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PREPARATION OF WINTER OIL FROM COTTONSEED OIL

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This invention relates to an improved process for preparation of "winter" oil from cottonseed oil.

Its principal object is to prepare an oil suitable for a salad oil for table use, that is, one which will not become cloudy due to deposition of stearine even at the lowest temperatures ordinarily occurring in homes.

A further object is to shorten the time heretofore consumed in the commercial process of cooling cottonseed oil and the subsequent crystallization of the stearine from same, with a corresponding reduction in the space required for the operation.

Our process consists essentially in "supercooling" the oil by cooling it so rapidly from a temperature at which the oil is perfectly limpid to a temperature a few degrees below the temperature at which the bulk of the stearine is to be crystallized out that the oil acquires a supercooled condition without separation of stearine, and then allowing the stearine to crystallize spontaneously from the oil without further refrigeration but allowing the temperature of same to rise slightly due to the heat evolved by the crystallization of the stearine, after which the stearine is separated leaving an olein having the desired properties for use as a salad oil.

The oil ordinarily used in this process is what is commonly known as "summer yellow" cottonseed oil, although bleached oils can also be used if desired. This is produced from ordinary crude cottonseed oil by the well known alkali refining treatment. It is important that the oil be free from moisture and suspended matter or dissolved soap, etc.; if it is perfectly clear at a temperature of 70° F., it will be satisfactory in these respects.

Ordinary refined cottonseed oil ("summer yellow oil") consists essentially of a mixture of glycerides of various higher fatty acids, some of which glycerides are liquid at ordinary temperatures and others are solid. The mixture, however, is a clear liquid at ordinary room temperatures. At low temperatures, however, some of the solid constituents crystallize out forming what is known as "stearine" and leaving a liquid portion known as "olein." The so-called stearine contains little, if any true stearic acid but consists mainly of glycerides containing palmitic acid. Temperatures below about 60° F. generally show some separation of stearine but the bulk of the stearine does not separate until the temperature is reduced to about 40–45° F., and in any event it is necessary to hold the oil at a crystallizing temperature for a considerable time before crystallization sets in to any great extent. This is due to supercooling to which the oil is easily subject. The olein constitutes the ordinary winter oil of commerce and, for commercial purposes, it is required to pass the "cold test" as described in the Rules of the National Cottonseed Products Association, wherein a four-ounce bottle of the oil must stand immersion in melting ice for five hours without becoming cloudy. Ordinary winter oils will develop cloudiness if held sufficiently long in this test, but not in the five-hour period prescribed. A further small quantity of stearine can be separated from such winter oils by subjecting them to a second crystallization at a lower temperature than that of the first crystallization, and the oil will then have a considerably longer "cold test" and it will stand a correspondingly lower temperature without clouding.

In making winter oil heretofore, the necessary refrigeration has usually been applied over substantially the whole period during which crystallization is actually taking place. The cooling has been accomplished, in one system, by first filling a large tank with the oil at a temperature of 70° F. to 90° F., at which temperature it is clear and limpid, and then passing cold brine through coils immersed in the body of the oil itself. Gentle agitation, either intermittent or continuous, is sometimes applied by blowing air through the oil during the first part of the chilling operation. In another system no coils are used in the oil itself but the tank containing the oil is surrounded by refrigerated air or cold water, and generally no agitation is used. Refrigeration in both procedures is continued until crystallization of stearine has progressed to the desired extent. In either case the oil remains substantially stationary, so the transfer of heat is slow, and these chilling processes on the large masses of oil commonly used usually require about thirty hours or more with the brine coil system, and several days with the refrigerated air system, to chill the oil from 70–90° F. to the crystallization temperature and to continue the refrigeration until the crystallization of stearine is substantially complete. The temperature at which most of the stearine crystallizes from summer cottonseed oil is usually about 42° to 45° F., although the stearine usually begins to crystallize at temperatures slightly below 60° F. The crystallized stearine is then separated, usually by filtration in a filter press followed by blowing with air, thus obtaining the olein for use as salad oil.

It is possible to recover commercially from average summer cottonseed oil in the above described manner, eighty-five per cent to eighty-eight per cent of marketable winter oil with twelve per cent to fifteen per cent of marketable stearine having a titer test of 44° to 45° C., but in some commercial operations only seventy-five to eighty per cent of winter oil is recovered, the remainder going into the stearine.

The brine coil system of cooling represented an advance over the older air cooling system in reducing the time required for cooling and crystallizing. Further attempts to shorten the time required have not been successful when working on large stationary masses of oil. If, in endeavoring to shorten the time required, the temperature of the refrigerating medium is maintained more than a few degrees below that of the oil, the effect is to overcool those portions of the oil nearest to the refrigerating medium so that they may actually freeze solid without allowing the stearine to separate in grains from the olein; in other cases, the result may be a rapid crystallization in the form of fine particles of stearine which cannot be filtered out. Agitation throughout the cooling and crystallizing period in large masses of oil likewise prevents crystallization of stearine in the form of large crystals which can be filtered.

In our process we cool the oil very rapidly, while it is moving over the refrigerating surface, so that a supercooling effect is obtained. The entire cooling takes place before there is any separation of stearine, or at the most only a very slight separation. We accomplish this preferably by passing the dry oil at a temperature of at least 60° F., but warm enough to be clear and limpid, continuously into and through a suitable heat exchanger supplied with a cooling medium such as cold brine, the temperatures and rates of flow of the two liquids being so regulated that the oil issues from the heat exchanger with the desired temperature. The oil should preferably be well agitated during its passage through the heat exchanger in order to secure uniform cooling and to prevent excessive local cooling of any portion of the oil. The cooling operation may thus be made continuous, and the oil may be completely cooled without exposure to the refrigerating surface for more than a minute or two, as only a small amount of oil is in contact with the refrigerating surface at any one time. In any event the cooling operation should not ordinarily extend over 30 minutes in order to assure the desired supercooling effect. The temperature to which the oil is cooled should be about 2° to 5° F. below the temperature at which the stearine will separate in large quantities or the temperature to which the oil will subsequently rise due to the heat of crystallization of the stearine. This latter temperature is referred to herein as the "temperature of graining." In this way all the refrigeration required for crystallizing the stearine in our process is applied to the oil before crystallizing actually begins. The best temperature to which the oil should be cooled is usually about 41° to 44°, depending on the nature of the oil and the amount of stearine which it contains, but not below 40° F., and can be readily determined in each case by a preliminary laboratory test or by observation of previous batches of the same oil. Care must be taken not to supercool the oil to an excessively low temperature, as this may cause the subsequent separation of stearine to occur too rapidly and in a form difficult to filter.

After cooling, the oil is accumulated in a suitable tank where it is held without further reduction of temperature, and with protection against changes of temperature due to other causes than the natural heat of crystallization, until the desired crystallization of stearine occurs, which requires from ten to twenty hours for completion. During the first few hours of this stage the oil is gently agitated, (usually about two to six hours, or until crystallization is well under way), after which it is found best to allow the oil to rest without further agitation until the crystallization of stearine is completed. During this stage the temperature will rise several degrees, sometimes as much as 5° F., due to the heat of crystallization. The separation of stearine is considered complete when no further rise of temperature takes place, and then the oil is ready for separating the stearine by filtration or centrifuging, etc.

In practice we find it best to maintain the atmosphere surrounding the tanks of oil for crystallizing at a temperature either the same as that of the oil or one or two degrees below the temperature of the oil, in order to assure that no access of heat to the oil takes place from outside sources during the crystallization; if this were not done, and if the surrounding air had a higher temperature than the oil, it would make necessary further cooling of the oil to offset the heating effect of the higher surrounding temperature during the subsequent crystallization. It is not intended however to impart any additional refrigeration to the oil at this stage.

We prefer to agitate the oil during the first portion of the crystallization stage with a gentle current of air or other gas, avoiding in all cases strong agitation. This is continued only until about the time when crystallization of stearine has produced noticeable turbidity, after which agitation is stopped. We find that if the oil has been cooled to as low as 41° F. about one hour of agitation is enough; if the oil has been cooled to 43°, we agitate about four hours; and if the oil has been cooled to about 44 to 45° we agitate as much as eight or ten hours. In any event the oil then remains quiet throughout the remainder of the crystallization period. Practically no temperature rise occurs during the period of agitation when conducted as here specified, but after this is stopped and rapid crystallization sets in the temperature gradually rises and may go up as much as 5° F.

The time required for crystallization can be considerably reduced by the process of "seeding" the super-cooled oil by adding a small amount, such as about two to five per cent for example, of the crystallized oil from a previous batch, or an equivalent quantity of crystallized stearine from a previous batch, which would amount to not over one per cent of the stearine. This should be added to the oil immediately after cooling. The agitation is then limited to about one hour and the separation of stearine is completed in a much shorter time than otherwise. Oftentimes the time is as short as seven hours.

The advantages of our new procedure are mainly the greatly reduced time for completion of the operation, the correspondingly reduced space required for production of a given amount of winter oil, and the reduced loss of refrigeration due to the greatly reduced time of chilling. It is novel, as far as we are aware, to find that cotton-

seed oil can be cooled rapidly from an initial temperature of 70–90° F., for example, to a temperature below the final crystallizing point of the oil without separating stearine, and then without further refrigeration to have the stearine crystallize spontaneously in a form which will permit a satisfactory rate of filtering. This rapid chilling is contrary to all previous experience and theories for the production of winter oil.

Having thus described our invention, what we claim and desire to secure by Letters Patent is:

1. A process for separating stearine from refined cottonseed oil which comprises cooling the oil from a temperature above its graining temperature at which it is limpid to a temperature at least 2° F. below its temperature of graining but not below 40° F., the rate of cooling being sufficiently rapid to avoid any substantial graining out of stearine during said cooling, then protecting the oil from any substantial change of temperature due to other causes than the natural heat of crystallization while stearine crystallizes from the oil, and separating the grained stearine from the olein.

2. A process for separating stearine from refined summer cottonseed oil which comprises cooling the dry oil while it is in motion, from a temperature above its graining temperature at which it is limpid to a temperature at least 2° F. below its temperature of graining but not below 40° F., the rate of cooling being sufficiently rapid to avoid any substantial graining out of stearine during said cooling, gently agitating the oil until substantial crystallization begins, protecting the oil from any substantial change of temperature due to other causes than the natural heat of crystallization until crystallization substantially ceases, and separating the grained stearine from the olein.

3. A process for separating stearine from refined summer cottonseed oil which comprises cooling the dry oil while it is in motion, from a temperature above its graining temperature at which it is limpid to a temperature at least 2° F. below its temperature of graining but not below 40° F., the rate of cooling being sufficiently rapid to avoid any substantial graining out of stearine during said cooling, then adding to the cooled oil grained cottonseed stearine in solid form in an amount not exceeding one per cent of the oil, gently agitating the oil until substantial crystallization begins, protecting the oil from any substantial change of temperature due to other causes than the natural heat of crystallization until crystallization substantially ceases, and separating the grained stearine from the olein.

4. A process for separating stearine from refined summer cottonseed oil which comprises cooling the dry oil while it is in motion, from a temperature at which it is limpid, but at least 60° F., to a temperature at least 2° F. below its temperature of graining, but not below 40° F., within a period not exceeding twenty minutes, the rate of cooling being sufficiently rapid to avoid any substantial graining out of stearine during the said cooling, gently agitating the oil until substantial crystallization begins, protecting the oil from any substantial change of temperature due to other causes than the natural heat of crystallization until crystallization substantially ceases, and separating the grained stearine from the olein.

5. A process for obtaining from refined summer cottonseed oil an olein suitable for use as a salad oil, which comprises supercooling the oil by continuously cooling a moving stream of the dry oil from a temperature at which it is limpid and above its graining temperature to a temperature from 2° to 5° F. below its graining temperature, the rate of cooling being sufficiently rapid to avoid any substantial graining out of stearine during said cooling, gently agitating the oil until substantial crystallization begins, protecting the oil from any substantial change of temperature due to other causes than the natural heat of crystallization until crystallization substantially ceases, and filtering the grained stearine from the olein.

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