Salad Oil Manufacture and Control[1]

T. J. WEISS, Hunt-Wesson Foods, Fullerton, California

Abstract

Cottonseed salad oil is normally prepared by winterization, a process whereby oil is chilled slowly to form crystals of disaturated triglycerides, which are then removed by filtration. Hydrogenated soybean oil is similarly processed. Unhydrogenated soybean, corn, and safflower oils do not require winterization. A recent approach is to winterize from solvent, resulting in increased salad oil yield. The main control method is the cold test, a measure of the time required for the oil to cloud in an ice bath. Crystal inhibitors, such as oxystearin or polyglycerol esters, are used to lengthen the cold test.

Introduction

SALAD OIL IS DEFINED as an edible oil which will not solidify at temperatures of 40–50F. The need for such oil in huge quantities is attributable principally to the use of mechanical refrigerators in the home and the requirements of the mayonnaise and salad dressing industry.

If an oil solidifies or even clouds at temperatures above 40F, the oil must be winterized. Winterization is the name given to the process of removing solid fatty matter which appears in vegetable oils at low temperature. The term originated from the early practice of allowing cottonseed oil to remain in outdoor storage tanks in winter and of filtering off the resulting solid fats. The oil thus obtained would remain clear and pourable at moderately low storage temperatures. The solidified fats or stearine, being harder, could be used in shortening bases.

The original intent was to retain fluidity and clarity during the colder months. Unwintered or summer oil offered no problem in warmer seasons. The advent of mechanical refrigeration made winterization a necessity. This was especially true as the mayonnaise industry expanded. Mayonnaise could not be made from oils which would crystallize in the refrigerator and cause breakage of the emulsion. New terminology came about because of this association with mayonnaise. Winterized oil became "salad" oil. Summer oil became "cooking" oil. The terms winter and summer are still used in cottonseed trading rules. Salad and cooking terms are used by refiners in describing finished oils.

Cottonseed oil is the oil most frequently involved in winterization. Recent developments have made use of lightly hydrogenated soybean oil (to about the same iodine value as cottonseed oil) and of oil blends as a source of salad oil through winterization (2–4).

Unhydrogenated soybean and safflower oils do not require winterizing. Corn oil does not precipitate solid fats, but a cloud of waxes does form on chilling and should be removed. Peanut oil gels on cooling and cannot be filtered to remove the solid material.

The process of winterization is essentially a fractional crystallization. Lard, oleo stock, coconut, and palm kernel oils and more than slightly hydrogenated soybean oils are also fractionated by similar means. Strictly speaking, these crystallizations are not and should not be called winterization. Although the physical nature of all these processes is the same, the resulting products do not fit the salad oil classification.

The increased demand for winterized oil made it impossible to rely on natural conditions for the required annual production of salad oil. Processors seized on the simplest expedient; they created winter conditions indoors. This method is still in use today.

Winterizing Without Solvent

Warm, dry, refined, and bleached cottonseed oil is pumped into tanks which are usually deep but narrow for greatest wall exposure. The room is held continuously at 42F. The warm oil soon cools and begins to deposit solid fats known as winter stearine. The oil is not agitated so that the transfer of heat from the oil is by convection alone. Cooling is of necessity a slow process, requiring two to three days.

It is the slowness of cooling that makes the entire procedure feasible. The oil gets quite viscous at low temperatures. Poor mobility of the triglyceride molecules limits the growth rate of the crystals. The optimum conditions for growth rate are obtained when the cooling rate matches molecular movement. This results in the growth of large crystals, which are best for filtration from the oil. Rapidly cooled oil, especially under agitation, would deposit small crystals which would easily block a filter cloth and result in an oil which might not be filterable. After the oil reaches the temperature of the cold room, it must be held for several hours to allow the stearines to precipitate more fully. The stearines are filtered off, usually by forcing the oil through filter cloth in a plate and frame press. Early installations relied on gravity feed to the presses. Later developments made use of compressed air at low pressures, usually 5 to 20 psi. Low-speed, positive displacement pumps have also been used. In all cases care must be taken not to break up the crystals excessively. Filtration is slow because of high oil viscosity, but forcing the oil by high pressure only presses the stearine crystals into the filter cloth, causing blocking of the filter and stopping of the oil flow. The filter area must be large. Two to three pounds of oil per hour per square foot of filter is a general guide. A tank of oil takes 16–36 hours to filter.

The stearine cake is removed by melting. The filter cloths are cleaned by passing warm oil through the presses. Cloths may also be steamed or blown with hot air.

More recently, jacketed enclosed tanks equipped with cooling coils and agitators have been used for winterization. Control of coolant temperature is critical and is defined in terms of differential between coolant and oil temperatures. Specifically a 25F-differential has been found to be appropriate for oil at 75F, the starting temperature for the entire process. The differential is reduced to 10F by the time the oil reaches 45F. If the coolant is allowed to become too cold in relation to the oil, a heavy layer of stearine will build up on the coils and effectively insulate them from the main body of oil. In some plants, agitation is used only until crystallization begins and is then discontinued. In others, agitation is continued throughout crystallization, but it must be slow and gentle in order to maintain the growth of large crystals. Attempts at forcing crystallization, by means of excessively cold coolant and rapid agitation, results in small crystals, which are virtually unfilterable. One advantage of using an inclosed tank for winterization is that air pressure can be applied directly to the tank for filtration.

A typical time-temperature sequence for winterization is given by Bailey as follows. Oil is pumped to the chilling tank at 70–80F. It is cooled to 55F in six to 12 hours. At this point crystals will begin to form in the average oil. The cooling rate is reduced so that temperature drops to 45F during the next 12 to 18 hours. Heat of crystallization is released as the crystals form. The oil temperature will rise 2–4F as a result of this and will then again decrease. Cooling is discontinued at 42F, and the oil is held at this temperature for several hours before filtration. The effectiveness of winterization depends on the length of time the oil is held at the minimum temperature. Since the oil is viscous and molecular movement is slow, crystals continue to grow long after the minimum temperature is reached. According to Bailey, holding the oil for 12 hours at 42F will usually result in a 20+ hour cold test. This, of course, will depend on the starting material.

---

[1] Presented at the AOCS Short Course, East Lansing, Aug. 29–Sept. 1, 1966.

In general, it is better to hold a chilled oil longer at 42F than to hold for a short time at lower temperatures (5).

Brown (6) has found that more readily filterable crystals and improved oil yield are obtained if 0.1% lecithin is added to the oil before chilling. The same claim has been made for oxystearin, a crystal inhibitor (7). Gooding and Rich (8) winterized the oil, adding lecithin after the first winterization and stearine removal. This is to increase the amount of stearines removed and thereby increase the cold test.

Another approach to increase winterization efficiency is to add 5 to 10% of a previously obtained stearine to 90 to 95% fresh oil. The larger amount of stearine to be crystallized supposedly increases the crystal size and thereby makes filtration faster and more efficient.

While it would appear that 8 to 12% stearine should be removed from the oil, ordinary winterization usually yields 20 to 30% stearine because of the entrainment of oil in the crystals. A normal cottonseed oil of 103–112 iodine value usually winterizes to a salad oil of 107–117 IV and a stearine with a 90–100 IV (9).

There are a number of modifications of the classic techniques. Eckey and Folzenlogen (10) have supercooled cottonseed oil in a heat exchanger to 40F and allowed the oil to warm slowly in holding tanks to the normal 42F. While this method would markedly shorten the chilling time, it would seem that the crystals would be small, profuse, and difficult, if not impossible, to filter. The use of a crystal modifier has been suggested as a means of forming more readily filterable crystals (11). Separation of stearine from the chilled oil by means of a centrifuge has been described by Little (12) and by Kirkpatrick (13). This method of separation is based on the difference in specific gravity between stearine and oil. Little cools the oil to the proper winterization temperature and removes the stearine before the crystals have grown to filterable size. Kirkpatrick cools the oil slowly to just below initial crystallization temperature (45–50F) and then rapidly to 30–35F by pumping it through a heat exchanger and immediately through the centrifuge. The main problem with centrifugal separation is in getting a satisfactory yield as the stearine tends to trap excessive amounts of oil.

Solvent Winterization

Close examination of winterization from the solvent reveals that it is essentially similar to winterization from oil. The viscosity of miscella (solution of oil in solvent) is considerably lower than that of oil. This leads to the possibility of faster crystal growth and more rapid separation of stearine. Continuous systems become practical; in fact, the one described by Cavanagh (14) is actually in commercial use.

Miscellas containing 30 to 60% by weight of oil in hexane is given by Cavanagh as the working range for material. Since stearine is more soluble in hexane than it is in oil, crystallization temperatures are lower. Miscella containing 50% oil is preferred. It is cooled rapidly through a heat exchanger to 20–26F or 8 to 12F. One or the other temperature range is reported to be critical, but the reason is unknown. At either temperature the chilling rate is then reduced to about 0.2°/min so that the miscella reaches 0–5F within 90 to 120 min. The system described by Cavanagh uses a series of winterizing cells, equipped with cooling coils and paddle-type agitators. The stearine precipitates out of the miscella, and the whole mass flows into a separating tank where the stearine settles out. It is pumped to one holding tank; stearine-free miscella is sent to another; both are stripped of solvent.

Cavanagh uses a unique system for separating the crystals from the top of the tank. Conventional filtration equipment and centrifuges are also useful in the separation from solvent. The low viscosity of the miscella minimizes separation problems. The yield by this method is excellent since the low viscosity also minimizes entrainment of oil by stearine. Yields are reported to be 8 to 12% of a stearine with an iodine value of 70 to 75.

TABLE I

Triglyceride Composition of a Typical Cottonseed Oil (I.V. 105.0)[a] (1)

Triglyceride	Composition
Trisaturated	0.1%
Disaturated mono-olein	5.9%
Disaturated monolinolein	7.3%
Mono-saturate mono-oleo Monolinolein	40.6%
Mono-saturate dilinolein	17.8%
Mono-oleo dilinolein	28.3%

[a] Reference 1.

An additional advantage of solvent winterization is that the oil need not be dried free of moisture, prior to winterization, as is required in nonsolvent processing. The low temperature required for solvent separation causes freezing of the water, and this is removed with the stearine. In fact, Cavanagh claims that minute quantities of moisture in the oil act as crystal nuclei for the stearine and are actually advantageous.

Rubin (15,16) used 80 to 90% oil in acetone or other water-miscible solvents. They chilled the miscella to 32F in a Votator, holding it at this temperature for 3–4 hr before separating the crystallized stearine by means of a centrifuge. The low solvent level was used to keep the volume of miscella low and the chilling less than would be needed for chilling to 0F.

It should be pointed out that the low solvent level also results in a viscous miscella which is not much better than a solvent-free system. The problems inherent in forcing crystallization in oil are found as well. Small profuse stearine crystals do not grow well in or separate well from viscous media. Yields are too low to be commercially practical.

Boucher and Skau have published a series of articles (17–19, 20–21) giving time, temperature, and oil concentration relationships for winterization of oils from acetone, hexane, and acetone-hexane by weight. It is claimed that hexane solutions supercool badly and require a lower temperature and longer time to produce readily separable crystals than does acetone. On the other hand, acetone alone would dissolve water and cause oil to separate from the solvent. Addition of 15% hexane prevents the separation with up to 1.4% moisture in the acetone, but there is still insufficient hexane to cause undesirable supercooling. Boucher and Skau have demonstrated that the removal of a given amount of stearine from oil in the acetone-hexane blend could be done in a shorter time by lowering chilling temperature or by increasing oil concentration in the solvent. They reported that chilling a 30% solution of oil in acetone-hexane removes the same amount of stearine in three hours at 10.4F, in 45 minutes at 8.6F, and in 33 minutes at 6.8F. A 40% oil concentration requires 45 minutes at 10.4F for the same stearine yield. Their curves also reveal that crystallization times of less than one hour are impractical. This observation is borne out by the fact that all known workable systems require holding times, at winterization temperature, of one and a half hours minimum in the absence of solvent.

Economics favors not only solvent winterization but solvent refining prior to winterization as well. Modern plant design starts with the solvent extraction of the oilseeds, followed by refining and winterization in the miscella, subsequent stripping of solvent, deodorization, and packaging in a continuous, automated system. The oil mill and refinery have become an integrated unit. While this type of plant is not commonplace, it seems likely that more will be built in the future.

Examination of the glyceride composition of cottonseed

TABLE II

Fatty Acid Composition of Typical Cottonseed Oil Winterization Products

Fatty acid (GLC)	Cooking oil	Salad oil	Stearine from oil	Stearine from Solvent
Myristic	0.8%	0.7%	0.6%	0.4%
Palmitic	24.2%	22.6%	32.4%	52.0%
Palmitoleic	0.6%	0.4%	0.3%	0.0%
Stearic	1.6%	2.8%	2.4%	1.6%
Oleic	21.0%	19.8%	17.2%	12.0%
Linoleic	51.8%	53.7%	47.1%	34.0%
Iodine value	107.8	111.4	98.0	71.1

(Continued on page 186A)