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WINTERIZATION PROCESS

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This invention relates to a novel process for the winterization of refined oils. More particularly this invention relates to the combined winterization of refined oils and of salad oils, mayonnaise and salad dressings containing these winterized oils. Still more particularly, this invention relates to the combined winterization of whole refined cottonseed oil with one or more unsaturated edible vegetable oils.

The winterization of oils involves the removal of higher melting glycerides from the oil and may be accomplished by cooling and filtration means, during which time the higher melting glycerides crystallize and are removed by filtration. It is necessary that the higher melting glycerides be removed when the oils are to be used primarily in food products such as salad oils and dressings because the higher melting glycerides tend to crystallize from the salad oils and dressings and the emulsions separate when these products are stored at refrigeration temperatures of approximately 40 to 45° F. which is the usual temperature maintained in home refrigerators in this country.

One of the methods by which oil may be winterized is by storing the oil in a chilling tank in a refrigerated room and allowing the cold air of the room to cool the oil at a rate which will allow the crystals of the higher melting glycerides to be formed and deposited.

Another method by which oil is winterized is to store the oil in chilling tanks which are equipped with coils through which refrigerant is passed thereby cooling the oil and allowing the crystals to form and be deposited in the tank.

In all artificial winterizations it is necessary that the temperature be very closely regulated and that the oil be cooled slowly in order to obtain filterable crystals. After the desired cooling temperature has been reached, the oil is held at this temperature for a predetermined period of time, usually about 12 hours or more. After holding the oil for the desired period of time, the oil is filtered under very rigidly controlled conditions to prevent disintegration of the crystals or clogging of the filtering medium. For example, filtration must be conducted at a low pressure (about 5 to 20 pounds gauge). Thus it will be seen that the time required for both crystallization and filtration of a batch will generally be about 2 days or more.

As will be seen from the foregoing, the artificial winterization step in the production of salad oils involves a considerable period of time, elaborate equipment and regulated conditions and consequently is one of the least satisfactory steps in the production of an edible oil.

The yield of a winterized oil, such as cottonseed oil is about 75–85% oil depending upon the iodine value of the starting oil, the time allowed for crystallization, the temperature of the crystallization and the degree of dryness obtained in the filter-cake or centrifugate. The higher melting glyceride precipitated during the winterization of cottonseed oil is known as cottonseed stearine which is a standard article of commerce. However, being a by-product material, it does not command a price equivalent to the parent oil and therefore the separated stearine always represents some degree of economic loss to the processor. Thus in the winterization of cottonseed oil, about 15–25% of cottonseed stearine is produced. In the winterization of soybean oil in accordance with the teaching of Gooding U.S. Patent No. 2,627,467 soybean stearine in the amount of 7–8% is obtained.

It is therefore an object of this invention to provide a process for substantially reducing the amount of stearine produced during the winterization process.

It is another object of this invention to provide a process whereby substantial economies are achieved in the production of winterized vegetable oils.

It is a further object of this invention to provide a salad oil containing the winterized oils of this invention.

These and other objects will be readily apparent from the following description.

According to the present invention, the amount of stearine usually produced during winterization is substantially reduced by mixing together two or more oils in a desired ratio prior to subjecting the oils to winterization. When the oils are combined prior to the winterization, it has been found that the amount of stearine and other undesirable by-products may be reduced to as little as 4–5% or even less.

It has also been discovered that when the two oils are winterized together in the desired ratio, an oil is obtained of sufficiently good cold test and one which will confer adequate resistance to freezing to salad dressings made from these oils at temperatures as low as 25° F. and even 15° F.

In carrying out the process of this invention, the glyceride oils which are to be winterized in accordance with this invention are refined in accordance with the conventional methods customarily employed. One or more of the oils may be lightly hydrogenated, if desired, prior to the winterization.

The desired oil-blend is prepared and is winterized and the solid components are separated by filtration, centrifugation, decantation or other suitable means. It is desirable to use different conditions of crystallization depending upon the degree of hydrogenation, if any, to which the second component oil is subjected. For example, a crystallization temperature of 40 to 55° F. and preferably 43 to 47° F. has been found satisfactory when one of the component oils is hydrogenated to an iodine value of 110 to 115. It has also been found desirable to permit the oil-blend to remain at the temperature to which it is cooled for a period of one day or longer. The temperature to which the oil is cooled is maintained during the separation of the solid components of the oil therefrom. After the combined winterization of the oils is completed by separation of the crystallized stearine the combined oil filtrate is deodorized.

The combined winterization of oils of this invention is applicable to a wide variety of oils such as cottonseed, soya, corn, peanut, milo, etc. and is also applicable to mixtures of three or more oils. For example, a mixture of whole cottonseed oil with soya oil may be winterized as may also cotton seed oil with a mixture of soya oil and corn oil, etc. While we do not wish to be bound by any suggested theory for determining the various oils which can be combined, it is believed that the species of crystallizable glycerides of the separate oils should not be the same. For example, the crystallizable glycerides of cottonseed oil are composed of large amounts of linoleic and palmitic acids in a glyceride distribution peculiar to the parent oil. The crystallizable glycerides of lightly hydrogenated soybean oil are made up of small amounts of linolenic acid, linoleic, and oleic acids, a little palmitic and more stearic acids, all in more or less random distribution among the glycerides. Thus the stearins from each of the component oils is of composition different from the other.

The process of this invention has been found to be particularly effective using a mixture of refined oils comprising whole cottonseed oil and soybean oil whether or not the

latter is lightly hydrogenated. The whole cottonseed oil should have an iodine value of between 106–113, a saponification value of 190–198, and a low free fatty acid content. The lightly hydrogenated soybean oil is produced, for example, in accordance with U.S. Pat. 2,627,467 by hydrogenating a refined soya bean oil to an iodine value of 100 to 120, preferably 110 to 120, at a temperature less than 245° F., preferably less than 225° F., and desirably 195° F. to 220° F.

The temperature at which the soya bean oil is produced should not exceed 245° F. Generally, the lower the temperature of hydrogenation below 245° F., the lower are the amounts of conjugated dienoic, trienoic and tetraenoic acid components, and the more adaptable is the resulting oil for salad oil uses, and such as a component in mayonnaise. Any hydrogenation catalyst which is effective for hydrogenation at temperatures of 245° F. or below is satisfactory for producing the soya bean oil. A nickel catalyst, such as that sold under the name of Rufert's catalyst and described in U.S. Patent 2,424,811 (July 29, 1947), if activated at 250° F. may be used in the customary amount (about 0.1% nickel on the amount of the oil hydrogenated) for hydrogenation conducted down to 210° F. The nickel copper catalyst described in U.S. Patent 2,320,063 granted on May 25, 1943, to The Best Foods, Inc., as assignee of C. J. Borkowski and J. L. Schille, may be used if hydrogenation is conducted at temperatures of 185° F. to 245° F. with optimum results obtained at 195° to 220° F. Generally, the higher the pressure, the greater is the rate of hydrogenation. Pressures of 40 to 50 p.s.i. have been found satisfactory although higher pressures, such as 100 p.s.i. may be used to advantage.

The ratio of the oils which are to be combined and winterized may be varied over wide ranges depending on the ultimate intended utility of the combined winterized oils. Oils normally difficult to winterize such as cottonseed oil, peanut oil etc. may be mixed with other vegetable oils so as to provide a mixture having at least 30% of the oil which is difficult to winterize. For example, when the combined winterized oil is to be employed as a salad oil in mayonnaise, a 45–55 mixture of whole cottonseed oil and a lightly hydrogenated soybean oil as discussed previously, has been found to be particularly satisfactory.

In Table I below the freeze resistance of mayonnaise made from the various combined winterized blends of cottonseed oil and soybean oil are set forth. The winterized oils were incorporated into mayonnaise and the cold-break resistance was compared with a control mayonnaise. The

composition of the control oil is a 50—50 blend of cottonseed oil and lightly hydrogenated soyabean oil, having an iodine value of 116, which have been winterized separately.

TABLE I

	Days at 15° F.			Days at 25° F., no break						
	One	Two	Three							
Blend of 50 parts cottonseed salad oil. and 50 parts lightly hydrogenated winterized soya salad oil.	OK	OK	OK	3	6	10	13	15	----	---
	OK	OK	B [1]							
55% hydrogenated soybean oil. and 45% whole CSO	B	B	B	3	6	10	13	15	20	27
	OK	B	B							
60% hydrogenated soybean oil. and 40% whole CSO	OK	B	B	3	6	10	13	15	20	27
	OK	B	B							
65% hydrogenated soybean oil. and 35% whole CSO	OK	OK	B	3	6	10	13	15	20	---
	OK	B	OK							
70% hydrogenated soybean oil. and 30% whole CSO	OK	OK	B	3	6	10	13	15	20	27
	OK	OK	B							

[1] Break.

Note.—Duplicate samples examined at intervals. Single samples examined at intervals and removed from test.

The test used in determining the freeze resistance of the mayonnaise blends requires that the finished mayonnaise at 25° F. be undamaged after 96 hours. It will thus be seen that the blends winterized according to the teachings of this invention compared very satisfactorily in freeze resistance with the blends produced by the prior art methods of winterization when the mayonnaise was kept at a temperature of 25° F. which is considerably lower than the temperature at which the mayonnaise would ordinarily be stored under conditions of home refrigeration.

In Table II, an evaluation of the product of winterization of various blends of oils produced according to the teachings of this invention and as compared with a control blend are set forth.

TABLE II

	M.P. °F.	Cold test	Solids content index				Stearine				Y, per cent
			0.0° F.	15.0° F.	25.0° F.	32.0° F.	M.P.	S.P.	I.V.	S.V.	
Control [1]	33.5	12–14 hrs	17.7	13.5	5.7	3.9	81.5	22.4	85.3	198.0	----
			17.5	13.3	6.8	5.2	----	----	----	----	16
50% soya oil [2]	36.5	----do	19.3	14.9	9.0	7.3	102.2	----	----	193.1	----
			19.8	15.5	9.3	7.6	102.7	----	----	193.7	4.6
55% soya oil [2]	39.0	Clear 6, cloud in 7 hrs	18.8	14.9	9.3	7.6	----	----	----	----	----
			18.7	14.9	9.4	7.8	105.8	25.3	92.6	194.3	3.7
60% soya oil [2]	39.0	Less than 15 min	19.3	15.2	9.9	6.4	----	----	----	----	----
			18.8								
			19.4	15.4	10.0	6.4	101.2	25.8	93.6	194.1	5.2
			18.9								
65% soya oil [2]	38.5	----do	18.6	14.8	9.7	6.2	10	----	----	----	----
			18.2								
			19.0	15.2	9.9	6.4	99.1	25.3	92.8	193.4	6.5
			19.0								
70% soya oil [2]	73.0	----do	17.6	14.9	9.2	5.7	----	----	----	----	----
			19.9								
			17.5	13.9	9.0	5.5	102.6	25.2	93.1	193.1	6.9
			18.8								

[1] The control sample comprised equal parts of separately winterized cottonseed and separately winterized lightly hydrogenated (116 I.V.) soya salad oils. The values given refer to the stearine obtained from the separately winterized cottonseed oil.

[2] The percent of hydrogenated soya oil (I.V. 116) refers to the composition of the blend prior to winterization. The balance of the blend was whole cottonseed oil.

It will be noted that the separately winterized and blended oils at the lower temperatures are not superior to the new oils obtained by the combined winterization. The control oil differs from the new oils in solids content to the greatest degree at 32° F. and at 25° F., temperatures at which the new oils have been found to be satisfactorily stable in mayonnaise.

The cold test values were determined by observing the elapsed time to the cloud point of the oil at 32° F. It will be noted that in some of the tests the blends had a cold test of as little as 15 minutes. However, despite such a low value for the cold tests, the blends were eminently satisfactory for use as the salad oil in mayonnaise and had adequate freeze resistance.

Heretofore, the standard cold test (Official Method Cc 11–42 of the American Oil Chemist's Society) of the salad oil has been the criterion for determining the relative freeze resistance of the mayonnaise produced from the tested oil, and it has been generally accepted that an oil of a cold test of 5½ hours (freedom from clouding at 32° F.) is the bare minimum value for satisfactory use in mayonnaise. Likewise, it was believed that the higher the cold test, the greater is the resistance to freezing of the mayonnaise. Contrary to this teaching, it has been found that the higher the cold test, the greater is the resistance to freezing of the mayonnaise. Contrary to this teaching, it has been found that cold test values of the salad oils of this invention bear little or no relationship to the ability of a mayonnaise to resist freezing at 25° F. and that the cold test values do not serve as an index of stability to lower temperatures of mayonnaise produced from such oils.

The more economic utilization of cottonseed oil in salad oils and dressings is likewise achieved by the combined winterization of whole cottonseed oil with one or more oils of the class which do not contain appreciable quantities of stearines crystallizable at usual winterization temperatures. This class includes whole soya, corn, milo, peanut, safflower, sunflower, sesame and the like. A greatly reduced quantity of stearine separates from cottonseed oil in all such combined winterizations, more of the starting quantity of cottonsed oil remains in the winterized oils and less loss accrues through undesired stearine by-product.

Example I

10,350 lbs. (45%) of bleached cottonseed oil (109.0 I.V.) and 12,650 lbs. (55%) of lightly hydrogenated soybean oil (I.V. 116.9) were thoroughly mixed and charged to each of two winterizing cells of 23,000 lbs. capacity. The starting temperature was 128° F. The initial cooling rate was approximately 10° F. per hour. At 55° F. the oil was still clear. During the initial cooling the oil mixture was agitated with air introduced from a perforated pipe at the inner bottom of the winterizing cell. Upon reaching 50° F. crystallization began and was evidenced also by a slow increase in temperature to 52° F. over a period of 12 hours. Cooling was resumed by circulating 38° F. brine through the cooling coils within the cell. After 48 hrs. and at a temperature of 44° the mixture of oil and crystals was filtered through a chilled plate-press. Filtration of 46,000 lbs. was completed in less than 8 hrs.

There was obtained—

42,170 lbs. salad oil
4,043 lbs. stearine (8.7%)

The chill test of the salad oil was 9 to 10 hours and it was found to have an iodine value of 113.4.

The stearine had an I.V. of 99.0; a melting point of 97.2; saponification value of 194.2.

The solids content indexes were:

50°	10.0
70°	6.7
80°	5.1
92°	2.8

Mayonnaise prepared from the oil produced in this example remained stable for more than 10 days at 25° F.

Example II

60,000 lbs. of an oil blend was prepared from 37½ parts of whole cottonseed oil of I.V. 110.3 and 62½ parts of lightly hydrogenated soya oil of I.V. 115.6 which was prepared according to U.S. 2,627,467 and distributed to three winterizing cells.

The cells were cooled to 45° F. in about 34 hrs. by circulated brine cooling and held at this temperature for another 36 hrs. Filtration was completed in 7 hrs. with the following results.

Salad oil filtrate—54,480 lbs. (I.V. 113.3).
Stearine—5,020 lbs. (8.5%), I.V. 98.9; sap. value 194.9.

Mayonnaise made from the salad oil of Example II in half-pint jars resisted freeze-break for more than 48 hrs. at 15° F. and for more than the required 96 hrs. at 25° F.

Example III

Other combinations of oils upon winterization gave the following results.

Starting mixture		Salad oil filtrate cold test (cloud point)	Products		
			Stearine		
Whole cottonseed oil, percent	2d component (balance)		Iodine value	Melting point	Percent yield
50	Whole soya oil	6 hrs.	97.8	112.6	1.0
35	do	3 hrs.			Nil
50	Whole milo oil	4 hrs.	65.8	140.3	2.2
35	do	3 hrs.	101.4	144.5	2.3
50	Whole corn oil	6 hrs.	96.3	109.4	1.5
35	do	5 hrs.	94.2	117.7	0.5
50	Whole peanut oil	60 min.	84.8	110.8	0.6
35	do	30 min.	84.8	115.7	0.1

In addition to use in mayonnaise, the oils produced by the combined winterization may be incorporated in other pourable or semi-pourable dressings such as french dressing, salad dressing etc. The keeping quality of pourable or semisolid dressings prepared from salad oils produced by combined winterization may be improved by the incorporation of stabilizing agents and preservatives as disclosed in U. S. 2,910,368.

Having thus provided a written description of the present invention and provided specific examples thereof, it should be understood that no undue restrictions or limitations are to be imposed by reason thereof but that the present invention is defined by the appended claims.

I claim:

1. A process for the combined winterization of refined oils which comprises first mixing together at least two oils selected from the group consisting of cottonseed, soya, corn, peanut and milo in a ratio of 30:70 to 70:30, then subjecting said mixture of oils to winterization and recovering the blend of winterized oils.

2. A process for the combined winterization of whole cottonseed oil and a lightly hydrogenated soybean oil which comprises mixing said oils together in a ratio of 30:70 to 70:30, subjecting said mixture to winterization and recovering the mixture of winterized oils.

3. A process for the combined winterization of whole cottonseed oil and a lightly hydrogenated soybean oil which comprises mixing said oils together in an amount of

30 to 55% cottonseed oil with the balance being said soybean oil and then subjecting said mixture to winterization and recovering the mixture of winterized oils.

4. A process for the combined winterization of whole cottonseed oil and a lightly hydrogenated soybean oil having an iodine value of 100 to 120 which comprises mixing said oils together in an amount of 30 to 55% cottonseed oil with the balance being soybean oil and then subjecting said mixture to winterization recovering the mixture of winterized oils.

5. The method according to claim 8 wherein said soybean oil is hydrogenated to an iodine value between 110–120.

6. A process for the combined winterization of whole cottonseed oil and another whole oil normally subjected to winterization comprising mixing said oils together in an amount of 30 to 55% whole cottonseed oil with the balance being said another oil, subjecting said mixture of oils to winterization and recovering the mixture of winterized oils.

7. A process for the combined winterization of refined oils which comprises mixing together an oil selected from the group consisting of peanut oil and cottonseed oil and an oil selected from the group consisting of corn oil, milo oil and soya oil in a ratio of 30:70 to 70:30, subjecting said mixture to winterization and separating the precipitated solids from the winterized oils and recovering the mixture of winterized oils.

8. A process for the combined winterization of whole cottonseed oil having an iodine value of between about 106 to 113 and a lightly hydrogenated sybean oil having an iodine value of about 100 to 120 which comprises mixing said oils together in about a 50–50 ratio, subjecting said oils to winterization and recovering the mixture of winterized oils.

9. A process for the combined winterization of whole cottonseed oil having an iodine value of about 106 to 113 and a lightly hydrogenated soybean oil having an iodine value of about 100 to 120 which comprises mixing said oils together in a ratio of between 50 to 70% soybean oil and 50 to 30% cottonseed oil, subjecting said oils to winterization and recovering the mixture of winterized oils.

10. The method according to claim 9 wherein said soybean oil is hydrogenated to an iodine value of between about 110 to 120.

References Cited in the file of this patent

UNITED STATES PATENTS

2,607,695	Ayers et al.	Aug. 19, 1952
2,627,469	Melnick et al.	Feb. 3, 1953