

Some Observations on the Edible Oil Industry of Europe

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THE writer had the opportunity, in the summer of 1930, of visiting a number of the larger oil plants in England and the continent. Some of the observations made will, I hope, be of interest to members of the Society.

The visitor from this country is at once impressed by the geographical concentration of the crushing plants in Europe and in England. The operations are conducted principally in a few deep water ports and the plants are generally of very large capacity. You can get some idea of this if you will imagine that we produced no cotton-seed and imported it from abroad and that our crushing operations were concentrated in a few ports such as New York, Norfolk, New Orleans and San Francisco. Plants in Europe are much larger than observed on this side and give one an impression of magnitude that is not gained from an inspection of mills in this country. The most modern equipment is used for unloading seed from the boats and transferring it to the mills. The construction and design of the more recent mills excels that of any that I have seen in this country. Careful consideration has been given to sanitation, and attractiveness of design has not been overlooked.

Observations were confined mainly to refining processes, but a few notes on the crushing and extraction of oil from seeds may be of interest. The types of machinery used vary considerably due to the great variety of seeds handled—viz., soya beans, peanuts, copra, palm kernels, sesame, sunflower, rape, etc. This is particularly true of the decorticating machinery. The type of hydraulic press used in this country is used there and is known as the Anglo-American press. It is used there for peanuts, cotton-seed and the smaller seeds. The curb press is used for palm kernels. In some plants a new type of automatic press is used for making a first pressing. The virgin oil produced is of very fine quality, but the cake contains so much oil that it

must be subsequently pressed in a hydraulic press or extracted by solvents. Extraction of oil by solvents has been developed to a high degree. The plants observed had been in operation for a number of years and seemed to work very satisfactorily. The advantage of the solvent plants as compared with pressing was admitted to be small. Saving in labor and the higher yield of oil was largely offset by the cost of solvent and left only a small margin in favor of extraction. The quality of extracted oils is considered to be equally as good as of pressed oils and in some cases better.

My interest was chiefly in refining equipment and processes. Vegetable oil refining, while conducted in much the same way as in this country, presents many new features, due largely to the greater varieties of oils handled. When I speak of American practice I have cotton-seed oil in mind. Other oils are, no doubt, handled on this side in much the same way as in Europe. The European refining practice might well be termed "wet" refining and our method of refining cotton-seed oil "dry" refining. Their object is to secure a liquid soapstock which will settle at the bottom of the refining tank with the clear oil above it and as small a layer of emulsion as possible between them. In refining cotton-seed oil in this country we admit the impossibility of securing a liquid soapstock and accept the principle that the soapstock produced will be an emulsion more or less solid in consistency. We seek to minimize the refining loss by controlling as carefully as possible temperature, agitation and other conditions of refining so as to produce as small an amount of soapstock or emulsion as possible. The refined cotton-seed oil produced is comparatively dry.

The European idea of wet refining is, I think, perfectly logical for the clean types of oils which they are handling. In most cases the crude oils are heated to 65° C.-85° C. and suffi-

cient caustic added to neutralize the free fatty acid. Very quickly a "break" occurs and the agitation is discontinued. Hot water is sprayed over the surface of the oil. This dissolves and carries down the soapstock which has been formed. The tank is allowed to stand preferably over-night. The liquid soapstock and emulsion layers are drawn off into separate receivers. The refined oil must be washed with hot water until all dissolved soap is removed. When this is accomplished the oil is saturated with water.

This procedure naturally requires special equipment. Extra tank capacity is needed for washing the oil. It must be dried by heating and stirring vigorously under vacuum. Consequently a European refinery must contain a series of vacuum drying tanks. It is generally believed over there that there is an advantage in bleaching under vacuum; therefore, bleaching tanks are usually closed and provided with some form of equipment for producing vacuum.

A striking difference in general arrangement of refineries was noted in several plants. The central bay of the refinery was kept open. The equipment was installed on the various floors on each side of the open space. This made it possible to observe practically all the equipment in the refinery from any point around the opening. It also made it possible for heat and odors to escape quickly.

I had heard that the Europeans preferred small refining units. In many cases this was true, but in some of the more modern plants I found equipment quite as large as used in this country—refining tanks with a capacity of 30-35 metric tons and deodorizers handling 15-17 metric tons.

The European chemists have been actively engaged in research to improve refining methods. A review of some of their efforts will, I think, be of interest. The processes suggested can be discussed under two headings:

- (1) Deacidification.
- (2) Distillation.

Deacidification is the term used for process for reducing rather than eliminating free fatty acid before neutralizing. It is a logical line of thought for them to follow as large quantities

of oil are handled which contain relatively high percentages of free fatty acids. Obviously the reduction of the free fatty acid or its practical elimination would effect a large saving in refining loss.

Several of the processes designed for this purpose have been patented. I would like to discuss briefly the ones which I consider more interesting.

The process of the Naamlooze Vennotschap Ant, Jurgens (German Patent 238812, Dec. 6, 1924), has for its object the deacidification of crude vegetable or animal oils. The process is conducted in a closed column containing suitable filling material for breaking up the stream of oil and exposing a great surface. The column is connected in series with condensers and suitable vacuum pumps. The oil entering at the top of the column is preheated in the terms of the patent not to exceed 250° C. It percolates over the filling bodies and comes into intimate contact with the superheated steam or indifferent gas which is admitted at the bottom. The steam or gas carries off the free fatty acid and the glyceride flows downward and is eventually pumped out of the column after having passed through coolers. In the patent application an example is given of the treatment of a lot of soya bean oil containing 70% F.F.A. The F.F.A. content was reduced to 6.7%. No description was given of the result which might be secured when handling a crude oil of, say, 7% F.F.A. The equipment and process is similar to that used in many French refineries for deodorization.

Another interesting process is that patented by R. Craig and C. E. L. Shawfield (assigned to Lever Bros., Ltd., English patent 242316, May 9, 1924). The apparatus covered can be used both for deacidification and deodorization. The main feature of it is a horizontal cylinder made of an alloy containing 90% aluminum and 10% copper. It is provided with a system of baffles which greatly lengthen the distance covered by the oil in flowing from one end to the other. A manifold supplies steam to every part of the path covered by the oil. The cylinder is heated by a series of gas jets placed beneath it in a suitable setting. Oil to be treated is first filtered with Fuller's earth at 95° C. It is then

passed through the apparatus, where it is heated to a temperature of 245-285° C. and superheated steam is supplied to the distributor previously mentioned. A high vacuum is maintained. Most of the free fatty acid and much of the flavor and color are removed. It is claimed that the use of caustic soda is not necessary for producing an oil of high quality, but this is doubtful. The apparatus is also used for the deodorization of oils which have previously been neutralized with caustic soda, particularly such oils as cotton-seed, which cannot be deacidified in the same way as the cleaner oils, such as cocoanut, palm, peanut and soya.

Another interesting process for deacidification is that of A. Von Sigmond (German patent 410170, May 15, 1918). This closely resembles the ordinary deodorizing process. Oil to be deacidified is treated in a closed vessel under a high vacuum at a temperature of 90-150° C.

The last process of this kind which I wish to describe is the Wecker Process, which is intended for both deacidification and distillation. It was the most interesting and promising of the processes studied, and for that reason I wish to describe it at some length. I had the opportunity of observing the process in successful operation at the plant of Messrs. Carl Hagenbucher & Sons, Heilbronn, Wurtemberg. The inventor, Dr. Ernst Wecker, is manager of this company. The patents have been transferred to the Veredelungsgesellschaft für Oele und Fette, Frankfurt-a-Main, which is associated with the I. G. Farbenindustrie. The patent (German patent 397332, March 21, 1923) was first published in 1924. The apparatus is adapted for both deacidification of oils and distillation of fatty acids, and operates continuously. For instance, it can be used for deacidifying oil, containing, say, 6-7% F. F. A. and reducing it to .5% F. F. A. or less, if desired. It can also be used for the distillation of split acids, containing, say, 96% F. F. A. For obvious reasons the writer hasn't the permission to discuss the details of the apparatus. It is sufficient to say that in spite of the high temperatures employed, no pyrogene decomposition occurs. In respect to heat economy, the apparatus is admirably worked out, effective

use having been made of heat interchangers and of the counter-flow principle.

The apparatus was constructed for handling about 1 ton per 1 hour per unit. Five to six minutes is required from the time the oil first enters the apparatus until it leaves it. The time varies with the amount of deacidification to be accomplished. With oils containing 20% or more of free fatty acid, the time required for treatment is greater and the capacity of the apparatus consequently reduced. The operation of the apparatus is simple, the controlling instruments can be observed from one point. One man can supervise several units.

The operation is much the same, whether the object is (a) deacidification or (b) distillation. The material to be treated is passed through continuously. In the case of oils to be deacidified, the free fatty acids are distilled off and condensed. The deacidified oil passes through the apparatus into coolers and then to storage. When the apparatus is used for distillation, most of the material is distilled off and only the residue of neutral oil and impurities passes through the apparatus to the cooler. This is sometimes split again and redistilled. The distilling apparatus is maintained under a high vacuum. Great stress is laid in the patent on the advantage of using wet steam for distilling the fatty acids instead of dry or superheated steam. It is maintained that distillation is promoted by the action of the fine droplets of water being vaporized in contact with the free fatty acids.

There appears to be no loss through decomposition. Very little neutral oil is carried over with the free fatty acid. The distilled fatty acids were of excellent color. The apparatus when used for distillation is far superior to the pot stills employed in this country.

Careful observations were made on a series of tests in which the Wecker apparatus was used for deacidifying peanut oil. The crude oil contained 5.5% free fatty acid. This was reduced to .5%. At the same time the oil was practically deodorized and most of the color removed. The treated oil left the Wecker apparatus at a temperature of 85° C. It was transferred to a refining tank and neutralized without further cooling. It was handled in the man-

ner previously described for wet refining. The refining loss was 1%. The subsequent work of bleaching and deodorizing was rendered very easy by the treatment in the Wecker apparatus. Dr. Wecker recommends the use of caustic soda for complete neutralization and also as a means of purification for which no substitute has been found. I think that most of us will agree with him that caustic soda is essential in the treatment of most oils.

I consider the Wecker process as a marked

There is much yet to be learned about the subject before a perfectly satisfactory product can be turned out.

A variety of designs for deodorizing plants was observed. The more recent installations make use of the booster for producing high vacuum. Most of you are familiar with the Lurgi system of deodorizing which has been extensively advertised in this country.

Continuous deodorizing is conducted in several ways. The Craig process has been dis-

CONSUMPTION FOR 1,000 KG. RAW OIL

	Wages for 1-4 App.	Consumption of Heat in kg. Coke	Steam	Power	Cooling Water	Residue F.F.A. After Treatment
Palm kernel, 5.8%						
F.F.A.	1 work hour	30 kg	120 kg	6 KW h ^x	6 m ³	0.5 %
Groundnut, 3.9%						
F.F.A.	1 work hour	35 kg	160 kg	6 KW h ^x	6 m ³	0.4 %
Groundnut, 10%						
F.F.A.	1 work hour	35 kg	170 kg	6 KW h ^x	6 m ³	0.5 %
Sesame, 3.2%						
F.F.A.	1 work hour	35 kg	160 kg	6 KW h ^x	6 m ³	0.35%
Soyabean, 1.1%						
F.F.A.	1 work hour	35 kg	150 kg	6 KW h ^x	6 m ³	0.2 %

^xIncluding power needed for heating purposes.

These figures are completed by the following data regarding yield and quality of the products obtained.

EVERY 100 KG. RAW OIL YIELDS:

	Deacidi- fied Oil	Content of F.F.A.	Output of F.F.A.	Content of Neutral Oil in Fatty Ac.	Total Waste	%
Palm kernel, 5.8% F.F.A.....	94.30 kg	0.50%	5.80 kg	8-9%	No	100
Groundnut, 3.9% F.F.A.....	96.30 kg	0.40%	3.70 kg	4-5%	No	100
Groundnut, 10% F.F.A.....	90.20 kg	0.50%	9.80 kg	3-4%	No	100
Sesame, 3.2% F.F.A.....	97.00 kg	0.35%	3.00 kg	4-5%	No	100
Soyabean, 1.1% F.F.A.....	98.90 kg	0.20%	1.10 kg	12%	No	100

advancement in knowledge of processing oils and fatty acids.

The following service data cover the yield and consumption of materials per 1,000 kg. raw neutral oil. This is submitted through the courtesy and with the permission of the Veredelung:

The subject of selective hydrogenation has not received the same consideration in Europe as it has in this country. Hydrogenated fats are used principally in margarine. There does not seem to be the same need in margarines as in vegetable shortenings for selectively hydrogenated products.

Refiners of whale oil have developed special processes for treating this obstinate material.

cussed. A number of French refineries work along the lines of the Naamlooze process previously mentioned.

The Lurgi Society has also developed an improved pot still which makes use of the booster for securing high vacuum and thereby lowering the temperature of distillation. It also introduces a new system of securing the high temperature necessary for this type of still. In place of the direct fire system generally used the Lurgi Society has substituted high pressure steam coils. Steam is generated at the ordinary pressure—viz., about 7 atmospheres—and then raised to 30 atmospheres by the use of an electrically driven compressor. The temperature of

the steam rises with the pressure, and it is possible to raise the temperature within the still to 220° C. This is a novel and clever application of thermo-dynamics to the problem of securing high temperatures.

Several of the research laboratories visited were splendidly equipped, not only for carrying out chemical research, but also semi-commercial investigations. Every possible type of plant equipment built on a small scale had been provided.

Pine Nut Oil*

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PINE nut oil from *Pinus monophylla* is described by *Blasdale*¹ as "a brown drying oil with an unpleasant odor and taste." It is classed by *Lewkowitsch*² as a drying oil, although the iodine number is but 101.3. This figure has been confirmed by *Adams and Holmes*³ and by the writer. On the other hand, quince seed oil (from *Cydonia Vulgaris*) with an iodine number of 113-120 is classed as a non-drying oil. Here was an opportunity to investigate an anomalous situation.

A quantity of the nuts or seeds was obtained through the courtesy of the Forest Service in Nevada. These were ground, extracted in *Drechsel* extractors with ether, and about 21 per cent of a light yellow oil of a balsamic taste and odor was obtained. This oil showed the following characteristics:

Drying quality: Submitted to the usual Archbutt test—(drying on a glass plate at 50°) it was not dry after 30 days' exposure. The oil about the stopper of the container did not dry after months of exposure as much as cottonseed; it resembled olive more closely in this regard. This test would alone decide the question.

Table I. *Chemical & Physical Characteristics*

Specific gravity 20° C.	0.911
Acid value Mg KOH per gram oil. . .	5.4
Reichert-Meissl value.	0.89
Saponification value.	183.9
Unsaponifiable matter, per cent. . .	1.95

¹J. Am. Chem. Soc., 17, 935 (1895). ²"Analysis of Oils, Fats and Waxes," *Ind. & Eng. Chem.*, 5, 285 (1913).

*Original Contribution, received July 27, 1932.

It would not be possible in an article of this length to present a comprehensive description of European refining practice so I have attempted to point out some of the more interesting points of difference between their practice and our own and also to present a few of the more promising ideas which have been advanced for improving methods of refining. I came away from Europe with a high respect for the ability of their oil chemists and a keen appreciation of their liberality in affording opportunities for observation.

Iodine number Hanus.	102.1
Thiocyanogen-iodine number.	78.8
Saturated acids corrected per cent	8.1
Unsaturated acids corrected per cent	84.5
Iodine number of unsaturated acids	122.6
Glycerine per cent	8.9

The fatty acids were determined by saponification of the oil with an excess of a known quantity of N/2 alcoholic potassium hydroxide: the excess of hydroxide was neutralized with a known quantity of N/2 hydrochloric acid. The solution was evaporated to dryness and the residue weighed. This contained the potassium soaps, a known quantity of potassium chloride, glycerine and unsaponifiable matter. The latter was extracted with ether, which, on being evaporated, left a heavy terpene- or sesquiterpene-like oil. The glycerine was determined by the acetin method on a fresh sample. Knowing the amount of potassium in the soap, the fatty acids combined with it could be determined: thus the fatty acids, glycerine and unsaponifiable matter were determined.

The saturated fatty acids were determined by the well-known lead-salt-ether method. Their methyl esters were prepared and fractionated, using 37.5 grams. From these three fractions the following percentages were obtained:

Saturated Acids: myristic C ₁₄	5.0 per cent
palmitic C ₁₆	2.7 per cent
stearic C ₁₈	0.4 per cent

No evidence of a low boiling methyl ester was found, hence lauric and lower acids are absent.