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PROCESS FOR PREPARING SUSPENSIONS OF SOLID TRIGLYCERIDE AND LIQUID OIL

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The present invention relates to a process for producing fluid to pasty suspensions of solid glycerides in liquid fatty esters, and more particularly to a process of producing a fluid to pasty fat mixture containing a high proportion of glyceride in solid form and having pumpable fluid consistency over the normal range of room temperature.

It is common practice to convert liquid glyceride oils to non-fluid plastic condition by incorporating or forming therein relatively small amounts of saturated glycerides. This invention is especially concerned with processes whereby these and other mixtures of normally solid and normally liquid fatty esters may be prepared with such consistency that they may readily be pumped by standard pumping equipment, and will retain such consistency after storage for considerable periods under normal storing conditions. Mixtures of such ingredients, when prepared by conventional methods, either do not acquire a pumpable consistency, or are incapable of maintaining that consistency, acquiring instead a non-fluid plastic consistency.

It is therefore an object of the present invention to provide a process whereby pumpable suspensions of solid glycerides in liquid fatty esters, having a high degree of uniformity as regards the distribution of solid in liquid, and capable of retaining pumpable consistency over the normal range of room temperature, may readily be obtained.

Other objects will become apparent from the description of the invention which follows.

Briefly the process of the present invention comprises the steps of converting the solid glyceride into the beta crystalline form, hereinafter more fully described, suspending the converted solid glyceride in liquid fatty ester, and at any stage in the process, subdividing the solid glyceride into finely divided form.

It is known that glyceride fats may be crystallized in any one of a plurality of forms depending inter alia on the temperature and speed at which crystallization is permitted to take place. In this connection the reader is referred to Ferguson and Lutton, Chem. Rev., 29, 355 (1941) and Lutton, J. A. C. S., 67, 524 (1945) for a review of related details, especially those concerning means for identification of the various crystalline forms and their physical characteristics. Although these references deal principally with substantially pure trisaturated glycerides, the information given can be used as a guide in determining data encountered in dealing with solid triglycerides produced commercially as by hydrogenation of natural fats and oils.

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It will be apparent from the above referred to articles that the identifying terminology for the crystalline forms of fats may vary depending on the investigator, and in order that there may be no misunderstanding regarding the meaning of the terms herein used the following definitions of the crystalline forms are submitted.

The alpha crystalline form is the least stable, lowest melting and least dense of the forms. An X-ray diffraction pattern reveals a single short spacing at 4.14 Angstrom units, indicating hexagonal arrangement of the long chain axes in the crystals. Its macroscopic appearance is translucent.

The beta-prime form is usually intermediate between the alpha form and beta form (described below) in relative stability, melting point and density. It is in general characterized by two short spacings in the X-ray diffraction pattern, one at 4.2 Angstrom units and the other at 3.78 Angstrom units. The macroscopic appearance of the beta-prime crystalline form is more opaque than alpha but less opaque than beta.

The beta form is the most stable, most dense, highest melting of the polymorphic forms for many hard fats. Its X-ray diffraction pattern is characterized by multiple short spacings, at 5.3, 4.6, 3.9 and 3.7 Angstrom units and its macroscopic appearance is opaque, very white, and sometimes powdery.

For purposes of illustrating the variation in melting points of the crystalline forms, pertinent melting point data on some of the more common triglycerides are given below, a description of determination methods being set forth in the Lutton reference from which certain data have been taken.

Triglyceride	Melting Point, °C.		
	Alpha	Beta-Prime	Beta
Trilaurin [1]	14	34	43.9
Trimyristin [1]	32	44	55.5
Tripalmitin [1]	44	55.5	65.5
Tristearin [1]	54	64	73.1
Hydrogenated peanut oil—8 iodine value	51.5	63.0	67.8
Hydrogenated soybean oil—3 iodine value	51.6	64.0	70.2
Hydrogenated lard—1 iodine value	50.0		65.8
Hydrogenated linseed oil—14 iodine value	50.5	61.5	68.0

[1] Lutton, J. A. C. S. 67, 524 (1945).

As indicated above and as set forth in the reference articles mentioned, critical control must be exercised in order that a solid fat may have a desired crystalline form. Although the beta form is usually the most stable, solid triglycerides frequently do not exist in this form because solidi-

fication of the triglyceride has taken place under conditions which do not permit the formation of beta crystals and because solid transformation from the alpha and beta-prime forms, in which the solid usually exists immediately after solidification, to the beta form is extremely slow at ordinary room temperature. Many normally occurring solid triglycerides, therefore, are constituted of a plurality of crystalline forms, the beta-prime form frequently predominating. Such solid fats, when reduced to a powder and suspended in liquid fatty esters in amounts from about 10 per cent upwards form suspensions which although fluid or pasty in nature and pumpable immediately after preparation, tend to set up or become plastic or solid and non-pumpable during storage at ordinary temperature, and those suspensions containing 40–60 per cent solid for example do so quite rapidly at temperatures around 90° F. Moreover this marked tendency of the suspension to change from a fluid to a non-pumpable plastic or solid state is aided by alternate heating and cooling of the suspension within normal atmospheric temperature range such as 70° to 100° F. Thus, such suspensions may be said to lack stability against marked change in consistency within the aforementioned temperature range for example. Corresponding suspensions in liquid fatty esters of powdered solid triglycerides in beta crystalline form, on the other hand, possess greatly improved stability against such changes in consistency as is more fully pointed out below, and in accordance with our invention fluid to pasty suspensions of as much as about 60 per cent solid fat in about 40 per cent liquid fatty ester possessing stability against change to a non-pumpable consistency over the normal range of room temperature may be prepared.

The consistency of the products prepared in accordance with the present invention has been conveniently measured with the aid of an A. S. T. M. type penetrometer employing a cone shaped aluminum needle having the following characteristics:

Angle, 90°

Maximum diameter, 3.2 cm.

Total weight, 78.3 gms., including cone, shaft and 50 gram wt.

In actual test the needle is brought to the surface of the sample, released for 5 seconds and the penetration in tenths of a millimeter is read from a dial indicator. Consistency as measured by this method was coordinated with viscosity in centipoises and a penetration of 100 corresponds to a viscosity of about 300,000 centipoises. Any material having a penetration as determined above of 100 or more is sufficiently fluid to be pumped with any of the commercially available pumps specifically designed for handling viscous materials, and the term "pumpable" as used herein is to be understood as designating a penetration of at least 100 and a viscosity of not substantially more than 300,000 centipoises.

Stability against marked change in consistency and pumpability within the range of temperatures ordinarily encountered in storage and transportion is commercially highly desirable in the case of cooking fat compositions containing saturated triglyceride fats and which, for convenience of handling for example, are prepared in a flowable condition. To obtain products having stability with respect to consistency and pumpability when the solid fat content is in excess of 10–15 per cent for example, it is essential that the solid fat be in the beta crystalline form, otherwise normal changes in temperature especially those encountered during transportation in the summer months, will cause a firming of the product to the point where it cannot be pumped or otherwise handled as a fluid.

Although the invention may be practiced with pure triglycerides, such as triolein and tristearin, more practical applications involve the use of natural fatty esters. As the liquid constituent any of the naturally occurring liquid glyceride oils such as cottonseed oil, soybean oil, peanut oil, linseed oil, sunflower seed oil, corn oil, olive oil, rapeseed oil, fish oil, and the like, or normally liquid fractions obtained from glyceride oils, or normally liquid mono- and di-glycerides, such as monolinolein, monolinolenin, diolein, dilinolein and the like may be employed. In addition other normally liquid fatty esters such as sperm oil, methyl oleate, palmityl oleate, benzyl laurate, ethyl laurate and diethylene glycol monolaurate may be employed. In general the liquid constituent may be any normally liquid oleaginous ester of a mono- or polyhydric alcohol and a saturated or unsaturated fatty acid containing at least 8 and preferably at least 12 carbon atoms in the molecule, and the term 'liquid fatty ester" as used herein is to be so construed.

As the solid constituent, completely saturated triglyceride fats containing major amounts (e. g. more than 40 per cent) of tristearin and/or tripalmitin and/or other normally solid triglyceride having strong beta-forming tendencies such as symmetrical palmitodistearin for example, and including substantially completely hydrogenated triglyceride fats from the following vegetable and animal oils or their mixtures may be used: sunflower seed oil, linseed oil, hazelnut oil, soybean oil, peanut oil, olive oil, corn oil and lard. These solid fats may be employed in combination with each other or with hydrogenated fats such as cottonseed, palm, tallow, whale, fish, and the like, which have a lesser tendency to form beta crystals on crystallization. More specifically, mixtures containing major amounts of tristearin or tripalmitin, such as a substantially completely hydrogenated mixture of soybean oil and cottonseed oil, may be used in the practice of the invention, and fats which have been subjected to molecular rearrangement and hydrogenation processes whereby a major amount of tristearin, tripalmitin or other suitable solid triglyceride is caused to be present in the rearranged product may also be employed.

The following test, designed to determine the beta-forming characteristics of a fatty material, may be employed as a guide in the selection of normally solid triglycerides for use in making pumpable suspensions in accordance with the present invention.

Several thin-wall glass capillaries (about 1 mm. outside diameter) are charged with about a centimeter length of the molten fat to be tested. The alpha softening point and the maximum melting point are then determined by methods based on those methods used by Lutton (J. A. C. S., 67, 524 (1945)).

To determine the alpha softening point a sample in its capillary tube is first heated to 100° C. then is chilled for 2 seconds at 0° C. The sample is then thrust into a bath held at a temperature in the range of the expected softening point. The procedure is repeated employing baths at various trial temperatures. On each trial it is noted whether there is or is not increased translucence

of the fat sample. The recorded alpha softening point is the average of the lowest temperature where increased translucence is observable and the highest temperature at which it is definitely not observable.

To determine the maximum melting point the sample of fat in its capillary is melted at 100° C., then chilled at 0° C. for 2 to 60 seconds. The sample is then held for 4 hours at 1° to 2° C. below the alpha softening point to effect solid transformation to a higher melting form and for 15 to 20 hours at a temperature 10° C. higher to insure transformation to the higher melting form, then is transferred to a melting point bath and the complete melting point determined at a heating rate of 0.2° C. per minute.

With the data thus obtained the beta-forming characteristics of the fatty material and its suitability for use in making pumpable suspensions may be determined as follows.

One of the capillary samples is melted at 100° C., then chilled at 0° C. for 2 to 60 seconds. The sample is stored for 2 hours at 1° to 2° C. below the alpha softening point, then for 4 hours from 1.5° to 2.5° C. above the average of the alpha softening point and the maximum melting point to effect recrystallization. An X-ray diffraction pattern of the sample is then obtained and if this pattern indicates that the sample is 85 per cent or more in the beta crystalline form, the hard stock is suitable for use in the present invention for producing pumpable suspensions.

It is to be understood that throughout the specification and claims the expression of the relative amount of any particular crystalline form in a fatty material is based on the X-ray diffraction pattern of the crystals present.

Within reasonable limits the degree to which solid fats are hydrogenated for use in the invention is not critical, altho it is preferable that the iodine value be below 20. This preference is of especial importance if the solid is to be converted to the beta crystalline form from the alpha or beta-prime form by solid transformation of the crystals because the presence of unsaturated constituents tends to reduce the rate at which such solid transformation takes place and reduces the incipient melting point of the glyceride so that it tends to fuse together at temperatures required for reasonably rapid transformation.

The invention will be more fully understood from the following examples.

Example I.—Peanut oil, hydrogenated to about 3 iodine value was rapidly chilled and solidified on a cooled roll, then reduced to flake form. This product was placed on a tray to a depth of about 2 inches. The tray was charged to an oven held at 62° C. (just below the beta-prime melting point of 63° C.). After about 7½ hours the temperature of the flakes had risen to about 61° C. The temperature of the oven was then gradually reduced so as to permit the stock to cool to about 49° C. over a period of about 30 minutes. The flakes were then permitted to cool to room temperature over a period of about 10 minutes. By X-ray analysis the product was judged to be about 90% in the beta crystalline form.

The heat treated flakes were then passed through a hammer mill and ground to a powder, about 0.3 pound of Dry Ice per pound of flakes being added with the flakes to absorb the heat developed in grinding and thus prevent melting the flakes and destroying the crystalline form produced in the solid phase transformation. Screen analyses showed that 94% of the powdered

material passed through a 60 mesh screen and that about 11% passed through a 200 mesh screen, about 70% being from about 100 to about 150 mesh in size.

Suspensions were then prepared by gently mixing 50% in one case and 60% in another case of this powder with 50% and 40% respectively of unhydrogenated refined and bleached peanut oil. All products possessed pumpable consistency as prepared and also possessed such consistency after storage for 10 days at 70–100° F. as indicated by the following penetration data. A comparable suspension formed from 60% of the powdered solid triglyceride in its original unconverted crystalline form (designated as 60% U in the table of penetration data) was pumpable immediately after preparation but became very firm and non-pumpable when stored for 10 days at 70–100° F.

	Samples stored 10 days at—								
	70° F.			90° F.			100° F.		
Percent solid triglyceride	50	60	60U	50	60	60U	50	60	60U
Penetration	>250	199	4	>250	111	2	>250	104	3

If desired the suspensions may be modified by the addition of insoluble non-triglyceride solid materials adapted to provide special effects, such as flavoring, keeping quality, or fortification of vitamin content, for example. Specifically, sugar or salt may be incorporated and suspended. For example, one of the important uses of suspensions of the character herein concerned is in the manufacture of peanut butter to inhibit oil separation during storage. As an example of this use 50 parts by weight of finely divided salt (sodium chloride finer than 60 mesh; between 100 and 200 mesh preferable) are added to 100 parts by weight of a 50-50 suspension of the powdered solid triglyceride in liquid triglyceride of Example I. The mixture remains as a uniform suspension throughout the normal range of room temperature and is adapted to be pumped by means of suitable proportioning pumps onto the peanuts in the peanut grinding mechanism. It has been found that such suspensions of salt and solid triglyceride in liquid triglyceride may contain as much as 70 per cent total solids without being too viscous for pumping and if the solid triglyceride constituent has been converted to the beta crystalline form, the suspension with salt will also have heat stability as shown by the following penetration data on a suspension of 50 parts of salt more than 50% of which was about 150 mesh or smaller in size, and 100 parts of a 50-50 mixture of the suspension of Example I.

	Samples stored 10 days at—		
	70° F.	90° F.	100° F.
Penetration	207	163	140
Above samples then stored at 70° F. for 1 day.			
Penetration	214	142	113
Above 70° F. samples then returned to original storage.			
Penetration	214	152	130

Comparable salt-containing mixtures, which had been made from a solid triglyceride that had not been heat treated to effect crystal conversion to the high melting beta phase but which initially were readily pumpable, became characteristically firm and substantially solid, showing penetrations of 8 and less after 6 days' storage at 90 and 100° F.

Example II.—Flakes of hydrogenated cottonseed oil (8 iodine value) and of hydrogenated soybean oil (8 iodine value), prepared by passing the liquid fats in liquid condition over a chilled flaking roll, were heat treated separately three days at 60° C., two hours at 49° C. and one hour at 38° C. After the flakes were cooled to room temperature they were separately pulverized to about 100–200 mesh by passage through a micropulverizer, about 30% carbon dioxide ice being employed as refrigerant. By examination of X-ray patterns the hydrogenated cottonseed oil was judged to be about 50% in the beta form and 50% in the beta-prime form and the hydrogenated soybean oil was judged to be 100% beta.

A mixture of 10% of the powdered cottonseed oil hard stock and 90% of the powdered soybean oil hard stock was then mixed with refined and filtered peanut oil and sodium chloride in the proportions of 1:1:1 by weight to form a suspension. This suspension was initially pumpable and as will be noted from the following penetration data it retained its pumpable consistency over a temperature range of 70–100° F.

Penetration after 1 day at—			Penetration after 1 week at—		
70° F.	90° F.	100° F.	70° F.	90° F.	100° F.
220	175	135	206	121	111

Example III.—50 parts by weight of the hydrogenated soybean oil and 50 parts by weight of the hydrogenated cottonseed oil used in Example II were melted together and solidified in flake form by passage over a refrigerated flaking roll. The flaked product was tempered 3 days at 60° C., 2 hours at 49° C. and one hour at 38° C., then cooled to room temperature. This material was then pulverized as in Example II to about 100–200 mesh. From the X-ray patterns it was judged that the crystalline form of the product was about 90% beta and 10% beta-prime.

A suspension of the powdered hard stock together with sodium chloride was made with refined and filtered peanut oil, the proportions being 1:1:1. The suspension as initially made was pumpable and, as indicated by the following penetration data, it retained its pumpable consistency over a temperature range of 70–100° F.

Penetration after 1 day at—			Penetration after 1 week at—		
70° F.	90° F.	100° F.	70° F.	90° F.	100° F.
361	305	271	390	287	277

Variations of details and limitations given in the above examples are permissible without departing from the spirit of the invention and the discussion that follows is devoted to a fuller explanation of the scope of applicants' contribution.

Instead of effecting conversion of the crystalline form of the hard fat by solid phase transformation as indicated in the above examples, the beta crystalline form may be obtained by melting the hard fat and permitting crystallization to take place at a temperature slightly above the beta-prime melting point. In the case of the hydrogenated peanut oil used in Example I for example, such crystallization temperature would be about 64° C. The resulting converted solid, preferably after cooling substantially to room temperature, may be ground to a powder and suspended in the manner indicated in the example.

Another way of obtaining the solid fat in the beta crystalline form is to dissolve it in 10 to 20 parts for example of a hydrocarbon fat solvent such as petroleum ether, hexane or the like, then chill the solution and permit the solid fat to crystallize. Beta crystals are formed without difficulty. The precipitated solid is thereafter separated from the mother liquor as by filtration and if residual solvent in the fat is undesired it may be removed by evaporation at a temperature below the melting point of the fat. Thereafter the solid fat is converted to suitable finely divided form and suspended in liquid fatty ester.

When crystallization in beta form by solid phase transformation is practiced as in the examples, the physical form (i. e. cake, flake, powder) in which the solid fat exists during the heating step is not critical and we do not wish to be limited in this respect. It will be apparent, however, that the rate of heat diffusion varies with particle size and that the time required to effect substantially complete conversion of the crystals to the beta form will accordingly increase with increase in the size of the particles of the solid material. Cakes of the solid material may be stored at a suitable temperature to effect conversion to the beta crystalline form, but of course a sufficiently long time of storage must be provided to permit the penetration of the heat to the center portions of the cake. Therefore we find it preferable to heat treat the solid material in particulate form, such as in flake, powder, or granular form, whereby the time of treatment at the conversion temperature may be reduced due to the more rapid diffusion of heat throughout the solid material as a whole. A particularly suitable practice is to spray or atomize the melted solid fat into a cool atmosphere to form a powder, then classify the powder according to particle size and segregate the desired suspendible portion, and subject this portion to solid phase crystal transformation to form beta crystals. In this practice caution should be observed to avoid the use of transformation temperatures at which substantial fusion of the powder and subsequent caking may result. It is also possible and entirely practical to obtain suitable conversion of the crystalline form by storage of flaked, granular, powdered or caked material in containers such as sacks in a tempering room controlled at a suitable temperature.

The temperature at which the solid material is preferably heated to effect solid phase conversion to the beta form will of course be dependent on the melting point of the crystalline form of the solid being treated. Solid phase transformation to the beta crystalline form takes place at a rate which increases with increase in temperature, the maximum rate of change occurring at or slightly below the melting point of the exist-

ing phase. In the event that the heat treatment is performed at the melting point of the existing phase (e. g. beta-prime), or in the event that the solid being treated is constituted of a mixture of glycerides as in a substantially completely hydrogenated natural glyceride, then some of the glycerides may exist at that temperature in the fluid state. With fluid present, caking may result on cooling, and in addition precautions should be taken during the cooling cycle to obtain beta crystals from the liquid as more fully pointed out below.

To avoid localized overheating and fusion of the solid, adequate control over the temperature of the heating medium should be exercised. Very good results have been obtained by having the solid material in flake or granular form and by forcing suitably heated air (e. g. at a temperature slightly below the melting point of the existing crystalline phase) through a layer of the material supported in a wire basket or on a suitable conveyor. This practice is especially adaptable for continuous operation wherein the solid is passed through suitably controlled heating and cooling zones.

The period of time required for crystal transformation is also variable depending on the temperature at which the transformation is permitted to take place and on the purity of the material being treated. The presence of unsaturated constituents, for example, has been found to retard the rate of transformation appreciably and whereas pure tristearin may transform from the beta-prime form to the beta form in a matter of seconds at the beta-prime melting point, a mixture of glycerides such as substantially completely hydrogenated peanut oil may require as much as 15 minutes heat treatment at the optimum temperature to obtain maximum conversion.

If the material being subjected to solid phase transformation is relatively pure and substantially no fusion of the solid material occurs during the heat treatment then, after the desired conversion has taken place, the material may be cooled without precaution. However, in the event that fused material is present it is essential to cool slowly to permit crystallization of the melted portion into its beta crystalline form. If the fused glyceride is cooled too rapidly, undesirable alpha or beta-prime crystals may be formed. In a practical application of the invention on substantially completely hydrogenated oils in which some melted glyceride exists at the optimum beta transformation temperature, the period in which the mass is cooled to a temperature below the alpha melting point is preferably extended over a period of about 30 minutes or more depending, for example, on the size of the batch and on the cooling facilities. After the temperature of the mass has passed below the alpha melting point, the rate of cooling is not critical.

In order to realize fully the improved results of our process a substantial amount of the fat should of course be converted to the beta crystalline form. We prefer to conduct the heat treatment so that at least 85 per cent of the solid material, as judged by the relative intensities of short spacing lines in the X-ray diffraction pattern, is so converted. Such high beta conversion is especially desirable in the preparation of pumpable compositions containing 50–60 per cent normally solid triglyceride. When compositions containing lower proportions of solid triglyceride, such as 25–30 per cent, are contemplated, the amount of convertion to the beta crystalline form may be somewhat lower, but preferably at least 70 per cent.

The term "crystallizing" as employed in claiming the process is intended to include within its scope any of the aforementioned or accepted methods of forming crystals, or of altering the form thereof as in solid phase transformation.

If the converted solid fat is not in form which may be directly suspended in the liquid fatty ester, it may be reduced to powder of suitable size to permit such suspension. A hammer mill has been found suitable to effect desired grinding of the tempered material to a fine powder although any other suitable grinding equipment may be used. However, care must be observed in this grinding operation, so as to avoid a development of heat of sufficient magnitude to cause the hard stock to melt. In order to avoid this, the grinding mechanism is preferably refrigerated or an inert volatile refrigerant such as Dry Ice may be added to the grinding mill with the tempered flakes and ground therewith.

Although the transformed solid material may be ground to the desired particle size in dry form, wet grinding may also be employed by admixing the solid material with the liquid fatty ester in suitable proportion and passing it through a wet grinding mill such as the ordinary type of ointment mill. The heat developed in the grinding operation is more readily dissipated when a liquid vehicle is present.

Within the reasonable limits herein contemplated the particle size of the converted solid fat in the suspension is not critical and has no significant effect on the consistency of the product. However, if the particles are coarse, e. g. larger than 60 mesh, the product, although more fluid, tends to be grainy in appearance and may be subject to separation of solid and liquid by gravity. If, on the other hand, the particle size of the solid triglyceride is very small, such as about 350 mesh, then the suspensions may be unduly viscous, especially if the solid triglyceride is present in the higher range of proportions herein contemplated. The terms "finely divided" and "powder" have been chosen to define a particle size from about 60 mesh to about 350 mesh and these terms are to be interpreted accordingly in the claims. A preferable degree of subdivision is from about 60 mesh to about 200 mesh.

In the event the material is prepared in the desired particle size in dry form, it may then be added to the liquid fatty ester and incorporated therewith in any suitable manner. It is preferable to add the powdered solid to the liquid and stir gently to avoid lump formation and excess incorporation of air which tends to stiffen the product. However, amounts of air as high as 15 per cent have been found to be not seriously harmful. In fact, the presence of some air or other inert gas such as nitrogen or carbon dioxide in the product has the advantage of rendering the product more stable with regard to pumpable consistency in the ordinary range of room temperature and of giving it a whiter appearance. However, a governing factor relative to incorporation of gases, aside from the tendency of the gases to stiffen the oleaginous product, is the ultimate use to which the product is to be put and it is to be borne in mind that any free oxygen may tend to cause rancidity or off-flavor where unsaturated and oxidizable materials are present.

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

1. A process of preparing a uniform suspension of finely divided, high melting, normally solid triglyceride fat in liquid fatty ester, said suspension having pumpable and flowable consistency throughout a temperature range of 70° to 100° F., a penetration of at least 100 and a viscosity of not substantially more than 300,000 centipoises, which comprises the steps of crystallizing at least 70% of a normally solid, high melting triglyceride fat in the beta crystalline form, thereafter admixing the thus crystallized and separately formed solid triglyceride fat with liquid fatty ester to form a suspension in which the solid fat constitutes from about 10% to about 60% by weight of the fatty mixture, and at any stage in the process subdividing the solid triglyceride fat to a particle size from about 60 mesh to about 350 mesh.

2. A process of preparing a uniform suspension of finely divided, high melting, normally solid triglyceride fat in liquid fatty ester, said suspension having pumpable and flowable consistency throughout a temperature range of 70° to 100° F., a penetration of at least 100 and a viscosity of not substantially more than 300,000 centipoises, which comprises the steps of admixing a separately formed, normally solid, high melting triglyceride fat, of which at least 70% is in the beta crystalline form, with liquid fatty ester to form a suspension in which the solid fat constitutes from about 10% to about 60% by weight of the fatty mixture, and at any stage in the process subdividing the high melting solid triglyceride fat to a particle size from about 60 mesh to about 350 mesh.

3. A process of preparing a uniform suspension of finely divided, high melting, normally solid triglyceride fat in liquid fatty ester, said suspension having pumpable and flowable consistency throughout a temperature range of 70° to 100° F., a penetration of at least 100 and a viscosity of not substantially more than 300,000 centipoises, which comprises the steps of heating a normally solid, high melting triglyceride fat to a temperature below its melting point at which solid phase transformation to the beta crystalline form occurs until conversion of at least 70% of the solid fat to the beta crystalline form is effected, thereafter admixing the thus crystallized and separately formed solid triglyceride fat with liquid fatty ester to form a suspension in which the solid fat constitutes from about 10% to about 60% by weight of the fatty mixture, and at any stage in the process subdividing the solid triglyceride fat to a particle size from about 60 mesh to about 350 mesh.

4. The process of claim 1 in which the solid fat is subdivided to a particle size from about 60 mesh to about 200 mesh.

5. The process of claim 3 in which the solid fat is subdivided prior to conversion thereof to the beta crystalline form.

6. The process of claim 3 in which at least 85% of the solid fat is converted to beta crystalline form.

7. The process of claim 3 in which the solid fat is subdivided after conversion of the same to the beta crystalline form and is cooled during the step of subdivision.

8. The process of claim 3 in which the liquid fatty ester is triglyceride oil.

9. A process of preparing a uniform suspension of sodium chloride and finely divided, high melting, normally solid triglyceride fat in liquid triglyceride fat, which comprises heating hydrogenated peanut oil having an iodine value not substantially greater than 20, and being predominantly in beta prime crystalline form, to a temperature below its beta prime melting point, thereby transforming at least 85% of the crystals to beta crystalline form, reducing the transformed hydrogenated peanut oil to suspendible form from about 60 to about 200 mesh in size, and admixing the separately formed and subdivided solid triglyceride and sodium chloride with liquid peanut oil to form a suspension in which the solid fat constitutes from about 10% to about 60% by weight of the fatty mixture.

10. The process of claim 9 in which the relative proportions of hydrogenated peanut oil to liquid peanut oil to sodium chloride are about 1:1:1.

11. A process of preparing a uniform suspension of finely divided, high melting, normally solid triglyceride fat in liquid fatty ester, said suspension having pumpable and flowable consistency throughout a temperature range of 70° to 100° F., a penetration of at least 100 and a viscosity of not substantially more than 300,000 centipoises, which comprises the steps of heating a normally solid, high melting triglyceride fat to a temperature below its melting point at which solid phase transformation to the beta crystalline form occurs until conversion of at least 85% of the solid fat to the beta crystalline form is effected, reducing the converted fat to suspendible particles from about 60 to about 200 mesh in size, and thereafter admixing the separately converted and reduced solid triglyceride fat with liquid fat ester to form a suspension in which the solid fat constitutes from about 10% to about 60% by weight of the fatty mixture.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,395,934	Stockton	Nov. 1, 1921
1,911,222	Bollens et al.	May 30, 1933
2,442,532	Eckey	June 1, 1948