Iwanoff[9] showed that the amount of moisture in the atmosphere under which a flax plant is raised influences the unsaturation of the oil deposited during growth. Linseed oil obtained from flaxseed grown under humid conditions had iodine numbers of 167.8 to 170.6, while under arid conditions of growth, the same variety gave an oil having an iodine number of 181.8 to 187.0. Considering the above factors, unsaturation in linseed oil is influenced by environment for the percentage of each of the three unsaturated fatty acids differed for the same variety of flaxseed grown in different geographical localities.

It is naturally expected that of two oils, one of lower and the other of higher iodine number, the oil having the higher iodine number would contain a lower percentage of saturated fatty acids. However the oil extracted from the Bison variety of low iodine number, grown in the same vicinity with the Abyssinian Yellow variety contained a smaller percentage of saturated fatty acids than the oil from the latter. The percentage of saturated fatty acids in the Bison oil was more nearly uniform and did not vary as much as in the oil obtained from the Abyssinian Yellow seed.

SUMMARY

All seed oils of the Bison flax variety contained a higher percentage of oleic acids and a lower percentage of linolenic acids than the Abyssinian Yellow flax variety. Abyssinian Yellow seeds yielded oils which contained a relatively low percentage of oleic acid and a higher per cent of linolenic acid than the Bison variety grown in the same locality.

The amount of unsaturation in an oil, or its iodine number, appears to be a varietal characteristic, and the components of the oil are blended by nature to give a certain degree of unsaturation which can be varied by growing the flax plants under different environmental conditions.

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EDIBLE OIL DEODORIZING EQUIPMENT AND METHODS:

A Short Historical Sketch

By ALAN PORTER LEE and WALTER G. KING, JR.*

Abstract

Edible oil deodorizing equipment and processes have been developed as a result of advances in hot-pressing and extraction of oil-bearing materials. Early methods included masking of odors by means of aromatics, washing out of odors and neutralization or destruction of odors by means of various chemical treatments.

Commercial deodorization methods involve steam distillation in vacuo. Processes have been developed independently in several European countries and in the United States. Most efficient equipment uses low absolute pressures and high temperatures in equipment constructed of corrosion-resistant metals and supplied with suitable recording instruments.

Current trend is toward continuous processes with automatic control.

THE deodorizing of oils for edible purposes was an unknown, even an unnecessary art, until comparatively recent years. The ancients were acquainted with olive oil and undoubtedly with coconut oil, but in early practice both of these oils were expressed by means of cold-pressing processes, from fresh raw materials and both therefore were entirely suitable for edible uses without further processing of any sort.

As the world progressed, new methods of pressing developed and it was discovered that cooking or heating oil-bearing raw materials before pressing, results in marked increase in the yield of oil. Further developments in agronomic economics brought about storage of oil-containing seed or fruit from one harvest period until the arrival of the next, as well as shipment of the materials over great distances before expression of the oil. All these factors contributed to reduction in the average quality of the expressed oil from the standpoint of edibility. Both storage and cooking before pressing cause increase of free fatty acid in the oil. The latter operation, particularly, may account for the presence of small amounts of aldehydes, ketones and other bodies which impair the flavor of the freshly-expressed oil, but which are not soluble in the oil at the low temperatures employed in cold-pressing. The same impurities result from slight decomposition of the seed and of its contained oil during storage and shipment.

In a cold-pressed oil are to be found minute quantities of phosphatides, vitamines and other unsaponifiable organic materials which appear to act as inhibitors of or buffers against fermentative decomposition of the oil with attendant increase of free fatty acids and subsequent onset of rancidity. In hot-pressed oils, on the other hand, the beneficent effect of such substances apparently is decreased, either by the presence of other substances which accelerate fermentative action or by the presence of increased volume of the same phosphatides, which, although acting protectively when present in small amounts, may themselves become subject to fermentation when their relative volume is greater, as in hot-pressed oils. It is possible, also, that certain protective vitamines present in cold-pressed oils, may be destroyed or may lose their protective capacity when subjected to the temperatures incidental to the hot-pressing operation.

Hot-pressing of oils results also in the presence in the oil of larger amounts of the natural coloring matters of the oil-bearing fruit, the solubility of such coloring matters in the oil during the pressing operation generally being directly proportional to temperature. The pres-

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ence of increased amounts of coloring matters tends to modify the flavor of the oil from the bland, smooth flavor of a cold-pressed oil toward the sharper, more acrid flavor of the hot-pressed type. The development of the hot-press made possible also the utilization of various new materials as sources of edible oils, such as the cottonseed, flaxseed, rapeseed, soybean and maize germ. Cold-pressed oils from such sources have in some instances enjoyed limited use for edible purposes, but the great commercial exploitation of such oils is attributable mainly to the development of hot pressing methods.

Solvent-extraction methods of obtaining oils from seeds were developed in the same era which saw the rapid growth of hot-pressing methods. Oils produced by solvent-extraction require the same deodorizing treatment as hot-pressed oils. The characteristics of hot-pressed and solvent-extracted oils created demand for processes and methods to render these oils truly edible. Among the first means in general use were found two methods which were diametrically opposed in principle. The first[1] consisted of washing the oil with a moderately strong solution of sulfuric acid (in small percentage), washing out all traces of the acid, then filtering the oil to remove the albuminous and other materials which had been coagulated or charred by action of the acid. Earliest date of use of this process is unknown, but records exist of its use in England by Gower in 1790. The second refining method[2] was the forerunner of those most in use at the present time and consisted of neutralization of free fatty acids by means of the hydroxides or carbonates of alkalies or alkaline earths. This method was used in France, England and Germany as early as 1855[3] and was applied to cottonseed oil in the United States in the late seventies or early eighties of the nineteenth century.

In this country, alkaline neutralization of the free acids made possible the adaptation of cottonseed oil to edible uses, principally because the alkali, in addition to removing the free acids, acted as a strong bleach, removing a large part of the dark red coloring matter and leaving the oil with a straw-yellow color. This oil was suitable for admixture with olive oil or with lard and these were the principal uses of cotton oil for several years after first discovery of its value as an edible oil in the United States.

In European countries, knowledge of the possibility of improvement of the flavor of oils by means of blowing a current of steam through them seems to have antedated similar knowledge in the United States by a considerable period of time. France, Germany and Belgium were leaders in the development of the deodorizing process, which was possibly first suggested by methods of steam distillation of essential oils or by the blowing of oils with air for polymerization, or by both these processes. When steam deodorization was adopted in the United States, the great improvement in the flavor of cottonseed oil which resulted was undoubtedly responsible for the remarkable growth in favor of this oil for edible purposes. Once started in the United States, progress in deodorizing methods in this country for a time outstripped that in Europe, probably because the American taste demanded a bland, sweet oil, while the European preferred oil with some vestiges of the natural flavor remaining.

Growth of the margarine trade, however, which has been much more rapid in Europe than in America, resulted in the development of improved deodorizing methods abroad, until, at the present day it may be said justly, that the best European procedure compares very favorably with the leading processes of this country. As in many other process operations, American tendencies in deodorizing have been toward large-scale equipment, while the Europeans have in the past devoted more attention to refinements of design and operation as applied to equipment of smaller capacity. It is only in very recent years, since Americans have demonstrated the possibility of applying the same refinement of design and control of operation to large-scale apparatus, that some of the Europeans have followed suit and now offer very effective equipment in the larger capacities.

The basic mechanism of the deodorizing process is simple, consisting of fractional distillation from the oil of those bodies (present in almost infinitesimal amounts) which constitute flavoring and odorous substances. The ideally pure fatty oil would resemble nothing quite so much as a white Russian mineral oil. Some years ago one of the authors of this paper was given a sample of such mineral oil as his first sample of an unknown fatty oil. The material had a bland, slightly nutty flavor, but iodine number and saponification number, each of zero, quickly revealed the hoax which was being perpetrated upon many unsuspecting Canadian purchasers of so-called "cooking oil." The object of the deodorizing step in a refining process is to distill away the minute traces of odorous and flavoring substances to the greatest possible extent, with minimum attendant injury to the oil during treatment. The substances to be removed are small quantities of free fatty acid and of various ketonic and aldehydic compounds identified. Lewkowitsch[4] states that methylnonyl ketone and methylheptyl ketone have been isolated from the deodorizing distillate of coconut oil.

Some early attempts were made to deodorize oils by dry distillation at atmospheric pressure.[5] The addition of injection of steam to facilitate the distillation through reduction of the partial pressure of the substances to be distilled offers such an obvious advantage that it was quickly resorted to wherever deodorizing was undertaken. In addition to its primary purpose, the steam served a second, that of preventing oxidative decomposition of the oil, which it accomplished by means of its exclusion of atmospheric air.

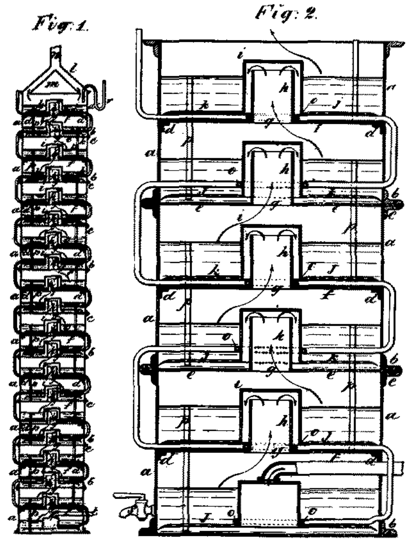
From the basic requirements we can construct a hypothetical primitive deodorizing equipment, which will consist of an open cylindrical tank equipped with means of heating and with, at its bottom, a steam nozzle or perforated steam pipe connected with a steam boiler. Such tanks have been used for the renovation of rancid butter, a process analogous to deodorization of oils.

Advancing from the primitive deodorizer, we find that inventive trends have followed comparatively few avenues of progress. Superheating of the steam; addition of vacuum apparatus; the use of higher and still higher temperatures; of lower and still lower absolute pressures; introduction of baffles of various designs to secure intimate contact between steam and oil; variations in design of heating apparatus, of entrainment breakers, of catchalls, of steam distributors and of vacuum apparatus; construction of the equipment from various inert or noncorrosive metals and the development of continuous apparatus; will cover in a general way all deodorizing developments to date. In addition there have been some attempts to deodorize by addition of odor-killing or masking substances or by washing out the odors and flavors through the use of various solvents. There were also early attempts to adsorb odors in some one of numerous adsorbents or to destroy them through the action of various chemicals. There has been some use of inert gases other than steam as deodorizing agents. None of these undertakings has achieved any general commercial success, steam distillation in vacuo being the process used practically universally today.

Early deodorizing attempts are typified by the following examples: A United States patent of 1854[6] proposed to purify oil by shaking it with one-fourth its volume of alcohol, then settling. Masking of odors has been attempted with ethyl nitrite, oil of mirbane, coumarin, menthol, thymol, etc. For adsorption of odors, such substances as charcoal, elm-bark,[7] clays and clay composition have been proposed. Washing materials for removal of odors have included borax, tartaric acid, alum, manganic chloride and many others. Reduction of aldehydes and ketones by boiling with sodium bisulfite is claimed to furnish effective deodorization of rancid oils.[8]

In reviewing progress in modern deodorizing methods we can conveniently consider separately the work done in various countries, starting with the outstanding European countries and completing the survey with the United States. Dates given are in many cases approximate, in others accurate.

1. France. Deodorization of fatty oils in France apparently was started near the middle of the nineteenth century. Cassgrand[9] demonstrated in 1854 the cleansing effect of a current of steam on fats. French Patent No. 15394 of Dec. 17, 1855, to Bardies, Moutié et Mme. Decoudun, covered distillation of fatty acids under vacuum, likewise steam distillation of odors from fats in vacuo. Wurtz and Wilm[10], in 1873 treated colza oil with a current of steam at 116-120° C. Among the leading refiners of France was the firm of Rocca, Tassy et DeRoux of Marseilles, who refined coconut oil, palm and palm-kernel oils and arachis oil from Africa. As early as 1906 this firm was deodorizing coconut oil in aluminum deodorizing vessels, using superheated steam. The deodorizing was performed under atmospheric pressure. By arrangement with the Marseilles firm their process was established, with imported equipment, in the United State at Portland, Oregon, prior to 1910. Similar equipment was later adopted by a large coconut butter company at Chicago. Emilien Rocca of the Marseilles firm patented a continuous deodorizing apparatus in Germany in 1900, in the United States in 1902.[11] Rocca's apparatus was described in his patents in two forms, the first following the well-known rectification column-still design and the second the principle of the diffusion battery used in beet sugar mills. In both forms the principles of counter-current flow and intimate contact between the oil and the steam were employed. Pressures in Rocca's apparatus were atmospheric.



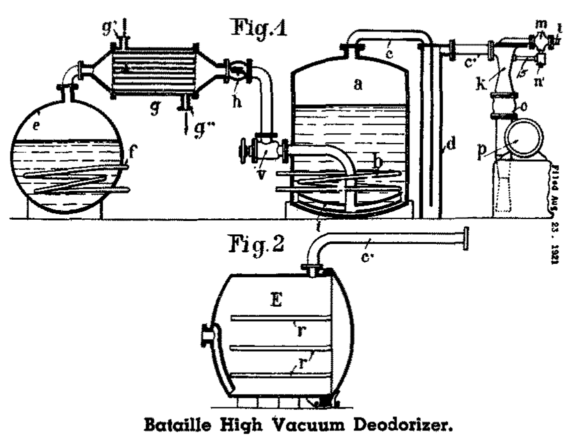
Rocca Continuous Deodorizing Plant.

The Societe Anonyme des Parfums Naturels de Cannes in 1894 patented[12] a process of treating oil with steam without permitting condensation. The oil was first mixed with a very volatile solvent, then sprayed into a vessel where it was treated with steam. After deodorization the oil was cooled in an atmosphere of inert gas.

As early as 1891, E. Bataille of Paris marketed deodorizing equipment designed to operate under pressure lower than atmospheric. Features of Bataille's equipment included superheating of low-pressure steam by means of shell-and-tube exchange with high-pressure steam; a rotary condenser for the vacuum equipment, in other words a single high-speed centrifugal-type pump for removal of condensing water, non-condensables and condensate. Bataille's deodorizing apparatus was generally tin-lined, giving effective corrosion resistance at the low temperature of its operation. Although others had previously employed the steam-jet ejector for production of vacuum in various types of stills and evaporators, Bataille appears to have been the first to apply the booster ejector to deodorizing in conjunction with barometric or rotary jet condensers. His patents (France 1914, U. S. 1921)[13] describe the production by this means of absolute pressures lower than the vapor pressure corresponding to the temperature of the condensing water. This type of equipment is now employed on deodorizers by the majority of the leading oil refiners of Europe and America. Effective absolute pressures of from 3 to 8 millimeters of mercury are thus obtainable in the deodorizing vessel under conditions of actual operation.

2. Germany. Early experimentation in Germany in the field of edible oil deodorization with steam was not very fertile. In 1893 the Fabriques de produits chimiques de Thann et Mulhouse (Upper Alsace) patented[14] in Germany the treatment of oils with steam in vacuo or in an atmosphere of inert gas, for deodorizing. This process is reputed to have produced for years the best grades of coconut butter in Europe.

At about the time of the world war, the standard German deodorizing equipment was modeled closely along the lines of the French-built equipment of Bataille and consisted of a steam-jacketed tin-lined vessel with one or two gross kilos charge capacity, equipped with barometric



Bataille High Vacuum Deodorizer.

or surface condenser and reciprocating vacuum pump. The Germans were probably the first to add a vacuum cooling tank, also tin-lined, generally equipped with water-jacket and agitator. German practice included also fire-heated steam superheaters. German edible oil of this period was not ranked exceptionally high in quality, particularly in comparison with the best American or French oils.

During the world war, when Germany was called upon to make her fat economy entirely internally maintained, German deodorizing methods and equipment advanced rapidly. Gensecke[15] and Brucke of Metallurgi Gesellschaft developed the famous Lurgi deodorizing apparatus. This equipment follows Bataille in the use of the booster steam jet or thermo-compressor for the purpose of obtaining low absolute pressures, but goes a step beyond Bataille in using a two-vessel system and passing the expanded steam from the thermo-compressor through a preliminary deodorizing tank somewhat in the manner of Rocca's diffusion battery system. The Lurgi system has been extensively adopted in Europe, is available in large capacities and is reputed to produce edible oil of quality equally as good as any produced in the United States. Recently patented improvements in the Lurgi system include rather elaborate internal baffle construction designed to promote thorough admixture of the steam with the oil. The Lurgi system describes the employment of very high-pressure steam from a special boiler or a steam compressor, as the means of obtaining the high temperature necessary for modern deodorizing.

Another modern German system is that of Wecker[16], which is a continuous process. The art of deodorization of oils in principle and practice is only a special division of that of the distillation of fatty acids. The Wecker process, controlled by the great "I. G.," German dyestuff and chemical manufacturers, was first described for removal of free fatty acids from neutral oil by distillation, but is applicable to the usual distillation of fatty acids or to the deodorization of oils. Wecker's plan of operation includes spraying water or other liquid into the oil or fatty acids to be treated, then passing the mixture into a heated reaction chamber where the volatile portions are distilled by injection of superheated steam under high vacuum. The process is continuous and the equipment modern. We do not know positively of Wecker plants devoted to deodorizing alone, but it is claimed that the equipment is capable of distilling a small percentage of free fatty acids from an oil and deodorizing the oil at the same time. The Wecker plants built for fatty acid distillation have made extensive use of cast aluminum for the reaction chamber, which is heated directly by oil or gas flame, and of rolled and drawn aluminum sheet and tubing for the condensation equipment.

The Friedrich Krupp Grüsonwerke (Krupps) have been offering deodorizing equipment for export, but it is believed that their designs are produced by the Lurgi organization, under a working agreement.

3. Belgium. At about the beginning of the present century, the Societe Anolyme des usines J. E. Bruyn in Termonde, Belgium, patented in France[17] a counter-current column apparatus for deodorization of oils in which the screen-plate principle was employed. The oil was fed at the top of the column, or below the first plate and descended as a rain of drops through the perforated plates, counter-current to a stream of superheated steam which was admitted through a perforated pipe at the bottom of the tower. Bruyn's apparatus was designed for atmospheric pressure operation, as was Rocca's. All of these tower-type continuous deodorizing equipments seem to have lost favor in Europe when vacuum processes were adopted.

4. Austria. A European deodorizing method, of interest chiefly because it was used in the United States for some years, was developed by E. Khuner und Sohne of Vienna, manufacturers of coconut butter. The Khuner process employed superheated steam at high temperatures in atmospheric pressure deodorizing vessels. The vessels were small, only about 1,100 pounds working capacity each, were jacketed and heated by hot mineral oil circulated through the jackets by means of thermosyphon action. Each pair of deodorizing vessels was equipped with a separately-fired steam superheater and with a mineral oil heater, each heater in a separate brick setting. Heating surface for steam and for mineral oil was in the form of plain smooth-surfaced helical coils. Steam off-takes from the vessels were small; were joined in a common header leading to a helical coil submerged in water. This resulted in some condensation of the effluent steam and consequent operation of the vessels at from ¼ lb. to ½ lb. below full atmospheric pressure. Deodorization required about ten hours, for even such small batches. When the deodorization was complete, steam was shut off and the oil forced by means of CO_2 pressure through a coil pipe cooler immersed in water.

This process was used for deodorization of coconut oil by India Refining Company of Philadelphia from about 1909 to 1919. The installation consisted of ten deodorizers, five separately fired steam superheaters and five independent mineral oil heaters, total output 27,500 pounds in 24 hours! Unlike most European apparatus of its time, the Khuner equipment was not tin-lined, probably because of the high temperatures employed in its operation.

5. England. Progress in deodorization in England has paralleled closely that of other European countries. Lake[18], in 1885, proposed a method of refining and deodorizing involving the combined action of superheated steam and various chemicals, alkalies and acids. In 1895, Morgans[19] proposed to blow air through oil previously emulsified with water, thus producing two layers, one of clear edible oil and the other of milky water containing the odorous and other elements removed from the oil by the treatment.

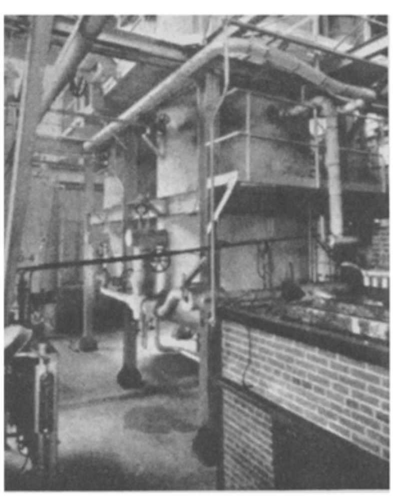
With the development, about the close of the last century, of such large margarine and coconut butter plants as Otto Monsted, Ltd., The Maypole Dairy Co., and Loders & Nucoline, Ltd., methods of deodorizing employing superheated steam and vacuum equipment were developed, particularly for coconut oil. Loders & Nucoline, Ltd., utilized a semi-continuous triple effect tower system, using low-level condensers and wet-vacuum pumps. The same company developed the use for deodorizing purposes of exhaust steam from prime movers, employing an elaborate system of oil separators for purification of the steam.

At about 1912 the advantages were being recognized more and more of noncorrosive equipment in production of oils of finer flavor and superior keeping qualities. Rocca in France had already used aluminum in atmospheric pressure type deodorizers and tin-coated vacuum vessels were being used in France and Germany. In this era the Aluminum Plant & Vessel Co. of England produced a complete vacuum deodorizing plant of aluminum construction, using boiler-pressure steam in aluminum coils in the deodorizers for oil heating, superheated steam spray coils for adjunctive steam and an aluminum vacuum cooler with aluminum water coils.

In recent years the Birmal Chemical Engineers of Birmingham, England, have produced an all-aluminum continuous fatty acid distillation plant somewhat similar in external appearance to the Wecker equipment. As noted above, such equipment in highly suitable for deodorizing purposes. At least one installation has been made in the United States.

6. United States. In this country deodorizing progress for a time seemed to lag behind that of Europe insofar as quality of product was concerned, but was always until recently far in advance as regards volume of material handled in a single operation.

The first important deodorization work in the United States was done by Henry Eckstein, at the time Superintendent of the N. K. Fairbank Co., of Chicago. Eckstein in 1891 demonstrated the great improvement in flavor of refined cottonseed oil which resulted from heating the oil to 325-350° F. and blowing a current of live steam through it. With tank capacities of from 10,000 to 30,000 pounds, the operation could be completed in from five to ten hours. Eckstein used no vacuum and did not superheat the blowing steam. His practice was followed by many large American cottonseed oil refiners for at least thirty years after its discovery and is probably in use today, over forty-five years later, in numerous South American and Central American plants. For



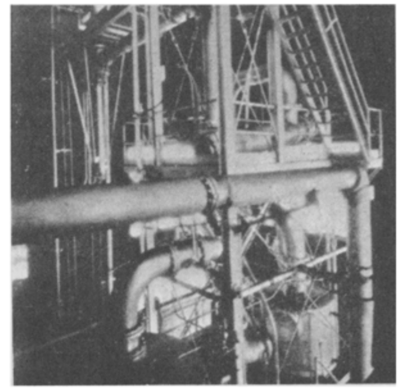
Interior View—Modern Deodorizing Plant.

the Fairbank Company, which later became a unit of the American Cotton Oil Company, many improvements in the Eckstein process were made by Boyce, Cluff, and others, including the addition of vacuum equipment and superheating of the steam.

The most successful American deodorizing process undoubtedly has been that of David Wesson, which was introduced by the Southern Cotton Oil Co. in 1900. Wesson and his company relied on keeping the process secret rather than on patents, so that details of the process are not generally known. It is believed that the Wesson process was one of the first to employ vacuum methods in the United States, if not the first. It may suffice to say that for many years the quality of Wesson's oil was unrivaled and that it is still a standard for edible oils throughout the world.

Until 1914 or 1915 the usual American equipment for deodorizing cottonseed oil consisted of a simple tank equipped with open and closed steam coils and with a chimney-like outlet in the top for exit of the steam and gases. The steam was seldom superheated. In a few instances large steam-driven wet-vacuum pumps were used in conjunction with low-level condensers and in at least one instance within the writer's knowledge, such a pump was used as a direct exhauster of superheated steam without intermediate condensation of any sort. One large Midwestern refiner employed deodorizing tanks of horizontal cylindrical construction with steam superheating and vacuum equipment. A prominent cotton oil company of northern Texas utilized vacuum and superheated steam type deodorizing equipment imported from Europe, including vacuum cooling tanks, and produced oil of exceptionally high quality.

Between 1914 and 1920 several large refiners adopted a method of deodorizing which involved circulating the oil through a tubular direct-fired heater and a vacuum tank while passing steam into the tank through a perforated coil or "sparger." This is known as the direct-heat system and is popular with many refiners. The problems of modern deodorizing are centered in three features: high temperatures; low absolute pressures; selection of materials of construction of greatest resistance to corrosion as a means to improvement in stability of the oils and fats deodorized.



Condensing Equipment Modern Fatty Acid Distillation Plant.

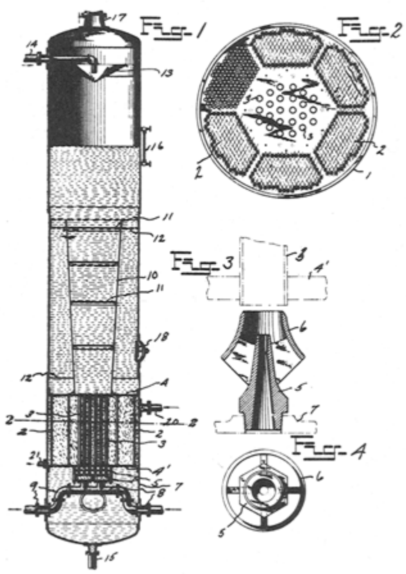
Many refiners have decided that the direct-heat vacuum system furnishes the most efficient solution to these problems, at any rate to the first two of them.

During the same war period above referred to, coconut oil refining gained considerable prominence in the United States and improvement in deodorizing methods was eagerly sought. In 1917 Allbright and Lee designed and installed at Milwaukee the first vacuum deodorizer of non-corrosive metal (aluminum) manufactured in America. During 1918 sheet aluminum was not available on account of the war and the same engineers installed several plants with tin-lined steel vacuum deodorizers, aluminum cooling coils and tin-lined cooling vessels. These were necessarily plants with vessels of small capacity. In 1919 sheet aluminum again became available and in that year and in 1920 several additional vacuum plants were built of that metal.

At about the same time Gibson built for the Southern Cotton Oil Company a complete deodorizing plant with vacuum tanks of glass-enameled steel. Heating was by circulation of externally heated mineral oil through steel jackets welded to the outside of the tanks. All interior fittings in the deodorizing tanks were of silver, gold and gold plate.

In 1920 the Bataille high-vacuum equipment was introduced into the United States, since which time most large refiners and many smaller ones have adopted the thermo-compressor or "booster" as a means of obtaining high vacuum in the deodorizing vessel. The reciprocating or rotary air pump for removal of noncondensables has been supplanted almost entirely by the two-stage steam-jet ejector with inter-cooler, either surface or barometric, serving the same purpose. Heating of the oil in deodorizing is accomplished by the direct-heat system above-mentioned, by circulation of externally-heated petroleum oil through a jacket on the deodorizing tank or by circulation of very high pressure steam from a special boiler through coils in the deodorizer, returning the condensate to the boiler. One large manufacturer circulates heated mineral oil through a series of coils in the deodorizing tank rather than through a jacket on the tank.

Cassidy[20] circulates oil in the vacuum deodorizing tank by jet ac-



Cassidy High Pressure Steam Deodorizing Equipment.

tion of the deodorizing steam. The oil circulates through tubes which are heated by contact of high pressure steam in a central drum of the deodorizing vessel.

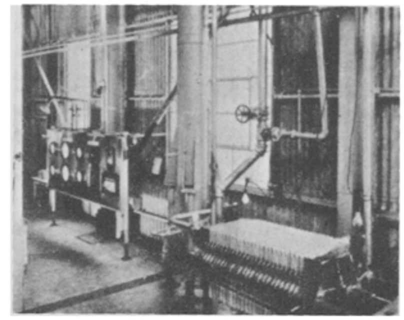
Cooling of oil after deodorizing is almost invariably accomplished in modern plants by means of double-tube or shell-and-tube heat exchangers. Some attempts have been made to utilize incoming oil as the cooling medium for outgoing finished oil, but in batch operations this procedure has not had marked success, probably because of the difficulty of closely controlling the time required for deodorization. Some manufacturers transfer the deodorized oil through a cooler and filter press to vacuum holding tanks in which it is stored under vacuum until needed for packing and shipping.

An interesting modern plant designed by Lee and King is believed to be the first in the world in which the oil contacts no metal other than 18/8 stainless steel while being heated, deodorized and cooled. Tank capacity is 25,000 pounds of oil per charge. The direct-heat system is used, with certain improvement modifications for which patent applications have been filed. Deodorizing vessels are stainless-clad, welded; pipelines, pumps, heating tubes, shell-and-tube cooler are all of solid 18/8 stainless steel construction. Pipelines are all designed with long-radius bends made to templates and assembled with Vanstone joints. The final filter press has plates cast of aluminum. Vacuum equipment includes surface condenser, barometric jet condenser, thermocompressor and two-stage steam-jet ejector air pump, maintaining absolute pressure of from 3 to 5 millimeters of mercury in the deodorizing vessels during operation.

Steam is superheated in a seamless low-carbon steel coil built into the same setting with the stainless steel oil heater, in which mineral oil is utilized as fuel. The deodorizing vessels are fitted with special entrainment-prevention chambers in their upper portions immediately below the nozzle outlets to which the thermocompressors are attached. Total entrainment losses have been reduced to the order of 0.05%. Baffled expansion chambers reduce the velocity of vapors leaving the thermocompressors.

Complete instrumentation of the plant includes six-point recording potentiometer, twelve-point indicating potentiometer, total steam and distillation steam flowmeters and pressure gauges, recording vacuum gauges, indicating mercury type absolute pressure gauges at several points, draft gauge and fuel and water pressure and level gauges. These instruments facilitate control of operation and insure unformity of quality in production.

CONTINUOUS DEODORIZING PLANTS. The continuous deodorizing equipment of Rocca and of Bruyn have been mentioned already, as have the continuous distillation plants (suitable for deodorizing oils) of Wecker and of Birmal Chemical Engineers, examples of each of the latter two having been installed in the United States. Within the past two years at least one continuous deodorizing plant (Binder & Carleton) has been installed in this country, utilizing electric heat and superheated steam in a counter-flow column apparatus. Elaborate heat-exchange apparatus will, it is claimed, so reduce the heat consumption that the electric heat-



Control Board and Finishing Filter Press Modern Deodorizing Plant.

ing will be economical with average current costs. The steam is superheated by electric heat, likewise.

Another continuous fatty acid distillation process recommended by its proponents as suitable for continuous deodorization is that of Tolman and Goranflo.[21] The basic elements of this system comprise a method of heating the incoming stock by means of exchange with superheated steam in a shell-and-tube heat exchanger, recirculation of the superheated steam through a direct-fired heater, make-up of additional steam needed through the medium of a thermocompressor and securing of intimate contact between oil and distillation steam by means of screen-tray or bubble-cap-tray counter-current tower apparatus. Operation of the plant in fatty acid distillation is under moderate vacuum, which can, of course, be increased for purposes of deodorization.

Summarizing, the methods of continuous deodorization include the moderately antique methods of Rocca and Bruyn, the modern methods of Wecker, Birmal, Tolman-Goranflo, and the electric heat method of Binder-Carleton. Lee's improved direct-heat method of batch deodorization is also suitable for adaptation to continuous deodorization.

The trend toward continuous deodorization and modern developments in the field of automatic instrumental control of thermal reactions will doubtless result in giving us completely automatic operation of deodorization equipment at some not-far-distant date. Quality of finished product will continue to be judged, however, at least in part, by the organoleptic methods of smelling and tasting.

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The Seed Oil of the Hackberry

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THE hackberry, or sugarberry*, is the fruit of *Celtis occidentalis* L., a tree which bears a superficial resemblance to the elm and, like it, is one of the Ulmaceae. This tree attains its best development in the deep alluvial soils of the river bottoms, but thrives on gravelly and stony upland sites, especially limestone outcrops. It is widely scattered, its range extending from southern Quebec to eastern Washington and Oregon, southward to Florida and westward to eastern Texas, New Mexico and Nevada. A smaller, more dainty edition of this genus is the southern hackberry (*C. mississippiensis,* Bosc.) whose habitat is the south central states and Mexico. The fruit of the species in question is a cherry-like, slightly ovoid drupe whose color, at first an ocher-yellow, eventually becomes a purple black. It matures in autumn and persists into winter. The nutlet or pit is oblong-oval, thick-walled and light brown in color. From data obtained in this Laboratory (*1*) it appears that the shell constitutes three-fourths of the weight of the pit; that the latter contains 37 per cent mineral matter and 11.33 per cent (petrolic) ether extract and the "meat" itself 43.15 per cent of fatty matter. That its popular name should reflect the chief saccharine characteristic of the fruit whose pleasant taste suggests the flavor of figs is not surprising in view of the fact that reducing sugars account for approximately one-third of its composition (*2*).

If the seed oil of *C. occidentalis* L., or that of any other species of this genus, has ever been subjected to critical study, then this fact escaped notice during the course of the survey of the literature which was made preliminary to the inception of an investigation, the results of which are herein recorded. Some work, however, has been done on the fruit and the pit itself. Over eighty years ago Payen (*3*) reported on his studies of the pit of *C. cordata,* a species name which is probably here synonymous with *C. occidentalis.* The outstanding fact resulting from his researches is the preponderance of the element calcium which, he observed, exists here as the carbonate. More recently Yanovsky and associates (*2*) examined the whole fruit. They confirmed the findings of Payen in respect to the high calcium carbonate content of the pits, extended the analysis to include its other inorganic constituents and investigated the nature of the organic acids of the pulp.

*This term is used in its popular sense. What is called here a berry is, botanically, a drupe.

This investigation was carried out for the purpose of determining whether the genetic relationships existing among the Ulmaceae might be reflected in chemical similarities in composition of the seed oils of two of its genera, Ulmus and Celtis.

The unusual composition of the seed oil of the elm, *Ulmus americana* L., reference to which has already been made in this Journal (*4*), raised the hope that equally interesting facts might be brought to light in this instance also.

The fruit from whose pit the fatty oil was extracted† was collected during the months of October to December, 1935, on the campus of the University of Wisconsin and on private property in a suburb of the city lying over a limestone outcrop. Extraction of the oil was made with purified petroleum ether (b.p. 60-70°) by a two-step operation: a preliminary extraction of the cracked whole pit followed by re-extraction of the comminuted partially de-fatted residue. Removal of the solvent under reduced pressure in the presence of carbon dioxide and filtration gave an oil, 12 yellow in color, whose physical and chemical characteristics were found to be as follows (Table I):

TABLE I

Analytical Constants of Hackberry Seed Oil.

Specific gravity 25°/25°	0.9204
Refractive index 25°	1.4794
Iodine number (Wijs)	150.0
Thiocyanogen number	81.97
Saponification number	191.1
Reichert-Meissl number	0.0
Polenske number	0.3
Hydroxyl number	4.9
Soluble acids (per cent butyric)	0.08
Insoluble acids (per cent corr.)	91.97
Unsaponifiable matter (per cent)	1.35

†Grateful acknowledgment is made to the NYA for supplying the labor necessary in the collection of the fruit and the subsequent recovery of the pits of which approximately 5 kgs. were obtained.

The presence of both low and medium molecular weight fatty acids in any noticeable amounts seems to be contra-indicated by the low order of magnitude of Reichert Meissl and Polenske numbers, a view which is supported by a normal saponification value. Hydroxy-acids, likewise, are not conspicuous. On the other hand, the ratio between thiocyanogen and iodine numbers furnish a clew to the probable presence of not only oleic acid but at least one other of a higher degree of unsaturation, such as linoleic, a fact which was subsequently demonstrated.

Identification of the unsaturated fatty acids was effected by means of their bromo-derivates as well as their oxidation products. No hexabromide derivative of linolenic acid was obtained by brominating a 2-g. sample at -10°. It is true that a voluminous precipitate was obtained at this temperature but analysis proved it to be the tetrabromo-derivate of linoleic acid. It had the same composition as that fraction which was insoluble in cold petroleum ether (m. p. 114.3; Br theoretical 53.3%, observed 53.8%). Its precipitation at this point was probably due to the unexpected solubility conditions in cold ethyl ether which had been created because of its abundance. That there are involved here the two isomeric forms of this acid seemed not improbable,