

Fat fractionation

- 1 This invention relates to a process for the dry
fractionation of a vegetable fat capable of crystallizing
in several forms at temperatures around ambient temperature,
to the fractions obtained and to their use.
- 5 Natural fats are complex mixtures of triglycerides of
which the composition includes solid saturated fatty acids
and liquid unsaturated fatty acids having different chain
lengths. The composition of the fatty acids and their
distribution within the triglyceride molecules are particular
10 to each fat and determine its properties and, in particular,
its melting behavior. Since natural fats are mixtures,
(eutectic) interaction phenomena occur within a certain
temperature range, modifying the physical state of the
constituents, the triglycerides being capable in addition
15 of crystallizing in several forms.
- Vegetable fats, for example cocoa butter, generally
show these crystalline polymorphism phenomena around ambient
temperature in cases where the disaturated-monounsaturated
and monosaturated-diunsaturated triglycerides are pre-
20 ponderant. Apart from cocoa butter, these fats are not
directly used in foods because of their plastic character
and it has been proposed to fractionate them to enable
them to be used either as table oil or as confectionery
fat, for example as a substitute for cocoa butter.
- 25 The confectionery/chocolate industry uses large
quantities of cocoa butter because of its unique physical
properties which are highly valued in that industry: it is
solid up to 25°C, melts rapidly in a narrow temperature
range between 35 and 37°C (particularly in the mouth, which

1 produces sensations of melting and taste) and hardens
rapidly with controllable shrinkage on cooling (which
enables it to be removed from moulds). Accordingly,
consideration has been given to the use of other vege-
5 table fats available in greater abundance as a substitute
fat because the climatic zones in which cocoa can be
grown are restricted and the supply of cocoa butter is
limited. Fats of the type in question have to show
physical behavior very similar to that of cocoa butter
10 to be compatible therewith. With the object of producing
such substitutes, vegetable fats, particularly palm oil,
are industrially fractionated by various methods:
- A first method using solvents comprises cooling a
solution of the fat in an organic solvent, for example
15 acetone, hexane, isopropanol, 2-nitropropane,
dichlorodifluoromethane, trichlorotrifluoroethane,
separating the stable β and β' crystalline forms
required by filtration and eliminating the solvent by
evaporation. This method may be applied to natural
20 oil, for example as described in GB-PS 827 172, or
to a concrete fraction obtained after a first dry
fractionation step. To increase the yield of table oil
whilst at the same time producing a fraction suitable
for use in confectionery, this method has been applied
25 to an oil that has undergone catalytic interesterification
(or transesterification), i.e. an internal rearrangement
of the fatty acids in the triglyceride molecule, for
example as described in French Patent Application
2 427 386. Another process described, for example, in
30 US-PS 3 431 116 comprises selectively hydrogenating a
stearin obtained as by-product in the manufacture of
table oils from cotton seeds to convert the linoleic
acid into oleic acid before the fractionation treat-
ment with solvents. Although all these known processes
35 enable fractions suitable for use in confectionery to be

1 obtained, they have the disadvantage of using
chemical agents and/or solvents which have to be treated
and recycled and also eliminated from the fats.
This requires heavy installations which are not always
5 available in the regions where the starting material
is gathered and involves considerable energy consumption.
- A second method carried out in aqueous medium is
based on the wettability of the crystals in the
presence of a surfactant which enables an aqueous
10 suspension of crystals to be separated from the liquid
oil, the crystals being subsequently collected by
centrifuging. This method has the disadvantage of
producing a large volume of waste waters.
- A third method comprises dry fractionation. In this
15 method, which is described for example in German
Patent Application 29 16 604, the entirely liquid fat
is treated in a controlled manner and, after the molten
mass has been seeded with β crystals of the fat at 45°C,
the melt is cooled and the crystals formed are filtered.
20 The operation is carried out a second time at a lower
temperature. This method does not prevent the
occlusion of part of the liquid fraction in the crystal
aggregates, which leads either to a fraction that
does not have the required characteristics or to a
25 low yield at the separation stage. This confirms the
opinion generally held among experts that it has not
hitherto been possible to obtain a concrete fraction
compatible with cocoa butter by dry separation from
palm oil in a yield comparable with that obtained where
30 solvents are used.

The present invention seeks to provide a process for the
dry fractionation of vegetable fats capable of crystallizing
in several forms at temperatures around ambient temperature
which gives concrete fractions compatible with cocoa butter
35 in a yield comparable with that obtained by fractionation

1 with solvents without having any of the disadvantages
attending the known processes of aqueous or solvent fraction-
ation.

5 The process according to the invention comprises the
following steps:

- (a) a pre-refined fat (as herein defined) is heated to
convert it entirely into liquid form,
- (b) the liquid of step (a) is cooled to a temperature
3 to 10°C above its solidification temperature,
- 10 (c) it is then seeded with β or β' crystals obtained from
said fat or from cocoa butter and stabilized in the
required form by storage,
- (d) the suspension of step (c) is progressively cooled at
a rate of 0.5 to 1.5°C per hour to a fractionation
15 temperature in the range from 15 to 35°C and
- (e) the suspension of step (d) is filtered under a pressure
rising to at least 20 kg/cm² at the fractionation
temperature and a concrete fraction (C_1) having an
iodine value of at most 50 and a fluid fraction (F_1)
20 having a higher iodine value than the concrete fraction
(C_1) are collected, the difference between the iodine
value of the fraction (C_1) and that of the fraction
(F_1) being at least 10, and if necessary the sequence
of operations from the fluid fraction (F_1) is repeated
25 until the desired concrete fraction is obtained.

The vegetable fats capable of being fractionated in
accordance with the invention are those which show a
crystalline polymorphism around ambient temperature, i.e.
between 20 and 35°C. Vegetable fats such as these, which
30 are known generically as vegetable tallows, include shea
butter (karite, *Butyrospermum parkii* or *Bassia parkii*),
sal butter (*Shorea robusta*), Borneo tallow (*Shorea*
stenoptera), kokum butter (*Garcinia indica*), mango kernel butter
(*Mangifera indica*), mowra fat (*Madhuca latifolia* and
35 *Madhuca longifolia*), palm oil (*Elaeis guineensis*), etc.

- 1 The expression "pre-refined fat" means that the fat
has been subjected to
- the removal of mucilages to insolubilize the phospho-
 lipids, the polysaccharides, the gums, the resins,
5 etc.... which are separated by centrifuging,
 - neutralization to eliminate most of the free fatty
 acids in the form of soaps to a free fatty acid content
 of less than 0.2% and preferably less than 0.1% by
10 weight. The fractionated fat is preferably refined
 in known manner by the following successive additional
 operations:
 - decoloration by passage in molten form over active
 carbon or other adsorbents, for example bleaching earths
 based on activated aluminium silicate, such as mont-
15 morillonite or bentonite, and filtration to remove the
 chlorophyll and the carotenoid pigments and
 - deodorization by stripping with steam in vacuo at a
 temperature below 260°C and preferably at a temperature
 below 200°C.
- 20 All the pre-refining and refining steps have to be
carried out in such a way as to avoid isomerization of the
triglycerides.

 The process according to the invention may be carried
out in accordance with schemes 1 and 2 of the accompanying
25 drawing (Figures 1 and 2) which represent two embodiments
of the invention respectively comprising one or two
fractionation cycles.

Scheme 1

 This embodiment comprises a single fractionation
30 cycle and is particularly suited to vegetable tallows, such
as mango butter, sal butter, mowra fat or kokum butter,
which are rich in saturated and monounsaturated acids
and which have a particular configuration of the triglycerides
characterized by a high percentage of 2-monounsaturates
35 and a low percentage of trisaturates. This composition makes

1 them fairly similar to cocoa butter from the chemical
and physico-chemical viewpoints.

As indicated in Table I below, which gives their
composition in % of the fatty acids and their 2-mono-
5 unsaturated triglycerides in % of the triglycerides,
these fats contain more linoleic and oleic acid and,
correlatively, more monosaturates-diunsaturates than cocoa
butter. The fractionation described hereinafter reduces
the content of these triglycerides in the concrete
10 fraction (C_1), bringing its physico-chemical properties
into line with those of cocoa butter and making it
compatible with cocoa butter. The fraction (1) may there-
fore be used as an ingredient of a cocoa butter substitute
in chocolate.

Table I

15		<u>Mango</u>	<u>Sal</u>	<u>Mowra</u>	<u>Kokum</u>
	Palmitic acid	8-9	6-7	16-24	2-3
	Stearic acid	40-42	42-44	20-25	52-58
	Oleic acid	40-42	38-42	43-45	38-40
	Linoleic acid	5-8	4-7	9-14	2-3
20	2-monounsaturates	46-50	64-79	26-30	73-78

The fractionation cycle comprises the following steps:

- 1a) The refined fat is heated to convert it entirely into
liquid form, i.e. to a temperature of from 60 to 80°C,
25 depending on the fat used, for example to around 60°C
for sal butter and to around 70°C for mango butter.
- 1b) The liquid fat is cooled to a temperature 5 to 10°C
above its solidification temperature, i.e. generally
to 30-45°C.
- 1c) The cooled liquid fat is seeded with stabilized β
30 crystals to direct crystallization towards the form-
ation of a concrete fraction (C_1) of high melting
point or stearin. The β crystals consist essentially
of saturated fatty acid triglycerides. They constitute
35 the polymorphic form of those triglycerides which is
stable and has a higher melting point or a higher
melting range. The first

- 1 seed crystals may be obtained by straightforward crystallization of the molten fat at 30-35°C and filtration.

5 Once production has started, β crystals emanating from the stearin (C_1) obtained during the fractionation of a preceding batch may be used for seeding a batch. These β crystals may emanate from cocoa butter or preferably from fractionated fat. Fractionation may have been carried out by the dry method or using a solvent, 10 the solvent having been subsequently eliminated. Whatever their origin, the β crystals will preferably have been stored for at least 4 weeks at 30-35°C. The crystals are added in a quantity of from 0.05 to 0.2% by weight either as such or in the form of a 5-15% by weight 15 suspension in the corresponding fluid fraction (F_1). The suspension is slowly stirred in order thoroughly to mix the crystals.

1d) The suspension obtained in step 1c) is progressively cooled at a rate of 0.5 to 1.5°C/hour and preferably at a rate of 20 1°C/hour to facilitate the formation of coarse crystals down to the fractionation temperature which is preferably in the range from 20 to 35°C. Alternatively, the suspension may be left standing for 8 to 15 hours at the seeding temperature and then cooled under the conditions 25 described above. The suspension may also be cooled as described above and then left standing for 8 to 15 hours at the fractionation temperature. The same operation may be carried out at an intermediate temperature by interrupting cooling.

30 Crystallization is carried out either in tanks or on trays.

1e) Fractionation is carried out by means of a hydraulic press, for example a frame press. To this end, the suspension obtained in step 1d), which has assumed a 35 paste-like consistency due to the crystal aggregates, is

1 transferred to cloths or "press bags" which are
arranged in stacks, each bag being separated from the
adjacent bags by metal plates provided with drainage
grooves. Alternatively, it is possible to use a semi-
5 continuous automatic press of the type used for
separating cocoa butter from the nibs, suitably modi-
fied for example, by replacing the metal filters
by cloths having a mesh width of at most 2 microns.

10 In this step, the pressure should be applied in
stages and should reach a level of at least 20 kg/cm^2
to obtain as complete as possible an expulsion of the
fluid fraction. Preferably, the pressure is increased
in stages of around $6-7 \text{ kg/cm}^2$, the pressure applied
being maintained for at least 5 minutes, to at least
15 30 kg/cm^2 , for example to 45 kg/cm^2 , the maximum
pressure being maintained for at least 1 hour, for
example 1-2.5 hours. The operation lasts at most 1.5
to 3 hours.

20 Depending on the composition of the fat used, the
concrete fraction (C_1) obtained represents from 10 to 90%
by weight of the starting fat and its iodine value is
in the range from 18 to 50. The iodine value depends
upon the degree of unsaturation of the fatty acids contained
in the triglycerides and is the number of grams of iodine
25 fixed by 100 g of fat. In the present case, it indicates
the degree of separation of the concrete fraction from
the fluid fraction. Preferably the IV of the concrete
fraction (C_1) is from 33 to 42. The fluid fraction (F_1)
or olein has an iodine value of from 45 to 70.

30 The fluid fraction (F_1) may be used in foods either
as such or after slight hydrogenation, for example as table
oil.

Scheme 2

35 This embodiment comprises two successive fractionation
cycles and is particularly suited to palm oil which has a
comparatively higher content of triunsaturated triglycerides
having a high melting range. Table II below gives its
composition in % of the fatty acids and also the number of

- 1 2-monounsaturated and trisaturated triglycerides in % of the triglycerides.

Table II

	Palmitic acid	41-49
5	Stearic acid	4-6
	Oleic acid	27-53
	Linoleic acid	5-10
	2-monounsaturates	40-45
	Trisaturates	6-9

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In this case, the fractionation of step 1e) of the first cycle (according to scheme 1) makes it possible to separate a concrete fraction (C_1) having an iodine value of 18-24 in a yield of 10 to 12% by weight. This fraction (C_1) has a melting range of 45-55°C. It may be used for hardening shortenings or margarines or may even form part of cosmetic or pharmaceutical products.

The fluid fraction (F_1) undergoes a second fractionation cycle under the following conditions:

- 20 2a) The fluid fraction (F_1) having an iodine value of greater than 55 and preferably from 56 to 56.5 represents from 90 to 93% by weight of the starting fat. The starting fat is heated to a temperature of from 40 to 60°C and preferably to a temperature of approximately 45°C to convert it completely into liquid form.
- 25 2b) The liquid obtained in step 2a) is cooled to a temperature around 30°C which is 3-5°C above the solidification temperature of the fraction (F_1).
- 30 2c) The cooled liquid is seeded with stabilized β' crystals to direct crystallization towards the formation of a desired intermediate concrete fraction (C_2). The β' crystals consist essentially of 2-monounsaturated triglycerides which may be obtained by the crystallization of a fraction (F_1) heated to approximately 45°C and cooled to a temperature of 20-30°C, followed by
- 35

1 filtration. Once production has started, β'
 crystals emanating from a fraction (C_2) of a
 preceding batch will preferably be used for seeding
5 a batch. Preferably, these crystals will have been
 stabilized by storage for at least 4 weeks at 20-30°C.
 They are added as such in a quantity of from 0.05
 to 0.2% by weight or in the form of a 5-15% by
 weight suspension in the corresponding fluid
 fraction (F_2) with slow stirring.

10 2d) The suspension obtained in step 2c) is
 cooled at a rate of 0.5-1.5°C/hour and preferably
 at a rate of 1°C/hour to 15-20°C, preferably after
 it has been left standing for 8 to 15 h at the
 seeding temperature.

15 The suspension is then left standing for 72-120
 hours at 15-20°C and preferably at 15-18°C.

2e) The suspension is fractionated as indicated above
 in reference to scheme 1 under the following
 particular conditions: the pressure is increased in
20 stages of 6-7 kg/cm², the pressure applied in each
 stage being maintained for at least 5 minutes and
 preferably for 10 to 15 minutes. The maximum
 pressure is at least 30 kg/cm² and preferably 45 kg/cm²
 and is maintained for at least 1h and preferably for
25 around 2h.

 In the present case, it is important to ensure that
 the temperature does not exceed 18°C, particularly at the
 beginning of fractionation. This is because excessive
 heating during pressing could result in partial melting
30 of the crystals which would in turn result in a reduction
 in yield and would promote the subsequent appearance of
 undesirable crystal forms.

 The foregoing procedure gives a concrete fraction (C_2)
 having physico-chemical characteristics very similar to
35 those of cocoa butter, and preferably having an iodine value
 of 36-38, in an overall yield of around 30% by

1 weight, based on the fat used, and a fluid fraction (F_2)
having an iodine value of 65-68.

5 In one preferred embodiment, it is possible to improve the
overall yield of concrete fraction (C_2) to 33% by leaving
the fluid fraction (F_2) to crystallize for 2 to 3 days
at approximately 15°C and recovering a concrete fraction
(C_3) by pressing under the conditions applied to the
fraction (F_2); the fraction (C_3) may be added to the
following batch (F_1) in step 2e).

10 The concrete fraction (C_2) melts at body temperature
and, accordingly, has many uses in confectionery and
biscuit manufacture as a substitute for cocoa butter, in
cosmetics, for example as a cream, lotion or lipstick base,
and in the pharmaceutical field as a medicament support,
15 for example as a suppository. In chocolate manufacture,
it may be used on its own or in combination with a
vegetable tallow in a fat composition intended as a
substitute for cocoa butter. In a fat composition such
as this, the vegetable tallow will preferably be in the
20 form of one or more concrete fraction(s) (C_1), for example
sal butter or mango butter. Thus a fat composition suitable
for use in confectionery/chocolate manufacture as a substitute
for cocoa butter may contain from 0 to 60% by weight of one
or more concrete fraction(s) (C_1) and from 40 to 100% of
25 a concrete fraction (C_2). The concrete fraction(s) (C_1)
will advantageously represent from 10 to 60%, for example
50%, by weight of the composition. The confectionery/
chocolate product may comprise a fat phase which contains
from 10 to 20% by weight of the fat composition intended
30 as a substitute for cocoa butter and from 80 to 90% by
weight of cocoa butter.

The fluid fraction (F_2) may be used as table oil,
more particularly as frying oil.

35 The invention is illustrated by the following Examples
in which the percentages are percentages by weight. In
the Examples, I.V. is the iodine value as determined by
titration with sodium thiosulfate of the iodine released

1 by the addition of potassium iodide to a solution contain-
ing the fat and iodine chloride (reagent) by comparison with
a reference solution of the reagent without the fat
5 (Method 2.205, I.U.P.A.C. "Standard Methods for the Analysis
of Oils, Fats and Derivatives", Pergamon Press, 6th Edition,
1979).

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- 1 In the following Comparison Example, an iodine
value balance was made. This operation comprises
comparing the I.V. of the starting fat with the weighted
sum of the I.V.'s of the concrete fraction (C) and the
5 fluid fraction (F) obtained:

$$I.V. = I.V_C \cdot C + I.V_F \cdot F$$
 C and F being, respectively,
the percentages of concrete fraction and fluid fraction
obtained by fractionation.

- 10 The suitability of a concrete fraction (C_1) or (C_2)
to be mixed with cocoa butter is verified
- by studying its behavior on cooling, giving its
solidification point, under clearly defined conditions
(Shukoff's method, reference temperature 0°C,
15 I.U.P.A.C., Method 2.132),
 - by determining its melting curve, giving the solid
fat index (S.F.I.) or the percentage of solid fat
present in the partially melted fat at a given
temperature, by pulsed NMR (nuclear magnetic resonance of
20 the proton) (I.U.P.A.C., 1st Supplement, 1982
Method 2.323) or by dilatometry (I.U.P.A.C., 6th
Edition, 1979, Method 2.141),
 - by its use in chocolates in conjunction with cocoa
butter either on its own or in admixture with another
25 concrete fraction.

The following Comparison Example shows the yields
and I.V.'s obtained for the fraction (C_2) of palm oil
in accordance with the present invention (A) by
comparison with the fractionation of palm oil with
30 hexane (B) and acetone (C), respectively, under the
following conditions:

- (B) The fluid fraction (F_1) emanating from fractionation
in accordance with scheme 2 above is heated to 45°C
and dissolved in hexane in a ratio by weight hexane
35 to olein of 3:1, after which the solution is cooled to

- 1 -10°C and left to crystallize for 24h at -10°C, the
 suspension is stirred for 5 minutes at 3-4 r.p.m.
 4h before the separation of the crystals and the
 crystals are filtered using a frame filter. The
5 crystals are collected and melted and the solvent
 evaporated.
- (C) The procedure is as in (B) except that a ratio by
 weight of acetone to fluid fraction (F_1) of 6:1 is
 used and the suspension is crystallized for 24h at 5°C.
- 10 Where solvents are used, as in (B) and (C) above,
 separation of the crystals may be carried out in any
 apparatus suitable for liquid/solid separation without
 the application of pressure, for example in a filter
 or a centrifugal separator.

15

Example 1

Preparation of palm olein (F_1)

- 100 kg of palm oil freed from mucilages and
 neutralized (containing at most 0.1% of free fatty acids),
20 I.V. 53, are heated to 60°C and then cooled to 45°C.
- The oil is seeded at 45°C with 100 g (0.1%) of
 β crystals of concrete palm fraction (stearin) prepared
 as described hereinafter. After it has been transferred
 to a tank, the suspension is progressively cooled to 30°C
25 in 12h. The suspension of the crystals formed is filtered
 in a frame press. The pressure cycle used is 2h at 30°C,
 the maximum pressure of 30 kg/cm² being reached in 35 mins.
 at intervals of 7 mins. 90 kg of clear fluid fraction
 (olein F_1) having an I.V. of 56-56.5 and 10 kg of a
30 concrete fraction (stearin C_1) having an I.V. of 22-24,
 representing 10% of the oil used, are collected. The
 iodine value of the stearin (C_1) depends upon the separation
 efficiency: thus, a low iodine value is indicative of
 a high separation yield. A precise iodine value for the
35 olein (F_1) is merely indicative of orientation of the

1 crystallization process without detriment to the yield.

Preparation of the concrete fraction (C_2)

90 kg of the preceding fraction (F_1) are heated to 45°C and seeded with 90 g (0.1%) of stabilized β' crystals (fraction C_2) prepared as described above. After the crystals have been mixed with the liquid, the suspension is cooled for 10h to 26-27°C. It is then transferred in batches of 25 kg onto trays at that temperature, cooled to 18°C in 8h and left to crystallize for 4 days at 18°C, care being taken to ensure that the temperature of the trays stays between 17 and 19°C.

The suspension is then transferred to press bags which are stacked in a hydraulic press and collectively subjected to a pressure increasing progressively in stages of 6-7 kg/cm² every 15 minutes to 20 kg/cm², i.e. in 45 minutes, which corresponds to a reduction of the charge of around 30%. The duration of the increase in pressure naturally depends upon the type of press used and its geometry. The pressure is then increased in stages of 6-7 kg/cm² every 15 minutes to 45 kg/cm², i.e. in 1h and pressing is continued at that pressure for 2h. A concrete fraction (C_2), I.V. 36-38, yield 30%, and a fluid fraction (F_2) having an I.V. of 65-67 are obtained.

Preparation of β and β' crystals of palm stearin

25 The β seed crystals are prepared by tempering the palm oil for 2 weeks at 30-35°C. The crystals formed are separated by filtration and left standing for 2 weeks at 30-35°C.

To prepare the β' seed crystals, the fluid fraction (F_1) is tempered for 2 weeks at 20-28°C. The crystals formed are separated by filtration and left standing for 2 weeks at 20-28°C.

Once production has started, part of the stearin fractions (C_1) and (C_2), respectively, may be removed from the following batches and tempered as indicated above for

- 1 at least 2 weeks in order to collect stabilized β and β' crystals, respectively, the crystals then being left standing for 2 weeks.

Comparison Example

- 5 A refined palm oil having an I.V. of 53 is fractionated
- (A) by the method of Example 1,
 - (B) as previously described, the last step using hexane,
 - (C) as previously described, the last step using acetone,
 - (D) in accordance with Example 3 of German Patent

- 10 Application 29 16 604.

The yields based on the fat used and the I.V.'s of the various fractions are shown in Table III below:

(31)

Table III

	A		B		C		D	
	I.V.	Yield %	I.V.	Yield %	I.V.	Yield %	I.V.	Yield %
Starting palm oil	53		53		53		53	
1st pressing or filtration for D								
Stearin (C_1)	22-24	10	22-24	10	22-24	10	31 ¹	16.7
Olein (F_1)	56.5		56.4		56.5		58 ²	
2nd pressing or filtration for B, C & D								
Stearin (C_2)	36-38	33	38-40	35.	36	40	33 ³	52
Olein (F_2)	65-67		68		70		60 ³	48
Overall yield		30 ⁴		32		36		43

1 Legend:

1. The I.V. of the stearin is too high and shows that a certain quantity of olein (F_1) is still present due to the poor separation
- 5 2. On the basis of the yields indicated, evaluation of the I.V.'s gives an I.V. of 58 for the olein (F_1).
3. On the basis of the I.V.'s indicated, the I.V. balance gives :
$$58 = .33.C + 60.F \text{ with } C+F = 1, \text{ i.e. } C = 0.07$$
- 10 In fact, only 7% of stearin (C_2) having an iodine value of 33 is obtained.
4. The overall yield may be increased to 33% if the olein (F_2) is left to crystallize for 2 to 3 days at 15°C. A concrete fraction (C_3) (I.V. 52-53, yield 15%) and a fluid fraction (F_3) (I.V. 67-68, yield 85%) are thus recovered by pressing under the conditions described in Example 1 in reference to the preparation of the concrete fraction (C_2). The concrete fraction (C_3) may be added to the following batch (F_1).
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Table IV below shows the S.F.I. and Shukoff values of the concrete fractions (C_2) obtained by methods A, B and C compared with those obtained for cocoa butter.

Table IV

		Concrete fraction (C ₂) acc. to A	Concrete fraction (C ₂) acc. to B	Concrete fraction (C ₂) acc. to C	Cocoa butter
I.V.					
S.F.I. (%) at temperature (°C)	25	36-38	38-40	36	36
	30	54	52	62	63
	35	32	25	35	41
	40	5	2	5	0
		0	0	0	
Shukoff cooling curve (0°C)					
Minimum temperature (°C) in time (mins)		18.0 27	14.7 37	18.4 30	17.7 39
Maximum temperature (°C) in time (mins.)		20.6 50	18.3 70	22.1 52	20.6 60
Δ temperature (°C)		2.6	3.6	3.7	2.9
Δ time (mins.)		23	33	22	21
Δ temperature/Δ time (°C/mins.)		0.11	0.11	0.17	0.14

1 EXAMPLES 2-3

The analytical values for these Examples (S.F.I., yield, I.V., Shukoff cooling curve) are set out in Table V below.

- 5 2. 100 kg of refined sal butter are heated to 60°C and, after cooling to 30°C, are seeded with 1 kg of a suspension containing 10% of β crystals of sal stearin in liquid sal olein, these crystals having been stabilized as indicated in Example 1. The suspension obtained is
- 10 left standing for 12h at 30°C and cooled to 26°C in 4h. The suspension is fractionated in 2h as described in Example 1 in reference to the preparation of the fraction (F_1), except that the operation takes place at 26°C and the maximum pressure is 35 kg/cm². 80 kg of a concrete
- 15 fraction (C_1) are obtained.
3. 1 kg of refined mango kernel butter is heated to 68°C, cooled to 30°C and seeded with 1 g (0.1%) of β crystals of mango stearin stabilized as described in Example 1. The suspension is cooled for 5h to 23°C and then kept for 15h
- 20 at 23°C. The suspension is fractionated in 2h as described in Example 1 in reference to the preparation of the fraction (F_1), except that the operation takes place at 23°C and the maximum pressure is 36.5 kg/cm². 670 g of a concrete fraction (C_1) are obtained.

Table V

1	Example	2	3
	S.F.I. at 25 (°C)	74	45
	30	69	37
	35	41	9
5	40	0	0
	Yield of stearin (%)	78	70
	I.V. stearin	33	42
	olein	45	61
	Shukoff cooling curve (0°C)		
10	Minimum temperature (°C) in time (mins.)	18.8 43	18.0 32
	Maximum temperature (°C) in time (mins.)	25.1 51	26.4 44
	Δ temperature (°C)	6.3	8.4
	Δ time (mins.)	18	12
	Δ temperature/Δ time (°C/min.)	0.35	0.7

15

EXAMPLE 4

4 chocolate masses are prepared from the following ingredients.

The foreign fat consists of: 100% of concrete fraction (C₂) of Example 1 (I); 10% of concrete fraction (C₁) of Example 2/90% of concrete fraction (C₂) of Example 1 (II) and 40% of concrete fraction (C₁) of Example 2/60% of concrete fraction (C₂) of Example 1 (III).

<u>Ingredient, % by weight</u>		<u>Milk chocolate</u>	<u>Plain chocolate</u>
25	Sugar	43.3	48.7
	Nibs (Ghana)	12.0	45.2
	Milk powder (26% fat, based on dry matter)	27.5	-
	Cocoa powder (12-14% cocoa butter, based on dry matter)	-	0.7
30	Deodorized cocoa butter	11.8	-
	Foreign fat (corresponds to approx. 16-18% of the total fat)	5.0	5.0
	Lecithin	0.4	0.4
35	Vanillin (traces)	-	-

- 1 The various ingredients, except the lecithin, the
vanillin and some of the fat, are mixed at 40°C in such
a way that the mixture contains from 27 to 29% by weight
of fat, after which the mixture is finely ground in a roll
5 mill. The ground mixture is then conched for 24h at 55°C,
the remainder of the fat being added in portions. The
vanillin and then the lecithin are added just before the
end of conching. After conching, the milk chocolate
is cooled to 27°C and the plain chocolate to 29°C,
10 followed by tempering at 29°C and at 30-31°C, respectively.

The compatibility of the foreign fat with the cocoa
butter is evaluated by the following tests:

Mould releasability

- 15 Metal moulds are filled with 100 g of the tempered
chocolate and placed in a refrigerated chamber at 4°C.
After about 5 mins, the moulds are inverted and vibrated
and the time (mins.) which the chocolate takes to separate
from the mould is recorded.

Fat bloom

- 20 Fat bloom is the appearance of a whitish layer of fat at
the surface of the chocolate. This undesirable phenomenon
occurs more readily the more the foreign fat interacts
with the cocoa butter. It is thus a good indication of
its compatibility. The chocolate is subjected to 30
25 tempering cycles comprising 12h at 15°C, then 12h at 28°C
and finally another 12h at 15°C and the results obtained
are recorded on a scale of 1 to 5 as follows:

- 1 = no change (shiny appearance)
2 = slightly clouded
30 3 = clouded
4 = fat bloom
5 = considerable bloom

- The number of tempering cycles after which there is
a change of 1 unit on the scale is also noted. 1/18, 2/6,
35 3/6 for example signifies that, after 18 cycles, the

sample has a rating of 2, after 6 more cycles it moves onto 3 and remains at 3 after 30 cycles.

The results are shown in Table VI below:

Table VI

Sample	Milk chocolate		Plain chocolate	
	Whitening	Mould separation time (mins.)	Whitening	Mould separation time (mins.)
I	1/30	26.5	1/24;3/6	19.3
II	1/30	31.3	1/24;3/6	21.6
III	1/30	27.9	1/18;2/6;3/6	20.6
Cocoa butter	1/30	25	1/20;2/4;3/6	20



28

1 CLAIMS

1. A process for the dry fractionation of a vegetable fat capable of crystallizing in several forms at temperatures around ambient temperature with a view to obtaining a concrete fraction compatible with cocoa butter, characterized by the following steps:
- 5 (a) a pre-refined fat (as herein defined) is heated to convert it entirely into liquid form,
- 10 (b) the liquid of step (a) is cooled to a temperature 3 to 10°C above its solidification temperature,
- 15 (c) it is then seeded with β or β' crystals obtained from said fat or from cocoa butter and stabilized in the required form by storage,
- 20 (d) the suspension of step (c) is progressively cooled at a rate of 0.5 to 1.5°C per hour to a fractionation temperature in the range from 15 to 35°C and
- 25 (e) the suspension of step (d) is filtered under a pressure rising to at least 20 kg/cm² at the fractionation temperature and a concrete fraction (C₁) having an iodine value of at most 50 and a fluid fraction (F₁) having a higher iodine value than the concrete fraction (C₁) are collected, the difference between the iodine value of the fraction (C₁) and that of the fraction (F₁) being at least 10, and if necessary the sequence of operations from the fluid fraction (F₁) is repeated until the desired concrete fraction is obtained.
- 30 2. A process as claimed in Claim 1 comprising a single fractionation cycle, characterized in that the fat used is a vegetable tallow, particularly mango or sal butter, and in that, in step c), the liquid of step a) is seeded with from 0.05 to 0.2% by weight of stabilized β crystals of said fat or of cocoa butter obtained by storage for at least 4 weeks at 30-35°C.
- 35 3. A process as claimed in Claim 2, characterized in that, in step d), the suspension is left standing for 8 to 15 hours



at the seeding temperature, at the fractionation temperature or at an intermediate temperature.

4. A process as claimed in Claim 2, characterized in that, in step e), the suspension of step d) is filtered under a pressure increasing progressively in stages of approximately 6-7 kg/cm², the pressure applied being maintained for at least 5 minutes, to a maximum pressure of at least 30 kg/cm², the maximum pressure is maintained for at least 1 hour and a concrete fraction (C₁) having an iodine value of from 33 to 42, which represents the desired concrete fraction, is collected.

5. A process as claimed in Claim 1 comprising two successive fractionation cycles, characterized in that the fat used is palm oil, in that in step c) of the first fractionation cycle the liquid is seeded with stabilized β crystals and in that the second fractionation cycle comprises the following steps applied to the fraction(F₁):

2a) the fluid fraction (F₁) having an iodine value of greater than 55 is heated to a temperature of from 40 to 60°C to convert it completely into liquid form,

2b) the liquid of step 2a) above is cooled to a temperature 3 to 5°C above its solidification temperature,

2c) the cooled liquid is seeded with stabilized β crystals of palm oil obtained by storage for at least 4 weeks at 20-30°C,

2d) the suspension of step 2c) above is progressively cooled at a rate of 0.5 to 1.5°C/hour to a temperature of 15 to 20°C and left standing at that temperature for 72 to 120 hours so that crystallization may continue,

2e) the suspension of step 2d) is filtered under a pressure increasing progressively in stages of approximately 6-7 kg/cm², the pressure applied being maintained for at least 5 minutes, to a maximum pressure of at least 30 kg/cm², the maximum pressure is maintained for at least 1 hour and a concrete fraction (C₂) having



(40)

- 25 -

an iodine value of 36-38, which represents the desired concrete fraction, and a fluid fraction (F_2) having an iodine value of 65-68 are collected.

- 5 6. A process as claimed in Claim 5, characterized in that, in step c) of the first fractionation cycle, the liquid is seeded with from 0.05 to 0.2% by weight of stabilized β' crystals of palm oil and in that, in step 2c) of the second fractionation cycle, the liquid is seeded with from 0.05 to 0.2% by weight of stabilized β' crystals
- 10 of palm oil.
7. A process as claimed in Claim 5, characterized in that in step 2d) of the second fractionation cycle, the suspension is left standing for 8 to 15 hours at the seeding temperature before it is cooled.
- 15 8. A process as claimed in Claim 5, characterized in that the fluid fraction (F_2) is left to crystallize for 2 to 3 days at approximately 15°C, in that the suspension obtained is filtered under the fractionation conditions of step 2e) of the second fractionation cycle and in that a concrete
- 20 fraction (C_3), which is added to the suspension of step 2d) during a second fractionation cycle applied to a subsequent batch, and a fluid fraction (F_3) are collected.
9. A concrete fraction (C_1) of mango or sal butter obtained by the process claimed in Claim 2.
- 25 10. A concrete fraction (C_2) of palm oil obtained by the process claimed in Claim 5.
11. A fat composition suitable for use in confectionery/ chocolate manufacture as a substitute for cocoa butter containing from 0 to 60% by weight of one or more concrete
- 30 fraction(s) (C_1) of the type claimed in Claim 9 and from 40 to 100% of a concrete fraction (C_2) of the type claimed in Claim 10.
12. A confectionery/chocolate product of which the fat phase contains from 10 to 20% by weight of the fat
- 35 composition claimed in Claim 11 and from 80 to 90% by weight

41

- 26 -

of cocoa butter.

13. A table oil or frying oil containing a fluid fraction (F_1), (F_2) or (F_3) obtained by the process claimed in Claim 2, 5 or 8.

