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## (54) Title: PROCESS FOR DRY FRACTIONATION OF A PALM OIL OLEIN

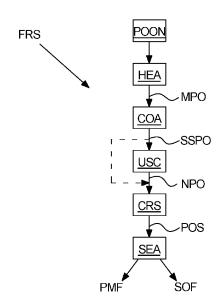


Fig. 1

(57) Abstract: A process for dry fractionation of a palm oil olein (POON) is disclosed, the process comprising the steps of providing the palm oil olein (POON) as a melted palm oil olein (MPO). cooling the melted palm oil olein (MPO) to obtain a supersaturated palm oil olein (SSPO), subjecting at least a part of the supersaturated palm oil olein (SSPO) to ultrasonic treatment, crystallization, in a crystallizer (CRS), of the supersaturated palm oil olein (SSPO) having been subjected to ultrasound to obtain a palm oil olein slurry (POS), separating the palm oil olein slurry (POS) to obtain a palm oil mid fraction (PMF) and a super ole in fraction (SOF). Also, a fractionation system (FRS) and a dry-fractionated non-detopped palm oil mid fraction (PMF) are disclosed.



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#### PROCESS FOR DRY FRACTIONATION OF A PALM OIL OLEIN

## FIELD OF INVENTION

The invention relates to the field of dry fractionation of palm oil olein, particular to a process of dry fractionation of palm oil olein assisted by ultrasound treatment, a fractionation system therefore, and a palm oil mid fraction.

## **BACKGROUND**

Practionation of palm oil olein to obtain a palm oil mid fraction is a process

performed at a large scale. Over time, a tradition of adding PPP-triglycerides to the palm oil olein before fractionation has developed in order to overcome the difficulty of separating the two main components (POO-triglycerides and the POP-triglycerides). Thereby, the process could obtain a palm oil mid fraction with an acceptable yield and quality, while using the economically more viable dry

fractionation, as long as the excess PPP-triglycerides were removed during a so-called de-topping step after the main fractionation. This is especially true for chemically neutralized palm oleins, which are known to crystallize significantly slower than their physically refined counterparts.

Thus, the method known in the art for fractionation of palm oil olein comprises

several fractionation steps and a blending step and therefore requires a large number of separate tanks to handle the different components of the fractionation process.

One example of this process is given in the article "Principles of palm olein fractionation: a bit of science behind the technology" by Gijs H. Calliauw, Véronique Gibon and Wim F.J. De Greyt, from Lipid Technology, July 2007, Vol. 19, No. 7, pages 152-155.

A further example involving fractionation is JP 2014/162859 A. Examples display crystallization times of 34-40 hours.

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A further example of fractionation of palm oil olein is described in Hashimoto, S., Nezy, T., Arakawa, H., Ito, T., Maruzeni, S. "Preparation of Sharp-Melting Hard Palm Midfraction and Its Use as Hard Butter in Chocolate", J. Am. Oil. Chem. Soc., Vol. 78, No. 5, 2001, page 455-460. The method described starts from POO IV56 and obtains PMF IV45 by using a detopping step after the main fractionation together with a further fractionation. A 45 hour long cooling profile is used in the process described.

Thus, the processes described in the art are all lengthy and capacity consuming processes.

However, fractionation facilities have a certain capacity, and the problem remain that the fractionation of palm oil olein still takes up a large part of the limited capacity.

Thus there is a need for a process of fractionating palm oil olein taking up a smaller part of the limited capacity in the fractionation facilities and at the same time producing a high quality end product.

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#### **SUMMARY**

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The present invention solves the identified problem with fractionation of palm oil by utilizing ultrasound after the initial main fractionation step. It has been realized by the inventor that by changing the known process in this way palm oil olein can elegantly be fractionated to obtain a palm-middle fraction with low content of PPP, omitting the traditional de-topping step to bring down the PPP-content. Additionally, by applying the ultrasound treatment a super olein with a low 3-MCPD content (< 0.5 ppm) is produced via this non-seeded-one-step fractionation route, which has not traditionally been possible.

Thus, the present invention minimizes the number of tanks needed at the fractionation facilities to run the process, it saves time and makes logistics easier and results in a high yield of the end product of high quality with a low 3-MCPD content.

The invention relates in a first aspect to a process for dry fractionation of a palm oil olein, the process comprising the steps of providing the palm oil olein as a melted palm oil olein, cooling the melted palm oil olein to obtain a supersaturated palm oil olein, subjecting at least a part of the supersaturated palm oil olein to ultrasonic treatment, crystallization, in a crystallizer, of the supersaturated palm oil olein having been subjected to ultrasound to obtain a palm oil olein slurry, separating the palm oil olein slurry to obtain a palm oil mid fraction and a super olein fraction.

According to an embodiment of the invention the step of cooling the palm oil olein is performed in said crystallizer, wherein at least part of the supersaturated palm oil olein is fed from the crystallizer to an ultrasonic emitting apparatus arranged external to said crystallizer, and wherein the palm oil olein having been subjected to ultrasonic treatment in said ultrasound emitting apparatus is fed back into said crystallizer.

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The invention relates in a further aspect to a fractionation system for fractionating a palm oil olein, the fractionation system comprising

an inlet for receiving the palm oil olein as a melted palm oil olein,

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a cooling arrangement for cooling the melted palm oil olein into a supersaturated palm oil olein,

an ultrasound emitting apparatus arranged to subject at least a part of the supersaturated palm oil olein to ultrasonic treatment,

a crystallizer for crystallizing the supersaturated palm oil olein having been subjected to ultrasonic treatment to obtain a palm oil olein slurry,

a separation arrangement for separating the palm oil olein slurry into a palm oil mid fraction and a super olein fraction.

The invention relates in an even further aspect to a dry-fractionated non-detopped palm oil mid fraction, wherein the palm oil mid fraction has a content of PPP-triglycerides below 2.0 percent by weight of the palm oil mid fraction.

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## **FIGURES**

The invention will now be described with reference to the figures where

5 Figure 1 illustrates a process for dry fractionation of a palm oil olein according to an embodiment of the invention,

Figures 2A-2C illustrate ways of obtaining a melted palm oil olein according to various embodiments of the invention,

Figures 3A-3C illustrate different combinations of cooling, ultrasonic treatment, and crystallizing and according to various embodiments of the invention,

Figure 4 illustrates a process of operating several crystallizers with only one ultrasound emitting apparatus according to an embodiment of the invention, and

Figure 5 illustrates a series of fractionations performed according to an embodiment of the invention.

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#### DETAILED DESCRIPTION

Definitions

As used herein, the term "fatty acid" encompasses free fatty acids and fatty acid residues in triglycerides.

As used herein, "%" or "percentage" all relates to weight percentage, i.e. wt.% or wt.-% if nothing else is indicated.

As used herein, the singular forms "a", "an" and "the" include plural referents unless the context clearly dictates otherwise.

As used herein, "at least one" is intended to mean one or more, i.e. 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, etc.

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As used herein, the term "triglycerides" may be used interchangeably with the term 'triacylglycerides' and should be understood as an ester derived from glycerol and three fatty acids. "Triglycerides" may be abbreviated TG or TAG. A single triglyceride molecule, having a specific molecular formula, is of either vegetable or non-vegetable origin.

As used herein, the term "palm oil olein" is intended to mean an olein fraction based on a fractionation product from a fractionation of palm oil into a palm oil olein fraction and also a palm stearin fraction. It is to be understood that the palm oil olein may comprise some amount of oils or fats coming from other sources than palm oil. Such other oils may have been added with a specific purpose or may be present due to e.g. process limitations. Such process limitations may include, as an example, a situation where a first fat other than palm oil is fractionated using a fractionation system, and the palm oil thereafter is fractionated on the same fractionation system to obtain the palm oil olein. Residues of the first fat may then often be present in the fractionation system; in some cases residues of up to 5 percent of the first fat have

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been reported. In one embodiment, the palm oil olein comprises no more than 10 percent by weight of oils from other sources than palm oil, such as no more than 5 percent by weight. In one embodiment the palm oil olein has an iodine value above 50, comprises between 20 and 40 percent of POP-triglycerides by weight of the palm oil olein, comprises between 20 and 40 percent of POO-triglycerides by weight of the palm oil olein, and comprises between 0.01 and 2.0 percent of PPP-triglycerides. While the process of the invention may comprise a subsequent fractionation obtaining a "subsequent palm olein", such subsequent palm olein is to be distinguished, by the use of "subsequent", from the "palm oil olein" referred to above. Throughout the application the terms "palm oil olein" and "palm olein" may be used interchangeably.

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As used herein, the term "cooling" is intended to mean a process of lowering the temperature. This may be done in various ways, but typically includes contacting the melted palm oil olein with cooled surface, e.g. such that one side of the surface is contacted with the palm oil olein, while the opposite surface is contacted with a coolant.

As used herein, the term "fraction" is intended to mean a product of a fractionation process. The fractionation product, i.e. the fraction, may or may not be further treated in various other ways. However, it is to be understood that if a certain fraction undergoes a further fractionation, two new, separate fractions are obtained. In other words, when a fraction is subjected to a further fractionation, the original fraction ceases to exist, whereas the two new, separate fractions have replaced it. In some cases, the term "fraction" may be omitted, e.g. the terms "stearin" and "stearin fraction" may be used interchangeably, also the terms "olein" and "olein fraction" may be used interchangeably.

As used herein, the term "supersaturation" is intended to have its ordinary meaning of a supersaturated solution, i.e. intended to mean a solution that contains more of the

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dissolved material (solute) than could be dissolved by the solvent under normal circumstances.

As used herein, the term "dry fractionation" is intended to mean a fractionation without the use of a solvent. Solvents, such as e.g. hexane, is used in solvent fractionation to disperse the crystals of the solid fraction in or to flush the liquid part of the oil out.

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As used herein, the term "melted" is intended to refer to a completely melted fraction, i.e. a fraction where all triglycerides are completely melted and hence which do not have any content of crystalline triglycerides. The melted palm oil olein may be obtained by melting a palm oil olein, or by obtaining the melted palm oil olein from a source of melted palm oil olein.

As used herein, the term "ultrasonic treatment" is intended to mean a process of treating the supersaturated palm oil olein with ultrasound, specifically by subjecting the supersaturated palm oil olein to ultrasound. The ultrasonic treatment induces crystalline nucleation in the supersaturated palm oil olein in a relatively effective and homogenous way. Throughout the application the terms "ultrasound" and "ultrasonic" may be used interchangeably.

As used herein, the term "ultrasound emitting apparatus" is intended to mean an apparatus capable of emitting ultrasound usable in the ultrasonic treatment of the invention. The ultrasound emitting apparatus may e.g. be an ultrasonic cell, an ultrasonic flowcell, a sonotrode, etc.

As used herein, the term "crystallization" is intended to mean maintaining the supersaturated palm oil olein having been subjected to ultrasonic treatment and also the supersaturated palm oil olein, if any, having bypassed the ultrasonic treatment in order to allow crystallization. According to the invention, the step of crystallization is carried out in the crystallizer, i.e. all of the supersaturated palm oil olein is

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crystallized in the crystallizer. Throughout the application the terms "crystallizing" and "crystallization of" may be used interchangeably.

As used herein, the term "palm oil olein slurry" is intended to mean a slurry based on palm oil olein, i.e. a portion of palm oil olein comprising both crystalline palm oil olein and also liquid palm oil olein thus together having a slurry like state. In other words, the term "slurry" is a partly melted composition where at least some crystals are present. Thus, a "slurry" may also be understood as a partly melted suspension, partly molten suspension or a paste.

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As used herein, the term "palm oil mid fraction" is intended to mean the highest melting fraction of the dry fractionation of a palm oil olein. It is noted that when referring to a "palm oil mid fraction" herein it is the palm oil mid fraction obtained by dry fractionation of a palm oil olein, unless otherwise specifically mentioned. Thus, the "palm oil mid fraction" should be distinguished from a "subsequent palm oil mid fraction", which is the hard melting fraction of a subsequent fractionation of the palm oil mid fraction.

As used herein, the term "super olein fraction" is intended to mean the lowest melting fraction of the dry fractionation of a palm oil olein.

As used herein, the term "crystallizer" is intended to mean a vessel for performing crystallization. The crystallizer may be a dynamic crystallizer, i.e. a crystallizer with agitation, or static crystallizer without any agitation. Crystallizers are often provided with a cooling arrangement, e.g. in the form of a water jacket, but this is not necessary in all embodiments.

As used herein, the term "de-topping" is intended to mean any process of removing PPP-triglycerides from the palm oil mid fraction. It may typically be a de-topping fractionation, which is a process of fractionating at an elevated temperature of the palm oil mid fraction, such as around 25 to 30 degrees Celsius in order to remove

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PPP-triglycerides. The elevated temperature at which the fractionation de-topping is performed is due to the relatively high melting point of PPP-triglycerides.

As used herein, the term "iodine value" is intended to mean an iodine value obtained according to IUPAC 2.205/4-7th edition, unless otherwise specified. The iodine value may also be referred to as "Wijs iodine value", "Wijs IV", or simply "IV".

As used herein, the term "3-MCPD" is intended to mean 3-MCPD (3-chloropropane-1,2-diol), 2-MCPD (2-chloropropane-1,3-diol) and esters thereof.

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fraction.

**Abbreviations** 

P = palmitic acid/palmitate

O = oleic acid/oleate

St = stearic acid/stearate

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POP-triglyceride = 1,3-dipalmitate-2-oleateglycerol

POO-triglyceride = 1-palmitate-2,3-dioleateglycerol

OOO-triglyceride = 1,2,3-trioleateglycerol

XYZ-triglyceride = 1-X-2-Y-3-Zglycerol of the fatty acids X, Y, and Z

IV = iodine value

The invention relates to a process for dry fractionation of a palm oil olein, the process comprising the steps of

providing the palm oil olein as a melted palm oil olein,

- cooling the melted palm oil olein to obtain a supersaturated palm oil olein, subjecting at least a part of the supersaturated palm oil olein to ultrasonic treatment, crystallization, in a crystallizer, of the supersaturated palm oil olein having been subjected to ultrasound to obtain a palm oil olein slurry,
  - separating the palm oil olein slurry to obtain a palm oil mid fraction and a super olein

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One significant advantage of the invention may be that a superior palm oil mid fraction may be obtained in a relatively fast process without any need for performing a subsequent de-topping to remove PPP-triglycerides. Resulting advantages thereof include significant capacity savings due to the need for de-topping being eliminated, with resulting costs savings.

Thus, advantages of the invention include that superior yield, increased capacity in the fractionation facilities and improved quality of the end product may be achieved without the addition of any PPP-triglyceride source to the palm oil olein, and that, consequently, a following de-topping step to remove the added PPP-triglycerides may be avoided. In other words, by using the ultrasonic treatment according to the invention, the need for adding PPP-triglycerides to the palm oil olein in order to achieve an acceptable fractionation is overcome. This may lead to significant capacity, yield, and cost savings, for example by eliminating the need for performing a de-topping. Moreover, these advantages may be realized without adding any triglycerides portion to the palm oil olein before the fractionation. E.g., the production for a given production capacity is optimized.

A further advantage of the invention is that a palm oil mid faction may be obtained having superior steep melting characteristics. The superior steep melting characteristics require a relatively low content of PPP-triglycerides, as such highmelting triglycerides distort and compromise the steep melting characteristics. By using ultrasound treatment, addition of PPP-triglycerides may be avoided, and thus the superior steep melting characteristics may be obtained even without any detopping of the PPP-triglycerides from the palm oil mid fraction. This is especially an advantage when using the palm oil mid fraction in tempered confectionary products, as a high content of PPP-triglycerides, e.g. above 3 percent by weight of the palm oil mid fraction, may make it difficult to temper the confectionary product.

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It should be understood that the high melting fraction of this process (i.e. the obtained palm oil mid fraction) may be subjected to further fractionation step(s) according to traditional procedures and/or according to a process of the invention.

It should be understood that in the step of subjecting at least a part of the supersaturated palm oil olein to ultrasonic treatment is intended to mean that at least some of the supersaturated palm oil olein is subjected to ultrasonic treatment. In some embodiments, all of the supersaturated palm oil olein is subjected to ultrasonic treatment, whereas in other embodiments only a part of the supersaturated palm oil olein is subjected to ultrasonic treatment.

According to an embodiment of the invention the process comprises a further step of melting the palm oil olein in order to obtain the melted palm oil olein.

The ultrasonic treatment and the cooling may be applied both external to the crystallizer and inside the crystallizer, according to different embodiments.

According to an advantageous embodiment of the invention the step of ultrasonic treatment is performed external to said crystallizer.

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For example, the ultrasonic treatment could be performed before reaching the crystallizer, i.e. by cooling the melted palm oil olein prior to the crystallization. Such pre-cooling of the melted palm oil olein could for example be obtained by using a tubular cooler arranged external to the crystallizer.

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In another example, the melted palm oil olein is fed to the crystallizer where it is cooled, and then afterwards pumped to an ultrasound emitting apparatus arranged external to the crystallizer, and then back into the crystallizer for the step of crystallization. I.e. in such example, the palm oil olein is pumped in a loop from the crystallizer to the ultrasound emitting apparatus and then back to the crystallizer.

According to a further advantageous embodiment of the invention, the step of cooling the palm oil olein is performed in said crystallizer. It should be understood that since the palm olein is provided as a melted palm oil olein, it is also the palm oil olein in the form of the melted palm oil olein that is being subjected to the cooling step.

Thus, the steps of cooling and crystallizing are each performed in the crystallizer. The ultrasonic treatment may, for example, also be performed inside the crystallizer by positioning an ultrasound emitting apparatus inside the crystallizer.

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In another example, the ultrasound emitting apparatus is positioned externally to the crystallizer, and the supersaturated palm oil olein is pumped from the crystallizer, after the step of cooling, to the ultrasound emitting apparatus, and then back into the crystallizer for the step of crystallization. It should be understood that since the palm olein is provided as a melted palm oil olein, it is also the palm oil olein in the form of the melted palm oil olein that is being subjected to the cooling step.

According to an even further advantageous embodiment of the invention, the step of cooling the palm oil olein is performed externally to said crystallizer.

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According to a still further advantageous embodiment of the invention, the step of cooling the palm oil olein is performed in said crystallizer, wherein at least part of the supersaturated palm oil olein is fed from the crystallizer to an ultrasound emitting apparatus arranged external to said crystallizer, and wherein the palm oil olein having been subjected to ultrasonic treatment in said ultrasound emitting apparatus is fed back into said crystallizer. It should be understood that since the palm olein is provided as a melted palm oil olein, it is also the palm oil olein in the form of the melted palm oil olein that is being subjected to the cooling step.

One very important advantage of this embodiment is that the step of cooling may be applied as a batch process in the crystallizer, i.e. the cooling may be distributed over

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a period of time long enough to avoid needs for very intense cooling, which puts significant demands on the cooling system, cooling fluids, and which is often a much less energy-efficient process.

- According to an embodiment of the invention, the step of cooling is performed external to said crystallizer, for example in tubular cooler, whereas said step of ultrasonic treatment is performed external to the crystallizer, e.g. in an ultrasound emitting apparatus arranged between the tubular cooler and the crystallizer.
- According to a further embodiment of the invention, the step of cooling and the step of ultrasonic treatment are each performed in said crystallizer. For example, the ultrasonic treatment may take place in an ultrasound emitting apparatus at least partly submerged in the palm oil olein inside the crystallizer.
- According to a still further embodiment of the invention, the step of cooling takes place in said crystallizer, whereas the step of ultrasonic treatment is performed externally from the crystallizer. For example, the ultrasonic treatment may take place in an ultrasound emitting apparatus arranged in a loop from the crystallizer, through the ultrasound emitting apparatus, and back to the crystallizer.

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According to an embodiment of the invention, the ultrasonic treatment is applied for a period of between 1 and 120 minutes. The ultrasonic treatment may comprise one or more sequences of ultrasound being applied for a period of between 1 and 120 minutes.

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According to an embodiment of the invention, the ultrasonic treatment is applied with a power of between 100 and 20,000 (twenty thousand) Watts per liter of palm oil olein.

According to an embodiment of the invention, the ultrasonic treatment is applied with an absolute pressure of 0.5-6 bar.

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According to an embodiment of the invention, the ultrasonic treatment is applied with a frequency 12-100 kHz.

5 According to an embodiment of the invention, the ultrasonic treatment is applied to

10-100 percent by weight of the palm oil olein.

According to an embodiment of the invention, the ultrasonic treatment is applied for

a period of between 1 and 120 minutes, with a power of between 100 and 20,000

(twenty thousand) Watts per liter of palm oil olein, with an absolute pressure of 0.5-6

bar, with a frequency 12-100 kHz, and to 10-100 percent by weight of the palm oil

olein.

According to an embodiment of the invention, the ultrasonic treatment is applied by

means of a flow cell or vibrating pipe.

According to an embodiment of the invention, the ultrasonic treatment is applied by

means of a flow cell or vibrating pipe to an amount of supersaturated palm oil olein

of 5-50 tons per hour.

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The palm oil olein is cooled to obtain the supersaturated palm oil olein.

According to an advantageous embodiment of the invention, the supersaturated palm

oil olein has a temperature of between 10 and 37 degrees Celsius.

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According to an embodiment of the invention, the supersaturated palm oil olein has a

temperature of between 12 and 30 degrees Celsius, such as 15 and 22 degrees

Celsius.

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According to an embodiment of the invention, the palm oil olein is provided as a batch of palm oil olein and wherein the batch of palm oil olein is crystallized in the crystallizer during the step of cooling the palm oil olein.

5 Addition of PPP-triglycerides may be avoided due to the application of ultrasonic treatment.

According to an advantageous embodiment of the invention, the palm oil olein has a content of PPP-triglycerides below 1.0 percent by weight of the palm oil olein.

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However, in further embodiments of the invention the concentration of PPPtriglycerides in the palm oil olein may be even lower. For example, the palm oil olein may have a content of PPP-triglycerides below 0.9 percent by weight of the palm oil olein, or even below 0.8 percent by weight of the palm oil olein. In a further example, the palm oil olein may have a content of PPP-triglycerides below 0.7 percent by weight of the palm oil olein, below 0.6 percent by weight of the palm oil olein or even below 0.5 percent by weight of the palm oil olein, such as even below 0.2 percent by weight of the palm oil olein or below 0.1 percent by weight of the palm oil olein. Still even lower contents of PPP-triglycerides may be used, e.g. between 0.1 and 0.4 percent by weight of the palm oil olein.

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According to a still further embodiment of the invention, the palm oil olein has a content of PPP-triglycerides of between 0.01 and 1.0 percent by weight of the palm oil olein, such as between 0.01 and 0.7 percent by weight of the palm oil olein.

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A relatively high yield may be obtained, i.e. a high weight ratio between the obtained palm oil mid fraction and the palm oil olein used. At the same time, a palm oil mid fraction having superior quality may be obtained directly after fractionation compared to directly after fractionation when PPP-triglycerides are added as is done in the traditional fractionation process known in the art.

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According to an advantageous embodiment of the invention the yield of the palm oil mid fraction relative to the palm oil olein is at least 20 percent by weight.

Thus, for each kilogram of palm oil olein used, at least 200 grams of palm oil mid fraction is obtained. In a further embodiment the yield may be at least 30 percent by weight. Thus, the yield of palm oil mid fraction may be between 20 and 60 percent by weight, such as between 30 and 50 percent by weight.

According to a further embodiment of the invention, the yield of the palm oil mid fraction relative to the palm oil olein is at least 25 percent by weight, such as between 28 and 40 percent by weight.

According to an even further embodiment of the invention, the yield of the palm oil mid fraction relative to the palm oil olein may be increase by between 20 and 50 percent by weight relative to a traditional fractionation process including de-topping.

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According to a still further embodiment of the invention, the yield of the subsequent palm oil mid fraction relative to the palm oil mid fraction may be increase by between 20 and 50 percent by weight relative to a traditional fractionation process including de-topping.

According to an embodiment of the invention, the palm oil mid fraction has a content of PPP-triglycerides below 10 percent by weight of the palm oil mid fraction.

According to a further embodiment of the invention, the capacity increase is increased by at least 20 percent compared to a process of using palm oil olein with a concentration of PPP-triglycerides above 0.8 percent by weight of the palm oil olein and using a subsequent detopping to remove PPP-triglycerides.

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According to a further advantageous embodiment of the invention, the palm oil mid fraction has a content of PPP-triglycerides below 2.0 percent by weight of the palm oil mid fraction.

- For example, the content of PPP-triglycerides may be below 1.5 percent by weight of the palm oil mid fraction, or below 1.0 percent by weight of the palm oil mid fraction, such as below 0.3 percent by weight of the palm oil mid fraction, or even below 0.1 percent by weight of the palm oil mid fraction. The content of PPP-triglycerides in the palm oil mid fraction may thus be between 0.01 and 2.0 percent by weight of the palm oil mid fraction. Alternatively, the content of PPP-triglycerides in the palm oil mid fraction may thus be between 0.01 and 1.5 percent by weight of the palm mid fraction.
- An important measure of the quality of the obtained palm oil mid fraction is the iodine value (IV).

According to an even further advantageous embodiment of the invention, the palm oil mid fraction has an iodine value below 50.

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In a further embodiment, the palm oil mid fraction has an iodine value below 47, such as below 45.

As an example, the palm oil mid fraction may have an iodine value between 30 and 50, such as between 40 and 50.

According to a still further advantageous embodiment of the invention, the difference between the iodine value of the palm oil mid fraction and the iodine value of the super olein fraction is at least 15, such as at least 16.

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For example, the difference between the iodine value of the palm oil mid fraction and the iodine value of the super olein may be between 15 and 25, such as between 15 and 20, such as between 16 and 20.

An important parameter may be the iodine value (IV) of the obtained super olein fraction.

According to an even further advantageous embodiment of the invention, the super olein fraction has an iodine value above 60, such as between 60 and 100.

According to an even further advantageous embodiment of the invention, the palm oil mid fraction comprises POP-triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction.

For example, the palm oil mid fraction may comprise POP-triglycerides in an amount of between 40 and 60 percent by weight of the palm oil mid fraction.

According to a still even advantageous embodiment of the invention, the palm oil mid fraction has a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction.

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According to an embodiment of the invention, the palm oil mid fraction has a content of PPP-triglycerides below 2 percent by weight of the palm oil mid fraction, an iodine value below 50, comprises POP triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction, and a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction.

According to an embodiment of the invention, the palm oil mid fraction has an iodine value below 50, comprises POP triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction, and a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction.

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According to a still further advantageous embodiment of the invention, the process of obtaining palm oil mid fraction from the palm oil olein is performed in a time period of less than 18 hours.

5 The obtained palm oil mid fraction may be used in a further fractionation.

According to an advantageous embodiment of the invention, the palm oil mid fraction is subjected to a subsequent dry fractionation obtaining a subsequent palm oil mid fraction and a subsequent palm olein.

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It should be understood that the palm oil mid fraction may in some cases be treated before being subjected to the subsequent dry fractionation. However, no de-topping of any kind needs to be applied when a palm oil olein with a low concentration of PPP-triglycerides is used, such as a concentration of PPP-triglycerides below 1.0 percent by weight of the palm oil olein.

In alternative embodiments, de-topping may be applied, when a combination of ultrasound treatment and a higher concentration of PPP-triglycerides in the palm oil

olein is used, such as above 1.0 percent by weight of the palm oil olein.

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According to a further advantageous embodiment of the invention, the subsequent fractionation is performed at a temperature of below 25 degrees Celsius. For example, the subsequent fractionation may be performed at a temperature between 18 and 25 degrees Celsius.

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According to an even further advantageous embodiment of the invention, the subsequent palm oil mid fraction has an iodine value below 40.

For example, the subsequent palm oil mid fraction may have an iodine value between 20 and 40, such as between 30 and 40.

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According to a still further advantageous embodiment of the invention, no detopping is used before the subsequent fractionation.

According to a still even further advantageous embodiment of the invention, the subsequent palm oil mid fraction has a content of PPP-triglycerides below 4.0 percent by weight subsequent palm oil mid fraction. For example, the subsequent palm oil mid fraction has a content of PPP-triglycerides below 2.0 percent by weight subsequent palm oil mid fraction.

According to an even further advantageous embodiment of the invention, the palm oil mid fraction is not subjected to any de-topping.

According to an embodiment of the invention, the step of separating the palm oil olein slurry is performed as a batch process.

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According to a further embodiment, the palm oil olein is neutralized. Thus, the palm oil olein is provided as a neutralized palm oil olein. E.g. the palm oil olein may be neutralized, bleached and deodorized, also known as a NBD palm oil olein.

According to a still further embodiment, the palm oil olein is refined. Thus, the palm oil olein is provided as a refined palm oil olein. E.g. the palm oil olein may be refined, bleached and deodorized, also known as a RBD palm oil olein.

According to an even further embodiment, the stearin fraction and the super olein fraction has a content of 3-MCPD of less than 0.5 ppm. 3-MCPD compounds (3-chloropropane-1,2-diol) are generally considered unattractive, and low content are considered advantageous. For example, the content of 3-MCPD in the obtained super olein fraction may be between 0.01 and 0.5 ppm, such as between 0.05 and 0.5 ppm. This may be especially attractive when using neutralized palm oil olein, such as NBD palm oil olein. The person skilled in the art will know that it is particularly

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difficult to maintain a low 3-MCPD level in the super olein fraction due to migration. The person skilled in the art will know how to measure 3-MCPD.

Various types of crystallizers may be usable with the present invention.

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According to an advantageous embodiment of the invention, the step of crystallization of the palm oil olein in the crystallizer comprises cooling the palm oil olein in said crystallizer. It should be understood that since the palm olein is provided as a melted palm oil olein, it is also the palm oil olein in the form of the melted palm oil olein that is being subjected to the step of crystallization.

In order to obtain the palm oil mid fraction, the palm oil olein slurry is separated.

According to an advantageous embodiment of the invention, the step of separating the palm oil olein slurry comprises filtering and pressing the palm oil olein slurry.

Ultrasonic treatment may also be used in combination with adding PPP-triglycerides.

According to an advantageous embodiment of the invention, the palm oil olein has a content of PPP-triglycerides above 1.0 percent by weight of the palm oil olein.

According to an advantageous embodiment of the invention, an amount of supersaturated palm oil olein corresponding to at least 10 percent by weight of the total amount of palm oil olein is subjected to ultrasound treatment.

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In a further embodiment, an amount of supersaturated palm oil olein corresponding to at least 20 percent by weight of the total amount of palm oil olein is subjected to ultrasound treatment, such as at least 30 percent by weight of the total amount of palm oil olein.

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In a further embodiment, the amount of supersaturated palm oil olein subjected to ultrasound treatment corresponds to between 10 and 100 percent by weight of the total amount of palm oil olein, such as between 30 and 100 percent by weight of the total amount of palm oil olein, such as between 50 and 100 percent by weight of the total amount of palm oil olein.

The invention relates in a further aspect to a fractionation system for fractionating a palm oil olein, the fractionation system comprising

an inlet for receiving the palm oil olein as a melted palm oil olein,

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a cooling arrangement for cooling the melted palm oil olein into a supersaturated palm oil olein.

an ultrasound emitting apparatus arranged to subject at least a part of the supersaturated palm oil olein to ultrasonic treatment,

a crystallizer for crystallizing the supersaturated palm oil olein having been subjected to ultrasonic treatment to obtain a palm oil olein slurry,

a separation arrangement for separating the palm oil olein slurry into a palm oil mid fraction and a super olein fraction.

In is noted that the inlet may be connected to any source for obtaining a melted palm oil olein, such as a tank. Thus, the source for obtaining the melted palm oil olein include non-melted or partly melted palm oil olein if these are then brought into a melted state by heating, or palm oil olein kept in melted state, e.g. in a heated tank.

In should thus be understood that the inlet is arranged to receive the palm oil olein as a melted palm oil olein. Also, it should be understood that the cooling arrangement is adapted and arranged to cool the melted palm oil olein received by the inlet into a supersaturated palm oil olein. Further, it should be understood that the crystallizer is adapted and arranged to crystallize the supersaturated palm oil olein having been subjected to ultrasonic treatment to obtain a palm oil olein slurry. Finally, it should be understood that the separation arrangement is adapted and arranged to separate the palm oil olein slurry into a palm oil mid fraction and a super olein fraction.

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According to an advantageous embodiment of the invention, the fractionation system further comprises a heating arrangement for melting the palm oil olein.

According to a further advantageous embodiment of the invention, the fractionation system is adapted to operate in accordance with the process of the invention or any of its embodiments.

The invention relates in a further aspect to a dry-fractionated non-detopped palm oil mid fraction, wherein the palm oil mid fraction has a content of PPP-triglycerides below 2.0 percent by weight of the palm oil mid fraction.

The dry-fractionated non-detopped palm oil mid fraction may be obtained by dry fractionation without any de-topping.

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According to an advantageous embodiment of the invention, the palm oil mid fraction has an iodine value below 50.

According to a further advantageous embodiment of the invention, the palm oil mid fraction comprises POP-triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction.

According to an even further advantageous embodiment of the invention, the palm oil mid fraction has a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction.

According to a still further advantageous embodiment of the invention, the palm oil mid fraction is obtainable by the process of the invention or any of its embodiments or by a fractionation system of the invention or any of its embodiments.

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Referring to figure 1A, a process for dry fractionation of a palm oil olein POON according to an embodiment of the invention is illustrated.

The process comprises the steps of providing the palm oil olein (POON) as a melted palm oil olein MPO. The melted palm oil olein (MPO) may be obtained by melting the palm oil olein (POON). Alternatively, the melted palm oil olein (MPO) may be obtained from a source of melted palm oil olein (MPO). Different options for obtaining a melted palm oil olein (MPO) are illustrated on figures 2A-2C. The palm oil olein (POON) may typically be obtained as the liquid fraction obtained by fractionation of palm oil.

The process furthermore comprises the step of cooling the melted palm oil olein (MPO), e.g. in a cooling arrangement (COA), to obtain a supersaturated palm oil olein (SSPO). The supersaturated palm oil olein (SSPO) may be obtained by cooling the melted palm oil olein (MPO) at a rate faster than the rate of precipitation to a temperature below the supersaturation threshold. The cooling may be provided in for example a tubular pre-cooler or a crystallizer (CRS). When using a crystallizer (CRS), the crystallizer used for the subsequent step of crystallization may be used. The melted palm oil olein (MPO) may be agitated for example mechanically and/or with a gas during cooling.

Furthermore, the process comprises the step of subjecting at least a part of the supersaturated palm oil olein (SSPO) to ultrasonic treatment. This may be performed in an ultrasound emitting apparatus (USC). The process then comprises the step of crystallization, in a crystallizer (CRS), of the supersaturated palm oil olein (SSPO) having been subjected to ultrasonic treatment to obtain a palm oil olein slurry (POS).

The ultrasonic treatment may be obtained by applying ultrasound in pulses or continuously to the supersaturated palm oil olein (SSPO). The ultrasound emitting apparatus (USC) is not necessarily a single isolated unit. It may be comprised in the cooling arrangement (COA) and/or the crystallizer (CRS) or the ultrasound emitting

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apparatus (USC) may comprise the cooling arrangement (COA) and/or the crystallizer (CRS). It may also comprise the pipes in which the supersaturated palm oil olein (SSPO) is transported from the cooling arrangement (COA) to the crystallizer (CRS). The ultrasonic treatment may thus be applied in different ways to the supersaturated palm oil olein (SSPO).

The ultrasonic treatment may for example be applied to the supersaturated palm oil olein (SSPO) before it reaches the crystallizer (CRS) in a flowcell using for example a sonotrode. Alternatively, the ultrasonic treatment may be applied to the supersaturated palm oil olein (SSPO) through the pipe walls. Ultrasonic treatment may also be applied in the crystallizer (CRS) and/or cooling arrangement (COA). This could for example be the case when a sonotrode is partly or wholly submerged directly in the crystallizer (CRS) and/or cooling arrangement (COA). Ultrasonic treatment may also be applied in an external loop connected to a cooling arrangement (COA) provided in the crystallizer (CRS).

The ultrasound emitting apparatus (USC) can be connected and applied in such a way that more crystallizers and fractionation plants can make use of the same ultrasound emitting apparatus (USC).

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The crystallizer may for example be a dynamic crystallizer (CRS), such as DeSmet L-Frac units or Tirtiux, DeSmet Mobulizer, or a static crystallizer (CRS), such as a DeSmet Statoliser.

- The process additionally comprises the step of separating the palm oil olein slurry (POS) in a separation arrangement (SEA) to obtain a palm oil mid fraction (PMF) and a super olein fraction (SOF). Separating the palm oil olein slurry (POS) to obtain a palm oil mid fraction (PMF) and a super olein fraction (SOF) may for example be accomplished by filtration, such as for example membrane filtration under pressure.
- The pressure may be applied continuously or pulsating using for example a gas or a

liquid. The separation may also be performed by using super-decanters, centrifuges or vacuum filtration.

Referring to figures 2A to 2C, methods of obtaining a melted palm oil olein (MPO) are illustrated according to different embodiments, either by using a heating arrangements (HEA) as in figures 2A and 2B, or by obtaining the melted palm oil olein (MPO) from a source of melted palm oil olein (MPO) as in figure 2C.

Figure 2A shows a container comprising palm oil olein (POON), which container has a heating arrangement (HEA) surrounding the container. Thus, the palm oil olein (POON) stored in the container may be kept at the intended temperature by means of the heating arrangements (HEA), specifically the palm oil olein (POON) may be kept at a temperature where it is melted, thus ensuring that no residual crystalline triglycerides remain in the palm oil olein (POON) at later stages.

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The melted palm oil olein (MPO) obtained from the container may then be pumped to the next stage, i.e. the step of cooling,

The embodiment of figure 2A may be used in the context of figure 1.

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- Figure 2B shows a container also comprising palm oil olein (POON), but without any integrated heating arrangement (HEA). Instead, the palm oil olein (POON) is pumped to an external heating arrangement (HEA) by the pump (PMP).
- When using this embodiment, it must be ensured that the palm oil olein (POON) in the container does not crystallize too much, since this would increase the viscosity to a level where the palm oil olein (POON) is not pumpable. In such cases, it may be necessary to install an auxiliary heating arrangement in or near the container to keep the palm oil olein (POON) sufficiently melted to have a pumpable viscosity.

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The embodiment of figure 2B may be used in the context of figure 1.

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- Figure 2C illustrates that a melted palm oil olein (MPO) is obtained from a source of melted palm oil olein (MPO).
- For example, in some cases, distributors of palm oil olein (POON) keep the palm oil olein (POON) at a melted state in order to ensure pumpability. Obtaining the palm oil olein (POON) therefrom may in some cases eliminate the need for melting the palm oil olein (POON) by means of a heating arrangement (HEA).
- However, even if obtaining the palm oil olein (POON) as a melted palm oil olein (MPO), it may still in some situations be beneficial to heat the palm oil olein (POON), either to keep the palm oil olein (POON) at a melted state, or to ensure that all of the palm oil olein (POON) is in the melted state.
- 15 The embodiment of figure 2C may be used in the context of figure 1.

A further alternative may be to use the crystallizer (CRS) as a heater, e.g. if the crystallizer (CRS) has an integrated water jacket, then controlling the water supplied thereto in order to ensure melting of the palm oil olein (POON).

Referring now to figures 3A-3C, different embodiments of ultrasonic treatment and crystallization are illustrated.

Figure 3A shows a melted palm oil olein (MPO) is cooled externally from the crystallizer (CRS) to obtain a supersaturated palm oil olein (SSPO). The cooling may e.g. take place in the cooling arrangement (COA) as shown in figure 3A.

The obtained supersaturated palm oil olein (SSPO) is then subjected to ultrasonic treatment, also externally from the crystallizer (CRS). The ultrasonic treatment may take place in an ultrasound emitting apparatus (USC), as illustrated on figure 3A.

Then, the obtained nucleated palm oil olein (NPO) is fed into the crystallizer (CRS), in which it is crystallized for a sufficient period of time. In some cases, the nucleated palm oil olein (NPO) is further cooled during the step of crystallization. The crystallizer may be a dynamic crystallizer, i.e. with some sort of agitation, or it may be a static crystallizer without any agitation.

Finally, palm oil olein slurry (POS) is obtained from the crystallizer (CRS).

The embodiment illustrated on figure 3A may be implemented in the embodiment of figure 1 and works well with the options illustrated on figures 2A-2C.

Figure 3B shows a further embodiment, where the melted palm oil olein (MPO) is fed into the crystallizer (CRS). Here it is cooled, e.g. by using an integrated cooling arrangement (not shown) of the crystallizer (CRS).

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After the supersaturated palm oil olein (SSPO) is obtained by the cooling, the ultrasound emitting apparatus USC starts to treat the supersaturated palm oil olein (SSPO) with ultrasound. In some cases, only a part of the supersaturated palm oil olein (SSPO) is subjected to ultrasonic treatment, whereas in other cases all of the supersaturated palm oil olein (SSPO) is treated.

Finally, after crystallizing the nucleated palm oil olein (NPO) obtained from the ultrasonic treatment, a palm oil olein slurry (POS) is obtained.

This embodiment may be implemented in the embodiment of figure 1 and works well with the options illustrated on figures 2A-2C.

Figure 3C shows another possible setup where the melted palm oil olein (MPO) is cooled in the crystallizer (CRS), e.g. by an integrated cooling arrangement (not shown).

palm oil olein (POON) used.

Subsequently some or all of the obtained supersaturated palm oil olein (SSPO) is fed through a loop comprising an ultrasound emitting apparatus (USC), which is arranged external to the crystallizer (CRS), and back into the crystallizer as nucleated palm oil olein (NPO). The amount of the supersaturated palm oil olein (SSPO) treated by ultrasound may vary due to the exact conditions, process equipment, etc. but may for example be between 30 and 100 percent by weight of the total amount of

If using a dynamic crystallizer, the nucleated palm oil olein (NPO) may typically be mixed with the remaining supersaturated palm oil olein (SPO), if any, and then crystallized; both in the crystallizer (CRS).

This embodiment may be implemented in the embodiment of figure 1 and works well with the options illustrated on figures 2A-2C.

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Referring now to figure 4, a further embodiment of the invention is illustrated, where four crystallizers (CRS) are operated asynchronously in cooperation with one ultrasound emitting apparatus (USC). It should be understood that each crystallizer (CRS) may be operated in accordance with the embodiment of figure 1.

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A melted palm oil olein (MPO) is obtained, e.g. as illustrated in figures 2A-2C, and fed first to one of the four shown crystallizers (CRS), e.g. the leftmost crystallizer (CRS). Each of the crystallizers comprises an integrated cooling arrangement (COA), which may e.g. be provided as a water jacket with cooled water.

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When the first (leftmost) crystallizer (CRS) is filled with melted palm oil olein (MPO), which may e.g. be obtained as illustrated in figures 2A-2C, and the melted palm oil olein (MPO) is cooled into a supersaturated palm oil olein (SSPO).

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The valve (VLV) controlling the first crystallizer (CRS) is then opened, and at least part of the supersaturated palm oil olein (SSPO) therefrom is fed through the ultrasound emitting apparatus (USC) and back into the same crystallizer.

The nucleated palm oil olein (NPO) obtained from the ultrasonic treatment is then, together with the remaining supersaturated palm oil olein (SSPO), if any, crystallized in the crystallizer (CRS) to obtain a palm oil olein slurry (POS).

Finally, the palm oil olein slurry may be fed to a separation process.

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After a delay in time relative to starting the process for the first crystallizer (CRS), the same process may be carried out for the next crystallizer (CRS), e.g. the second from the left; then after a further time delay the third from the left; and finally the rightmost crystallizer (CRS).

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Thus, a number of crystallizers (CRS) may operate in cooperation with the same ultrasound emitting apparatus (USC) provided that the valves (VLV) are operated to ensure the proper relative time delay between the operation of each crystallizer (CRS).

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Then, as an illustrative example, at a certain point of time, melted palm oil olein (MPO) may be fed to one crystallizer (CRS), while another crystallizer (CRS) may be cooling the melted palm oil olein (MPO), a third crystallizer (CRS) may be in the process of feeding supersaturated palm oil olein (SSPO) through the ultrasound emitting apparatus (USC) for ultrasound treatment, and a fourth crystallizer (CRS) may be in the process of crystallizing the palm oil olein being fully or partly treated by ultrasound.

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The principle of operating more than one crystallizer (CRS) asynchronously with only one ultrasound emitting apparatus (USC) may be used for two or three crystallizers (CRS), or in some cases also for more than four crystallizers (CRS).

Also, the more than one crystallizer (CRS) may operate with only one separation apparatus, such as only one filter and pressing apparatus.

Referring now to figure 5, a further embodiment of the invention is illustrated.

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First, a palm oil (OIL) is fractionated in a first fractionation (FRAC1), which is a dry fractionation, into a palm oil olein fraction (POON) and a palm stearin fraction (PST). This process is not necessarily a part of the invention, but illustrates obtaining a palm oil olein (POON) from palm oil (OIL).

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Then, in a second fractionation (FRAC2), which is a dry fractionation, the obtained palm oil olein (POON) is fractionated into super olein fraction (SUP) and a palm oil mid fraction (PMF). The second fractionation (FRAC2) is the process illustrated in figure 1. The notations "first" and "second" fractionation are merely meant to signify the sequence in figure 5.

Optionally included may be a subsequent dry fractionation (SFRAC) of the palm oil mid fraction (PMF) into a subsequent palm oil mid fraction (SPMF) and a subsequent palm olein (OLN).

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It is noted that in this embodiment the palm oil mid fraction (PMF) is subjected to the subsequent fractionation without any intermediate de-topping process of removing PPP-triglycerides.

In some alternative embodiments, the palm oil mid fraction (PMF) may be subjected to the subsequent fractionation after an intermediate detopping process of removing PPP-triglycerides, e.g. if a palm oil olein (POON) with content of PPP-triglycerides above 1.0 percent by weight of the palm oil olein (POON) is used.

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#### FIGURE REFERENCES

POON. Palm oil olein

MPO. Melted palm oil olein

5 SSPO. Supersaturated palm oil olein

CRS. Crystallizer

POS. Palm oil olein slurry

PMF. Palm oil mid fraction

SOF. Super olein fraction

10 FRS. Fractionation system

HEA. Heating arrangement

COA. Cooling arrangement

USC. Ultrasound emitting apparatus

SEA. Separation arrangement

15 NPO. Nucleated palm oil olein

VLV. Valve

FRAC1. First fractionation

FRAC2. Second fractionation

OIL. Palm oil

20 PST. Palm stearin fraction

SFRAC. Subsequent fractionation

OLN. Subsequent palm olein

SMPF. Subsequent palm oil mid fraction

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#### **EXAMPLES**

## Example 1 – no ultrasound

A fractionation of palm oil olein was performed according to the following experimental setup for examples 1-1 and 1-2.

A crystallizer of the type L-frac pilot dynamic crystallizer from DeSmet was used for the step of cooling and crystallizing.

A total volume of 12 liters of palm oil olein was treated. Approximately 1 liter of the treated palm oil olein was subjected to the filtering and pressing step of the fractionation.

The crystallizer was set with a stirrer speed of approximately 10 rpm during the steps of cooling and crystallizing.

The external cooling water applied to the crystallizer was set to have a temperature profile: First, 60 degrees Celsius for at least 1 hour, then gradually lowering the temperature from 60 degrees Celsius to 16.0 degrees Celsius over a period of 2 hours, then finally keeping the temperature at 16.0 degrees Celsius for about 16 to 18 hours. The palm oil olein obtained after this had a final temperature of 17.3 degrees Celsius at the time of fractionation.

The fractionation was performed using a high-pressure filter at 25 bar.

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A fractionation of palm oil olein was performed for example 1-3 according to the same experimental setup as examples 1-1 and 1-2, with the modifications of using 15.5 degrees Celsius as the final temperature of the external cooling water instead of 16.0 degrees Celsius. This results in a final oil temperature of 17.0 degrees Celsius at the time of fractionation.

No ultrasonic treatment was used for any of the examples 1-1, 1-2, or 1-3.

The results of the fractionation examples 1-1, 1-2, and 1-3 are given in table 1 below. The results include the yield, the composition, and the iodine value of the resulting olein and stearin fractions (corresponding to the super olein fraction and the palm oil mid fraction, respectively).

Example	1-1		1-2		1-3	
PPP-triglycerides	1.5% w/w		0.5% w/w		0.5% w/w	
Cooling temp [°C]	16		16		15.5	
	Olein	Stearin	Olein	Stearin	Olein	Stearin
yield [% w/w]	66.1	33.9	87.9	12.1	66.1	45.8
TAG PLiLi	2.8	1.2	2.4	0.5	2.8	1.7
TAG PLiO	13.6	5.9	12.2	2.6	13.6	8.5
TAG PLiP	9.5	8.1	8.9	8	9.5	8.2
TAG 000	5.4	2.7	5	1.8	5.4	3.7
TAG POO	31.1	14.4	28.4	7.9	31.1	20.3
TAG POP	19.3	44.9	23.6	57.3	19.3	36.8
TAG PPP	<0.1	3.8	<0.1	4.4	<0.1	1.2
TAG StOO	3.7	1.6	3.5	0.7	3.7	2.3
TAG POSt	3.9	8.9	5.1	10	3.9	7.7
Iodine value	63.61	44.84	60.94	37.2	63.61	52.08

Table 1: Results without use of ultrasonic treatment, and with and without using PPP-triglyceride addition. "Stearin" indicates the stearin fraction of palm oil olein, i.e. a palm oil mid fraction. "Olein" indicates the olein fraction of palm oil olein, i.e. a super olein fraction. The notation of e.g. "TAG POO" signifies POO-triglycerides.

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In the traditional process for fractionation of palm oil olein two major modifications are available in order to improve the yield and the quality of the product, namely

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addition of PPP-triglycerides in different amounts and adjusting the cooling temperature.

As can be seen from table 1, example 1-1 shows that an acceptable stearin fraction of a palm oil olein, i.e. a palm oil mid fraction, may be obtained using a palm oil olein with a relatively high content of PPP-triglycerides (here 1.5 percent by weight of the palm oil olein).

If the amount of added PPP-triglycerides is lowered but the cooling temperature kept the same it can be seen in example 1-2 that a fractionation with an acceptable quality of palm oil mid fraction may be obtained from palm oil olein with a low PPP-triglyceride content (0.5 percent by weight of the palm oil olein), however the corresponding yield of the palm oil mid fraction (12.1 percent by weight of the palm oil olein) is unacceptably low.

Example 1-3 illustrates that when slightly lowering the temperature of fractionation in order to increase the yield of the palm oil mid fraction, the increase of yield is obtained at the cost of the quality. The unacceptable low quality is illustrated as the low difference between the iodine values of the two obtained fractions, and of the lower concentration of POP-triglycerides in the stearin fraction compared e.g. to example 1-1.

Thus, example 1-1 represents a traditional approach to palm oil olein fractionation while both examples 1-2 and 1-3 represents economically unattractive alternatives due to the low yield and quality, respectively.

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## Example 2 - PMF IV 45 - With ultrasound

A fractionation of palm oil olein was performed in pilot and full scale according to the following experimental setup for example 2.

#### 30 Pilot scale

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A crystallizer of the type L-frac pilot dynamic crystallizer from DeSmet was used for the step of cooling and crystallizing.

A total volume of 12 liters of palm oil olein was treated. The palm oil olein had a content of PPP-triglycerides of 0.5 percent by weight of the palm oil olein. Approximately 1 liter of the treated palm oil olein was subjected to the filtering and pressing step of the fractionation.

The crystallizer was set with a stirrer speed of approximately 10 rpm during the steps of cooling and crystallizing.

The external cooling water applied to the crystallizer was set to have a temperature profile: First, 60 degrees Celsius for at least 1 hour, then gradually lowering the temperature from 60 degrees Celsius to 16.0 degrees Celsius over a period of 2 hours, then finally keeping the temperature at 16.0 degrees Celsius for 16 to 18 hours. The palm oil olein obtained after this had a final temperature of 17.3 degrees Celsius at the time of fractionation. The fractionation was performed using a high-pressure filter at 25 bar.

When the temperature of the palm oil olein reached a level of 18-19 degrees Celsius (within the supersaturated region), a volume corresponding to about 50 percent by weight of the supersaturated palm oil olein was pumped from the crystallizer, through an external loop comprising an ultrasound flowcell, and back into the crystallizer. A pumping rate of about 1.2 liters per minute was used.

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The ultrasound flowcell was configured to apply ultrasound in pulsed sequences of 1 second of ultrasound followed by 4 seconds of no ultrasound. The ultrasound was applied for a period of about 5 minutes to give a total of 60 pulses. The ultrasound amplitude was set to 30%, while the pressure was set to between 1 and 1.5 bar. The total ultrasound effect was 6 kilowatts, and an ultrasound frequency of 20 kilohertz was used.

The fractionation was performed using a high-pressure filter at 25 bar.

### Full scale

5 A 4 concentric dynamic crystallizers from DeSmet was used for the step of cooling and crystallizing.

A total volume of 64 tons of palm oil olein was treated. The palm oil olein had a content of PPP-triglycerides of 0.5 percent by weight of the palm oil olein. All 64 tons of the treated palm oil olein was subjected to the filtering and pressing step of the fractionation.

The crystallizer was set with a stirrer speed of between 10 and 3 rpm during the steps of cooling and crystallizing.

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The external cooling water applied to the crystallizer was set to have a temperature profile: First, 60 degrees Celsius for at least 1 hour, then gradually lowering the temperature from 60 degrees Celsius to 16.0 degrees Celsius over a period of 2 hours, then finally keeping the temperature at 16.0 degrees Celsius for 16 to 18 hours. The palm oil olein obtained after this had a final temperature of 17.3 degrees Celsius at the time of fractionation. The fractionation was performed using a high-pressure filter at 25 bar.

- When the temperature of the palm oil olein reached a level of 18-19 degrees Celsius (within the supersaturated region), a volume corresponding to 100 percent by weight of the supersaturated palm oil olein was pumped from the crystallizer, through an external loop comprising an ultrasound flowcell, and back into the crystallizer. A pumping rate of 30 tons per hour was used.
- The ultrasound flowcell was configured to apply ultrasound continuously. The ultrasound was applied for a period of about 120 minutes. The ultrasound amplitude

was set to 100%, while the pressure was between 1 and 1.5 bar. The total ultrasound effect was 6.5 megawatts, and an ultrasound frequency of 22 kilohertz was used.

The fractionation was performed using a high-pressure filter at 25 bar.

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Example 2					
PPP-triglycerides	0.5 % w/w				
	Pilot scale		Full scale		
	Olein	Stearin	Olein	Stearin	
yield [% w/w]	62.4	37.7	62	38	
TAG PLiLi	2.4	1.1	2.4	1.1	
TAG PLiO	13.1	5.9	13.1	5.6	
TAG PLiP	8.3	7.1	8.1	7.1	
TAG OOO	6.8	3.3	6.9	2.9	
TAG POO	33.7	16.2	32.5	14.3	
TAG POP	17.1	45.1	17.7	46.7	
TAG PPP	<0.1	1.575	<0.1	1.4	
TAG SOO	4.2	2.0	4.3	1.9	
TAG POS	3.4	9.4	3.8	10.4	
Iodine value	64.5	46.7	64.5	45.6	
3-MCPD level,	n.a.	n.a.	0.35	0.14	
[ppm]					

Table 2: Results with use of ultrasonic treatment, and without using PPP-triglyceride addition. "Stearin" indicates the stearin fraction of palm oil olein, i.e. a palm oil mid fraction. "Olein" indicates the olein fraction of palm oil olein, i.e. a super olein fraction. The notation of e.g. "TAG POO" signifies POO-triglycerides.

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As can be seen from example 2, a palm oil olein having a low content of PPP-triglyceride of 0.5 percent by weight of the palm oil olein is successfully fractionated to obtain a high yield of palm oil mid fraction having a superior quality. The high

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quality of the obtained palm oil mid fraction is illustrated by the large difference between the iodine values of the palm oil mid fraction (Stearin in table 2, IV 46.7 for pilot scale and 45.6 for full scale) and the super olein fraction (Olein in table 2, IV 64.5 for both pilot and full scale). Also the low content of POO-triglycerides in the obtained palm oil mid fraction and the relatively high content of POP-triglycerides in the stearin fraction is a measure of a high quality product. Thus, from example 2 it is evident that in both pilot and full scale it is possible to obtain a product of high quality while keeping the yield high. The 3-MCPD level was measured on the full scale experiment. The level of 3-MCPD in both the Stearin and Olein fraction was very low as can be seen in Table 2. The person skilled in the art will know that it is particularly difficult to keep the level of 3-MCPD low in the olein fraction because of migration.

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### **CLAIMS**

1. A process for dry fractionation of a palm oil olein (POON), the process comprising the steps of

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providing the palm oil olein (POON) as a melted palm oil olein (MPO),

cooling the melted palm oil olein (MPO) to obtain a supersaturated palm oil olein (SSPO),

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subjecting at least a part of the supersaturated palm oil olein (SSPO) to ultrasonic treatment,

crystallization, in a crystallizer (CRS), of the supersaturated palm oil olein (SSPO) having been subjected to ultrasound to obtain a palm oil olein slurry (POS),

separating the palm oil olein slurry (POS) to obtain a palm oil mid fraction (PMF) and a super olein fraction (SOF).

- 20 2. The process according to claim 1, wherein the step of ultrasonic treatment is performed external to said crystallizer (CRS).
  - 3. The process according to claim 1 or 2, wherein the step of cooling the palm oil olein (POON) is performed in said crystallizer (CRS).

- 4. The process according to claim 1 or 2, wherein the step of cooling the palm oil olein (POON) is performed external to said crystallizer (CRS).
- 5. The process according to any of the claims 1-3, wherein the step of cooling the palm oil olein (POON) is performed in said crystallizer (CRS), wherein at least part of the supersaturated palm oil olein (SSPO) is fed from the crystallizer (CRS) to an

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ultrasound emitting apparatus (USC) arranged external to said crystallizer (CRS), and wherein the palm oil olein having been subjected to ultrasonic treatment in said ultrasound emitting apparatus (USC) is fed back into said crystallizer (CRS).

- 5 6. The process according to any of the claims 1-5, wherein the supersaturated palm oil olein (SSPO) has a temperature of between 10 and 37 degrees Celsius.
  - 7. The process according to any of claims 1-6, wherein the palm oil olein (POON) has a content of PPP-triglycerides below 1.0 percent by weight of the palm oil olein.
  - 8. The process according to any of claims 1-7, wherein the yield of the palm oil mid fraction (PMF) relative to the palm oil olein (POON) is at least 20 percent by weight.
- 9. The process according to any of the claims 1-8, wherein the yield of the palm oil
  mid fraction (PMF) relative to the palm oil olein (POON) is at least 30 percent by weight.
- 10. The process according to any of claims 1-9, wherein the palm oil mid fraction (PMF) has a content of PPP-triglycerides below 2.0 percent by weight of the palm oil20 mid fraction (PMF).
  - 11. The process according to any of claims 1-10, wherein the palm oil mid fraction (PMF) has an iodine value below 50.
- 25 12. The process according to any of claims 1-11, wherein the difference between the iodine value of the palm oil mid fraction (PMF) and the iodine value of the super olein fraction (SOF) is at least 15, such as at least 16.
- 13. The process according to any of claims 1-12, wherein the super olein fraction30 (SOF) has an iodine value above 60.

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- 14. The process according to any of claims 1-13, wherein the palm oil mid fraction (PMF) comprises POP-triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction (PMF).
- 5 15. The process according to any of claims 1-14, wherein the palm oil mid fraction (PMF) has a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction (PMF).
- 16. The process according to any of claims 1-15, wherein the process of obtaining palm oil mid fraction (PMF) from the palm oil olein (POON) is performed in a time period of less than 18 hours.
- 17. The process according to any of the claims 1-16, wherein the palm oil mid fraction (PMF) is subjected to a subsequent dry fractionation (SFRAC) obtaining a subsequent palm oil mid fraction (SPMF) and a subsequent palm olein (OLN).
  - 18. The process according to claim 17, wherein the subsequent fractionation (SFRAC) is performed at a temperature of below 25 degrees Celsius.
- 20 19. The process according to claim 17 or 18, wherein the subsequent palm oil mid fraction (SPMF) has an iodine value below 40.
  - 20. The process according to any of claims 17-19, wherein no detopping is used before the subsequent fractionation (SFRAC).

21. The process according to any of claims 17-20, wherein the subsequent palm oil mid fraction (SPMF) has a content of PPP-triglycerides below 4.0 percent by weight

subsequent palm oil mid fraction (SPMF).

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22. The process according to any of claims 1-21, wherein the palm oil mid fraction (PMF) is not subjected to any de-topping.

- 23. The process according to any of claims 1-22, wherein the palm oil olein (POON) is neutralized.
- 5 24. The process according to any of claims 1-23, wherein the palm oil olein (POON) is physically refined.
  - 25. The process according to any of claims 1-24, wherein the super olein fraction (SOF) has a content of 3-MCPD of less than 0.5 ppm.
- 26. The process according to any of the claims 1-25, wherein the step of crystallization of the palm oil olein (POON) in the crystallizer (CRS) comprises cooling the palm oil olein (POON) in said crystallizer (CRS).

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- 15 27. The process according to any of the claims 1-26, wherein the step of separating the palm oil olein slurry (POS) comprises filtering and pressing the palm oil olein slurry (POS).
- 28. The process according to any of the claims 1-27, wherein the palm oil olein (POON) has a content of PPP-triglycerides above 1.0 percent by weight of the palm oil olein (POON).
  - 29. The process according to any of the claims 1-28, wherein an amount of supersaturated palm oil olein (SSPO) corresponding to at least 10 percent by weight of the total amount of palm oil olein (POON) is subjected to ultrasound treatment.
    - 30. A fractionation system (FRS) for fractionating a palm oil olein (POON), the fractionation system (FRS) comprising
- an inlet for receiving the palm oil olein (POON) as a melted palm oil olein (MPO),

a cooling arrangement (COA) for cooling the melted palm oil olein (MPO) into a supersaturated palm oil olein (SSPO),

- an ultrasound emitting apparatus (USC) arranged to subject at least a part of the supersaturated palm oil olein (SSPO) to ultrasonic treatment,
  - a crystallizer (CRS) for crystallizing the supersaturated palm oil olein (SSPO) having been subjected to ultrasonic treatment to obtain a palm oil olein slurry (POS),
- a separation arrangement (SEA) for separating the palm oil olein slurry (POS) into a palm oil mid fraction (PMF) and a super olein fraction (SOF).
  - 31. The fractionation system (FRS) according to claim 30, wherein the fractionation system (FRS) further comprises a heating arrangement (HEA) for melting the palm oil olein (POON).
    - 32. The fractionation system (FRS) according to claim 30 or 31, wherein the fractionation system (FRS) is adapted to operate in accordance with the process of any of claims 1-29.

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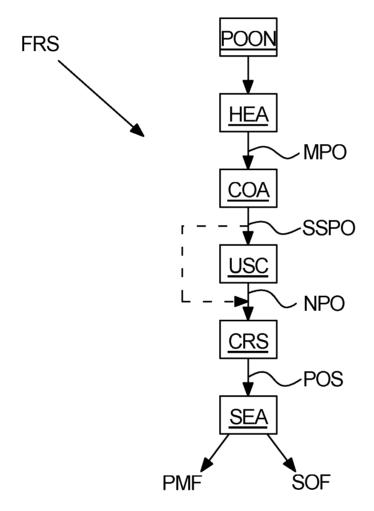
- 33. A dry-fractionated non-detopped palm oil mid fraction (PMF), wherein the palm oil mid fraction (PMF) has a content of PPP-triglycerides below 2.0 percent by weight of the palm oil mid fraction (PMF).
- 25 34. The palm oil mid fraction (PMF) according to claim 33, wherein the palm oil mid fraction (PMF) has an iodine value below 50.
  - 35. The palm oil mid fraction (PMF) according to claim 33 or 34, wherein the palm oil mid fraction (PMF) comprises POP-triglycerides in an amount of between 35 and 70 percent by weight of the palm oil mid fraction (PMF).

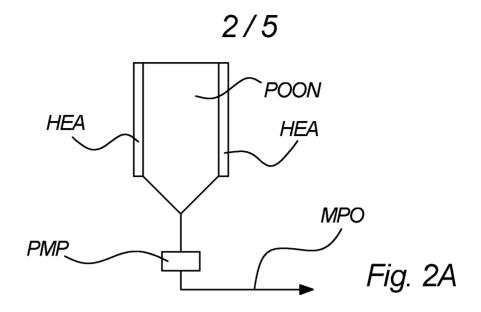
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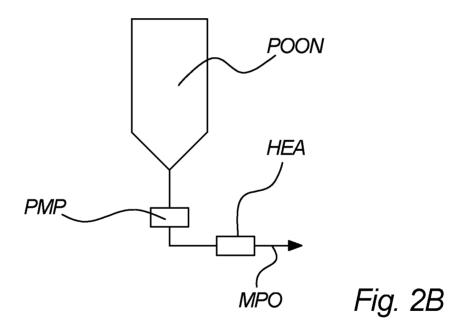
36. The palm oil mid fraction (PMF) according to any of claims 33-35, wherein the palm oil mid fraction (PMF) has a content of POO-triglycerides of less than 20 percent by weight of the palm oil mid fraction (PMF).

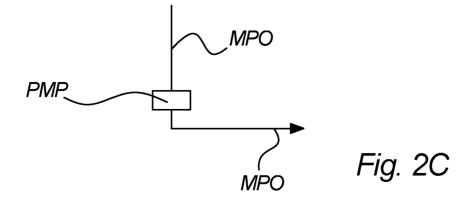
5 37. The palm oil mid fraction (PMF) according to any of claims 33-36, wherein the palm oil mid fraction (PMF) is obtainable by the process of any of claims 1-29 or by a fractionation system (FRS) according to any of claims 30-32.

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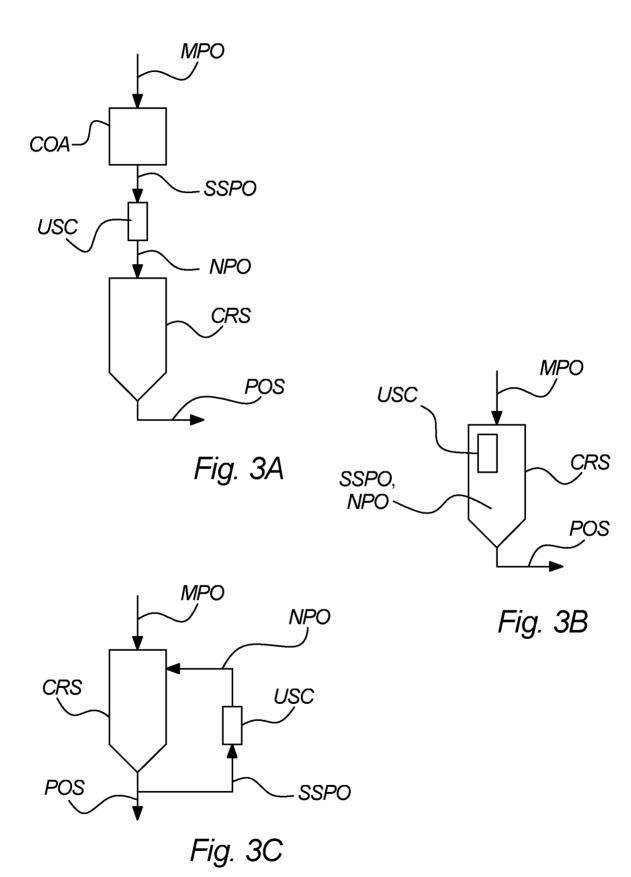












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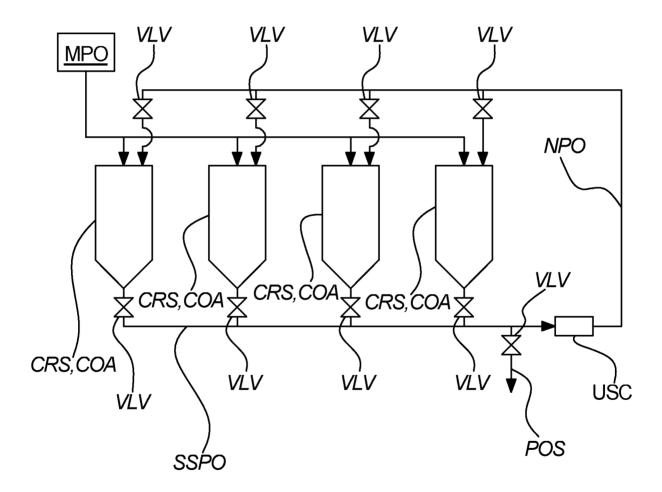
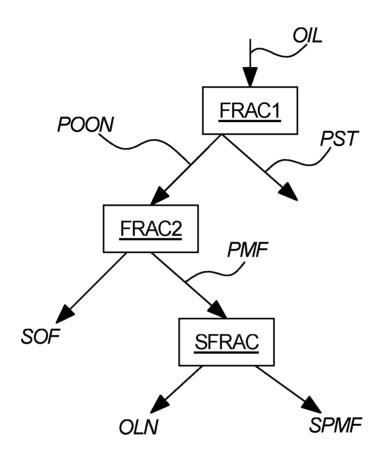


Fig. 4

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International application No.

## PCT/SE2017/051160

### A. CLASSIFICATION OF SUBJECT MATTER

### IPC: see extra sheet

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)

IPC: B01D, C11B

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

## SE, DK, FI, NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

# EPO-Internal, PAJ, WPI data, BIOSIS, CHEM ABS Data, COMPENDEX, EMBASE, INSPEC, MEDLINE, PUBCHEM

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Y	<del></del>	1-29
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$ \boxtimes $	Further documents are listed in the continuation of Box C.		See patent family annex.		
* "A"	Special categories of cited documents:  document defining the general state of the art which is not considered to be of particular relevance		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		
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Date	Date of the actual completion of the international search		Date of mailing of the international search report		
05-	05-07-2018		05-07-2018		
	Name and mailing address of the ISA/SE		Authorized officer		
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Facsimile No. + 46 8 666 02 86		Telephone No. + 46 8 782 28 00			

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Continuation of: second sheet	
International Patent Classification (IPC)	
C11B 7/00 (2006.01) B01D 9/00 (2006.01) C11B 3/16 (2006.01)	

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