weight percentage (7): myristic, 0.4; palmitic, 10.6; stearic, 2.4; saturated C_{20} and above, 2.4; unsaturated C_{16} and below, 1.0; oleic, 23.5; linoleic, 51.2; linolenic, 8.5. The principal differences from the cottonseed oil of Table I are a lower content of palmitic acid, a slightly higher content of linoleic acid, and the presence of linolenic acid in an appreciable amount.

Palm Oil

Palm oil is extracted from the fruit of the palm tree on plantations in the Netherlands East Indies, Malaya, and the Belgian Congo. It is an oleic-linoleic acid oil, orange in color, and semi-solid at room temperature. The constituent acids of a Malaya plantation oil (4) iodine number 53.8, are as follows in weight percentage: myristic, 2.5; palmitic, 40.8; stearic, 3.6; oleic, 45.2; linoleic, 7.9. It is considerably different from cottonseed oil, being mainly a glyceride of oleic and palmitic acid with considerably less linoleic acid than in cottonseed oil (7.9 compared to 47.8). The oleic and linoleic acid content promotes solubility; the oleic and palmitic acid content promotes detergency; and the palmitic and stearic acid content supplies firmness. Palm oil may be used alone or in blends with tallow and coconut oil.

Hydrogenated Whale Oil

Whale oil is usually extracted at sea by cutting up the blubber and boiling with water or heating in autoclaves. The oil must be hydrogenated to an iodine number of about 50 to prevent reappearance of odor on storage. The hardened fat which results from the saturation of the 20 and 22 carbon polyunsaturated components is not suitable for soap making alone but can be blended with softer oils or with coconut oil.

Summary

Four classes of glycerides have been discussed as raw materials for soap. They are the animal fats, for example tallow and grease; the lauric acid oils, for example coconut oil and babassu oil; the oleic-linoleic acid oils, for example cottonseed oil foots and palm oil; and the marine oils, for example hydrogenated whale oil. Soybean oil foots is still another class, representing the linolenic acid oils. No single fat has a fatty acid composition which would make it generally useful in soap making, and it is the usual practice to blend the raw materials.

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The Pretreatment of Soap Stocks

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IN the manufacture of soap in this country the basic fat stocks used for saponification are the various grades of tallow, greases, nut oils, such as coconut and babassu, marine oils (whale and menhaden), and soap stocks and acid oils produced by the refining of edible oils such as cottonseed, corn, peanut, soybean, etc. Formerly a fair quantity of palm oil was available, but since World War II imports of this stock have been, for all practical purposes, limited to that required for the manufacture of tin and tern plate. Tallow, greases, and nut oils form by far the greater proportion of soap stocks used today.

E. M. James

Tallows as produced by the packers or the renderer are graded according to their content of free fatty acid, FAC color, m.i.u. (moisture-insoluble-unsaponifiable), and titre (the solidification point of the fatty acids). In addition, choice or fancy tallows are graded according to the Lovibond color of a sample of the stock after caustic refining and earth bleaching in the laboratory.

These standards for the different grades of tallow are important for the following reasons. The percentage of free fatty acid has a direct bearing upon the glycerine yield when the stock is saponified, the m.i.u., particularly the m and i, represent non-fatty constituents which are lost in saponification and hence reduce the yield of soap. Hardness of the finished soap depends upon the titre, and the color of the original stock is reflected in the whiteness of the finished soap or in its brightness when a colored soap is produced.

The greases in general have higher limits for free fatty acid, m.i.u., and FAC color, and lower limits for titre. However choice white grease, which is usually an inedible lard, has limits on color, free fatty acid, and m.i.u. similar to those of the better grades of tallow.

The marine oils are usually bought on a basis of free fatty acid and m.i.u. Soap stocks are purchased on the basis of total fatty acid content (35% minimum) and acid oils on the same basis (85% minimum).

The higher grades of tallow and choice white grease are, as produced by the packers, often of edible quality. To prevent their use in edible products the government requires that they be denatured, either by the addition of a small percentage of a high boiling petroleum distillate, or a fractional percentage of brucine. Renderer's tallow, even the fancy grade, which is produced from scrap collected from butcher shops, etc., is not denatured.

Soap stocks are generally received by the soap manufacturer in tank cars or tank wagons although in certain cases they may arrive in steel barrels. Tallows and greases are almost always solid as delivered and must be melted by connecting the coils of the car or truck to a source of steam, often supplemented by open steam introduced into the bottom of the tank through a temporary line. Since the latter practice will always result in the condensation of water, the tanks are first sampled by means of a large tryer, and the stock is analyzed in the laboratory to determine whether it meets the specifications under which it was purchased and is then graded accordingly.

After the content of a tank of tallow or grease has been melted, it is pumped into cone-bottomed house storage tanks, and the car rubbed out as thoroughly as possible. In house storage the bulk of the moisture in the stock as received and that added in the form of open steam settles out and is withdrawn to the sewer from the cone bottom. Such drainings are usually passed through a catch basin to recover small amounts of fat which may be entrained, and the catch basin skimmings are returned to the melting-out house for further settling and treatment to recover the fat. The settled tallow or grease may either be pumped directly to yard storage or processed immediately for the soap kettle.

Settling in house tanks, while it removes the major portion of the moisture and dirt present in the stock as received, is seldom complete. In many cases the dirt, which consists mainly of organic matter such as animal tissue, very finely divided bone, etc., has strong emulsifying properties, and this may result in the formation of a stubborn emulsion layer between the water and fat which must be separately treated. In any event, the settled stock almost always contains some dirt, which settles to the bottom of the yard storage tanks and must be cleaned periodically at the cost of considerable labor. In addition, the presence of this organic dirt and moisture tends to split the fat and increase the free fatty acid, with consequent loss of valuable glycerine in the sludge and tank bottoms.

Recently some attempts have been made to centrifuge the settled fat, using a nozzle-type disc centrifugal which will allow continuous operation. The dirt can be effectively removed in this manner, but the moisture content is practically unaltered. However the residual water tends to settle rather rapidly and collect in the bottom of a yard storage tank as a muddy liquid instead of a sludge, and when the tank is cleaned, it can be run directly to the sewer through the catch basin, with practically no loss of fat.

Coconut or coconut type of oils are also usually received at the plant in a solid condition and must be melted out in a manner similar to tallow and grease. Marine oils, unless they have been purchased as hardened stocks, are liquid and do not present any special problem in unloading and handling. Acid oils are almost always liquid or very easily melted.

Soap stocks, from the refining of edible vegetable oils such as cottonseed, soybean, peanut, and corn oils, must be heated and often thinned with open steam to unload them. These materials containing large quantities of non-fat, usually in the form of decomposed phosphatides, meal, etc., are either acidified immediately with sulphuric acid to recover the fat as acid oil for subsequent splitting and distillation, or they may be used immediately as part of a soap kettle charge. If the soap stock is to be stored as such, it is usually completely saponified or "killed" in a soap kettle. The bulk of the impurities are destroyed by the caustic soda, and the stock may then be pumped to yard storage without danger of further decomposition.

Having thus briefly covered preliminary handling of soap stocks, we shall now discuss the processing of individual stocks before they are saponified.

Tallow and Greases

Since tallow or grease forms the bulk of the stock charged to the soap kettle, we shall cover their processing in considerable detail.

The better grades of tallow are usually earmarked for the production of toilet or white soaps. In this case it is advisable to have them as light in color as possible. Even though the finished soap may contain a dye, the color will be brighter and more appealing to the eye if light stocks are used.

Good grade tallows may be bleached with fuller's earth and filtered; they may be refined with caustic soda to remove acid as well as color; or they may be both caustic-refined and earth-bleached.

Caustic refining is carried out in a standard refining kettle. The most common type is a cylindrical, cone-bottomed tank, equipped with an agitator of the paddle type (preferably variable speed) steam coils, and often a steam jacket on the cone. The tank is also equipped with stock draw-off lines, which can be raised and lowered to facilitate decantation of the refined and settled stock from the foots or soap stock, and with a large valve at the apex of the cone for the removal of the soap. The capacity of the refining kettle is 60,000-90,000 pounds of crude fat, and sufficient head room is allowed to provide for the weight of the caustic soda solution added as well as the expansion of the fat at elevated temperatures.

The kettle is charged with stock which has been heated to 110-115°F. to make sure that it is all liquid, and a sample is taken to the laboratory for determination of the free fatty acid. From this information the refiner calculates the proper amount of caustic soda required to neutralize the acid and adds to this figure an excess of dry caustic of 15-25% of that required to neutralize the free fatty acid as oleic acid.

The calculated amount of caustic soda is then made up as a solution, the strength of which depends upon the judgment and predilection of the individual refiner but is usually 18-20° Be (12.67-14.36% NaOH).

The agitator is started at relatively high speed, and the caustic solution is added to the refining kettle. When the full amount has been run in, agitation at high speed is continued a few minutes to make sure that the caustic is dispersed as completely as possible. Neutralization of the free fatty acid is substantially instantaneous, and the contents of the kettle assume the appearance of a somewhat grainy water-in-oil emulsion. Steam is then admitted to the coils, and the agitation is materially reduced. As the temperature rises, the small masses of soap and water tend to coalesce into larger and larger flocks of soft soap stock. The refiner takes frequent hand samples from the kettle, from which he can judge the rapidity with which the soap stock settles out and the size of the flocks. When, in his judgment, a satisfactory settle is attained, the kettle is shut down and allowed to stand, preferably for a minimum of eight hours, to allow the soap stock to settle as completely as possible.

The temperature at which a hand sample settles well varies with individual stocks, but the maximum is approximately 150°F. When this temperature is attained, steam is shut off from the coils, even though the hand sample does not settle as well as it should, and agitation is continued at a slower speed. It may be necessary to continue agitation for 20-30 minutes with some stocks before the refining will finish properly.

Temperatures substantially in excess of 150°F. should be avoided since above this point there is a tendency toward dehydration of the soap stock, which will cause a considerable amount to rise to the surface of the fat in the kettle and result in increased refining losses, coupled with the considerable labor required to skim the dehydrated floating foots from the surface of the clear fat. After the contents of the kettle have settled for about eight hours, the clear neutral stock, which now has a free fatty acid content from 0.03-0.10%, is decanted through the swinging suction either to house or yard storage. The soap stock or foots remaining in the kettle is withdrawn through the foots valve and used as part of a charge for a kettle of lower grade soap, such as a built powder or laundry chips where color is of less importance.

Good refining operation will result in a loss to soap stock of approximately 2.5 times the free fatty acid of the crude stocks as oleic acid; i.e., from a tallow of an original free fatty acid of 4%, the yield of refined and partially decolorized fat will be 90%.

The question may be asked why the more efficient and well developed process of continuous refining is not used on tallow instead of the standard refining kettle which has been in use for more than 50 years. The answer is simple economics. The difference in value between the crude fat and the total fatty acid contained in the foots is not great enough to absorb the increased expense of continuous refining and allow a reasonable saving over kettle operation.

Although almost all grades of tallow and greases can theoretically be improved by caustic refining, in practice there is obviously a limit beyond which the yield of refined stock is too low to make the operation attractive. As the free fatty acid of the original crude increases, the yield of refined, decolorized stock decreases rapidly. For example, bearing in mind the free fatty acid loss ratio of 2.5, from a stock containing 15% FFA the yield of refined fat would amount to approximately 62.5%. Therefore stocks, such as the poorer grades of tallow and yellow greases, that can be used for products in which a light color of the saponification charge is less important, can be improved by either bleaching with fuller's earth or in some cases, chemicals. Recently another process, decolorization of these poorer grades of tallow by liquid-liquid extraction with propane, has been perfected.

Earth bleaching is carried out either at atmospheric pressures or under vacuum. The latter is the more efficient for a number of reasons, which will be explained below. Either bleaching method may be employed on caustic-refined fat as well as crude fat.

For atmospheric bleaching open tanks equipped with steam coils and constant speed agitators are used. Typical bleachers are cylindrical tanks having either a dish or a shallow cone bottom. The oil charge varies from 30,000-50,000 pounds, depending upon press capacity.

The fat is charged to the bleacher at a temperature of 120-150°F. and the earth added with the agitator running at full speed. The dosage will depend upon the quality of the stock to be decolorized. A good grade of refined tallow, for instance, will require 2.0-2.5% earth for maximum decolorization while darker crude stocks may need 5-6% of clay.

After the earth has been added to the fat, the temperature of the mixture is raised to a maximum of 200°F. by means of the closed steam coils. This temperature should not be exceeded since damage to the tallow may occur through oxidation. In some plants a maximum of 180°F. is specified.

The correctness of the dosage of earth is usually determined by filtering a hand sample of the contents of the bleacher through paper and reading the color. If the latter is too dark, more earth is added and agitation at heat continued.

When the desired color of the bleached fat is attained, the mixture is pumped through one or more filter presses of the plate and frame or recessed plate type. The filtered fat is recirculated until it is judged clear and is then sent to house storage. When the presses are full of earth, they are blown with air to remove free oil, and the cake is finally steamed to recover as much of the entrained oil as possible. Press steamings are sent to a settling tank, and the recovered fat is reprocessed.

Vacuum bleaching of soap fats is a comparatively recent development and came about as a result of the success of this process in the treatment of vegetable oils for shortening. The bleacher is a closed tank equipped with a constant speed agitator and a set of coils connected to both steam and water mains for first heating and then cooling the charge. The capacity of the bleaching vessel is 30,000-50,000 pounds with sufficient head room to take care of foaming caused by slightly wet stocks as well as the evolution of water from the bleaching clay. The source of vacuum is usually a two-stage ejector capable of maintaining 27-28.5 inches of vacuum on the system and equipped with a barometric condenser and hot well.

The melted fat and earth are charged to the bleacher, the system is closed tightly, and the pressure is reduced by the ejectors. Heating usually begins as soon as the system is closed, and bleaching is carried out as described above. More earth can be added to the bleacher by means of a hopper and a conduit. The latter is equipped with a gate valve or cock, using the vacuum to suck in the additional clay.

The temperature allowable in a vacuum bleacher is considerably higher than in the open type and may be as high as 240-250°F. The low pressure effectively protects the hot fat from oxidation, and the somewhat increased effect of the earth at these higher temperatures can thus be realized.

The vacuum bleacher usually has a catch-all tank installed between the vapor outlet and the ejectors, and this catch-all collects a considerable proportion of the hydrocarbon denaturant present in the white tallows. This hydrocarbon has been known to cause a flash fire when open bleachers are used and the room is not adequately ventilated.

When bleaching is completed, the steam is cut off and cold water is run through the coils until the bleached fat has been cooled to about 180°F. The vacuum is then broken and the charge filtered as described above.

Bleaching losses vary depending upon the type of earth used. Domestic clays, such as that from Pikes Peak, Ga., will hold one pound of oil for each three pounds of earth used. The high powered activated earths, such as the Filtrols, retain one pound of oil per two pounds of earth, but less is required to attain the same final color.

Various methods, such as solvent extraction of spent cake or boiling the latter with a solution of salt and soda ash to separate some of the absorbed fat by flotation, are used to recover at least a part of the fat lost in bleaching, but the quality of this recovered stock is always very poor, and it must usually be split and the fatty acid distilled if it is to be used in anything but very dark soap.

A number of chemical bleaches have been proposed for the decolorization of tallow and greases. Among these are chlorine dioxide, sodium peroxide, sodium hypochlorite, etc., but none of these have attained very wide usage. There are in addition many patents dealing with chemical bleaches, which those who are interested in pursuing the matter further are advised to consult since the subject cannot be adequately covered in a paper of this scope.

The Solexol Process

There is another process for the treatment of low grade tallow and yellow grease which has proven to be quite successful in recent years. This is the Solexol Process for the decolorization of dark colored stocks by means of liquid-liquid extraction with propane.

This process depends for its results upon an unusual property of propane. The solubility of tri-glycerides and fatty acids in liquid propane is complete at 120° F. and decreases as the temperature is raised until at 180°F. the dissolved material is almost completely precipitated. When a solution of yellow grease or dark tallow in propane is heated in the range of 150-170°F., the color bodies and oxidized fatty matter, being the least soluble constituents of the crude stock, become insoluble and precipitate from the solution. In practice the color bodies and oxidized fat carry with them some triglyceride, leaving a light colored mixture of glycerides and fatty acids dissolved in propane. The decolorized material is recovered from this solution by distillation. The evaporated propane is condensed at pressures well above its vapor pressure at condenser temperatures and recycled. The process is continuous and for most economical results should be operated 24 hours seven days per week.

A detailed discussion of the operation of the Solexol Process is far beyond the scope of this paper. E. B. Moore has published an excellent account of this process in the Journal of the American Oil Chemists' Society, XXVII, 75-80 (1950).

To date, three commercial units for the decolorization of tallow with liquid propane have been installed. Their capacities range from 150,000-250,000 of crude tallow per 24-hour day.

The process when operating on dark tallows or yellow grease is capable of turning out a lighter colored fat than can be obtained by any other decolorizing treatment now known except splitting and distillation. The two processes are completely different since the latter is a chemical-physical and the former a purely physical process. If the impurities removed from a Solexol processed tallow are re-mixed with the decolorized fat, the original stock is reconstituted.

It is possible to decolorize a dark tallow by the Solexol Process to a 7-11 red Lovibond on a $5\frac{1}{4}$-inch column, at a yield of treated stock of 97.5-98.0%. Of course the yield of finished product, assuming the most efficient operation, is dependent upon the percentage of oxidized fat, color-bodies, dirt, etc., in the crude material, but these yields are typical for yellow grease. Even lighter colors than this can be obtained at the expense of lower yields, but such operation is not economical since to date no satisfactory commercial use has been found for the separated color bodies or "bottoms." Their high proportion of oxidized fat makes them unsuited for splitting and distillation, and if they are saponified without further treatment, the soap produced is so dark as to be almost black.

Although the better grades of tallow can be decolorized to very light colors by the Solexol process, it is more economical to caustic-refine them as already described. The process is best fitted for those tallows and greases whose high free fatty acid makes caustic refining unprofitable. It has been successfully operated commercially on tallows containing 35 to 38% FFA. So far as I am aware, although reasonable decolorization has been obtained experimentally with lower grade greases and even with cottonseed acid oil, these materials are not being treated commercially.

Although a surprisingly white soap can be made from Solexol decolorized tallows, it should be emphasized that the process cannot change the basic chemical composition of the fat or raise the titre. Low titre stocks are not suitable for high grade toilet soaps, and this fact should be borne in mind in evaluating the process. The difficulty could be overcome if the stock were hydrogenated before decolorization to reduce its content of unsaturated acids. Such a step is quite possible, but hardening would raise the cost of treatment to uneconomical levels.

It has been observed that Solexol decolorized tallows and greases, if kept at temperatures of 140°F. and above, or if overheated in melting out yard tanks or tank cars will darken in color, and this darkening will be quite considerable. The reason for this phenomenon is obscure, and various antioxidants which have been tried have not been successful in wholly stabilizing the decolorized fat. That heat plays an important part in the color reversion is shown by the fact that if a sample of decolorized fat is chilled immediately, it may be kept in solid form for months. When cautiously melted at low temperatures it will be found to be unchanged in color. In passing, it may also be noted that the color reversion of Solexol-treated fat seems to be peculiar to animal fat since crude cottonseed or soybean oils, after decolorization with propane, are remarkably stable in color even when held for some time at temperatures of 180-200° F. This color reversion is the reason why hydrogenation of decolorized tallows is to date impracticable; the stock tends to darken very fast at converter temperatures.

If Solexol decolorized tallow is saponified either immediately or within a few hours after treatment, reversion will be at a minimum and the finished soap will be very light in color. Hence this material lends itself very well to continuous saponification by the various processes which are available commercially.

Hydrogenation of Soap Stocks

Inedible lard (choice white grease) and soft oils, particularly marine oils, are commonly hardened by treating with hydrogen in the presence of a nickel catalyst. The metallic nickel is usually supported on kieselghur. Hardening of marine oils raises the melting point and hence the titre of the stock and also partially deodorizes the fat by destroying the fishy odor always associated with these oils.

The proper degree of hydrogenation of the unsaturated glycerides is usually determined by the drop in refractive index as observed by means of a Zeiss butyro refractometer. This drop in refractive index is an approximate measure of the reduction in iodine value, and a curve can be drawn for any individual fat. A refractive index-iodine value curve is not too dependable except as a guide. Accurate results require determination of the I.V. of the finished charge.

Choice white grease does not present any problem in hardening, and the catalyst can be reused a number of times before losing its activity. Crude marine oils, on the contrary, contain a catalyst poison which either greatly reduces or destroys catalyst activity and must be given a preliminary bleach with from 3-5% of fuller's earth to remove this poison before charging to the converter. The catalyst poison may also be removed by refining the crude oils with caustic soda before hardening, but this procedure has the disadvantage that the soap stock produced is unsuited for soap making. Therefore, although marine oils are often caustic-refined before saponification, especially for use in white soaps, it is the hardened oils which are so treated. Caustic-refining of hardened marine oils, when employed, is carried out by approximately the same method and in the same equipment as that already described for white tallows.

There are many types of converters available for hydrogenating fat. A common form is a cylindrical vessel with a relatively small diameter in relation to its height, having a capacity of 15,000 to 35,000 pounds of fat. Since hydrogenation is an exothermic reaction, the converter is equipped with both heating and cooling coils. Agitation of the charge is usually accomplished by mechanical agitation and hydrogen or by hydrogen alone. Pressures vary between 20 and 50 psi.

The stock to be hardened is charged to the converter and the catalyst, usually mixed with a small amount of oil, is added. Or the catalyst and charge may be mixed in a mixing tank and then pumped to the converter. The tank is closed and hydrogen admitted to the bottom through a spider or sparge, usually from a hydrogen compressor connected to the gas holder. Pressure is allowed to build up to the desired point, and steam is turned into the coils. When the proper hardening temperature (180-200° C.) has been reached, steam is cut off. It is often necessary to use cold water in the coils to hold the temperature to the above limits.

The operator observes the progress of hydrogenation by taking frequent samples from a trycock, filtering a few drops and reading the refractive index. When the prescribed refractive index is obtained, hydrogen is shut off and the charge is cooled to filtering temperature. The catalyst is then removed by pumping the contents of the converter through a plate and frame or recessed plate filter press. In some instances hydrogen pressure is used to force the hardened stock through the press.

The press cake from fresh catalyst can be used for hardening a second charge of oil. Often one batch of catalyst is used several times before it must be discarded. The filtered hardened stock may be refined if necessary, charged directly to a soap kettle, or sent to yard storage.

Nut Oils, Coconut, Palm Kernel, Babassu

These oils, if they are of good grade, are often merely filtered or lightly bleached with fuller's earth and charged directly to the soap kettle. When destined to be used in white soaps, they may be refined in the kettle as described above for the treatment of white tallows. The principal difference in their treatment is that a somewhat smaller excess of dry caustic (10-15%) is employed. Foots from the refining are used in lower grade soaps. Yields are approximately the same as in the refining of white tallows. Again, crudes having very high FFA are not refined but are saponified directly for darker soaps.

For white soaps, refined nut oils are often bleached with fuller's earth or a mixture of fuller's earth and activated carbon in the equipment and by the method described above for white tallows.

Miscellaneous Stocks

Miscellaneous soap stocks, such as the acid oils produced by the caustic soda refining of edible vegetable oils, fat recovered by the treatment of spent bleaching earth, garbage greases, etc., are usually split and distilled. These operations require a rather special pretreatment, which is more properly considered under the subject of "fat splitting and distillation" than in this paper.