

PRODUCTION METHOD FOR FRACTIONATED SAL FAT

Technical field

The present invention relates to a production method
5 for a fractionated sal fat which is usable as a cocoa
butter substitute, particularly, to a production method for
a fractionated sal fat usable as a high quality cocoa
butter substitute from a low quality crude sal fat with
quality deterioration.

10

Background Art

Traditionally, as a fat and oil usable as an
alternative for cocoa butter which is a representative fat
and oil for chocolate, a cocoa butter substitute which is a
15 fat and oil mainly containing symmetrical triglycerides,
1,3-disaturated-2-unsaturated triglyceride (SUS,S:
saturated fatty acid, U: unsaturated fatty acid), has been
used. Representative examples of the saturated fatty acids
constituting the symmetrical triglyceride include palmitic
20 acid (P), stearic acid (St), and arachidic acid (A), and
representative examples of the unsaturated fatty acids
constituting the symmetrical triglycerides include oleic
acid (O). POP is obtained mainly from fractionated fat of
Palm oil or Chinese tallow, and POST is obtained from
25 illipe butter, mowrah butter, and the like and fractionated

fat thereof. StOSt is obtained from kokum fat, mango kernel fat, shea butter, Sal fat, and fractionated fat thereof.

5 Since sal fat contains a large amount of SOS (main components are StOSt and AOSt), a refined sal fat or a fractionated sal fat is one of increasingly important raw materials for the manufacture of cocoa butter substitutes.

10 Sal fat, which is also called as sal seed fat, is a fat extracted from seeds of shrubs, naturally grown Shorea robusta, by mainly using hexane and is one kind of a group of fats that are collectively called as Borneo tallow fats. Since the fat in Shorea robusta seed is easily hydrolyzed with a hydrolyzing enzyme, it is desirable to extract the fat immediately after collection of the seeds. However, in 15 actuality, it is not necessarily possible to immediately extract the fat due to working conditions and transportation conditions at the collection area. Therefore, a so-called low quality crude fat in which 20 hydrolysis has proceeded to an excessive degree is often found in obtained crude sal fats. Meanwhile, it has been known that triglycerides containing dihydroxystearic acid as a constituent fatty acid (hereinafter referred to as DHS-TG) and triglycerides containing epoxystearic acid as a 25 constituent fatty acid (hereinafter referred to as EP-TG)

exist in sal fat, and that a particularly large amount of DHS-TG is contained in the sal fat. Since the low quality crude fat contains 2 to 10 wt% of DHS-TG, and since even a refined sal fat and a conventional fractionated sal fat
5 ordinarily contain 1.0 wt% or more of DHS-TG, these fats are not suitable for the manufacture of a good quality cocoa butter substitute. More specifically, an increase in viscosity of a chocolate mass after tempering is caused during production of chocolates, leading to difficulty in
10 performing tempering and poor demolding after molding and subsequent cooling. As a result, there are great problems that the chocolate does not have a good gloss and a good melting speed in the mouth.

The following attempts have been conducted in the aim
15 of obtaining a high quality cocoa butter substitute by removing DHS-TG described above from a low quality crude sal fat.

J. Am. Oil. Chem. Soc. 62(7), 1126 (1985) relates to a method of removing DHS-TG and diglycerides (hereinafter
20 referred to as DG) by way of dry fractionation of sal fat or fractionation of sal fat with acetone as a polar solvent or a method of obtaining a sal fat fraction containing DHS-TG and DG with high concentration. A method of reducing DHS-TG and DG in a crystal part by way of fractionation
25 using acetone as a polar solvent and a method of reducing

DHS-TG and DG by absorbing them with a silica gel column are described. However, the former method is not effective enough to remove DHS-TG, and the latter method is not practical because an expensive silica gel column is
5 necessary and a large amount of a strong polar solvent is necessary in order to restore the silica gel column.

JP 53-111307 A describes such a method for removing DHS-TG and EP-TG that a refined sal fat by degumming and alkali deacidification is fractionated by using either
10 hexane as a nonpolar solvent or an aqueous detergent solution. However, the method merely enables to obtain a fractionated sal fat having a low SUS content.

JP 56-58444 A relates to hard butter comprising a sal fat fraction which is obtained by fractionating sal fat and
15 which has a SUS content of 80 to 90 wt%. In the method, fractionation is carried out by using a solvent such as hexane, and the solution is once relatively mildly cooled to remove precipitated crystals of a high melting part, followed by cooling again to low temperature, thereby
20 collecting precipitated crystals of a mid melting point fraction. However, the method has a problem of insufficient removal of DHS-TG because DHS-TG remaining after the first stage fractionation is crystallized together with the mid melting part by the second stage
25 fractionation in the method.

JP 2001-55598 A describes a method for removing DHS-TG by subjecting sal fat to a first stage fractionation by either dry fractionation or fractionation with hexane as a nonpolar solvent and subsequently subjecting the liquid part obtained in the first stage fractionation to a second stage fractionation using a polar solvent that is in an amount of 100 to 900 wt% with respect to 100 wt% of the liquid part. However, the method has a problem in terms of production efficiency since sal fat is subjected to solvent fractionation with a polar solvent after either dry fractionation or fractionation with hexane as a nonpolar solvent, and therefore, two types of fractionation equipments are required for both the dry fractionation and the solvent fractionation with a polar solvent or two types of solvents, both a nonpolar solvent and a polar solvent, are required.

Though the reduction method of DHS-TG is described in each of JP 53-111307 A, JP 56-58444 A and JP 2001-55598 A, none of which refers to reductions of diglycerides (hereinafter referred to as DG) and an asymmetrical triglycerides fraction (hereinafter referred to as SSU) that also influence on the quality of cocoa butter substitutes.

JP 56-58451 A discloses a cocoa butter substitute in which a SSU fraction which is one of tempering inhibiting

components is reduced, and it is also important to reduce the SSU fraction in order to obtain a high quality cocoa butter substitute.

From the above publications, it is understood that it is necessary to reduce not only DHS-TG but also DG and SSU fraction together with DHS-TG in order to obtain a high quality fractionated sal fat. However, conventional methods have their limits in reducing the components to an acceptable level so that the fractionated sal fat of good quality is obtained from a low quality sal fat efficiently.

Disclosure of the Invention

Problems to be Solved by the Invention

Thus, the objectives of the present invention are to provide a production method of a fractionated sal fat which can be used as a high quality cocoa butter substitute having sufficiently reduced DHS-TG, DG, and SSU fraction and being rich in SUS content from a low quality sal fat as a raw material, as well as to provide the fractionated sal fat of good quality.

Means for Solving the Problems

The present inventors have intensively studied on a method for reducing DHS-TG together with DG and SSU fraction and have found that it is possible to remove or reduce DHS-TG, DG, and SSU fraction and obtain a crystal

part being rich in SUS content when a total amount of the polar solvent used in a crystallization step for crystallization and a crystal part washing step carried out after the crystallization and filtration is 1000 wt% or
5 more with respect to 100wt% of the refined sal fat, and thereby the desired high quality cocoa butter substitute can be obtained. Thus, the present invention has been completed.

The present invention has been accomplished based on
10 the above-described findings, and as a first aspect, the present invention provides a production method of a fractionated sal fat, comprising preparing a refined sal fat and carrying out crystallization and washing of the crystal part by using a polar solvent to obtain the crystal
15 part as a hard butter component, wherein a total amount of the polar solvent used for the crystallization and the washing is 1000 wt% or more with respect to 100 wt% of the refined sal fat. As a second aspect, the present invention provides the production method of the fractionated sal fat
20 according to the first aspect, wherein, in the hard butter component, a content of triglycerides containing dihydroxystearic acid as a constituent fatty acid (DHS-TG) is 0.4 wt% or less; a content of diglycerides (DG) is 0.5 wt% or less; and a content of 1,2-disaturated-3-unsaturated
25 triglycerides (SSU) fraction is 2 wt% or less. As a third

aspect, the present invention provides the production method of the fractionated sal fat according to the first or second aspect, wherein, in the hard butter component, a content of 1,3-disaturated-2-unsaturated triglycerides

5 (SUS) is 85 wt% or more. As a fourth aspect, the present invention provides the production method of the fractionated sal fat according to any one of the first to third aspects, wherein the crystal part obtained by fractionation using the polar solvent is subjected to re-
10 fractionation using the polar solvent to obtain a crystal part as a hard butter component.

Effects of the Invention

It is difficult to stably obtain a cocoa butter
15 substitute from the sal fat which has unstable quality. Particularly, a low quality sal fat contains large amounts of triglycerides containing dihydroxystearic acid as a constituent fatty acid and diglycerides that exerts adverse influence on quality, especially on a crystallization
20 property, of the cocoa butter substitute.

The present invention provides a method for obtaining a high quality cocoa butter substitute using a low quality sal fat as a raw material, and the method enables to stably use sal fat which is a crude fat having unstable quality as
25 a raw material for the cocoa butter substitute.

Best Mode for Carrying Out the Invention

Hereinafter the production method of a fractionated sal fat of the present invention will be described. The
5 production method of the fractionated sal fat of the present invention is characterized in that a total amount of the polar solvent used in a crystallization step for crystallization and a crystal part washing step performed after the crystallization and filtration to 1000 wt% or
10 more with respect to 100 wt% of the refined sal fat prepared from a crude fat extracted from seeds of Shorea robusta and fractionating the refined sal fat by using the polar solvent, and thereby DHS-TG, DG, and SSU fraction are reduced and a crystal part having SUS content of 85 wt% or
15 more is obtained.

The method for preparing refined sal fat from a crude sal fat can be such an ordinary method that the crude sal fat is step by step subjected to degumming by adding
phosphoric acid in a preferable amount of 0.1 to 0.4 wt% to
20 the crude sal fat, alkali deacidification, washing with hot water after deacidification, dehydration under reduced pressure, bleaching, and deodorization. It is possible to omit a part of the steps depending on quality of the crude fat, but degumming, alkali deacidification, hot water
25 washing, and dehydration under reduced pressure are

indispensable in the case of a low quality crude sal fat, since DG, EP-TG, and DHS-TG contained in the crude fat are partially reduced by performing the steps. When these steps are not carried out, contents of DG, EP-TG, and DHS-TG are too large, and it is difficult to reduce the components to the acceptable ranges by subsequent solvent fractionation.

In addition, deodorization conditions in the deodorization step are important. A preferable deodorization temperature is 230°C to 260°C, preferably 230°C to 250°C, most preferably 230°C to 240°C, and a preferable vacuum degree upon deodorization is 5 Torr or less, preferably 2 Torr or less. The deodorization temperature of more than 250°C is undesirable because SUS fraction tends to be partially isomerized to generate the SSU fraction during the deodorization step. Also, the deodorization temperature of less than 230°C and the vacuum degree of more than 5 Torr are undesirable because removal of free fatty acids in fat becomes insufficient.

The low quality crude sal fat containing 2 to 10 wt% of DHS-TG can not be acceptable raw material for a hard butter component with good quality because 1 to 4 wt% of DHS-TG remains in the refined sal fat prepared by the above-described methods, and the contents of DG and SSU fraction in the refined sal fat are also out of the

acceptable level. In such a case, it is necessary to carry out solvent fractionation by the method of the present invention described below.

To the refined sal fat which has been completely
5 molten by heating to 50°C to 70°C, a polar solvent in an amount of 400 wt% or more, preferably 400 to 3000 wt% with respect to 100 wt% of the fat, is mixed, followed by gradual cooling to 0°C to 20°C with stirring in a crystallization tank equipped with a cooling device with a
10 coolant and a stirring device. A crystallization time required is about 0.5 to 3 hours though it is varied depending on a coolant temperature and a kind of polar solvent. After completing crystallization, a crystal part and a filtrate part are separated from each other by
15 filtration under reduced pressure. The separated crystal part is washed by mixing with a fresh polar solvent which has been previously cooled to a temperature ranging from the crystallization temperature to 10°C lower than the crystallization temperature and which is in an amount that
20 a total amount of the polar solvent used in the crystallization step and the washing step becomes 1000 wt% or more, i.e., preferably 0 to 2600 wt%, with respect to 100 wt% of the refined sal fat, and then separating into a crystal part and a filtrate part again by filtration. In
25 the thus obtained crystal part, DHS-TS, DG, and SSU

fraction are reduced, and SUS component is concentrated.

The total amount of the polar solvent used for the crystallization and the washing is 1000 wt% or more, preferably 1000 to 3000 wt%, most preferably 1100 to 2100

5 wt%. When the total amount is less than the lower limit, the reductions of DHS-TG, DG, and SSU fraction are undesirably insufficient. When the total amount exceeds the upper limit, production efficiency of the fractionation is undesirably reduced due to too large amount of the polar
10 solvent to be used, and an evaporation load undesirably becomes too large in a solvent removing step after the fractionation.

The following two methods 1) and 2), in a broad sense, are applicable as a fractionation method for a refined sal
15 fat in which a total amount of the polar solvent is set to 1000 wt% or more with respect to 100 wt% of the refined sal fat. The former method is suitably employed for a more efficient reduction of DHS-TG, DG, and SSU fraction.

1) To 100 wt% of the refined sal fat that has been
20 completely molten by heating to 50°C to 70°C, 400 to 900 wt% of the polar solvent is mixed, the mixture is cooled to 0°C to 20°C with stirring in a crystallization tank equipped with a cooling device using a coolant and a stirring device. After completing the crystallization, a
25 crystal part and a filtrate part are separated from each

other by filtration under reduced pressure or the like.

The separated crystal part is washed by mixing with a fresh polar solvent which has been previously cooled to a temperature ranging from the crystallization temperature to

5 10°C lower than the crystallization temperature and which is in an amount of 100 to 600 wt% with respect to 100 wt% of the refined sal fat, and then separating into a crystal part and a filtrate part again by filtration. In a crystal fraction obtained by removing the solvent from the thus-
10 obtained crystal part by an ordinary method, DHS-TS, DG, and SSU fraction are partially reduced. However, re-fractionation of the crystal fraction is carried out because the reductions of these components are insufficient at this stage in the case of the fractionation of a refined
15 sal fat prepared from a low quality crude sal fat.

After the crystal fraction obtained as described above is completely molten by heating to 50°C to 70°C, the polar solvent in an amount of 400 to 900 wt% with respect to 100 wt% of the crystal fraction is mixed, the mixture is
20 gradually cooled to 0°C to 20°C with stirring in a crystallization tank equipped with a cooling device using a coolant and a stirring device. After completing the crystallization, a crystal part and a filtrate part are separated from each other by filtration under a reduced
25 pressure or the like. The separated crystal part is washed

by mixing with a fresh polar solvent which has been previously cooled to a temperature ranging from the crystallization temperature to 10°C lower than the crystallization temperature and which is in an amount of 100 to 600 wt% with respect to 100 wt% of the refined sal fat, and then a crystal part and a filtrate part are again separated from each other by filtration under a reduced pressure or the like. In the crystal part obtained by the re-fractionation, DHS-TS, DG, and SSU fraction are further reduced, and the SUS component is further concentrated. Therefore, the crystal part is usable as a high quality cocoa butter substitute after ordinary solvent removal, bleaching and deodorization. The crystal part subjected to the re-fractionation can be a crystal part that is obtained by the first stage fractionation and contains the polar solvent, and, in such a case, the crystal part is completely molten by heating to 30°C to 50°C, followed by mixing with the fresh polar solvent and fractionation.

It is important that the total amount of the polar solvent used in the above-described two-stage fractionation is 1000 wt% or more with respect to 100 wt% of the refined sal fat for reducing DHS-TG, DG, and SSU fraction to the acceptable level.

Apart from the above-described two-stage fractionation method, the following procedure can be applied in the case

of a one-stage fractionation method.

2) To 100wt% of the refined sal fat that has been completely molten by heating to 50°C to 70°C, 400 to 3000 wt% of the polar solvent is mixed, the mixture is gradually cooled to 0°C to 20°C with stirring in a crystallization tank equipped with a cooling device using a coolant and a stirring device. After completing the crystallization, a crystal part and a filtrate part are separated from each other by filtration under reduced pressure or the like.

The separated crystal part is washed by mixing with a fresh polar solvent which has been previously cooled to a temperature ranging from the crystallization temperature to 10°C lower than the crystallization temperature and which is in an amount of 0 to 2600 wt% with respect to 100 wt% of the refined sal fat, and then separating into a crystal part and a filtrate part again by filtration. It is necessary that a total amount of the polar solvent used for the crystallization and the washing of the crystal part is also 1000 wt% or more with respect to 100 wt% of the refined sal fat. In a crystal fraction that is obtained from the thus-obtained crystal part by ordinary solvent removal, bleaching and deodorization, DHS-TS, DG, and SSU fraction are sufficiently reduced, and SUS component is sufficiently concentrated. Therefore, the crystal fraction is usable as a high quality cocoa butter substitute.

Examples of the polar solvent to be used in the present invention include acetone, metylethylketone, diethylketone, ethyl alcohol, n-butyl alcohol, and the like. Especially, acetone is suitably used. Desired water content in the polar solvent is 2 wt% or less, preferably 1 wt% or less. It is not preferred that the water content exceeds 2 wt% because solubility of the fat and oil is decreased to make it difficult to increase a fat and oil content with respect to the solvent.

The fractionated sal fat of the present invention obtained by fractionation using 1000 wt% or more of the polar solvent with respect to 100 wt% of the refined sal fat preferably have a DHS-TG content of 0.4 wt% or less, a DG content of 0.5 wt% or less, and SSU faction content of 2 wt% or less.

The DHS-TG content of the fractionated sal fat obtained by the present invention is 0.4 wt% or less as described above, preferably 0.3 wt% or less, more preferably 0.2 wt% or less. It is not preferable that a content of DHS-TG exceeds 0.4 wt% because an increase in viscosity of a chocolate mass after tempering becomes significant during tempering process which make chocolate molding and subsequent demolding after cooling difficult in the case of being used for chocolate.

DHS-TG contained in the fractionated sal fat obtained

by the present invention includes three components of 9,10-dihydroxystearoyl-distearin (StDSt), 9,10-dihydroxystearoyl-stearoyl-arachidin (StDA), 9,10-dihydroxystearoyl-stearoyl-palmitin (StDP), and a total
5 amount of the three components is 0.4 wt% or less. It is preferable that: an StDSt content is 0.2 wt% or less; an StDA content is 0.1 wt% or less; and an StDP content is 0.1 wt% or less.

Also, the DG content is more preferably 0.2 wt% or
10 less, and the DG content exceeding 0.5 wt% is not preferable because an increase in viscosity of a tempered chocolate mass becomes significant during chocolate tempering process which make chocolate molding and subsequent demolding after cooling difficult.

15 Further, the SSU fraction content exceeding 2 wt% is not preferable because crystallization properties of the fat and oil is likewise so deteriorated that chocolate tempering workability becomes poor, thereby causing drops of heat resistance , shape keeping property, snappiness,
20 and melting speed in the mouth.

An SUS content of the fractionated sal fat of the present invention obtained by fractionation using 1000 wt% or more of the polar solvent with respect to 100 wt% of the refined sal fat is preferably 85 wt% or more. It is not
25 preferable that the SUS content is less than 85 wt% because

snappiness, heat resistance and shape keeping property of chocolate obtained by using the fractionated sal fat are not good enough, and sharp melting speed in the mouth is not achieved.

5 In the case of using the fractionated sal fat obtained by the present invention as a cocoa butter substitute, the fractionated sal fat can be used alone as a cocoa butter substitute, and fats and oils such as cocoa butter, palm oil, illipe butter, shea butter, mango kernel fat,
10 fractionated oils thereof, and interesterified oils can be mixed with the fractionated sal fat of the present invention.

 The fractionated sal fat of the present invention is usable as fats for confectionaries, breads, solid curry
15 sauces, deep frying, and the like in addition to use for chocolate.

Examples

 Hereinafter, the present invention will be described
20 in detail in conjunction with examples and comparative examples. In the following examples, "%" means "wt%" unless otherwise described.

<Example 1>

 A crude sal fat (containing 5.2% of DHS-TG) obtained
25 from seeds of Shorea robusta was subjected to degumming by

adding 0.2% of phosphoric acid to the crude sal fat. The degummed fat was subjected to alkali deacidification by an ordinary method, followed by bleaching treatment using bleaching earth and deodorization treatment according to an ordinary method (deodorization temperature: 250°C to 260°C, vacuum degree: 2 Torr or less), thereby obtaining a refined sal fat. As a result of analysis using high performance liquid chromatography (HPLC), the obtained refined sal fat contained 2.3% of DHS-TG, 3.3% of DG, and 62.0% of SUS fraction. As a result of analysis with a thin layer chromatography (TLC) developing method, SSU fraction content was 3.8%.

The refined sal fat was completely molten by heating to 60°C, and acetone (water content: 0.9%) in an amount of 567 wt% with respect to 100 wt% of the refined sal fat was mixed. After the mixture liquid was adjusted to 40°C, the mixture liquid was cooled to 13°C at a cooling rate of 2°C/min with stirring, followed by crystallization with stirring at 13±0.5°C for 0.5 hours. The mixture was separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The crystal part was washed with fresh acetone that was cooled to 10°C and in an amount of 200 wt% with respect to 100 wt% of the refined sal fat, and the mixture was again separated into a crystal part and a filtrate part by vacuum filtration under reduced

pressure. The filtrate part was added to the formerly separated filtrate part. The obtained crystal part and the filtrate part were subjected to solvent removal. A yield of a crystal fraction obtained from the crystal part was 62.8%, and a yield of a filtrate fraction obtained from the filtrate part was 37.2%. As a result of analysis by HPLC, the crystal fraction contained 1.8% of DHS-TG (StDSt: 1.0%, StDA: 0.5%, StDP: 0.3%), 1.3% of DG, and 82.6% of SUS fraction. As a result of analysis by a TLC developing method, SSU fraction content was 2.2%.

Subsequently, the crystal fraction obtained as described above was completely molten by heating to 60°C, and 567 wt% with respect to 100 wt% of the crystal fraction (356 wt% with respect to 100 wt% of refined sal fat) of acetone (water content: 0.9%) was mixed with the crystal fraction. After the mixture liquid was adjusted to 40°C, the mixture liquid was cooled to 18°C at a cooling rate of 2°C/min with stirring, followed by crystallization with stirring at 18±0.5°C for 0.5 hours. The mixture was separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The crystal part was washed with fresh acetone that was cooled to 15°C and in an amount of 200 wt% with respect to 100 wt% of the crystal fraction (126 wt% with respect to 100 wt% of refined sal fat), and the mixture was again separated into a crystal

part and a filtrate part by vacuum filtration under reduced pressure. The filtrate part was added to the formerly separated filtrate part. The obtained crystal part and the filtrate part were subjected to solvent removal. A yield
5 of a crystal fraction obtained from the crystal part was 42.7% with respect to the refined sal fat, and a yield of a filtrate fraction obtained from the filtrate part was 20.1% with respect to the refined sal fat.

As a result of analysis by HPLC, the fractionated sal
10 fat obtained by the re-fractionation contained 0.2% of DHS-TG (StDSt: 0.2%, StDA: 0.0%, StDP: 0.0%), 0.0% of DG, and 93.2% of SUS fraction. As a result of analysis by a TLC developing method, SSU fraction content was 1.4%.

<Example 2>

15 The refined sal fat prepared in Example 1 was completely molten by heating to 60°C, and acetone (water content: 0.9%) in amount of 567 wt% with respect to 100 wt% of the refined sal fat was mixed. After the mixture liquid was adjusted to 40°C, the mixture liquid was cooled to 13°C
20 at a cooling rate of 2°C/min with stirring, followed by crystallization with stirring at 13±0.5°C for 0.5 hours. The mixture was separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The crystal part was washed with fresh acetone that was
25 cooled to 13°C and in an amount of 767 wt% with respect to

100wt% of the refined sal fat, and the mixture was again separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The filtrate part was added to the formerly separated filtrate part. The obtained
5 crystal part and the filtrate part were subjected to solvent removal. A yield of the crystal part was 43.2%, and a yield of the filtrate part was 56.8%.

As a result of analysis by HPLC, the obtained fractionated sal fat contained 0.3% of DHS-TG (StDSt: 0.2%,
10 StDA: 0.0%, StDP: 0.1%), 0.0% of DG, and 92.5% of SUS fraction. As a result of analysis by a TLC developing method, SSU fraction content was 1.8%.

<Example 3>

The refined sal fat prepared in Example 1 was
15 completely molten by heating to 60°C, and acetone (water content: 0.9%) in an amount of 1233 wt% with respect to 100 wt% of the refined sal fat was mixed. After the mixture liquid was adjusted to 40°C, the mixture liquid was cooled to 10°C at a cooling rate of 2°C/min with stirring,
20 followed by crystallization with stirring at 10±0.5°C for 0.5 hours. The mixture was separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The crystal part was washed with fresh acetone that was cooled to 7°C and in an amount of 200 wt% with
25 respect to 100% of the refined sal fat, and the mixture was

again separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The filtrate part was added to the formerly separated filtrate part.

The obtained crystal part and the filtrate part were

5 subjected to solvent removal. A yield of the crystal part was 42.2%, and a yield of the filtrate part was 57.8%.

As a result of analysis by HPLC, the obtained fractionated sal fat contained 0.3% of DHS-TG (StDSt: 0.2%, StDA: 0.0%, StDP: 0.1%), 0.0% of DG, and 92.8% of SUS
10 fraction. As a result of analysis by a TLC developing method, SSU fraction content was 1.7%.

<Comparative Example 1>

The refined sal fat prepared in Example 1 was completely molten by heating to 60°C, and acetone (water
15 content: 0.9%) in an amount of 567 wt% with respect to 100 wt% of the refined sal fat was mixed. After the mixture liquid was adjusted to 40°C, the mixture liquid was cooled to 16°C at a cooling rate of 2°C/min with stirring, followed by crystallization with stirring at 16±0.5°C for
20 0.5 hours. The mixture was separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The crystal part was washed with fresh acetone that was cooled to 13°C and in an amount of 200 wt% (twice as much as the refined sal fat parts by weight) with
25 respect to 100 wt% of the refined fat, and the mixture was

again separated into a crystal part and a filtrate part by vacuum filtration under reduced pressure. The filtrate part was added to the formerly separated filtrate part. The obtained crystal part and the filtrate part were subjected to solvent removal. A yield of the crystal part was 48.0%, and a yield of the filtrate part was 52.0%.

As a result of analysis by HPLC, the obtained fractionated sal fat contained 1.2% of DHS-TG (StDSt: 0.6%, StDA: 0.3%, StDP: 0.3%), 0.9% of DG, and 83.2% of SUS fraction. As a result of analysis by a TLC developing method, SSU fraction content was 2.3%.

Analysis results of DHS-TG contents, StDSt contents, StDA contents, StDP contents, DG contents, SSU fraction contents, and solid fat contents of the purified sal fat and the fractionated sal fats of Example 1 and Comparative Example 1 are shown in Table 1. Measurement methods of the above contents are as follows.

<Measurement Methods>

(1) DHS-TG contents, StDSt contents, StDA contents, StDP contents, and DG contents: Measured by HPLC (column: tandem columns with ODS-A and Lichrosorb; elution liquid: acetone/acetonitrile = 80/20; flow rate: 0.9 ml/min; column temperature: 25°C; detector: differential refractometer).

(2) SSU fraction content: Measured by a TLC developing method (TLC plate: HPTLC-Kieselgel 160 F254; developing

solution: benzene/hexane/diethylether = 75/25/2; coloring: bromine to sulfuryl chloride). The SSU fraction content was calculated by the following expression:

$$\text{SSU fraction content (\%)} = \left\{ \frac{\text{SSU fraction content}}{\text{SSU fraction content} + \text{SUS fraction content}} \right\} \times 100.$$

(3) Solid fat content: Measured according to the solid fat content NMR method of the JOCS Standard Methods for the Analysis of Fats and Oils, provisional method 1-1996.

Aging was performed at 26°C for 40 hours.

10 (4) Melting point: Measured according to the JOCS Standard Methods for the Analysis of Fats and Oils 2.2.4.3-1996 softening point (Ring and Ball Method). Aging was performed at 25°C for 24 hours.

<Table 1>

	Refined sal fat	Example 1	Example 2	Example 3	Comparative Example 1
Amount of polar solvent to be used (wt%)	-	567	567	1233	567
Crystallization at first stage	-	200	767	200	200
Washing at first stage	-	*567	-	-	-
Crystallization at second stage	-	*200	-	-	-
Washing at second stage	-	-	-	-	-
Total amount of polar solvent to be used (wt%)	-	1249	1334	1433	767
DHS-TG content (wt%)	2.3	0.2	0.3	0.3	1.2
StDSt content (wt%)	1.0	0.2	0.2	0.2	0.6
StDA content (wt%)	0.7	0.0	0	0.0	0.3
StDP content (wt%)	0.6	0.0	0.1	0.1	0.3
SUS content (wt%)	62	93.2	92.5	93.8	83.2
DG content (wt%)	3.3	0.0	0.0	0.0	0.9
SSU fraction content (wt%)	3.8	1.4	1.8	1.7	2.3
Solid fat content (%); at 26°C for 40 hours					
30°C	-	70.5	70	71.2	41.6
35°C	-	4.1	3.8	4.8	5.5
40°C	-	0.9	0.8	1	1.7
Melting point (°C); at 25°C for 24 hours	-	34.7	34.6	34.8	33.5

Note) amount of solvent to be used: wt% with respect to 100 wt% of the refined sal fat

* Amounts of the solvents used for the crystallization and the washing at second stage were with respect to 100 wt% of the crystal part obtained by the first stage, which are equivalent to 356% and 126% with respect to 100 wt% of the refined sal fat, respectively.

As shown in Table 1, Examples 1 to 3, in each of which a total amount of the polar solvent to be used for fractionation was 1200 to 1500 wt%, showed that the DHS-TG content (wt%), the DG content (wt%), and SSU fraction content (wt%) were lower than those of Comparative Example 1, on the contrary SUS content was higher than that of Comparative Example 1. Therefore, as compared to Comparative Example 1, Examples 1 to 3 had higher solid fat contents and melting points, thereby clearly proving the usability as a high quality cocoa butter substitute. In Comparative Example 1, DHS-TG, DG, and the SSU fraction are not satisfactorily reduced, and the SUS content is a little lower, so that Comparative Example 1 was not usable as a good quality cocoa butter substitute.

<Example 4>

A cocoa butter substitute which was approximately equivalent to cocoa butter in physical properties was prepared by mixing 47 parts by weight of the fractionated sal fat obtained in Example 1 and 53 parts by weight of palm mid fraction (iodine value: 34.5; melting point: 31.6° C; POP content: 68.0%) prepared by an ordinary method, and functions as cocoa butter substitute was evaluated by measuring cooling curve, solid fat content, and melting point.

<Comparative Example 2>

A cocoa butter substitute which was approximately equivalent to cocoa butter in physical properties was prepared by mixing 47 parts by weight of the fractionated sal fat obtained in Comparative Example 1 and 53 parts by weight of palm mid fraction (iodine value: 34.5; melting point: 31.6°C; POP content: 68.0%) prepared by an ordinary method, and functions as cocoa butter substitute was evaluated by measuring cooling curve, solid fat content, and melting point.

Results of measuring cooling curves, the solid fat contents, and the melting points of Example 4 and Comparative Example 2 are shown in Table 2. The measurement methods are as described below.

<Measurement Methods>

(1) Cooling curve: Measured according to the British Standard Methods of Fats and Fatty Oils; Part 1; Physical methods; Section 1.13; Determination of cooling curve. More specifically, after melting the fractionated sal fat by heating at 60°C for 30 minutes or more, 75 g of the fractionated sal fat was poured into 100 ml test tube, followed by cooling in a constant temperature water bath at 16°C with stirring. Changes during the cooling were plotted in a graph by taking temperature in the vertical axis and elapsed time in the horizontal axis. The graph showed the temperature changes of the fractionated sal fat

during cooling and crystallization. The cooling curve is a chart showing temperature changes that the temperature of the fat drops along with the cooling of the molten fat, and then raises up with starting crystallization due to

5 crystallization heat to the highest temperature, and then drops from the highest temperature. The temperature at which the temperature of the fat starts to raise due to the crystallization heat is called the minimum temperature (T_{min}) and the highest temperature is called the maximum
10 temperature (T_{max}). As the difference between T_{max} and T_{min} (ΔT) is larger, the fat is preferable as a cocoa butter substitute because the larger difference means a better crystallization property and a good demolding property after chocolate tempering. Among cocoa butter substitutes,
15 it is one of the standards of high quality that ΔT of the fat is 4.5°C or more in the case of high quality cocoa butter substitute called Cocoa Butter Equivalent (CBE) which has physical properties similar to cocoa butter.

(2) Solid fat content: Measured according to the solid
20 fat content NMR method of the JOCS Standard Methods for the Analysis of Fats and Oils, provisional method 1-1996.

Aging was performed at 26°C for 40 hours.

(3) Melting point: Measured according to the JOCS
Standard Methods for the Analysis of Fats and Oils 2.2.4.3-
25 1996 softening point (Ring and Ball Method). Aging was

performed at 25°C for 24 hours.

<Table 2>

	Example 4	Comparative Example 2
Cooling curve		
T min (°C)	24.2	23.9
T max (°C)	29.0	27.8
ΔT	4.8	3.9
Solid fat content (%); at 26°C for 40 hours		
25°C	62.7	48.3
30°C	39.8	27.9
35°C	1.8	1.5
Melting point (%); at 25°C for 24 hours	33.3	33.3

As shown in Table 2, Example 4 had a larger cooling curve ΔT and exhibited a higher solid fat content as compared to Comparative Example 2. From the results, it was apparent that Example 4 which was obtained by using the fractionated sal fat produced in Example 1 of the present invention was a high quality cocoa butter substitute as compared to Comparative Example 2.

The present invention relates to a method for improving the quality of a low quality sal fat and, more specifically, to a method for improving the quality of a fractionated sal fat so that the fractionated sal fat exhibits a good crystallization property when used as a cocoa butter substitute.

WHAT IS CLAIMED IS:

1. A production method of a fractionated sal fat, comprising preparing a refined sal fat and carrying out crystallization and washing of a crystal part by using a polar solvent to obtain the crystal part as a hard butter component, wherein a total amount of the polar solvent used for the crystallization and the washing is 1000 wt% or more with respect to 100 wt% of the refined sal fat.

2. The production method of the fractionated sal fat according to claim 1, wherein, in the hard butter component, a content of triglycerides containing dihydroxystearic acid as a constituent fatty acid (DHS-TG) is 0.4 wt% or less; a content of diglycerides (DG) is 0.5 wt% or less; and a content of a 1,2-disaturated-3-unsaturated triglycerides (SSU) fraction is 2 wt% or less.

3. The production method of the fractionated sal fat according to claim 1 or 2, wherein, in the hard butter component, a content of 1,3-disaturated-2-unsaturated triglycerides (SUS) is 85 wt% or more.

4. The production method of the fractionated sal fat according to any one of claims 1 to 3, wherein the crystal part obtained by fractionation using the polar solvent is subjected to re-fractionation using the polar solvent to obtain a crystal part as a hard butter component.