1% of 20 degree silicate solution to weight the foots.

Above 3% F.F.A. content a crude oil is refined in two ways. First, with a fairly large percent of heavy lye, which is the practice when the oil is such that a bleachable yellow oil can be obtained. The second procedure is to make a yellow oil to be used for cooking oils or yellow compounds. In the first case the oil is best refined with approximately 8% of 16 degree lye or 9% of 14 degree lye. If the oil is to be used as yellow oil as noted in the second method of refining the lye may be used 6% of 16 degree, 7.5% of 14 or even 9% of 12 degree caustic. The losses in the use of the weaker lyes will usually run from 1 to 2% lower. In case the oil contains excessive amounts of extractive matter or settlings, silicate solution to the amount of 1% of 20 degree strength may be used. This increases the smoothness of the foots and cuts down the loss.

Crude cotton seed oil with a F.F.A. content ranging from 4% to 7% will generally produce unbleachable yellow oil, and is best refined with 16 and 18 degree caustic. An excess of 3% to 4% is all that is required. For example, an oil of 6.3% F.F.A. requires 8.2% of 16 degree lye to neutralize the acid. 11.5% of 16 degree or 10.5% of 18 degree caustic is ordinarily sufficient for refining this grade of oil.

Oil that has a F.F.A. content ranging from 7% to 12% is best refined with 18% degree caustic. This lye gives a heavy foots that still has enough solution to form a soft smooth soap. 4% excess is plenty on oils of this character as they more often produce dark



colored yellow oils.

From 12% to 18% F.F.A., 20 degree lye generally gives the best results. 25 degree lye is sometimes used, but as it gives a hard foots that does not settle or break out from the oil very well. Silicate should be used in all of the above cases if laboratory results show it will cut down the loss. The use of silicate does not always cut down the loss, but as a usual thing it settles the foots better. Silicate is always preferred when using 25 degree lye to furnish extra solution and to make a softer foots.

In any of the above cases, where an oil that is refined with the limits of lye as allotted by the Interstate Cotton Seed Crushers Association, will produce a bleachable yellow oil, but an excessive loss, it is generally better practice to refine with a lower loss and darker color and later double bleaching as required.

Oils above 20 to 25% F.F.A. are refined only on laboratory test. 25 degree lye is usually the best, but oil of this nature is best refined by mixing with a poor grade of refined oil, thus cutting down the concentration of the foots produced. Otherwise a very heavy and hard foots is formed from the high fatty acid and the strong lye. If 25% F.F.A. is reduced through the addition of refined oil until about 15% F.F.A. results 18 or 20 degree lye can be used as cited for oils of this grade.

Yellow Butter Oil can be produced from a prime oil or from an oil slightly off grade by setting the color of the crude. This is done by heating the crude oil in the refining kettle to about 222 degrees F. and agitate slowly for about an hour while bringing up to heat. Let the hot oil set over night, cool down to 95 or 100 degrees F. and refine with a weak lye. In addition use about 1.5% silicate solution.

A good oil is usually started at 80 or 85 degrees F.

In case an oil runs 10% F.F.A. a temperature of 90 degrees F. may be used at the start and the refining finished at 130 to 135 degrees F.

In any of the above cases the oil is to be stored it is graded by a sample from the refining kettle. The grade is determined according to color, flavor and bleach, using 6% of Standard English Earth in making the bleach. The three factors determine its class and what it can be used for. The refined oil is then drawn off and cleaned with .20% to .75% of fuller's earth or Filtercel, at a temperature ranging from 120 to 150 degrees F.

Should the oil be desired for use at once it may be bleached without cleaning. This saves the time of an extra handling and the expense of cleaning. The amount of earth used for bleaching depends on the ultimate use of the oil.

In purchasing crude oil it must grade prime unless the contract specifies basis prime or otherwise. The standard for a prime oil is 35y. 7.6 r. The flavor must be prime and the loss not exceed 9.00%. Oil darker than 16 red is open to rejection unless stipulated in the contract.

#### CORN OIL

Corn oil is peculiar to the fact that it has no set trading standard as yet. It is usually sold on a 3% F.F.A. basis. It must not contain over .5% of H<sub>2</sub>O and settlings, be prime in flavor, (whatever that denotes), and refine to a color of 35y 7r with a loss of 9.00%.

This oil is usually clear and free from settlings. The oil is hard to refine without the use of silicate but if proper care is exercised it can be done. The oil seldom runs over 3% F.F.A. When an oil of 2.5% to 3% F.F.A. is to be refined 14 to 16 degree lye is the best. Below that 12 degree lye is better. In some cases 10

degree lye will suffice, especially if the crude oil is light in color. 14 degree is better in the majority of cases, as the foots are very light and require weighting. The foots are very flocculent and will float at times, even if silicate is used. Refining by using the lower coil only for heating is best. Cool the coils after the refining is completed. .25% excess pure NaOH is the best amount of lye to use regardless of the strength. Silicate can be added, either first or after the coil is up to heat.

Corn Oil is generally a good bleaching oil so the refining for a yellow oil is not considered. Circumstances will govern the production of a yellow oil. After the refined oil is deodorized, a deposit settles out as a white flocculent substance. This can be removed by filtering at a temperature of about 60 degrees F, using Filter-Cel. This has to be removed or it kills the cold test of the natural winter oil.

#### COCOANUT OIL

Cocanut oil is easily refined, both by weak and strong lyes. There are two methods that produce good results.

When 14 degree lye is used an excess of .25% pure NaOH is figured above the amount required to neutralize the F.F.A. The F.F.A. being calculated as Oleic Acid. Start the refining at 90 degrees and finish at 110 to 120 degrees F. The foots are water-like, and contain a lot of coloring matter. Be sure to allow the oil plenty of time to settle well, as it is usually slightly wet.

When using 20 degree lye, 1% more 20 degree lye is used than the F.F.A. of the oil. For example, on 6% F.F.A. use 7% of 20 degree lye. Approximately one-half of the F.F.A. is added in the form of water. On 6% F.F.A. 3% water is added. This is run in first with the oil at a temperature of 90 to 95 degrees F. The lye is run

in directly after the water. The whole operation should be completed in forty five minutes. The heat of reaction is about all that is required as it will finish about 110 degrees F. This method gives a better bleaching oil and one that holds its color better on deodorization, the loss, however, is sometimes slightly higher than in the first procedure.

A great many refiners prefer the hot refining method on this oil. This method consists of heating the oil to 140° - 150° F at the start and finishing the refining without the application of more heat. Neutral cocoanut oil is very difficult to saponify with weak lye which makes possible the use of high heat. Ten or twelve degree lye is used and the excess employed varies from 0.05 to 0.15 pure caustic. The lye is stirred in rapidly and the agitation discontinued after about 10 minutes. No "break" occurs as the foots are liquid and settle rapidly forming a liquid layer at the bottom of the kettle. The losses with this method can be brought as low as 1.4 times the F.F.A. percentage.

This oil is usually bought on the F.F.A. content and allowances are made for the excess acid. The standard color for refined oil is 30y 6.0r and should not show any bloom. The refining loss, as figured for factory bookkeeping is set at 12% which produces 22.5 pounds of foots.

#### PEANUT OIL

There are two kinds of crude peanut oil and the methods of refining differ. Oriental peanut oil is usually clear, light in color, and often semi-refined. It is low in free acid. The other grade of oil is domestic and may consist of good or bad oil depending on the mill producing it.

Oriental peanut oil refines the best with 10 degree lye although 12 degree is often used. A very small excess of NaOH is required on this kind of oil, preferably not over 1%. About 1 to 2%.

silicate is also used, and may be added first or after heating as in the case of bean oil. I think first is preferable because the color of this oil is far above standard even if silicate does retard the removal of as much crude coloring matter. 70 to 80 degrees F is the best temperature to start the refining and 120 to 140 degrees F is suitable for finishing. The foots produced are light and fluffy so be sure to have the oil stand for about three hours after pumping into the refining kettle for this helps remove air and cuts down the foam. In the process of refining heat the oil to temperature with the bottom coil. After the refining is completed be sure to cool the coils or a part of the foots will come to the top. On Oriental peanut oil 2% fullers earth is usually enough for bleaching.

Domestic peanut oil if clean and made from ripe peanuts refines the same as the oriental oil. If it is made from green peanuts and thus contains extractive matter, or if it contains a lot of settlings 14 to 18 degree lye is the best to use. This is determined by test. Only a small excess of lye is needed if the oil is clear yet made from green peanuts. Domestic peanut oil is generally darker than Oriental, therefore, it is necessary to use slightly more lye than on the imported oil. The silicate percentage remains about the same.

I have never used the process of mixed lyes on peanut oil as described for bean oil but I think this would be a very desirable procedure. Some refiners give peanut oil a water wash of about 1.5% to 2% after refining with 12 to 14 degree lye. The water is run in at a temperature of 110 to 120 degrees.

The official grading of peanut oil is 50y 5 r for color. Flavor sweet and bland, not burnt or woody as in the case of Oriental oil. Loss 5%, and crude not containing over .5% of water and settlings. These rules hold except when oil is bought under other specifications.

SOYA BEAN OIL

Soya Bean Oil bears the distinction of being at the same time the easiest and the hardest of oils to refine. Without the use of silicate this oil is very hard to refine; with the use of silicate it is very easy.

The intense yellow color of Soya bean oil, often approaching a brown or green or a mixture of the two, requires different methods of refining. If the oil is brownish red and clear a very low percentage of 16 or 18 degree lye is the best. If the oil is murky, wet footsy, and like linseed in color, it requires strong lye, long agitation and plenty of solution. Otherwise the refined, cleaned, bleached and even the deodorized oil will have a deposit of coagulated albuminoids if allowed to settle.

For a clear oil of .7% to 1.5% F.F.A., 16 to 18 degree lye is best used in conjunction with .5% to 1% silicate solution of 20 degree strength. Some refiners add silicate first and agitate for 15 minutes at a temperature of 80 degrees F, then add the amount of lye as calculated. Others add lye first and agitate for 15 minutes, heat to 110 degrees F, then add the required amount of silicate and heating up to 120 to 130 degrees F to finish the refining. This last method produces two kinds of foots and will not settle as compact as the former. There is a layer of heavy smooth foots then a top layer of fluffy light foots which do not contain the same amount of silicate as the lower mass.

On good oils, that is oils that refine to an average color of 100y 13r to 50y 10r, the following method is better. Mix the lye and silicate together, 75% of required strength be lye and 25% 40 degree silicate. Use this mixture as straight lye. The temperature of starting is 80 degrees F and that of finishing is from 120 to 125 degrees F. The

loss is from 1% to 2% lower, the color is usually a little darker than that obtained by the other methods. This is not always true however due to the fact that a lower temperature is used in refining. With oil of the above grade use .5% excess 18 and 16 degree lye. This is sufficient for a clear oil, but on a wet and dirty oil 1% excess of each strength lye should be used.

In oils ranging from 1.5% to 3% F.F.A., 1% excess of 16 and 18 degree lye is sufficient even though the oil is slightly wet and murky.

With oils that are like linseed, or oils that are way off average quality use 1.5% to 2% excess of 16 or 18 degree lye.

In the last method of refining, the mixture is figured as 75% caustic solution and from this the lye figured just as though the silicate were not present. For example, an oil that requires 3% of 18 degree lye for proper refining would take 4% of the lye and silicate mixture.

The refined oil is cleaned with fullers earth or Filter-Cel, better with the latter. About .3% of Filter-Cel is used at a temperature of 140 to 150 degrees F. In bleaching English Earth and Filtchar, or domestic earths are used. As high as 8% fullers earth and 1% of filtchar being used to product suitable for compound. This oil is bleached better if mixed with peanut, cotton seed or corn oil before put in the bleaching kettle. Note: Yellow soya bean oil on deodorization will lighten up as much as one half of original color. Often it becomes greenish after this treatment, according to the origin of the oil.

Soya bean oil is bought on two refining losses as the standard. The two used as basis prices are 5% and 7%. The latter is the one in Universal use. The refined oil should have a color of 12 red.

The yellow of the color varies, often ranging from 140 to 40 in intensity. The contracts usually read for edible oil that the oil be pressed and not extracted, but the difference is a hard thing to determine. The oil must not contain over .5% of water and settlings.

F) REFINING RESULTS

The term refining loss really means a depreciation in value of a certain percentage of oil rather than an actual loss. While the refined oil is worth more than crude oil the soap stock formed brings considerably less. This soap stock contains from 16 to 24% of neutral oil which is emulsified in the mass and cannot be separated profitably at present. Some notion of the losses obtained can be gained from Table 5, which represents the minimum losses which can be expected on various crudes when good color is obtained.

Tables 6 and 7 are included to convey some idea of the operation of a refinery over a period of years and thru one typical season.



TABLE 5.

<u>OIL</u>	<u>F.F.A.</u>	<u>LOSS</u>
<u>Cotton Seed</u>	0.5	3.5
	1.0	5.0
	2.0	7.0
	3.0	8.5
	6.0	14.0
<u>Peanut</u>	0.5	2.0
	1.0	3.0
	1.5	3.75
	2.0	5.0
<u>Cocoanut</u>	0.5	1.0
	1.0	1.5
	2.0	3.0
	5.0	7.5
<u>Corn</u>	1.0	4.0
	1.5	5.5
	2.0	7.5
	3.0	9.0
<u>Soya Bean</u>	7.0	3.5
	1.0	4.5
	1.5	5.5
	2.0	6.5
	3.0	9.5

- - - - -

TABLE 6.

SUMMARY OF NINE SEASONS OF COTTON OIL REFINING

<u>Season</u>	<u>Cars</u>	<u>Lbs. Crude Oil</u>	<u>Lbs. Yellow Oil</u>	<u>Loss %</u>
1908 - '09	1262	74,551,130	69,366,679	6.95
'09 - '10	948	53,932,165	50,219,511	8.26
'10 - '11	996	58,268,240	53,877,475	7.53
'11 - '12	875	49,137,612	44,762,998	8.90
'12 - '13	804	48,689,361	44,770,038	8.05
'13 - '14	424	25,115,790	22,507,088	10.38
'14 - '15	272	16,081,505	14,662,240	8.76
'15 - '16	289	17,129,272	15,842,780	7.51
'16 - '17	277	16,527,475	15,599,840	5.61
<b>TOTAL</b>	<b>6147</b>	<b>359,432,550</b>	<b>331,608,719</b>	<b>7.74</b>

<u>Season</u>	<u>Lbs. Foots</u>	<u>Lbs. Lye</u>	<u>Loss%</u>	<u>%F.A. in Foots</u>	<u>% F.A. in Loss.</u>
1908 - '09	9,630,121	4,434,648	6.97	43.50	80.70
'09 - '10	7,270,068	3,194,622	8.15	47.05	82.85
'10 - '11	7,867,853	3,495,144	7.52	44.39	79.20
'11 - '12	7,515,157	3,185,975	8.81	48.75	82.30
'12 - '13	7,174,146	3,303,516	7.95	44.94	82.80
'13 - '14	4,464,975	1,868,845	10.33	49.10	84.20
'14 - '15	2,562,593	1,133,042	8.82	48.31	87.20
'15 - '16	2,217,557	931,065	7.51	50.37	86.80
'16 - '17	1,620,103	692,468	5.61	46.13	80.56
<b>TOTAL</b>	<b>50,332,573</b>	<b>22,239,325</b>	<b>7.81</b>	<b>46.17</b>	<b>83.51</b>

TABLE 7.

SUMMARY OF A TYPICAL SEASON OF  
COTTON OIL REFINING

Month:	Cars:	Crude Oil:	Yellow Oil:	Cleaned Yellow Oil:	Loss %:	Foots:
Sept.	29	1,656,727	1,521,694	1,515,988	7.26	259,253
Oct.	117	6,788,905	6,375,960	6,352,050	6.08	803,085
Nov.	167	9,598,580	9,046,320	9,012,396	5.75	1,051,860
Dec.	159	9,114,475	8,582,870	8,550,684	5.83	995,388
Jan.	184	10,709,295	9,991,345	9,953,878	6.70	1,327,335
Feb.	203	11,875,883	11,081,505	11,039,949	6.69	1,480,398
Mar.	131	7,594,400	7,047,405	7,020,977	7.20	1,014,289
Apr.	79	5,457,165	4,991,865	4,973,146	8.53	833,720
May	100	5,671,880	5,165,555	5,146,184	8.93	899,713
June	90	5,929,110	5,424,960	5,404,617	8.50	935,268
July	3	154,710	137,200	136,685	11.32	29,812
<b>TOTAL</b>	<b>1262</b>	<b>74,551,130</b>	<b>69,366,679</b>	<b>69,106,554</b>	<b>6.95</b>	<b>9,630,121</b>

Month:	Lye (Lbs.):	Loss %:	Water:	% 74° Caustic:	% Total F.A.	% F.A. In Loss:
Sept.	108,976	8.17	7,370	10.45	44.50	84.00
Oct.	375,529	6.30	52,600	11.13	43.58	83.30
Nov.	497,567	5.77	70,380	10.79	40.96	77.90
Dec.	466,099	5.81	65,750	13.39	42.06	78.90
Jan.	610,463	6.69	80,950	10.52	44.20	81.80
Feb.	689,693	6.66	93,900	10.50	42.45	79.40
Mar.	470,106	7.17	63,500	10.60	43.60	81.05
Apr.	371,621	8.48	53,550	10.35	44.50	80.00
May	397,413	8.86	53,250	10.73	46.65	83.20
June	434,884	8.44	49,300	11.01	44.34	82.50
July	12,297	11.32	1,100	11.69	47.26	80.50
<b>TOTAL</b>	<b>4,434,648</b>	<b>6.97</b>	<b>591,650</b>	<b>11.01</b>	<b>43.50</b>	<b>80.70</b>

D) BLEACHING

The color of finished products made from edible oils has always been an important consideration. The bleaching operation has for its primary object the regulation of color. Oils for some uses must be bleached as near water white as possible, while for other products the original color of the refined oil is desired. The standard instrument for estimating colors is the Lovibond Oil Tintometer with red and yellow glasses, reading thru a  $5\frac{1}{4}$ " column of oil.

Crude oils show a great variation in original color, from light yellow in Cochin Coconut Oils to black in off Cotton Oils. This original color has little bearing on the quality of the oil in the case of cotton as dark crudes often refine to lighter colors than some lighter crudes. Refined oil color standards will be found in Table 1. It is this refined oil that is bleached to the color desired for the particular product being made.

All edible vegetables are bleached by means of fullers earth and, or decolorizing carbon. Chemical bleaches are not applicable because of the effect on the flavor. Varying proportions of earth and, or carbon are used to get the desired decolorizing effect. Different earths and carbons produce different degrees of decolorization on different oils, so that the selection of the proper bleaching material is simply a matter of experiment. In general, fullers earths remove the yellows in the oil more efficiently than the reds, while with carbons the action is just the opposite.

The bleaching operation consists of the agitation of the oil and bleaching agent for about one hour in an agitator tank and the separation of the agent from the oil by filtration. Filter presses are almost universally used for this last operation. At the end of the

filtration the oil is blown from the press by means of compressed air, the press blown with steam to remove excess oil and the cake again blown with air. These blowing and steaming operations are for the purpose of lowering the oil retention of the cake. This retention of oil by the filter cake is a very important item in the cost of bleaching and many attempts are at present being made to increase the efficiency of bleaching materials in order to decrease the amount used and thus lower the net retention of oil.

Numerous domestic deposits of fuller's earth have been exploited but few of them yield an earth that is equal to English earth in all particulars. The factors that must be given consideration in selecting an earth for commercial use are listed below:

a) Bleaching Power - Compared with Standard Earth.

b) Oil Retention

Calculated as % Oil on Dry Cake and

Determined by Actual Commercial Test.

c) Flavor

Meaning Intensity of Argillaceous Flavor

Imparted to Oil.

d) Filtering Qualities

Rate of Flow Measurements During Commercial Run.

e) Fire Hazard

Some Earths Catch Fire When Blown in the

Press With Air.

In plant practice to bleach an oil for compound an average of three percent of earth is used. When it is necessary to use more than three percent it will usually be found economical to use some carbon; 0.1 to 0.2% carbon being the usual amounts employed. The spent earth and carbon is usually discarded although some advocate the recovery

and revivification of this material. Recovery has not yet been accepted as economical by many refiners.

Complete operating data will be found in Table 8 which was prepared from a series of exhaustive tests carried out on a commercial scale while bleaching compound. This compound was made from refined cotton seed oil and oleo-stearine and is typical of the animal fat compounds being made at the present time.

TABLE 8

OPERATING DATA - USING VARIOUS FULLERS EARTHS

Kind Clay	English	Regular Medina	Floridin	Ark.	Ill.	American	General Reduction	Special Medina
Weight Refined Oil, lbs.	17,600	17,600	17,600	17,600	17,600	17,600	17,600	17,600
Weight Stearine added, lbs.	2400	2400	2400	2400	2400	2400	2400	2400
Wt. Clay used, lbs.	800	800	800	800	800	800	800	800
Temp. Stearine Melted at °F	218	218	218	218	218	218	218	218
Temp. Bleaching at °F	220	220	220	220	220	220	220	220
Minutes Agitation in Bleach Tank with clay 25.0		40	20	40	10	50	20	25
Minutes Circulated thru Press & Back 25.0	25.0	35	25	75	20	20	20	20
Minutes to filter & Drop 20,000 lbs.	60	65	50	65	65	60	55	60
Pressure at Start Filtering lbs. Gage	5.0	4.0	6	12	3	8	6	5
Pressure at End Filtering lbs. Gage	12.0	9.0	16	18	10	25	12	9
Color on Unbleached Comp'd. 35 yel.	6.5 red	6.1	6.9	5.7	7.0	5.3	5.8	6.0 red
Color on Bleached Comp'd. 20 yel.	2.4	2.0	2.7	1.9	1.8	2.3	2.3	2.0 red
Time Air was on Press. Minutes	10	10	10	10	15	10	10	10
Pressure Air on Press, lbs. Gage	6 $\frac{1}{2}$	6	7	13	4	4	3	5
Time Steam was on Press. Minutes	30	30	30	30	30	30	30	30
Pressure Steam on Press, lbs. Gage	11	9	15	15	10	15	10	10
Time Air on Press at End, Minutes	20	10	20	20	20	15	15	20
Pressure Air on Press at End	3	4	4	3	2	6	4	3
Weight Spent Clay, lbs.	912	923	Caught Fire	970	1193	827	1278	935
Percent Oil in Spent Clay	16.82	27.08	----	11.95	34.23	18.25	32.5	24.61
Percent Moisture in Spent Clay	3.96	1.72	----	3.15	0.58	2.62	3.13	1.70
Weight Oil in Spent Clay	153.7	250.0	----	116.0	408.4	150.9	415.4	230.2
Percent Shrinkage of Compound in Clay	0.769	1.250	----	0.580	2.042	0.755	2.077	1.151
Relative Order of Flavor of Comp'd.	1	5	2	3	6	7	8	4
Relative Densities of Clays	0.8996	0.7536	0.5704	1.0724	0.6316	1.0496	0.6144	0.7264

E) DEODORIZATION

The last step in the actual processing of oils is the elimination of objectional odors and flavors and is known as deodorization. Virgin peanut oil and olive oil are the only ones not subjected to this process. Before the development of deodorization the vegetable oils found use in burning and lubricating compounds only and it has been largely in the last twenty years that the edible side of the industry has been developed.

Deodorization was first attempted by blowing steam thru the hot, refined and bleached oil in open tanks. From this beginning the history of deodorizing is one of gradual improvement thru the addition of better auxiliary equipment and the development of better control methods. It is interesting to trace this development from the open tanks using wet steam to the high vacuum, superheated steam equipment of today. This development occurred in the following steps.

1. Open Tanks - Wet Steam - Low Temperatures.
2. Open Tanks - Dry Steam - Higher Temperatures.
3. Covered Tanks- Vapor Pipe - Slightly superheated steam.
4. Covered Tanks- Fan in Vapor Pipe giving 2" Vacuum.
5. Specially Designed Closed Tank - Barometric Condenser - 12-15" Vacuum - Superheated Steam.
6. Specially Designed Closed Tank - Barometric Condenser - Vacuum Pump - 20 - 24" Vacuum.
7. Improved Closed Tank - Barometric Condenser - Surface Condenser - Vacuum Pump - 27-28" Vacuum - Superheated Steam.
8. Improved Closed Tank - Barometric Condenser - Surface Condenser - Vacuum Pump - 27-28" Vacuum - Superheater for Oil with Recirculation.

There are in use at present two general types of



deodorizing plants, the non-circulating and the circulating types. Figures 4 and 5 illustrate the equipment and arrangement of apparatus for these plants.

#### THE NON CIRCULATING TYPE

In this type of plant the oil remains in the tank during the entire process of deodorization. The general procedure for operating a plant of this type is listed below.

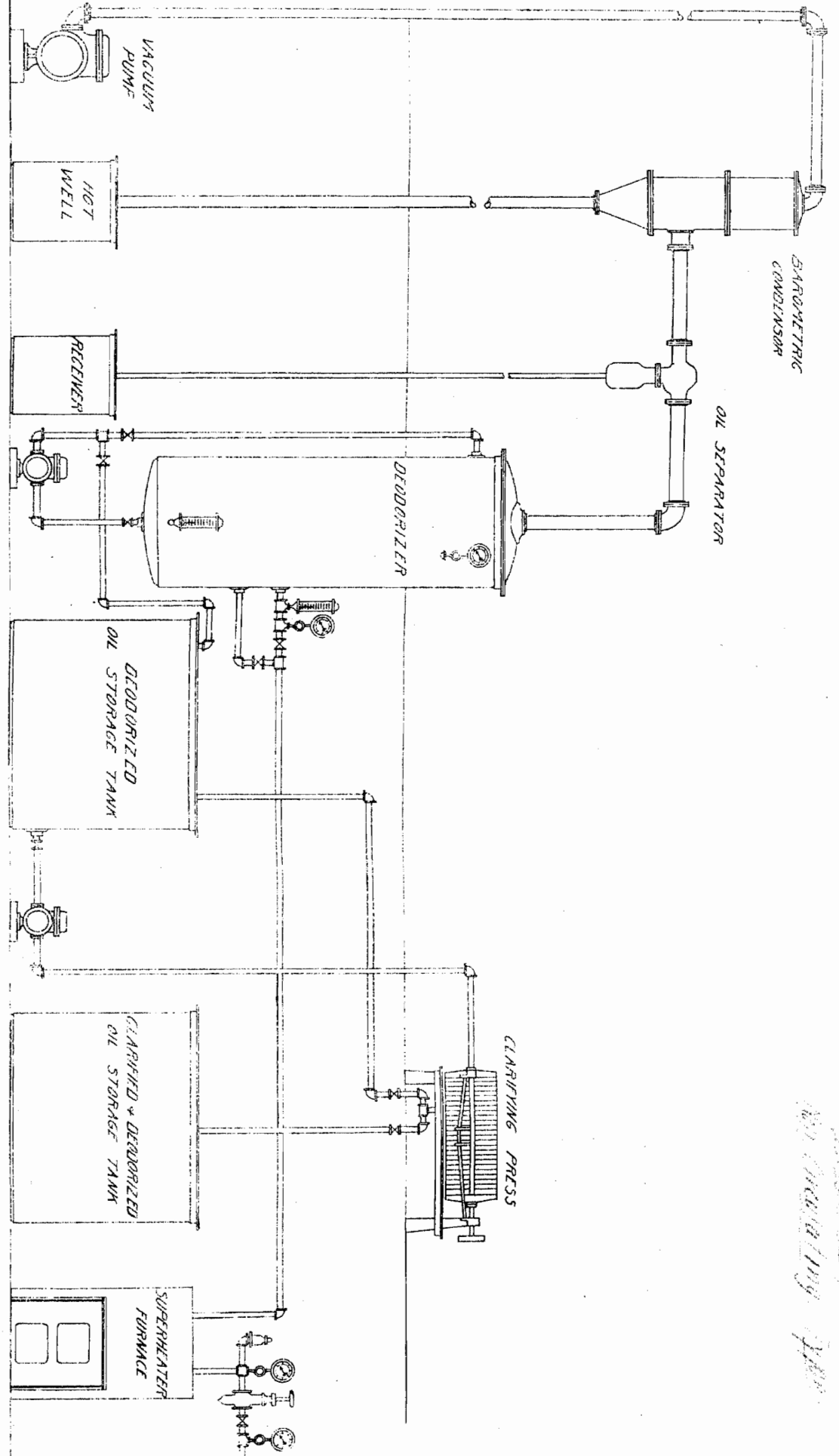
1. Charge tank with hot bleached oil to within  $1/3$  distance to the top.
2. Raise full vacuum on tank and heat by means of closed coil to  $250^{\circ}$  F with small amount of superheated steam on open coil.
3. Turn on open coil till oil splashes up to within about two feet of top of tank.
4. Cook. 4 to 8 hours with superheated steam depending on oil being processed. Keep temperatures within range given below.
 

Vegetable Compound	-	350 - 375 $^{\circ}$ F
Animal Compound	-	300 - 325 $^{\circ}$ F
Cotton Oil	-	350 - 375 $^{\circ}$ F
Corn Oil	-	325 - 350 $^{\circ}$ F
Peanut Oil	-	340 - 360 $^{\circ}$ F
Soya Bean Oil	-	350 - 370 $^{\circ}$ F
5. Turn off both open and closed coils, leaving vacuum on tank. Pump hot oil out of tank against vacuum thru cooling coils. Oil should be cooled to 120 - 130 $^{\circ}$  F before coming in contact with air.

#### THE CIRCULATING TYPE

This type of deodorizer was developed by David Wesson

*Figure 11  
 Deodorizing Plant  
 No. 1056*



**OIL DEODORIZING PLANT**

WURSTER & SANGER  
 CHEMICAL ENGINEERS

**1056**



of the Southern Cotton Oil Company. It has two advantages over the non-circulating type, i.e. it permits the use of higher temperatures and shows considerable economy in the use of steam. Steam consumption in deodorizing is the important cost item, the average steam-oil ratios of three types of plants being given below.

- |  |   |                                   |
|--|---|-----------------------------------|
| a) Non Circulating Plant<br>Barometric Condenser only        | ) | 3/4 to 1 pound steam per lb. oil. |
| b) Non Circulating Plant<br>Barometric and Surface Condenser | ) | 1/2 to 3/4 lb. steam per lb. oil. |
| c) Circulating Plant<br>Coal Fired Superheater               | ) | 1/3 to 1/2 lb. steam per lb. oil. |

In general the details of operation of this plant are quite similar to those already described for the non-circulating type. The main difference is the circulation of the oil thru the oil superheater during the actual deodorizing time. By this means direct heating of the oil is accomplished and a consequent saving in fuel realized. The following points should be carefully observed when operating a circulating plant.

- a) Start circulation of oil thru oil superheater before lighting superheater fire.
- b) Oil temperatures should approximate the following schedule.

<u>Time</u>	<u>Temp. of Oil</u>	
	<u>From Superheater</u>	<u>In Tank</u>
Start	Fire Lighted	200° F
1/2 Hr.	275° F	230
1st Hr.	360	250
2nd Hr.	420	300
3rd Hr.	475	350
4th Hr.	475	375
5th Hr.	410 Fire drawn	375
Finish	375	370

- c) At the finish the oil superheater coils should be thoroughly blown out into the deodorizing tank.

#### RESULTS OF DEODORIZATION

The deodorization process as practiced today is a highly specialized type of vacuum, steam distillation. Only a very small amount of true distillate is obtained; seldom over 1/10 of 1 percent. An average of 5/10 of 1 percent of oil is carried over by entrainment and in most plants recovered for soap oil. The bodies eliminated in deodorization belong mainly to the terpene group although some Free Fatty Acids and Fatty Acid Esters are distilled off. The varying flavors and odors met with in different crude oils are undoubtedly due to varying mixtures of terpenes and esters present. The problem of identifying the individual terpenes and esters in vegetable oil distillate is an extremely complicated one, and has received only casual attention by the research organizations of the industry.

The degree of deodorizing is determined approximately by tasting, the oil taster grading the oils by the presence or absence of certain flavors which he has learned to distinguish thru experience. The following grades are recognized.

Raw	- Seedy or Musty.
Undercooked	- Slight, distinctive flavor.
Neutral	- Bland, no flavor.
Overcooked	- Slight Acrid flavor.
Burnt	- Distinct scorched flavor.
Oxidized	- Distinct flavor akin to slight rancidity.
Rancid	- Distinct rancidity.
Hydrogenated	- Distinctive flavor of hydrogenated oils.

#### F) TYPICAL VEGETABLE OIL PRODUCTS

Table 9 gives complete specifications under which a number of popular vegetable oil products are manufactured.

TABLE 9.

<u>Product</u>	<u>Composition</u>	<u>F.F.A.</u>	<u>Color</u>	<u>Flavor</u>	<u>Uses</u>
Crisco	Hydrogenated C.S. Oil	0.10%	15y- 2.0r	Slight Hydrogenated	Shortening
Snowdrift	C.S.Oil and C.S. Stearine	0.10%	15y- 2.0r	Neutral	Shortening
Cottolene	C.S.Oil and Oleo-Stearine	0.10%	20y- 2.5r	Neutral	Shortening
Purity	C.S.Oil, Corn Oil and Beef Fat	0.10%	20y- 2.5r	Slight Beef Fat	Shortening
Wesson Oil	Winter Pressed C.S.Oil	0.05%	12y- 1.5r	Slight Overcooked	Salad
Union Pure	Winter Pressed C.S.Oil	0.10%	20y- 2.0r	Neutral	Salad
I X L	Summer C. S. Oil	0.10%	33y- 6.1 r	Neutral	Cooking
Mazola	Corn Oil	0.02%	20y- 4.0r	Slight Undercooked	Cooking & Salad
Amazo	Corn Oil	0.10%	20y- 3.5r	Slight Overcooked	Cooking & Salad
Acomine	Cococanut Oil	0.05%	5y- 1.0r	Neutral	Shortening
S.C.O. 88	C.S. Oil	0.05%	20y-2.0r	Neutral	Butter Oil
S.C.O. 77	C.S. Oil	0.05%	35y-9.0r	Neutral	Butter Oil