(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) **EP 1 889 898 A1**

(12) **EUROPEAN PATENT APPLICATION**
published in accordance with Art. 153(4) EPC

(43) Date of publication:
20.02.2008 Bulletin 2008/08

(21) Application number: **06731755.2**

(22) Date of filing: **13.04.2006**

(51) Int Cl.:
C11B 3/16 *(2006.01)*

(86) International application number:
PCT/JP2006/307819

(87) International publication number:
WO 2006/112347 (26.10.2006 Gazette 2006/43)

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU LV MC NL PL PT RO SE SI SK TR

(30) Priority: **14.04.2005 JP 2005117391**

(71) Applicant: **Adeka Corporation**
Tokyo 116-8554 (JP)

(72) Inventors:
- **NEZU, Toru,**
  c/o Adeka Corporation
  Tokyo 116-8554 (JP)
- **ITO, Tomonori,**
  c/o Adeka Corporation
  Tokyo 116-8554 (JP)
- **ARAKAWA, Hiroshi,**
  c/o Adeka Corporation
  Tokyo 116-8554 (JP)
- **TAKEBAYASHI, Shintaro,**
  c/o Adeka Corporation
  Tokyo 116-8554 (JP)

(74) Representative: **Forstmeyer, Dietmar**
BOETERS & LIECK
Oberanger 32
80331 München (DE)

(54) DRY FRACTIONATION METHOD FOR FAT

(57) A process for dry fractionation of fat and oil including the steps of melting fat and oil, crystallizing the melt by cooling to obtain a crystal slurry, separating the slurry into a first crystalline fraction and a first liquid fraction, and sweating the first crystalline fraction by heating while pressing to obtain a second crystalline fraction and a sweated-out second liquid fraction. A high purity crystalline fraction (high-melting components) and middle melting fraction can efficiently be separated from fat and oil without using a wetting agent or any special implement. The thus fractionated fat and oil are suited for use in chocolate, etc.

Printed by Jouve, 75001 PARIS (FR)

Description

Technical Field:

[0001] This invention relates to a process for dry fractionation of fats and oils.

Background Art:

[0002] In order to obtain a high purity crystalline fraction in dry fractionation of fats and oils, it is important to selectively crystallize desired triglyceride components and to minimize incorporation of a liquid phase into a crystalline fraction when the crystalline fraction is separated. Fat and oil, being a mixture of various triglycerides, triglycerides having close melting points are likely to form mixed crystals, and the composition of crystallized triglycerides varies depending on the crystallizing temperature. In lowered crystallizing temperatures, not only a desired triglyceride component but other triglycerides having lower melting points crystallize simultaneously, only to provide a product with a reduced purity of the desired triglyceride component. Accordingly, it is desirable to cause a desired triglyceride to crystallize slowly at a high temperature so as to selectively fractionate the desired triglyceride. Nonetheless, manufacturers often have to perform crystallization at a lower temperature in order to achieve an industrially practical crystallization rate.
Furthermore, because a crystallized phase obtained by dry fractionation is spherical agglomerates of fine crystals, a liquid phase tends to be entrapped inside the agglomerates or remain in the interstices between crystals, which reduces the purity of the crystalline fraction.

[0003] On the other hand, a so-called sweating operation is known as a technique for obtaining a high purity crystalline fraction, in which a crystalline fraction is heated to melt part of the crystals while sweating out a liquid phase remaining on and in the crystals.
Included in the related art utilizing the sweating operation is the process disclosed in Patent Document 1. The process comprises cooling a fatty acid mixture of solid fatty acids and liquid fatty acids to crystallize the solid fatty acids, mixing a wetting agent aqueous solution into the fatty acid mixture containing crystallized solid fatty acids, separating the resulting system into an aqueous phase having the solid fatty acids dispersed therein and a liquid fatty acid phase, and maintaining the aqueous phase having the solid fatty acids dispersed therein at or below the melting point of the solid fatty acids to cause the solid fatty acids to sweat out the liquid fatty acids. However, this process involves necessity to remove the wetting agent aqueous solution.
Patent Document 2 discloses a process in which crystals are sweated by heating while being supported by a screen-like supporting structure bent in a zig-zag. The process is disadvantageous in that a special implement is required and that a liquid phase is not sweated out unless a considerable amount of the crystals is melted.
Patent Document 3 teaches a dry fractionation process in which a crystalline fraction is heated to be melted only partially to cause sweating and then subjected to compression and filtration. According to the process, however, the fat and oil crystals are structurally weakened by the heating and easily crumbled by the subsequent compression operation to have extremely reduced filterability (separability of a liquid phase from the crystalline fraction). That is, it is difficult to achieve sufficient solid-liquid separation after sweating.
In addition to the poor separation efficiency, the process has poor time efficiency because, when the crystalline fraction or liquid fraction is again subjected to dry fractionation to obtain a middle melting fraction, the fraction cannot be transferred to the second fractionation step until the first fractionation step comes to a complete end.

Patent Document 1: JP-A-4-306296
Patent Document 2: JP-A-11-76701
Patent Document 3: JP-A-2004-123839

Disclosure of the Invention:

[0004] An object of the present invention is to provide a process for dry fractionation of fat and oil whereby a high purity crystalline fraction (high melting components) or middle melting fraction can be separated from fat and oil efficiently without requiring a wetting agent or a special implement.

[0005] The invention accomplishes the above object by the provision of a process for dry fractionation of fat and oil comprising melting fat and oil, crystallizing the melt by cooling to obtain a crystal slurry, separating the slurry into a first crystalline fraction and a first liquid fraction, and sweating the first crystalline fraction by heating while pressing to separate the crystalline fraction into a second crystalline fraction and a second liquid fraction.

Brief Description of the Drawings:

[0006]

Fig. 1 is a DSC melting pattern from which to decide the heating temperature of a first crystalline fraction.

Fig. 2 is a DSC melting pattern showing a plurality of melting peaks from which to decide the heating temperature of a first crystalline fraction.

Best Mode for Carrying out the Invention:

[0007] Any fats and oils except liquid oils can be applied to the invention with no restriction. Examples of useful fats and oils include those rich in symmetric triglycerides, such as palm oil, shea butter, sal fat, illipe butter, cacao butter, kokum butter, mango kernel oil; lauric fats and oils, such as coconut oil and palm kernel oil; animal fats and oils, such as beef tallow, lard, and milk fat; and their fractionated fats, hardened oils, and interesterified oils. Hardened oils prepared from liquid oils, such as soybean oil and rapeseed oil, are also useful. Fats and oils used to produce hard butter suitable for chocolate manufacture are particularly suited.

[0008] The present invention provides a process for dry fractionation of fat and oil including the steps of melting the above-described fat and oil, crystallizing the melt by cooling to obtain a crystal slurry, separating the slurry into a first crystalline fraction and a first liquid fraction, and sweating the first crystalline fraction by heating while pressing to separate the crystalline fraction into a second crystalline fraction and a second liquid fraction.

Classified according to melting point, the thus obtained second crystalline fraction, second liquid fraction, and first liquid fraction, correspond to high melting components, middle melting components, and low melting components, respectively.

[0009] In the first step of the invention, the fat and oil is melted. The temperature for melting depends on the kind of the fat and oil and is not particularly limited as long as the fat and oil melt.

[0010] The resulting melt is cooled to crystallize and separated into a first crystalline fraction and a first liquid fraction. The crystallizing temperature is such that the resulting system can be separated into a first crystalline fraction and a first liquid fraction.

The crystallizing method is not particularly restricted, provided that the method is applicable to dry fractionation. For example, crystallization is achieved by (1) cooling the system while stirring, (2) cooling the system under a static condition, (3) cooling the system under a stirred condition and then under a static condition, or (4) cooling the system under a static condition followed by fluidizing by mechanical stirring.

In using lauric fats and oils and fats and oils rich in symmetric triglycerides, particularly fats and oils used to produce hard butter for chocolate manufacture, it is difficult to precipitate a large quantity of easily filterable crystals under a stirred condition. Therefore, the method (3) or (4) is preferably adapted for crystallization. Crystallization does not have to be carried out batchwise and may be continuous crystallization or multistage batch crystallization (cascade crystallization).

[0011] It is preferred for the crystal slurry obtained by the crystallization step to have a solid fat content (SFC) of 1% to 65% at the crystallizing temperature. A crystal slurry with the solid fat content falling within that range exhibits good fractionation efficiency in separating into a first crystalline fraction and a first liquid fraction. When the solid fat content is less than 1% or more than 65%, the fractionation efficiency tends to be low.

When the aim of dry fractionation is to collect only a second crystalline fraction (high melting components), the solid fat content (SFC) at the crystallizing temperature is preferably from 3% to 40%, still preferably from 5% to 35%, to attain an increased yield.

When the aim of dry fractionation is to collect only a second liquid fraction (middle melting components), the solid fat content (SFC) at the crystallizing temperature is preferably from 20% to 65%, still preferably from 40% to 65%, to attain an increased yield.

It is preferred that the fat and oil crystals contained in the crystal slurry be spherical agglomerates of fine crystals. The particle size distribution (on volume basis) is preferably such that 99% or more of the fat and oil crystals fall within a range of from 5 to 1500 μm, still preferably 50 to 1000 μm, with the median diameter preferably ranging from 200 to 800 μm, still preferably 300 to 600 μm. When the fat and oil crystals are out of the above ranges, for example, when they are needle-like, or when 1% or more of the crystals have a particle size smaller than 5 μm, or when the median diameter is smaller than 200 μm, the slurry tends to have so poor filterability as to be difficult to separate into a first crystalline fraction and a first liquid fraction. When 1% or more of the crystals have a particle size greater than 1500 μm, or when the median diameter exceeds 800 μm, the crystals are apt to be crumbled by the pressing pressure to deteriorate filterability, also making it difficult to achieve separation into a first crystalline fraction and a first liquid fraction.

[0012] Separation of the crystal slurry into a first crystalline fraction and a first liquid fraction can be achieved by gravity filtration, suction filtration, pressure filtration, centrifugation, and the like. In order to carry out fractionation conveniently with minimum equipment in the dry fractionation, a pressure filtration process using pressure filtration equipment enabling pressing and fractionation, a filter press (membrane filter) with compression device (hereinafter, combined filter press), a belt press, etc. is preferred.

When the crystal slurry is extremely viscous due to a high solid fat content (SFC) at the crystallizing temperature or when the crystallized system looks like a block, a pressure filtration process is particularly suitable because the system is fluidized on pressure application.

The pressure to be applied in pressure filtration is preferably 0.2 MPa or higher, still preferably 0.5 to 5 MPa. The pressure to be applied is preferably increased gradually from the beginning to the end of the pressing. The rate of pressure increase is 1 MPa/min or less, preferably 0.5 MPa/min or less, still preferably 0.1 MPa/min or less. When the rate of pressure increase exceeds 1 MPa/min, the finally obtained second crystalline fraction may have a reduced purity.

[0013] The separation of the crystal slurry is preferably performed such that the ratio of the resulting first crystalline fraction to first liquid fraction may range from 5:95 to 90:10 by mass.

When the purpose is to separate only a second crystalline fraction (high melting components), the separation is preferably effected such that the first crystalline fraction to first liquid fraction mass ratio may range from 10:90 to 50:50, still preferably 10:90 to 40:60, to attain an increased yield.

When the purpose is to separate only a second liquid fraction (middle melting components), the separation is preferably effected such that the first crystalline fraction to first liquid fraction mass ratio may range from 50:50 to 90:10, still preferably 60:40 to 90:10, to attain an increased yield.

The fat and oil crystals of the resulting first crystalline fraction have practically the same size as those in the crystal slurry.

[0014] The first crystalline fraction thus obtained by the separation of the crystal slurry is then sweated by heating while pressing to separate the crystalline fraction into a second crystalline fraction and a second liquid fraction. The present invention is different from the conventional sweating operation, in which heating precedes pressing, in that sweating is carried out by heating while pressing, i.e., melting of the crystalline fraction by heating and separation of a sweated-out liquid fraction are conducted in parallel.

Compared with the conventional sweating operation, the sweating by heating simultaneously with pressing yields a second crystalline fraction with higher purity and at higher separation efficiency.

The reasons the sweating operation by heating while pressing achieves higher separation efficiency and higher crystalline fraction purity are as follows.

Firstly, to separate a sweated-out liquid little by little makes it possible to maintain the amount of crystals in the crystalline fraction at a high level and to hold the structure of the crystalline fraction strong and resistant to pressure.

Secondly, to maintain the amount of a liquid phase in the crystalline fraction at a low level results in shifting the solid-liquid equilibrium to solid, making it possible to minimize the amount of melting of the crystalline fraction.

The pressing pressure is preferably 0.02 to 2 MPa, still preferably 0.03 to 1.5 MPa, even still preferably 0.04 to 1 MPa. At a pressing pressure lower than 0.02 MPa, it tends to take longer time for a second liquid fraction to be sweated out and separated, and after separation into a second crystalline fraction and a second liquid fraction, middle melting components tend to remain in the second crystalline fraction, resulting in poor separation efficiency. Conversely, at a pressing pressure higher than 2 MPa, high melting components are liable to pass through the filter while the first crystalline fraction is sweated by heating and pressing, also resulting in poor separation efficiency between a second crystalline fraction and a second liquid fraction.

When there is a fear that the fat and oil crystals of the first crystalline fraction are crumbled by the pressure application due to reduction in pressure resistance with the sweating, which depends on the condition of the crystals, the pressing pressure may gradually be decreased from the beginning to the end of the sweating step.

[0015] The heating temperature of the first crystalline fraction is higher than the above-described crystallizing temperature of the fat and oil but not so high as to cause the crystalline fraction to melt completely. Preferably, the heating temperature is at or higher than the onset temperature (Ta in Fig. 1) and lower than the offset temperature (Tb in Fig. 1) of a melting peak observed when the first crystalline fraction is melted on a differential scanning calorimeter (DSC). When a plurality of melting peaks are observed, the melting peak of a component to be fractionated as a crystalline fraction is taken as a reference peak. For example, when a DSC chart shows a main melting peak and a shoulder as in Fig. 2, and a component having the main peak is desired as a crystalline fraction, the temperature Tac is taken as a reference temperature.

In this fractionation step, since fractionation is carried out through sweating by heating with pressing as stated above, the step is preferably performed by pressure filtration using pressure filtration equipment enabling pressing and fractionation, a combined filter press (membrane filter), a belt press, etc. is preferred.

The second liquid fraction may further be fractionated by the time of sweating out the second liquid fraction. A liquid fraction removed in the initial stage of the sweating contains a component having a relatively lower melting point than the other components of the second liquid fraction. Accordingly, the second liquid fraction collected after separation of the initial second liquid fraction contains a reduced amount of a relatively low melting component, i.e., with a higher purity. In order to obtain an even higher purity second liquid fraction, the heating temperature may be elevated stepwise over a period of from the start to the end of the sweating step by heating and pressing thereby to collect a plurality of second liquid fractions. A liquid fraction separated in the initial stage of sweating contains a component having a relatively lower melting point than the other components of the second liquid fraction. Accordingly, by elevating the heating temperature after the initial second liquid fraction is separated, a second liquid fraction with a reduced amount of a relatively low melting component, i.e., with a higher purity can be obtained.

In order to obtain an even higher purity second liquid fraction, the heating temperature may be elevated continuously over a period of from the start to the end of the sweating step by heating and pressing. Because a liquid phase is little by little removed from the crystalline phase through sweating induced by heating and pressing, the melting peak observed with the crystalline fraction on DSC gradually shifts toward higher temperatures. By elevating the sweating temperature continuously synchronously with the shift of the melting peak to higher temperatures, a second liquid fraction with a higher purity can be obtained.

[0016] The above fractionation is preferably performed such that the ratio of the resulting second liquid fraction to second crystalline fraction may range from 98:2 to 2:98, more preferably 95:5 to 5:95, most preferably 93:7 to 10:90, by mass. Where the ratio of the second crystalline fraction is less than 2, high melting components tend to dissolve in the second liquid fraction during sweating, which makes separation into a second crystalline fraction and a second liquid fraction difficult. Where the ratio of the second crystalline fraction is more than 98, this tends to give rise to the necessity to raise the temperature for heating the first crystalline fraction while pressing to induce sweating. As a result, middle melting components tend to dissolve in the second crystalline fraction, making separation into a second crystalline fraction and a second liquid fraction difficult.

The second crystalline fraction obtained by the fractionation process of the invention will then be described.

The second crystalline fraction obtained by the fractionation process of the invention is a concentrate of the crystalline phase (high melting components) of the first crystalline fraction. When, in particular, fats and oils used to make hard butter for chocolate manufacture are used as raw material, the second crystalline fraction is characterized by having concentrated tri-saturated glycerides and symmetric triglycerides compared with the first crystalline fraction. In this application, the second crystalline fraction obtained by the fractionation process of the invention preferably has a symmetric triglyceride content of 75% to 99% by weight, still preferably 80% to 95% by weight, even still preferably 85% to 95% by weight.

Applications of the second crystalline fraction include fats and oils for chocolate, white chocolate, buttercream, and filling cream; and starting fats and oils for margarine/shortening.

The second liquid fraction obtained by the fractionation process of the invention will be described.

The second liquid fraction obtained by the fractionation process of the invention is the first crystalline fraction having removed therefrom high melting components. When, in particular, fats and oils used to make hard butter for chocolate manufacture are used as raw material, the second liquid fraction is characterized by having a high symmetric triglyceride content with a low tri-saturated glyceride content. In this application, the second liquid fraction obtained by the fractionation process of the invention preferably has a symmetric triglyceride content of 50% to 99% by weight, still preferably 70% to 95% by weight, and a tri-saturated glyceride content of 5% or less, still preferably 3% or less, by weight.

Applications of the second liquid fraction include fats and oils for chocolate, white chocolate, buttercream, and filling cream; starting fats and oils for margarine/shortening; fats and oils for adjusting the hardness of chocolate; fats and oils for ice cream or for ice cream coating; and oils for O/W emulsified fats and oils, such as whipped cream.

Examples:

[0017] The present invention will now be illustrated in greater detail with reference to Examples, but it should be understood that the invention is not deemed to be limited thereto.

In Tables 1 through 5 below, DG stands for diglyceride; P, palmitic acid; S, stearic acid; A, arachidic acid; O, oleic acid; L, linoleic acid; s, saturated fatty acid; and u, unsaturated fatty acid. All the values given in Tables 1 to 5 are by mass percent.

EXAMPLE 1

[0018] A palm middle melting fraction weighing 500 g and having an iodine value of 45, which was obtained by dry fractionation of palm olein, was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 22°C while gently stirring for 8 hours to obtain a crystal slurry having an SFC of 10% (22°C). Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 60 to 800 μm with a median diameter of 650 μm. The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 22°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was raised to 40°C while applying a pressure of 0.5 MPa to the first crystalline fraction remaining in the membrane filter, and the first crystalline fraction was maintained in this condition for 8 hours to obtain a sweated-out liquid as a second liquid fraction and crystals remaining in the membrane filter press as a second crystalline fraction. A DSC chart of the first crystalline fraction showed an onset temperature of 25°C and an offset temperature of 48°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 16.9% by mass, 83.1 % by mass, 4.5% by mass, and 12.4% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 1 below. The second liquid fraction, which corresponded to middle melting components, contained 83.7% by mass sus and 1.4% by mass sss. The second crystalline fraction, which corresponded to high melting components, contained 60.5% by mass sus and 24.1 % by mass sss.

EXAMPLE 2

[0019] Palm olein weighing 500 g and having an iodine value of 65 was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 18°C while gently stirring for 5 hours to obtain a crystal slurry having an SFC of 10% (22°C). Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 60 to 700 μm with a median diameter of 400 μm. The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 18°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was raised to 35°C while applying a pressure of 0.5 MPa to the first crystalline fraction remaining in the membrane filter, and the first crystalline fraction was maintained in this condition for 8 hours to obtain a sweated-out liquid as a second liquid fraction and crystals remaining in the membrane filter press as a second crystalline fraction. A DSC chart of the first crystalline fraction showed an onset temperature of 25°C and an offset temperature of 45°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 13.5% by mass, 86.5% by mass, 3.6% by mass, and 9.9% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 2 below. The second liquid fraction, which corresponded to middle melting components, contained 76% by mass sus and 1 by mass sss. The second crystalline fraction, which corresponded to high melting components, contained 49.9% by mass sus and 43% by mass sss.

EXAMPLE 3

[0020] A palm middle melting fraction weighing 500 g and having an iodine value of 45, which was obtained by dry fractionation of palm olein, was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 22°C while gently stirring for 8 hours to obtain a crystal slurry having an SFC of 10% (22°C). Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 70 to 620 μm with a median diameter of 500 μm. The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 22°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was raised to 40°C, and a pressure of 0.5 MPa was applied to the first crystalline fraction remaining in the membrane filter. The liquid sweated out for the first one hour was collected as a second liquid fraction-1, and the liquid sweated out for an additional 7 hour period was collected as a second liquid fraction-2, and the crystals remaining in the membrane filter press as a second crystalline fraction. A DSC chart of the first crystalline fraction showed an onset temperature of 25°C and an offset temperature of 48°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, the second liquid fraction-1, and the second liquid fraction-2 were 14.8% by mass, 85.2% by mass, 3.6% by mass, 0.5% by mass, and 10.7% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 3 below. The second liquid fractions-1 and -2, both corresponding to middle melting components, contained 71.9% by mass and 88.6% by mass, respectively, of sus and 0.3% by mass and 3.3% by mass, respectively, of sss. The second crystalline fraction, which corresponded to high melting components, contained 44.9% by mass sus and 44.9% by mass sss.

COMPARATIVE EXAMPLE 1

[0021] A palm middle melting fraction weighing 500 g and having an iodine value of 45, which was obtained by dry fractionation of palm olein, was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 22°C while gently stirring for 4 hours to obtain a crystal slurry having an SFC of 4% (22°C). The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 22°C to obtain a first crystalline fraction and a first liquid fraction.

The first liquid fraction was completely melted and crystallized at 22°C while gently stirring for 11 hours to obtain a crystal slurry having an SFC of 6% (22°C). The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 22°C to obtain a second crystalline fraction and a second liquid fraction. The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 6.9% by mass, 93.1 % by mass, 12.5% by mass, and 80.6% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 4 below. The second crystalline fraction, which corresponded to middle melting components, contained 84.4% by mass sus and 2.2% by mass sss. The first crystalline fraction, which corresponded to high melting components, contained 77.1% by mass sus and 15.5% by mass sss.

COMPARATIVE EXAMPLE 2

[0022] A palm middle melting fraction weighing 500 g and having an iodine value of 45, which was obtained by dry fractionation of palm olein, was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 22°C while gently stirring for 8 hours to obtain a crystal slurry having an SFC of 10% (22°C). Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 60 to 800 μm with a median diameter of 650 μm. The crystal slurry was filtered and then pressed at 3 MPa using a membrane filter (combined filter press) in a thermostat kept at 22°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was raised to 40°C without applying a pressure to the first crystalline fraction remaining in the membrane filter. The first crystalline fraction was maintained in this condition for 8 hours and then pressed at 0.1 MPa to obtain a sweated-out liquid as a second liquid fraction and crystals remaining in the membrane filter press as a second crystalline fraction. A DSC chart of the first crystalline fraction showed an onset temperature of 24.5°C and an offset temperature of 47°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 11.6% by mass, 88.4% by mass, 7.8% by mass, and 3.8% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 5 below. The second liquid fraction, which corresponded to middle melting components, contained 80.5% by mass sus and 6.1% by mass sss. The second crystalline fraction, which corresponded to high melting components, contained 69.3% by mass sus and 19.4% by mass sss.

[0023]

TABLE 1

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC					
DG	4.5	3.5	4.3	8.3	2.7
LOO	0.9	0.1	1.0	0.2	0.3
PLO	5.7	1.0	7.1	0.2	2.0
PLP	9.7	6.8	10.5	2.8	8.3
OOO	2.4	1.6	2.6	4.4	0.9
POO	13.9	3.6	18.2	1.1	5.4
POP	47.9	63.9	43.6	44.0	65.7
PPP	1.4	5.9		24.3	0.6
SOO	1.3	0.2	1.6		0.3
POS	8.9	10.6	8.0	7.0	11.6
SPP	0.3	1.0		5.3	0.1
AOO	0.1		0.1		
SOS	1.3	0.9	1.3	0.6	1.5
SSP		0.2		1.3	
AOS			0.1	0.7	
SSS					
etc.	1.8	0.8	1.6	0.0	0.4

(continued)

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
Total	100.0	100.0	100.0	100.0	100.0
sss	1.7	7.0	0.0	30.9	0.8
sus	67.8	82.2	63.5	55.0	87.1
suu	20.9	4.8	26.9	1.4	7.7
uuu etc.	9.6	6.0	9.6	12.8	4.4
Total	100.0	100.0	100.0	100.0	100.0

[0024]

TABLE 2

	Palm Olein	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC					
DG	6.2	2.4	6.7	0.8	3.3
LOO	1.5	0.5	1.8	0.1	0.5
PLO	9.8	3.6	11.0	0.4	4.0
PLP	10.6	9.8	11.0	3.1	10.6
OOO	3.8	1.6	4.2	2.4	1.6
POO	24.5	10.3	26.8	3.2	11.7
POP	28.8	52.6	25.5	39.6	53.2
PPP	0.5	5.2		34.7	0.8
SOO	3.2	0.7	3.4	0.1	0.8
POS	6.7	10.1	5.4	6.4	10.2
SPP	0.1	0.8		6.8	0.2
AOO	0.2		0.3		0.1
SOS	1.0	1.2	0.8	0.8	1.8
SSP		0.2		1.5	
AOS			0.1		0.2
SSS		0.2			
etc.	3.2	0.8	3.3	0.1	1.2
Total	100.0	100.0	100.0	100.0	100.0
sss	0.6	6.4	0.0	43.0	1.0
sus	47.1	73.7	42.7	49.9	76.0
suu	37.6	14.6	41.1	3.7	16.4
uuu etc.	14.8	5.4	16.2	3.5	6.7
Total	100.0	100.0	100.0	100.0	100.0

[0025]

TABLE 3

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction-1	2nd Liquid Fraction-2
TGC						
DG	4.5	4.4	4.3	9.6	3.8	2.7
LOO	0.9	0.1	0.8	0.1	0.7	0.1
PLO	5.7	0.6	6.2	0.1	5.2	0.8
PLP	9.7	5.4	10.0	1.8	9.8	6.7
OOO	2.4	2.0	2.2	5.0	2.0	1.1
POO	13.9	2.2	16.0	0.4	13.7	3.0
POP	47.9	60.5	46.5	31.4	51.4	68.7
PPP	1.4	10.9		33.5	0.3	2.9
SOO	1.3	0.1	1.6		1.2	0.1
POS	8.9	9.9	8.8	6.4	9.3	11.6
SPP	0.3	2.3		9.1		0.4
AOO	0.1					
SOS	1.3	1.1	1.8	0.3	1.4	1.4
SSP		0.3		1.8		0.0
AOS						0.1
SSS				0.4		
etc.	1.8	0.2	1.8	0.1	1.4	0.3
Total	100.0	100.0	100.0	100.0	100.0	100.0
sss	1.7	13.5	0.0	44.9	0.3	3.3
sus	67.8	76.9	67.1	39.9	71.9	88.6
suu	20.9	2.9	23.7	0.5	20.0	4.0
uuu etc.	9.6	6.7	9.2	14.8	7.9	4.1
Total	100.0	100.0	100.0	100.0	100.0	100.0

[0026]

TABLE 4

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC				64.8	
DG	4.6	6.5	4.2	3.3	4.8
LOO	0.7	0.2	0.9	0.4	1.0
PLO	5.4	0.7	5.8	2.2	6.3
PLP	9.7	5.2	9.9	6.5	10.4
OOO	1.9	2.8	2.4	1.4	2.3
POO	13.3	2.8	14.2	5.4	14.9

(continued)

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
POP	50.0	55.3	48.8	64.7	47.0
PPP	0.6	11.8	0.2	1.7	0.1
SOO	0.9	0.2	1.1	0.3	1.2
POS	9.1	9.6	8.9	11.5	9.6
SPP	0.3	3.0		0.4	
AOO			0.1		0.1
SOS	1.2	1.0	1.2	1.4	1.1
SSP	0.1	0.7		0.1	
AOS				0.2	0.1
SSS					
etc.	2.2	0.2	2.3	0.5	1.1
Total	100.0	100.0	100.0	100.0	100.0
sss	0.9	15.5	0.2	2.2	0.1
sus	70.0	77.1	68.8	84.4	67.2
suu	19.6	3.6	21.1	7.9	22.5
uuu etc.	9.5	3.8	9.9	5.5	10.2
Total	100.0	100.0	100.0	100.0	100.0

[0027]

TABLE 5

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC					
DG	4.5	4.0	4.5	4.4	3.5
LOO	0.9	0.1	1.0	0.2	0.2
PLO	6.1	0.9	6.7	0.7	1.5
PLP	9.9	5.9	10.4	5.2	7.0
OOO	2.5	2.2	2.6	2.6	1.9
POO	15.4	3.4	17.1	2.9	5.0
POP	46.3	57.8	44.5	53.8	61.4
PPP	1.2	11.7		15.1	4.9
SOO	1.3	0.2	1.5	0.2	0.3
POS	8.5	9.8	8.3	9.2	10.7
SPP	0.3	2.8		3.5	1.1
AOO			0.1		
SOS	1.3	1.2	1.3	1.0	1.3
SSP				0.7	0.2

(continued)

	Palm Middle Melting Fraction	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
AOS	0.1		0.2	0.1	0.1
SSS					
etc.	1.6		2.0	0.3	1.1
Total	100.0	100.0	100.0	100.0	100.0
sss	1.5	14.4	0.0	19.4	6.1
sus	66.2	74.7	64.5	69.3	80.5
suu	22.8	4.5	25.3	3.8	6.7
uuu etc.	9.5	6.4	10.2	7.4	6.7
Total	100.0	100.0	100.0	100.0	100.0

[0028] Comparison between Example 1 and Comparative Example 1 reveals that the high melting fraction of Example 1 has a higher sss content than that of Comparative Example 1 obtained by a conventional fractionation process.
Comparison between Example 1 and Comparative Example 2 reveals that Example 1 produced a high melting fraction with a higher sss content and a middle melting fraction with a higher sus content than those of Comparative Example 2 in which the sweating step was not accompanied with pressing.
Comparison between Example 1 and Example 3 reveals that the second liquid fraction-2 obtained in Example 3 by fractionating the second liquid fraction by the sweating time in the sweating step is a middle melting fraction with a higher sus content than the second liquid fraction of Example 1.

EXAMPLE 4

[0029] Palm olein weighing 500 g and having an iodine value of 56 was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 18°C while gently stirring for 65 hours to obtain a crystal slurry having an SFC of 21%. Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 100 to 400 μm with a median diameter of 280 μm. The crystal slurry was filtered and then pressed at 1 MPa using a membrane filter press (combined filter press) in a thermostat kept at 18°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was continuously raised to 26°C over a 1 hour period and then up to 28°C over an 11 hour period while applying a pressure of 0.7 MPa to the first crystalline fraction remaining in the membrane filter press to obtain a sweated-out second liquid fraction and a second crystalline fraction remaining in the membrane filter press. A DSC chart of the first crystalline fraction showed an onset temperature of 25°C and an offset temperature of 36°C, and a DSC chart of the second crystalline fraction left after the sweating showed an onset temperature of 27°C and an offset temperature of 36°C.
The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 23.8% by mass, 76.2% by mass, 20.9% by mass, and 2.9% by mass, respectively.
The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 6 below. The second liquid fraction, which corresponded to middle melting components, contained 59% by mass sus and 0% by mass sss. The second crystalline fraction, which corresponded to high melting components, contained 86% by mass sus and 3.0% by mass sss.

EXAMPLE 5

[0030] Palm olein weighing 150 kg and having an iodine value of 56 was put in a jacketed glass crystallizer, completely melted at 60°C, and crystallized at 17°C while gently stirring for 48 hours to obtain a crystal slurry having an SFC of 22%. Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 60 to 820 μm with a median diameter of 450 μm. The crystal slurry was filtered and then pressed at 5 MPa using a membrane filter press (combined filter press) in a thermostat kept at 17°C to obtain a first crystalline fraction and a first liquid fraction. The temperature of the thermostat was raised to 28°C, and the first crystalline fraction remaining in the membrane filter press was heated at that temperature for 8 hours while applying pressure of 0.7 MPa to the crystalline fraction to obtain a sweated-out second liquid fraction-1. The pressing pressure was reduced to 0.5 MPa, and the heating was continued at 35°C for 16 hours to obtain a sweated-out second liquid fraction-2 and a second crystalline fraction-2 remaining in the membrane filter press. A DSC chart of the first crystalline fraction showed an onset temperature of 25°C and an offset temperature of 43°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction-2, the second liquid fraction-1, and the second liquid fraction-2 were 30.7% by mass, 69.3% by mass, 0.9% by mass, 17.3% by mass, and 12.5% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 7 below. The second liquid fractions -1 and -2 corresponded to middle melting components. The second liquid fraction-2 had an sus content of 87% by mass and an sss content of 1.2% by mass. The second crystalline fraction, which corresponded to high melting components, contained 53% by mass sus and 33% by mass sss.

EXAMPLE 6

[0031] Deacidified and bleached sal fat weighing 2 kg was completely melted at 60°C, transferred to a tray, and allowed to cool to 31 °C. Seed crystals were added and mixed in an amount of 0.01%, and the system was allowed to crystallize under a static condition at 31°C for 22 hours to obtain a high-viscosity crystal slurry having an SFC of 52%. Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 50 to 450 μm with a median diameter of 230 μm. The seed crystals were a slurry obtained by dissolving a previously fractionated crystalline fraction of sal fat in olive oil at a concentration of 20%, followed by cooling to 5°C. The high-viscosity crystal slurry obtained by crystallization was compressed in a press at 3 MPa in a thermostat set at 31 °C to obtain a first crystalline fraction and a first liquid fraction. The first crystalline fraction in the press was heated at a thermostat temperature of 35°C for 8 hours while being pressed at 0.5 MPa to yield a sweated-out second liquid fraction and a second crystalline fraction remaining in the membrane filter press. A DSC chart of the first crystalline fraction showed an onset temperature of 32°C and an offset temperature of 40°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 65% by mass, 35% by mass, 54% by mass, and 11 % by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 8 below. The second liquid fraction corresponded to middle melting components. This second crystalline fraction had an sus content of 91% by mass and an sss content of 0.2% by mass. The second crystalline fraction, which corresponded to high melting components, had an sus content of 51 % by mass and an sss content of 0% by mass.

EXAMPLE 7

[0032] Cacao butter weighing 2 kg was completely melted at 60°C, transferred to a tray, and crystallized by cooling in a refrigerator at 5°C for 4 hours and then allowed to crystallize under a static condition in a thermostat at 30°C for 40 hours to obtain a high-viscosity crystal slurry having an SFC of 60%. Particle size distribution analysis on the crystal slurry revealed that the particle sizes fell within a range of from 50 to 450 μm with a median diameter of 230 μm. The high-viscosity crystal slurry obtained by crystallization was expressed in a membrane filter press at 3 MPa placed in a thermostat set at 30°C to obtain a first crystalline fraction and a first liquid fraction. The first crystalline fraction in the membrane filter press was heated at a thermostat temperature of 33°C for 5 hours while being pressed at 0.5 MPa to obtain a sweated-out second liquid fraction and a second crystalline fraction remaining in the membrane filter press. A DSC chart of the first crystalline fraction showed an onset temperature of 28°C and an offset temperature of 38°C.

The yields of the first crystalline fraction, the first liquid fraction, the second crystalline fraction, and the second liquid fraction were 75% by mass, 25% by mass, 55% by mass, and 20% by mass, respectively.

The results of HPLC analysis on the triglyceride composition of each fraction are shown in Table 9 below. The second liquid fraction, which corresponded to middle melting components, had an sus content of 77% by mass and an sss content of 0.3% by mass. The second crystalline fraction, which corresponded to high melting components, had an sus content of 94% by mass and an sss content of 1.8% by mass.

[0033]

TABLE 6

	Palm Olein	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC					
DG	6.3	2.5	6.8	2.3	4.2
LOO	1.7	0.2	2.1	0.1	1.0

(continued)

	Palm Olein	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
PLO	11.2	2.2	14.3	1.5	7.8
PLP	10.1	9.1	9.2	8.8	11.2
OOO	4.1	1.3	5.6	1.0	3.0
POO	25.1	7.2	34.0	5.4	20.7
POP	29.0	62.4	16.3	65.5	39.5
PPP	0.5	2.2		2.5	
SOO	2.6	0.3	3.9	0.1	2.1
POS	5.2	10.4	3.2	10.8	7.5
SPP	0.1	0.4		0.4	
AOO	0.1	0.0	0.2		0.1
SOS	0.6	1.1	0.3	1.1	0.9
SSP		0.0		0.1	
AOS		0.1		0.1	
SSS					
etc.	3.5	0.6	4.1	0.5	2.0
Total	100.0	100.0	100.0	100.0	100.0
sss	0.5	2.6	0.0	3.0	0.0
sus	44.9	83.0	29.1	86.3	59.1
suu	39.0	9.8	52.3	6.9	30.7
uuu etc.	15.6	4.6	18.6	3.9	10.2
Total	100.0	100.0	100.0	100.0	100.0

[0034]

TABLE 7

	Palm Olein	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction-1	2nd Liquid Fraction-2
TGC						
DG	6.3	2.9	7.6	8.7	5.1	3.0
LOO	1.7	0.6	2.3	0.3	1.2	0.3
PLO	11.2	3.4	13.7	0.0	7.2	1.7
PLP	10.1	9.2	9.9	2.6	10.7	7.4
OOO	4.1	1.7	5.3	4.3	2.9	1.1
POO	25.1	9.1	31.3	0.4	17.6	4.9
POP	29.0	57.5	17.7	42.2	41.1	66.7
PPP	0.5	1.5	0.0	26.6	0.3	0.9
SOO	2.6	0.8	3.6	0.0	1.8	0.4
POS	5.2	10.4	3.3	7.1	8.2	11.6

(continued)

	Palm Olein	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction-1	2nd Liquid Fraction-2
SPP	0.1	0.4	0.0	5.9	0.1	0.2
AOO	0.1	0.0	0.3		0.1	0.0
SOS	0.6	1.3	0.5	0.8	1.1	1.4
SSP		0.0		0.1		0.0
AOS		0.1		0.1		
SSS						
etc.	3.5	1.2	4.6	1.0	2.7	0.5
Total	100.0	100.0	100.0	100.0	100.0	100.0
sss	0.5	1.9	0.0	32.6	0.3	1.2
sus	44.9	78.5	31.4	52.8	61.0	87.1
suu	39.0	13.2	48.8	0.4	26.8	6.9
uuu etc.	15.6	6.4	19.9	14.3	11.9	4.9
Total	100.0	100.0	100.0	100.0	100.0	100.0

[0035]

TABLE 8

	Sal fat	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid
TGC					
DG	2.5	1.8	6.4	1.3	5.3
LOO			0.6		0.6
PLO	0.5	0.1	0.8		0.6
PLP	0.1	0.1	0.4	0.1	0.2
OOO	2.5	1.0	5.2	0.5	4.2
POO	3.1	1.2	6.5	0.5	5.4
POP	1.5	1.4	3.4	0.8	2.8
PPP	0.1		0.2		
SOO	14.4	5.8	28.0	4.3	24.5
POS	11.8	10.9	12.6	10.2	12.8
SPP					
AOO	3.6	1.3	5.8		6.0
SOS	42.8	55.5	19.3	61.2	25.9
SSP	0.1	0.1			
AOS	12.4	15.8	6.9	16.5	8.2
SSS	0.3	0.3		0.2	
AOA	2.0	2.3	1.1	2.2	1.0
ASS					
etc.	2.3	2.4	2.8	2.2	2.5

(continued)

	Sal fat	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid
Total	100.0	100.0	100.0	100.0	100.0
sss	0.5	0.4	0.2	0.2	0.0
sus	70.6	86.0	43.7	91.0	50.9
suu	21.6	8.4	41.1	4.8	36.5
uuu etc.	7.3	5.2	15.0	4.0	12.6
Total	100.0	100.0	100.0	100.0	100.0

[0036]

TABLE 9

	Cacao Butter	1st Crystal-line Fraction	1st Liquid Fraction	2nd Crystal-line Fraction	2nd Liquid Fraction
TGC					
DG	2.1	1.2	4.6	1.1	3.6
LOO		0.1	0.4		0.5
PLO	0.8	1.0	3.0	0.2	3.2
PLP	1.7	1.5	4.4	0.5	4.0
OOO		0.4	1.3	0.2	1.3
POO	2.5	3.1	9.6	1.6	9.2
POP	18.0	15.5	25.4	11.5	24.1
PPP					
SOO	2.5	1.9	5.2	1.2	4.2
POS	40.0	38.5	31.9	39.6	32.9
SPP	0.4	0.2	0.1	0.4	0.1
AOO					
SOS	28.3	33.6	12.1	40.3	14.8
SSP	0.7	0.7	0.1	0.8	0.2
AOS	1.6	1.6	0.7	1.9	0.8
SSS	0.4	0.4		0.5	
AOA					
ASS					
etc.	1.0	0.3	1.3		1.3
Total	100.0	100.0	100.0	100.0	100.0
sss	1.5	1.3	74.6	1.8	0.3
sus	89.7	90.7	17.7	94.1	76.6
suu	5.8	6.0	7.6	3.0	16.6
uuu etc.	3.1	2.0	15.0	1.1	6.6
Total	100.0	100.0	100.0	100.0	100.0

[0037] The results of Example 4 show that a second crystalline fraction having an extremely high sus content can be

obtained in high yield by continuously elevating the heating temperature during sweating.
The results of Example 5 reveal that, by stepwise elevating the heating temperature during sweating, a second liquid fraction-2 having an extremely high sus content with an extremely low tri-saturated glyceride content can be obtained in high yield.
It is also seen from Examples 7 and 8 that a second crystalline fraction obtained by the dry fractionation process of the invention from a fat and oil having a high sus content has a further increased sus content.

Industrial Applicability:

[0038] The process of dry fractionation of fats and oils according to the present invention produces a high-purity crystalline fraction (high melting components) and a middle melting fraction from fat and oil efficiently without using a wetting agent or any special implement.

Claims

1. A process for dry fractionation of fat and oil comprising melting fat and oil, crystallizing the melt by cooling to obtain a crystal slurry, separating the slurry into a first crystalline fraction and a first liquid fraction, and sweating the first crystalline fraction by heating while pressing to separate the first crystalline fraction into a second crystalline fraction and a second liquid fraction.

2. The process for dry fractionation of fat and oil according to claim 1, wherein the crystal slurry had a solid fat content (SFC) of 1% to 65%.

3. The process for dry fractionation of fat and oil according to claim 1 or 2, wherein the pressing is at a pressure of 0.02 to 1 MPa.

4. The process for dry fractionation of fat and oil according to any one of claims 1 to 3, wherein the second liquid fraction is further fractionated by the time of sweating while the first crystalline fraction is heated and pressed to sweat out the second liquid fraction.

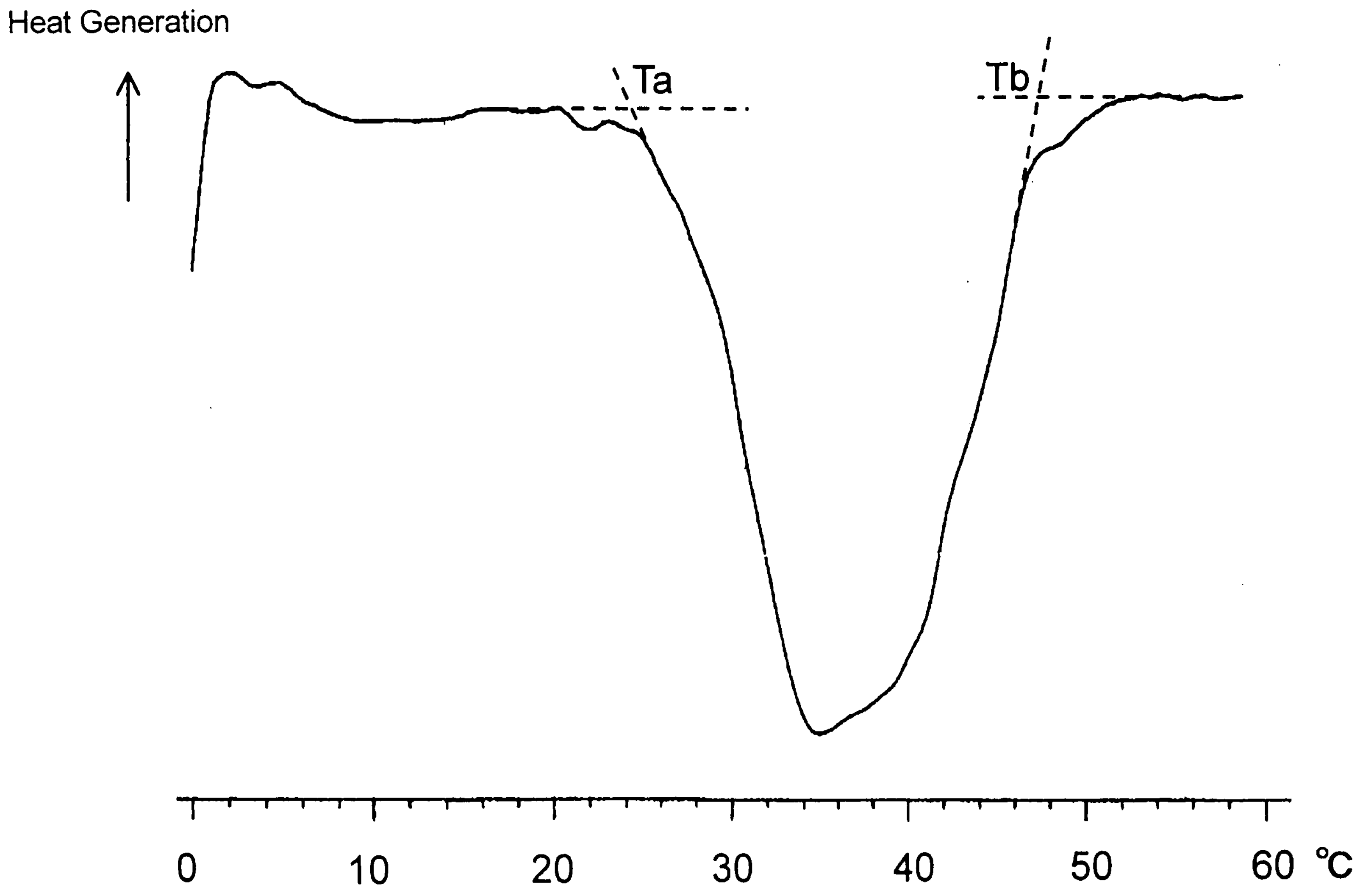
5. The process for dry fractionation of fat and oil according to any one of claims 1 to 4, wherein the heating temperature is stepwise elevated to obtain a plurality of second liquid fractions while the first crystalline fraction is sweated by heating and pressing.

6. The process for dry fractionation of fat and oil according to any one of claims 1 to 4, wherein the heating temperature is continuously elevated while the first crystalline fraction is sweated by heating and pressing.

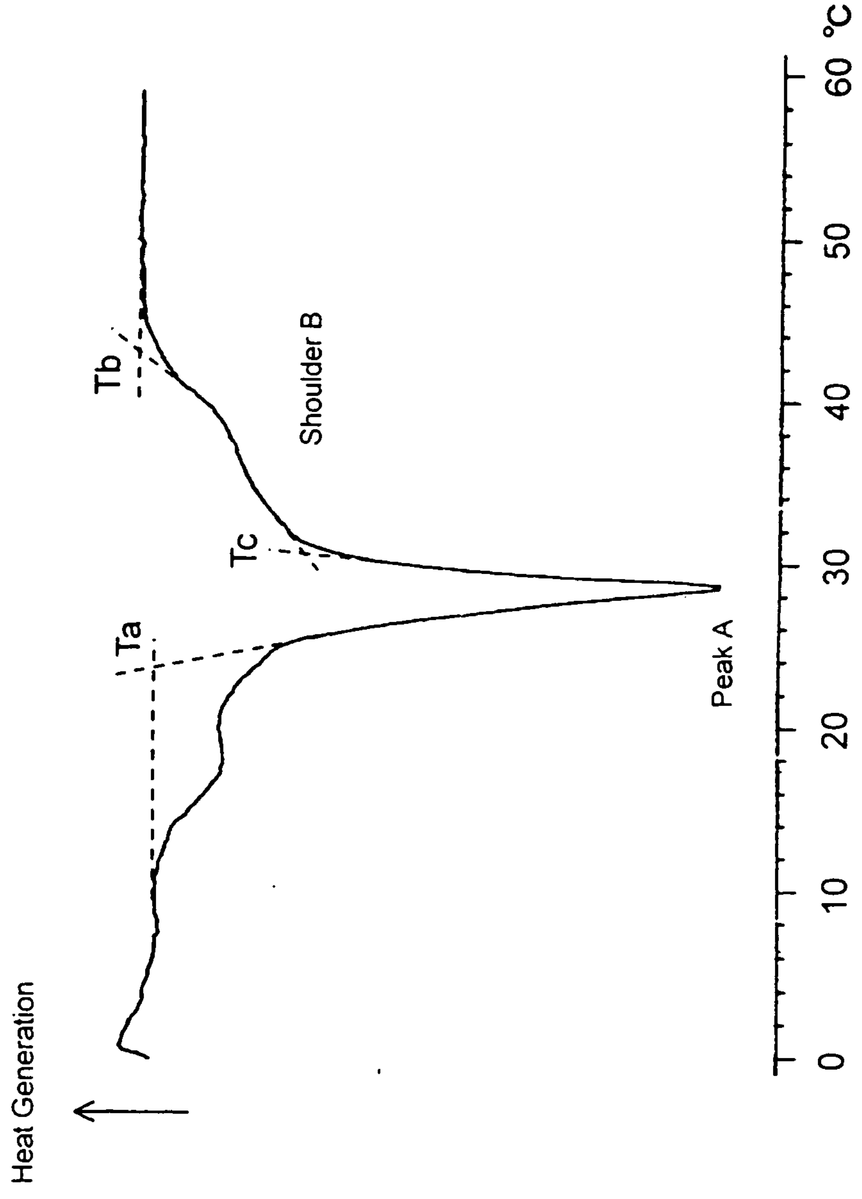
7. A second crystalline fraction obtained by the process for dry fractionation of fat and oil according to any one of claims 1 to 6.

8. A second liquid fraction obtained by the process for dry fractionation of fat and oil according to any one of claims 1 to 6.

〔Fig.1〕



[Fig.2]



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2006/307819

A. CLASSIFICATION OF SUBJECT MATTER
C11B3/16(2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11B3/16

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2006
Kokai Jitsuyo Shinan Koho	1971-2006	Toroku Jitsuyo Shinan Koho	1994-2006

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
CAplus(STN), JST7580(JDream2), JSTPlus(JDream2)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	BELKACEMI et al., Fractionation of Milk Fat by Falling Film Layer Crystallization, SEPARATION SCIENCE AND TECHNOLOGY, 2003, 38(12-13), pages 3115 to 3131	1-8
A	LÜDECKE et al., Scaleup in Suspension Crystallization of Two Multi Compound Fatty Acid Mixtures, ENGINEERING IN LIFE SCIENCES, 2003, 3(3), pages 154 to 158	1-8
A	PETERS-ERJAWETZ et al., Milk Fat Fractionation by Solid-Layer Melt Crystallization, JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY, 1999, 76(5), pages 579 to 584	1-8

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search 31 May, 2006 (31.05.06)	Date of mailing of the international search report 06 June, 2006 (06.06.06)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (April 2005)

REFERENCES CITED IN THE DESCRIPTION

Patent documents cited in the description

- JP 4306296 A **[0003]**
- JP 11076701 A **[0003]**
- JP 2004123839 A **[0003]**