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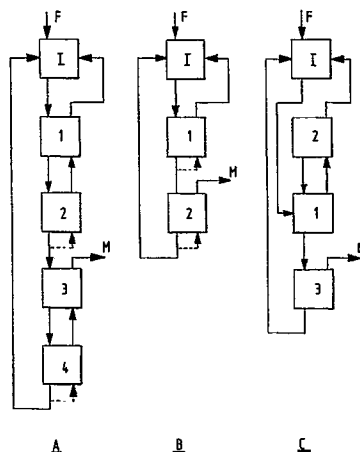
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NL-2517 GK The Hague(NL)(54) **Mid-fraction production by fractional crystallization with stearin and olein fraction recycling to interesterification.**

(57) The invention relates to a method for the production of mid-fraction by fractional crystallization of fatty substances, in which:

- i) a fatty substances comprising feedstock is subjected to a topping fractional crystallization treatment providing a stearin topping fraction and an olein topping fraction;
- ii) the olein topping fraction is subjected to a bottoming fractional crystallization treatment providing the mid-fraction and an olein bottoming fraction;
- iii) the stearin topping fraction and the olein bottoming fraction are combined and subjected to inter-esterification, and the interesterified mixture of fatty substances is recycled to the topping fractional crystallization treatment.

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The present invention relates to a method for the production of mid-fraction by fractional crystallization of fatty substances, including fats and glyceride oils. In particular, the invention relates to the production of mid-fraction by topping fractional crystallization and bottoming fractional crystallization, in which the stearin topping fraction and olein bottoming fraction formed as byproducts are mixed and interesterified, and the interesterified mixture is recycled as additional feedstock.

Natural glyceride oils and fats comprise a great many different triglycerides, the physical properties of which to a large extent are determined by the chain lengths and the degrees of unsaturation of the fatty acid moieties. To make natural glyceride oils or fats more suitable for particular applications it is often required to separate them into fractions characterized by fatty acid glyceride distributions which are homogeneous with respect to the melting behaviour.

The fractional crystallization may be carried out on the oils and fats as such (dry fractionation) or after mixing with a solvent (solvent fractionation). In the fractional crystallization treatment the oil is cooled to a temperature at which only a higher melting triglyceride fraction crystallizes, followed by separation of the crystallized solids (stearin fraction) from the liquid fraction (olein fraction) e.g. by filtration or centrifugation.

In the production of mid-fraction the oil or fat is first subjected to a topping fractional crystallization treatment providing a stearin topping fraction and an olein topping fraction. This olein topping fraction is subjected to a bottoming fractional crystallization treatment at a lower crystallization temperature, providing the aimed mid-fraction and an olein bottoming fraction.

It is understood that the topping fractional crystallization treatment and the bottoming fractional crystallization treatment may comprise more than one consecutive fractional crystallizations in which the olein fraction and/or stearin fraction are passed to the next or former fractional crystallization.

Mid-fraction is a desired starting oil or fat blend for producing margerines. Mid-fraction possesses a good consistency and a good melting performance imparting the margerines obtained good organoleptic properties and a suitable consistency at a temperature within the range of 15-25°C. However, the production of mid-fraction inevitably results in the formation of two byproducts, namely a stearin topping fraction and an olein bottoming fraction. These fractions are generally less valuable, so that the method for the production of mid-fraction is not optimal from an economic point of view.

The invention is based on the finding, that when the feedstock comprising fatty substances, used for the production of mid-fraction has a fatty acid composition of the triglycerides which is similar to that of the mid-fraction produced, and the stearin topping fraction and the olein bottoming fraction are mixed and this mixture is randomly interesterified, this interesterified mixture should have a fatty acid composition similar to that of the feedstock and may be recycled, substantially avoiding the formation of byproducts.

Accordingly, the present invention provides a method for the production of mid-fraction by fractional crystallization of fatty substances, in which:

- i) a fatty substances comprising feedstock is subjected to a topping fractional crystallization treatment providing a stearin topping fraction and an olein topping fraction;
- ii) the olein topping fraction is subjected to a bottoming fractional crystallization treatment providing the mid-fraction and an olein bottoming fraction;
- iii) the stearin topping fraction and the olein bottoming fraction are combined and subjected to interesterification, and the interesterified mixture of fatty substances is recycled to the topping fractional crystallization treatment.

A requisite for the method according to the invention is, that the stearin topping fraction and the olein bottoming fraction are subjected to a random interesterification. Therefore, the method according to the invention is not suitable for the production of mid-fraction comprising specific symmetrical or asymmetrical triglycerides.

As feedstocks any fatty substance may be used, such as semi-solid glyceride oils and fats of vegetable, animal or marine origin, such as fully or substantially hydrogenated palm oil, palm kernel oil, tallow, butter fats, fish oils, sunflower oil and their solvent or dry fractionated stearin or olein fractions. Preferred feedstock components comprise hydrogenated palm oil, hydrogenated palm kernel oil, fractionated palm oil stearin, sunflower oil, such as palm oil hardened to a melting point of 58°C (C₁₆ 40-50%; C₁₈ 60-50%), hardened palm oil stearin (C₁₆ 50-85%; C₁₈ 50-15%), hydrogenated palm kernel oil hardened to a melting point of 39°C (C₁₂ 45-55%; C₁₄ 12-18%; C₁₆ 5-10%; C₁₈ 12-25%), hydrogenated palm kernel oil stearin hardened to a melting point of 41°C (C₁₂ 50-60%; C₁₄ 15-25%; C₁₆ 5-10%; C₁₈ 5-12%), and sunflower oil hardened to a melting point of 69°C (C₁₆ 5-10%; C₁₈ 95-90%). Using these feedstock components any practical distribution of fatty acids in triglycerides of mid-fraction may be formulated.

Using these hardened feedstock components, for mid-fraction obtained by solvent fractionation, preferably the mid-fraction obtained has a H₃ (≥C₅₀) of less than 13, preferably less than 11, and a H₂ M

value (C_{44} , C_{46} , C_{48}) which is as large as possible, generally higher than 60, preferably higher than 64, and a $H_3 + H_2 M$ value which is as large as possible, generally larger than 70.

For mid-fraction obtained by dry fractionation the H_3 value is generally less than 16, preferably less than 15. The $H_2 M$ value is generally larger than 45, preferably larger than 50. The $H_3 + H_2 M$ value is generally larger than 65.

The interesterification used in the method according to the invention comprises any suitable interesterification process, by which the fatty acids of the triglycerides are randomly exchanged. Any prior art interesterification process might be used, such as the process disclosed in EP-A-76,682. Preferred is a catalyst comprising an alkaline metal or hydroxide. Preferably the catalyst system comprises sodium or sodium hydroxide, glyceride and water in a weight ratio of 1/2/3 to 1/2/7. The catalyst is generally added in a concentration of at least 0.03% by weight sodium hydroxide based on the oil. The information of the European patent application EP-A-76,682 is included by reference.

For an optimal distribution of the fatty acids over the triglycerides, used in the feedstock, it is preferred that the feedstock as well as the stearin topping fraction and olein topping fraction are subjected to the interesterification.

Depending on the separation efficiency, the solid particle content, the crystallization temperature and the cooling rate used in the fractional crystallization of the topping fractional crystallization treatment and bottoming fractional crystallization treatment, these treatments may comprise more than one, such as two consecutive fractional crystallizations.

When the topping fraction of crystallization treatment comprises two or more consecutive fractional crystallizations, a higher midfraction yield may be obtained when, according to a preferred embodiment the stearin topping fraction originates from the second consecutive topping fractional crystallization, and the olein topping fraction originates from the first consecutive topping fractional crystallization.

When the bottoming fractional crystallization treatment comprises two consecutive fractional crystallizations, it may be preferred for obtaining better midfraction quality when the olein bottoming fraction originates from the first consecutive bottoming fractional crystallization, and the mid-fraction originates from the second consecutive bottoming fractional crystallization.

If the olein topping or bottoming fraction comprises a relatively high solid content, it is preferred that this fraction is partly recycled to the topping or bottoming fractional crystallization treatment whereby the fraction is diluted. Preferably the recycling ratio of the recycled olein fraction is about 10-60%, more preferably 25-50%.

The consecutive topping and bottoming fractional crystallizations may be carried out as countercurrent fractional crystallizations, such as disclosed in the earlier European patent application to be included.

Impurities present in the feedstock or formed during the production of mid-fraction, such as partly saturated triglycerides or diglycerides, may accumulate in the recycled stearin topping or olein bottoming fractions. Therefore, it is preferred that intermittently or continuously a minor part of the stearin topping fraction and/or olein bottoming fraction is not recycled but disposed. The disposed fraction may comprise up to about 5% by weight of the recycled olein topping or bottoming fraction. Alternatively these impurities may be removed intermittently or continuously from the recycled fractions, for instance by adsorption.

The method according to the invention for the production of mid-fraction will be illustrated hereafter by way of non limiting examples. The two processes used are shown in the annexed single drawing in which each box refers to a fractional crystallization and the subsequent separation of the stearin fraction from the olein fraction.

In a batchwise process it is possible to use only one crystallizer and various storage tanks in which the olein and stearin fractions obtained in the topping fractional crystallization treatment and bottoming fractional crystallization treatment are temporarily stored.

EXAMPLE I

Mid-fraction was produced according to method A. Feedstock (F) comprising 47% fully hydrogenated palm oil and 53% fully hydrogenated palm kernel oil was subjected to interesterification (I) using 0.02% by weight metallic sodium.

In the two consecutive countercurrent fractional crystallizations 1 and 2 (crystallization temperature 44°C and 40°C , respectively) of the topping fractional crystallization treatment the solid particle content (SPC) was 11%. In the two consecutive fractional crystallizations 3 and 4 (crystallization temperature 35°C and 31°C , respectively) of the bottoming fractional crystallization treatment the SPC was 17%. The stearin topping fraction and the olein bottoming fraction were recycled to the interesterification and mixed with the feedstock in a ratio of 60/40. Initially the mid-fraction had a H_3/H_2M value of 12/55 and after steady state of

12/54.

For comparison purposes in each fractional crystallization the separation efficiency was kept at 0.5.

Table 1 shows the fatty acid composition (%) of the mid-fraction and of the feedstock.

5 **Table 1**

10 **Fatty acid composition (%) of the midfraction and the feed
after 10 cycles of interesterification and fractionation.**

Chain length in carbon numbers	Feed	Mid
8	2.2	2.1
10	2.0	2.2
12	26.2	26.0
14	8.9	9.0
16	25.5	25.7
18	34.5	34.7
20	0.3	0.3

30 **EXAMPLE II**

Feedstock comprising 7% fully hydrogenated, dry fractionated palm oil stearin, 33% fully hydrogenated palm oil and 60% fully hydrogenated palm kernel oil was used for the production of mid-fraction using method B.

35 In the fractional crystallization of the topping fractional crystallization treatment SPC was 8%, and in the fractional crystallization of the bottoming fractional crystallization treatment SPC was 18%. The topping stearin fraction 1 and the bottoming olein fraction 2 were recycled to the esterification and mixed with the feedstock in a ratio of 71/29.

At a separation efficiency of 0.5 the mid-fraction had a H₃/H₂M value of 15/45.

The fatty acid composition (%) of the mid-fraction obtained and of the feedstock is shown in Table 2.

Table 2

5 Fatty acid composition (%) of the mid-fraction and the feed
after 10 cycles of interesterification and fractionation.

Chain length in carbon numbers	Feed	Mid
8	2.9	2.7
10	2.3	2.2
12	29.3	29.4
14	9.6	9.1
16	24.6	25.0
18	31.3	31.6

EXAMPLE III

25 The same process as used in Example II was used, but the olein fractions were recycled at a recycling ratio of 50% on the feed. In the topping fractional crystallization treatment SPC was 5.4% and in the bottoming fractional crystallization treatment SPC was 12%. The mid-fraction obtained had a H₃/H₂M value of 15/45.

EXAMPLE IV

35 Feedstock, comprising 1,5% fully hydrogenated sunflower oil, 46,5% fully hydrogenated palm oil, 26% fully hydrogenated palm kernel oil and 26% fully hydrogenated palm kernel stearin was processed according to method A. In the topping fractional crystallization treatment the SPC in the crystallizations 1 and 2 was 8%. In the bottoming fractional crystallization treatment the SPC of the fractional crystallizations 3 and 4 was 16%. The stearin topping fraction and olein bottoming fraction were recycled and mixed with the feedstock in ratio of 62/38. At a separation efficiency of 0.5, the H₃/H₂M value of the mid-fraction was 12/49.

40 Table 3 shows the fatty acid composition (%) of the mid-fraction and of the feedstock.

Table 3

5 Fatty acid composition (%) of the mid-fraction and the feed after 15 cycles of interesterification and fractionation.

Chain length in carbon numbers	Feed	Mid
8	1.8	1.8
10	1.7	1.8
12	27.5	27.4
14	10.3	10.4
16	25.6	25.3
18	33.1	33.3

EXAMPLE V

25 A feedstock comprising 42% hydrogenated palm kernel stearin, 53% hydrogenated palm oil and 5% hydrogenated sunflower oil, was subjected to solvent fractionation in acetone in a ratio of 1/5 wt/wt. In the topping fractional crystallization treatment SPC was 29% and the crystallization temperature about 30°C. In the bottoming fractional crystallization treatment SPC was 33% (on present fat) and the crystallization temperature was about 20°C. The stearin topping fraction 1 and the olein bottoming fraction 2 were recycled and after removal of the acetone, mixed with the feedstock in a ratio of 47.5/52.5, and thereafter interesterified. The fatty acid composition of the mid-fraction resembles closely the composition of the feedstock, see Table 4.

35 Table 4

The fatty acid composition (%) of the mid-fraction and the feedstock after 10 cycles of interesterification and fractionation.

Chain length in carbon numbers	Feedstock	Mid
8	1.1	1.3
10	1.2	1.3
12	23.8	23.8
14	10.0	9.4
16	28.0	28.5
18	35.5	35.7

The H3/H2M value of the mid-fraction was 14/59. From time to time it was necessary to remove diacylglycerols by adsorption on silica. The time period between the removal procedures of these impurities

was depending on the composition of the feedstock.

EXAMPLE VI

- 5 A feedstock comprising 85% palm oil and 15% solvent fractionated palm oil stearin was used in method B. The mid-fraction obtained had a constant composition of 1.2% S₈, 67% S₂ O, 6.3% S₂ Li and 25.5% others.

EXAMPLE VII

- 10 Midfraction was produced according to method C. Feedstock comprising 43% fully hydrogenated palm kernel oil was subjected to interesterification (I) using 0.02% metallic sodium. In the two consecutive countercurrent fractional crystallizations 1 and 2 (crystallization temperatures of 44 and 48 °C, respectively) of the topping fractional crystallization treatment the solid particle content (SPC) was 18.5%. In the
15 bottoming fractional crystallization 3 (crystallization temperature of 38 °C) of the bottoming fractional crystallization treatment the SPC was 30%. The stearin topping fraction and the olein bottoming fraction were recycled to the interesterification and mixed with the feedstock in a ratio of 50/50. In the steady state the H₃/H₂M ratio was 12/52. For comparison purposes the separation efficiency was kept at 0.5. Table 5 shows the fatty acid composition (%) of the midfraction and of the feedstock. Usually, method C may be used in
20 systems in which the SE is still high at a high SPC.

Table 5

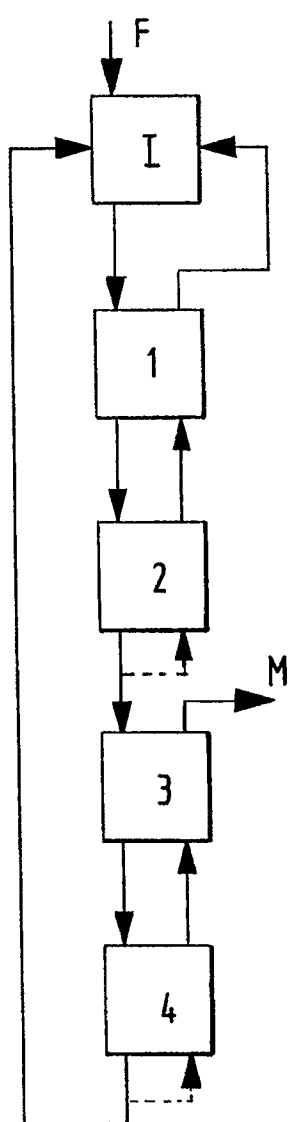
- 25 Fatty acid composition (%) of the feed and of the midfraction after 10 cycles of interesterification and fractionation (method C).

30 Chain length in carbon numbers	Feedstock	Mid
8	2.2	2.1
35 10	2.1	2.0
12	27.1	27.4
14	8.9	9.2
40 16	25.5	26.1
18	33.5	33.3
20	0.3	0.3

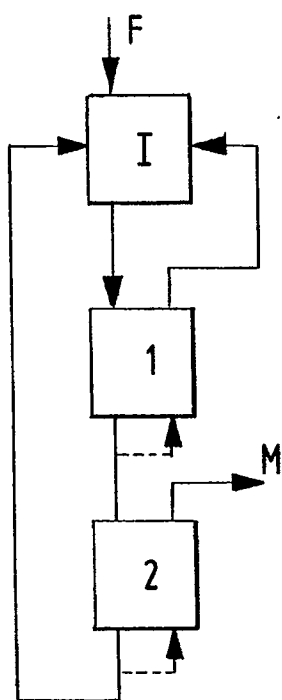
45 Claims

1. Method for the production of mid-fraction by fractional crystallization of fatty substances, in which:
 - 50 i) a fatty substances comprising feedstock is subjected to a topping fractional crystallization treatment providing a stearin topping fraction and an olein topping fraction;
 - ii) the olein topping fraction is subjected to a bottoming fractional crystallization treatment providing the mid-fraction and an olein bottoming fraction;
 - 55 iii) the stearin topping fraction and the olein bottoming fraction are combined and subjected to interesterification, and the interesterified mixture of fatty substances is recycled to the topping fractional crystallization treatment.
2. Method as claimed in claim 1, wherein the feedstock and the mixture of stearin topping fraction and olein bottoming fraction are subjected to interesterification.

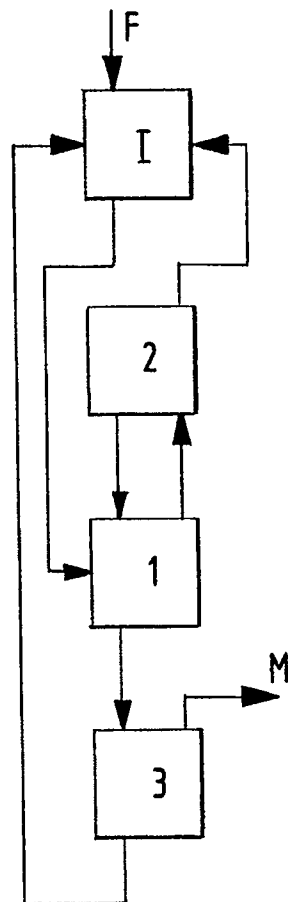
3. Method as claimed in claims 1 or 2, wherein the topping fractional crystallization treatment comprises at least two consecutive topping fractional crystallizations.
4. Method as claimed in claim 3, wherein the stearin topping fraction originates from the second consecutive topping fractional crystallization, and the olein topping fraction originates from the first consecutive topping fractional crystallization.
5. Method as claimed in claims 1-4, wherein the bottoming fractional crystallization treatment comprises at least two consecutive bottoming fractional crystallizations.
6. Method as claimed in claims 5, wherein the olein bottoming fraction originates from the first consecutive bottoming fractional crystallization, and the mid-fraction originates from the second consecutive bottoming fractional crystallization.
7. Method as claimed in claim 1-6, wherein the olein topping fraction is partly recycled to the topping fractional crystallization treatment.
8. Method as claimed in claim 7, wherein the recycling ratio of the olein topping fraction is about 10-60%, preferably about 25-50%.
9. Method as claimed in claims 1-8, wherein the olein bottoming fraction partly recycled to the bottoming fractional crystallization treatment.
10. Method as claimed in claim 9, wherein the recycling ratio of the olein bottoming fraction is about 10-60%, preferably about 25-50%.
11. Method as claimed in claims 3-10, wherein the consecutive topping and/or bottoming fractional crystallizations comprise countercurrent fractional crystallizations.
12. Method as claimed in claims 1-11, wherein the fractional crystallizations are selected from solvent crystallization and dry crystallization.
13. Method as claimed in claims 1-12, wherein impurities are removed from the stearin topping fraction and/or olein bottoming fraction.
14. Method as claimed in claims 1-13, wherein a minor part of the stearin topping fraction and/or olein bottoming fraction, generally up to 5%, is disposed.



A



B



C



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which under Rule 45 of the European Patent Convention
shall be considered, for the purposes of subsequent
proceedings, as the European search report

Application number
EP 91 20 1120

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	GB-A- 590 916 (PROCTOR & GAMBLE CO.) * Page 1, lines 39-41, page 1, line 101 - page 2, line 34; page 3, figure B; page 8, lines 20-47; claims 1,2,9 *	1,2, 12	C 11 B 7/00 C 11 C 3/10
X	EP-A-0 185 524 (UNILEVER PLC) * Page 2, lines 24-30; page 3, lines 23-35; page 4, lines 1-16; page 5, lines 6-14,28-30; example 1; claims 1-3 *	1,2, 12,13	
Y	--	3-6	
-	./.		
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 11 B C 11 C A 23 D
INCOMPLETE SEARCH			
The Search Division considers that the present European patent application does not comply with the provisions of the European Patent Convention to such an extent that it is not possible to carry out a meaningful search into the state of the art on the basis of some of the claims. Claims searched completely: Claims searched incompletely: 4,6 Claims not searched: Reason for the limitation of the search: Technical impossibility and inconsistency with respect to figure 1/1 A. If in both claims 4 and 6 the words "first" and "second" are interchanged, these claims become consistent.			
Place of search THE HAGUE		Date of completion of the search 29-07-1991	Examiner KANBIER
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			



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Application number

EP 91 20 1120 -2-

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
Y	JOURNAL OF THE AMERICAN OIL CHEMISTS SOCIETY, vol. 62, no. 2, February 1985, pages 417-421; H. TRAITLER et al.: "Palm oil and palm kernel oil in food products" * Abstract; figure 3; page 418 under "Utilization of PO fractions": "High melting stearin" page 418 and "Palm mid-fraction" page 419 *	3-6	
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A	EP-A-0 109 721 (UNILEVER PLC) * Page 3, line 27 - page 4, line 13; page 5, lines 29-32; page 6, lines 13-31; claims 7,12 *	2, 7-10, 12	TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
