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(54) **Title:** METHOD FOR PRODUCTION OF EDIBLE WATER-IN-OIL EMULSION

(57) **Abstract:** The present invention provides a method for production of water-in-oil emulsions (in particular low-fat water-in-oil emulsions (<50% oil)), without the requirement of making a premix of the emulsion, and without the requirement to heat an entire premix, and wherein the emulsion can be made in a single process step. It is especially an object of the present invention to produce low-fat water-in-oil emulsions (<50% oil) in a single step method. In this method a distributive and dispersive mixing apparatus of the Controlled Deformation Dynamic Mixer type is used to create a water-in-oil emulsion, and wherein the mixer is suitable for inducing extensional flow in a liquid composition, and wherein the mixer comprises closely spaced relatively moveable confronting surfaces at least one having a series of cavities therein in which the cavities on each surface are arranged such that, in use, the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus.



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METHOD FOR PRODUCTION OF EDIBLE WATER-IN-OIL EMULSION

FIELD OF THE INVENTION

The present invention relates to a method for the production of an edible water-in-oil
5 emulsion, by using a Controlled Deformation Dynamic Mixer.

BACKGROUND TO THE INVENTION

Mixing can be described as either distributive or dispersive. In a multi-phase material
10 comprising discrete domains of each phase, distributive mixing seeks to change the
relative spatial positions of the domains of each phase, whereas dispersive mixing
seeks to overcome cohesive forces to alter the size and size distribution of the domains
of each phase. Most mixers employ a combination of distributive or dispersive mixing
although, depending on the intended application the balance will alter. For example a
15 machine for mixing peanuts and raisins will be wholly distributive so as not to damage
the things being mixed, whereas a blender/homogeniser will be dispersive.

Fat continuous food products are well known in the art and include for example
shortenings comprising a fat phase and water-in-oil emulsions like margarine
20 comprising a fat phase and an aqueous phase. Margarine traditionally contains about
80% of an edible fat phase, and 20% of an aqueous phase, that is dispersed as small
droplets in the continuous edible fat phase. Other examples of water-in-oil emulsions
are low-fat spreads, in which the proportion of edible fat phase is lower and aqueous
phase is higher than margarine, for example about 10 to 40% edible fat phase and
25 about 60 to 90% aqueous phase. Such emulsions as described here are commonly
known to the consumer as a food product and can usually be spread on bread or toast
or the like, or can be used as a frying medium for cooking.

The fat phase of margarine and similar edible fat continuous spreads is often a mixture
30 of liquid oil (i.e. fat that is liquid at ambient temperature) and fat which is solid at
ambient temperatures. The solid fat, also called structuring fat or hardstock fat, serves
to structure the fat phase (for example in a shortening as well as in a water-in-oil
emulsion) and helps to stabilize the aqueous phase, if present, by forming a fat crystal
network. For a margarine or spread, ideally the structuring fat has such properties that

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it melts or dissolves at mouth temperature upon consumption. Otherwise the product may have a heavy and/or waxy mouthfeel.

5 Important aspects of a fat continuous spread like margarine and low fat spread, are for example hardness, spreadability and ability to withstand temperature cycling. Temperature cycling means that the product is subjected to low and high temperatures (e.g. when the consumer takes the product out of the refrigerator and leaves it for some time at the table to use it). This may have a negative influence on the structure of the spread (like for example destabilisation of the emulsion or oil-exudation).

10

Generally edible fat continuous food products like for example margarines and similar edible fat continuous spreads are prepared according to known processes that encompass the following steps:

- 15 (i) Making a pre-emulsion of an aqueous phase and an edible fat phase. As indicated before, the fat phase usually contains a mixture of liquid oil (often containing C₁₆-C₁₈ mono- or polyunsaturated fatty acids) and structuring fat (often containing C₁₆-C₁₈ saturated fatty acids).
- (ii) The emulsion is heated for pasteurisation, leading to complete melting of the edible fat phase.
- 20 (iii) The warm emulsion is cooled under high shear to induce crystallization of the structuring fat to create an emulsion. A fat crystal network is formed to stabilize the resulting emulsion and give the product some degree of firmness. Common equipment in this step is a scraped surface heat exchanger, wherein the wall of the heat exchanger is at a temperature lower than -10°C. The saturated fatty acids crystallise on
25 the cold wall, and these solids are scraped from the wall. The water-in-oil emulsion is formed in this step, leading to a fine dispersion of aqueous phase droplets (size usually smaller than 10 micrometer) in the continuous fat phase. The droplets are stabilised by small saturated fat crystals on the interface of the droplets.
- (iv) The crystal network is further modified to produce the desired firmness, confer
30 plasticity and reduce the water droplet size. This step is often done in a tube containing a mixer.
- (v) The emulsion is filled in its packaging for further distribution to consumers.

These steps are usually conducted in a process that involves apparatus that allow
35 heating, cooling and mechanical working of the ingredients, such as the churn process

or the votator process. The churn process and the votator process are described in the Ullmans Encyclopedia, Fifth Edition, Volume A 16, pages 156-158.

5 The choice of fats that can practically be used as structuring agent is rather limited. If the melting point of the structuring agent is too high the melting properties in the mouth are unsatisfactory. If on the other hand, the melting point is too low, the emulsion stability will be negatively affected.

10 Triacylglycerols (TAG) are the major constituents of natural fats and oils and are esters of glycerol and fatty acids. The chemical structure of the fatty acid and the distribution of the fatty acids over the glycerol backbone determine (at least partly) the physical properties of a fat. The physical properties of fats, like for example the solid fat content (SFC) expressed as N-value, can be modified by altering the chemical structure of the fat. Well known techniques that are widely used include hydrogenation and
15 interesterification.

EP 194 812 A2 discloses a cavity transfer mixer (CTM). Also WO 96/20270 describes a 'Cavity Transfer Mixer', comprising confronting surfaces, each having a series of cavities formed therein in which the surfaces move relatively to each other and in which
20 a liquid material is passed between the surfaces and flows along a pathway successively passing through the cavities in each surface. Generally, the cross-sectional area for flow varies by a factor of less than 3 through the apparatus.

WO 96/20270 also describes a novel mixer, hereinafter referred to as a 'Controlled
25 Deformation Dynamic Mixer' (CDDM). In common with the CTM, this type of mixer has stator and rotor elements with opposed cavities which, as the mixer operates, move past each other across the direction of bulk flow through the mixer. It is distinguished from the CTM in that material is also subjected to extensional deformation. The extensional flow and efficient dispersive mixing is secured by having confronting
30 surfaces with cavities arranged such that the cross sectional area for bulk flow of the liquid through the mixer successively increases and decreases by a factor of at least 5 through the apparatus. The CDDM combines the distributive mixing performance of the CTM with dispersive mixing performance.

EP 199 937 A1 describes a mixing device containing a member with cavities rotating within a cylinder containing cavities as well. A fat phase and an aqueous phase are separately cooled and plasticised in the A-unit and C-unit of a votator. Subsequently the phases are mixed in the mixing device, retaining fat crystal structure.

5

WO 2010/089320 A1, WO 2010/089322 A1, and WO 2010/091983 A1 disclose specific types of a distributive and dispersive mixing apparatus of the CDDM type or CTM type, comprising two confronting surfaces having cavities therein. These specific types may be used for the treatment of emulsions.

10

US 2010/220545 A1 discloses a mixer with distributive and dispersive action, that can be used for emulsification.

15

WO 2007/105323 A1 discloses a rotor-stator mixer with the intention of forming a water-in-oil emulsion fuel. This device is dispersive in character.

JP 7-194301 describes a mixing apparatus used for making emulsions like cream, dressings, and spreads.

20 US 6,468,578 B1 discloses the use of a cavity transfer mixer for creating an emulsion of water droplets in a continuous fat phase. In this method a cream of oil in an aqueous phase is produced which is pasteurised, followed by cooling. Inversion to a water-in-oil emulsion then is conducted in a cavity transfer mixer. In this case the scraped surface heat exchanger is replaced by a cavity transfer mixer as disclosed in EP 199 398 A1.
25 The amount of fat phase in this product is from 50 to 85% by weight.

US 5,837,307 discloses a method for making edible fat-continuous emulsions with less than 30% fat, using a cold process. The temperature at mixing of the aqueous phase and the fat phase should not be higher than the melting temperature of the fat phase.

30

The disadvantages of the standard process are that it consumes a lot of energy in heating and cooling. The complete pre-emulsion is heated, and subsequently cooled, which leads to a lot of energy dissipation. Additionally producing a low fat (less than 50% by weight of fats and oils) water-in-oil emulsion spread in a single process step,
35 having the right emulsion structure and product consistency is difficult.

SUMMARY OF THE INVENTION

Therefore it is an object of the invention to provide a method for production of water-in-oil emulsions, without the requirement of making a premix of the emulsion, and without
5 the requirement to heat an entire premix, and wherein the emulsion can be made in a single process step. It is especially an object of the present invention to produce low-fat water-in-oil emulsions (<50% oil) in a single step method.

10 We have now determined that this objective can be met by a method wherein the water-in-oil emulsion is formed in a distributive and dispersive mixing apparatus of the Controlled Deformation Dynamic Mixer type to create a water-in-oil emulsion, and wherein the mixer is suitable for inducing extensional flow in a liquid composition, and wherein the mixer comprises closely spaced relatively moveable confronting
15 surfaces at least one having a series of cavities therein in which the cavities on each surface are arranged such that, in use, the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus.

The advantage of this method is that energy can be saved, because not the entire
20 premix needs to be pasteurised. A pre-emulsion of aqueous phase and fat phase does not need to be produced, hence it is sufficient if only the aqueous phase is pasteurised before mixing with the fat phase in the CDDM to produce a fat-continuous emulsion. Additionally the fat-continuous emulsion is produced in a single process step, and the right consistency and hardness of the emulsion can be achieved without the need to
25 process it any further in another process step. This process requires less unit operations than conventional processes, which makes the process simpler.

Accordingly the invention provides a method for production of an edible water-in-oil emulsion,
30 wherein the emulsion comprises an aqueous phase, a structuring fat and a liquid oil, comprising the steps:
a) melting a structuring fat; and
b) introducing the molten structuring fat from step a), a liquid oil and an aqueous phase
35 into a distributive and dispersive mixing apparatus of the Controlled Deformation

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Dynamic Mixer type to create a water-in-oil emulsion,
and wherein the mixer is suitable for inducing extensional flow in a liquid composition,
and wherein the mixer comprises closely spaced relatively moveable confronting
surfaces at least one having a series of cavities therein in which the cavities on each
5 surface are arranged such that, in use, the cross-sectional area for flow of the liquid
successively increases and decreases by a factor of at least 5 through the apparatus.

DESCRIPTION OF FIGURES

10 Figure 1: Schematic representation of a Cavity Transfer Mixer (CTM); 1: stator, 2: annulus; 3: rotor; with cross-sectional views below.

Figure 2: Schematic representation of a Controlled Deformation Dynamic Mixer (CDDM) ; 1: stator, 2: annulus; 3: rotor; with cross-sectional views below.

Figure 3: Schematic representation of a preferred embodiment of the CDDM
15 apparatus, cross-sectional view (direction of bulk flow preferably from left to right).

Figure 4: Schematic representation of a preferred embodiment of the CDDM
apparatus, cross-sectional view (direction of bulk flow preferably from left to right).

20 DETAILED DESCRIPTION OF THE INVENTION

Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art.

All percentages, unless otherwise stated, refer to the percentage by weight. The
25 abbreviation 'wt%' or '% (w/w)' refers to percentage by weight.

In the context of the present invention, the average particle or droplet diameter is generally expressed as the $d_{3,3}$ value, which is the volume weighted geometric mean particle or droplet diameter. The normal terminology for nuclear magnetic resonance
30 (NMR) is used to measure the parameters $d_{3,3}$ and sigma (or alternatively $\exp(\sigma)$) of a log-normal oil droplet size distribution. Sigma is the standard deviation of the logarithmic of the droplet diameter $d_{3,3}$.

In case a range is given, the given range includes the mentioned endpoints.

An edible or a food product in the context of the present invention encompasses, but is not limited to, food products including spreads, salad dressings, dairy products, beverages, dietetic foods, dietary supplements, pharmaceutical compositions, and others. The products may contain ingredients common in the art and may be made by methods common in the art.

The terms 'fat' and 'oil' are used interchangeably. Where applicable the prefix '*liquid*' or '*solid*' is added to indicate if the fat or oil is liquid or solid at ambient temperature as understood by the person skilled in the art. The term '*structuring fat*' refers to a fat that is solid at ambient temperature. The term '*liquid oil*' refers to an oil that is liquid at ambient temperature.

Edible fats contain a large number of different triacylglycerols (TAGs) with varying physical properties. The TAGs in edible fats are composed of fatty acids with an even number of carbon atoms in the chains, generally varying between 4 and 24 in number. Common fatty acids from vegetable origin are C10, C12, C14, C16, C18, C20 and C22, and most common TAGs are composed of these fatty acids. Moreover each fatty acid can contain up to three double bonds at certain positions in the chain. Especially fish oil contains a high number of unsaturated fatty acids with more than one unsaturated bond in the chain. The terms '*triacylglycerols*', '*TAGs*', and '*triglycerides*' may be used interchangeably in here.

Ambient temperature is considered to be a temperature between about 20°C and about 25°C, preferably between 20°C and 25°C, preferably between 20°C and 23°C.

Cavity Transfer Mixers (CTMs)

Similar as in WO 96/20270, CTMs are defined as mixers comprising confronting surfaces, at least one of the surfaces, preferably both surfaces, having a series of cavities formed therein in which the surfaces move relatively to each other and in which a liquid material is passed between the surfaces and flows along a pathway successively through the cavities in each surface. The cavities are arranged on the relevant surfaces such that shear is applied to the liquid as it flows between the surfaces. The cavities are arranged on the respective surfaces such that there is a relatively small change in the effective cross sectional flow area as the material passes

through the mixer. In such mixers, primarily distributive mixing is obtained. Generally the cross-sectional area for flow varies by a factor of less than 3 through the apparatus. Shear is applied by the relative movement of the surfaces in a generally perpendicular direction to the flow of material there between.

5

Here we exemplify CTMs by reference to Figure 1 which displays an axial section and four transverse radial sections through a CTM configured as a 'concentric cylinder' device and comprising an inner rotor journalled within an outer stator. Briefly, the axial section shows the relative axial positions of rotor and stator cavities which are time invariant, whereas the transverse sections (A-A, B-B, C-C, D-D) demonstrate the axial variation in the available cross-sectional area for material flow axially:

- A-A through the stator cavities in positions in which those stator cavities are confronted by 'rotor rings', ie the circumferentially extending rings which separate successive rings of rotor cavities;
- 15 ▪ B-B between the stator cavities and the rotor cavities in positions in which the former are confronted by the latter;
- C-C through the rotor cavities in positions in which those rotor cavities are confronted by 'stator rings', ie the circumferentially extending rings which separate successive rings of stator cavities;
- 20 ▪ D-D between the rotor cavities and the stator cavities in positions in which the former are confronted by the latter.

The key feature to note is that there is little variation in the cross-sectional area for flow as the material passes axially down the device.

25

Controlled Deformation Dynamic Mixers (CDDMs)

Similar as in WO 96/20270, CDDMs are distinguished from CTMs by their description as mixers: comprising confronting surfaces, at least one of the surfaces, preferably
30 both surfaces, having a series of cavities formed therein in which the surfaces move relatively to each other and in which a liquid material is passed between the surfaces and flows along a pathway successively through the cavities in each surface and is subjected to extensional deformation and/or shear deformation and preferably both extensional and shear deformation. The cavities are arranged on the relevant surfaces
35 such that shear is applied by the relative movement of the surfaces in a generally

perpendicular direction to the flow of material there between. In addition to shear, significant extensional flow and efficient distributive and dispersive mixing may be secured by providing an apparatus having confronting surfaces and cavities therein in which the cavities are arranged such that the cross sectional area for flow of the liquid
5 successively increases and decreases by a factor of at least 5 through the apparatus.

Here we exemplify CDDMs by reference to Figure 2 which displays an axial section and four transverse radial sections through a CDDM configured as a 'concentric cylinder' device comprising an inner rotor journalled within an outer stator. Briefly, the
10 axial section shows the relative axial positions of rotor and stator cavities which are time invariant, whereas the transverse sections (A-A, B-B, C-C, D-D) demonstrate the axial variation in the available cross-sectional area for material flow axially :

- A-A through the stator cavities in positions in which those stator cavities are confronted by 'rotor rings', ie the circumferentially extending rings which separate
15 successive rings of rotor cavities;
- B-B between the stator cavities and the rotor cavities through the annulus formed in those positions in which the 'rotor rings' are confronted by the 'stator rings';
- C-C through the rotor cavities in positions in which those rotor cavities are confronted by 'stator rings', ie the circumferentially extending rings which separate
20 successive rings of stator cavities;
- D-D between the rotor cavities and the stator cavities in positions in which the former are confronted by the latter.

Clearly there is a significant variation in the cross-sectional area for flow as the material
25 passes axially through the annulus formed between the 'rotor rings' and the 'stator rings' (BB), and between confronting rotor cavities and stator cavities (D-D).

By comparison of Figure 1 and Figure 2, it will be understood that CDDMs are distinguished from CTMs by the relative position of the rotor and stator and consequent
30 incorporation of an extensional component of flow. Hence CDDMs combine the distributive mixing performance of CTMs with the dispersive mixing performance of multiple expansion-contraction static mixers.

Method for production of water-in-oil emulsion

The invention provides a method for production of an edible water-in-oil emulsion, wherein the emulsion comprises an aqueous phase, a structuring fat and a liquid oil, comprising the steps:

- 5 a) melting a structuring fat; and
b) introducing the molten structuring fat from step a), a liquid oil and an aqueous phase

into a distributive and dispersive mixing apparatus of the Controlled Deformation Dynamic Mixer type to create a water-in-oil emulsion,

- 10 and wherein the mixer is suitable for inducing extensional flow in a liquid composition, and wherein the mixer comprises closely spaced relatively moveable confronting surfaces at least one having a series of cavities therein in which the cavities on each surface are arranged such that, in use, the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus.

15

As has been indicated before, structuring fat refers to a fat that is solid at ambient temperature. The structuring fat serves to structure the emulsion by providing at least part of the structuring fat for the emulsion. Preferably the structuring fat is in the form of an edible fat powder, prior to being molten in step a).

20

The main components of structuring fat are triacylglycerols. The structuring fat may originate from one or more natural fats. Structuring fat as commercially available may comprise minor amounts of other components like for example monoglycerides that are naturally present and may likewise be present in the solid particles.

25

- The structuring fat may be a single fat or a mixture of different fats. The structuring fat may be of vegetable, animal (e.g. dairy fat) or marine origin. Preferably at least 50 wt% of the structuring fat (based on total amount of structuring fat) is of vegetable origin, more preferably at least 60 wt%, even more preferably at least 70 wt%, still more preferably at least 80 wt%, even still more preferably at least 90 wt% and even still more further preferably at least 95 wt%. Most preferably the structuring fat essentially consists of structuring fat of vegetable origin.
- 30

- Preferably the natural fat is selected from the group consisting of palm fat, allan
35 blackia, pentadesma, shea butter, coconut oil, soybean oil, rapeseed oil and dairy fat.

More preferably the natural fat is selected from the group consisting of palm oil, palm kernel oil, palm oil fraction, palm kernel fraction, coconut oil and dairy fat fraction. Even more preferably the natural fat is selected from the group consisting of palm oil, palm kernel oil, palm oil fraction, palm kernel fraction and coconut oil.

5

The various fat sources may be fully hardened by full hydrogenation, they may be fractionated, intra-esterified, and/or inter-esterified.

10 The structuring fat may comprise minor amounts of other components like for example monoglycerides that are naturally present in the fat. In addition to these naturally present components the edible fat powder may comprise additional components like for example emulsifier or liquid oil. It will be appreciated that care must be taken to prevent the properties of the structuring fat to be detrimentally affected. For example, the presence of liquid oil may affect the ability structure the emulsion, depending on the
15 natural fats and the liquid oil as well as the amounts thereof. It is within the reach of the skilled person to determine without undue burden how much of the additional components may be present using common general knowledge.

As the purpose of the structuring fat is to provide structure to the spread it may be
20 preferred not to include too many and/or too much of additional components that do not primarily add to the structuring ability of the structuring fat, like for example protein and carbohydrates. Preferably the structuring fat comprises not more than 20 wt% of protein and/or carbohydrates, more preferably not more than 15 wt%, even more preferably not more than 10 wt%, and still more preferably not more than 5 wt%. Most
25 preferably no protein and carbohydrates are present. Moreover, preferably the amount of water is not more than 20 wt%, preferably not more than 10 wt% and more preferably not more than 5 wt%. Most preferably no water is present in the structuring fat.

30 To optimize the structuring capacity and/or impression of the emulsion in the mouth upon consumption, structuring fats having a certain solid fat content are preferred. Therefore, the structuring fat as present in the solid particles preferably has a solid fat content N10 from 50 to 100%, N20 from 26 to 95% and N35 from 5 to 60%. The N-value expresses the solid fat content (SFC) at a certain temperature (in °C).

35

The structuring fat preferably has a solid fat content N10 selected from the list consisting of 45 to 100%, 55 to 90% and 65 to 85%;
N20 selected from the list consisting of 25 to 80%, 40 to 70% and 45 to 65%;
N35 selected from the list consisting of 0.5 to 60%, 0.5 to 20%, 0.5 to 14%, 15 to 50%
5 and 30 to 45%.

Preferred solid fat content profiles of the structuring fat are:

- N10 from 45 to 100%, N20 from 25 to 80% and N35 from 0.5 to 60%;
N10 from 55 to 90%, N20 from 40 to 70% and N35 from 0.5 to 20%;
10 N10 from 55 to 90%, N20 from 40 to 70% and N35 from 15 to 50%;
N10 from 65 to 85%, N20 from 45 to 65% and N35 from 0.5 to 14%; and
N10 from 65 to 85%, N20 from 45 to 65% and N35 from 30 to 45%.

In order to ensure that the structuring fat is liquid when introduced into the mixing
15 apparatus or before being mixed with the liquid oil phase, preferably in step a) the
melting temperature is at least 50°C, preferably at least 60°C, preferably at least 70°C,
and preferably maximally 90°C. At these temperatures all crystals of the triacylglycerols
have become liquid, there are no nuclei for solidification any more at these melting
temperatures.

20

Before introduction into the mixing apparatus, then preferably the structuring fat is
cooled again, otherwise the structuring fat may not solidify rapidly enough in the mixing
apparatus, and hence the fat-continuous emulsion may remain too soft after
production. Hence preferably the temperature of the structuring fat when introduced
25 into the mixer in step b) is maximally 70°C, preferably maximally 60°C. Preferably,
depending on the melting temperature of the structuring fat, the temperature at which
the liquid structuring fat is introduced into the mixing device is a few degrees above the
melting temperature, preferably 3 to 5°C above the melting temperature. The
temperature of the structuring fat is related to the temperature of the aqueous phase,
30 when the temperature of the aqueous phase is lower, the temperature of the structuring
fat can be higher. Alternatively the structuring fat may be cooled after step a) by mixing
with the liquid oil, before being introduced into the mixing device.

Preferably the temperature of the liquid oil when introduced into the mixer in step b) is
35 lower than 20°C, preferably lower than 15°C, preferably lower than 10°C. Preferably

the temperature of the liquid oil is between 12 and 18°C. The temperature is chosen such that the liquid oil does not start to crystallise before being introduced into the mixing apparatus. Additionally, preferably the temperature of the aqueous phase when introduced into the mixer in step b) is lower than 20°C, preferably lower than 15°C, preferably lower than 10°C.

The temperature of structuring fat, of liquid oil, and of aqueous phase when introduced into the mixing apparatus is important, as together they determine the crystallisation rate of the structuring fat, when the warm structuring fat is brought into contact with the aqueous phase and the liquid oil in the mixing apparatus. If the temperature of one the phases is not correct, then the viscosity of the mixture inside the mixing apparatus may be too low or too high, because the amount of solid fat crystals may be too low or too high. The temperature of the phases may also influence the stability of the emulsion that is produced in the method of the invention.

In order to create production flexibility, the wall of the mixing apparatus may be cooled by external means. This is only optional though, if the temperature of the phases is chosen correctly, and the weight ratio of the phases is chosen correctly, then external cooling means are not required. Increasing the cooling capacity may lead to the ability to produce low fat or very low fat water-in-oil emulsions.

Preferably the weight ratio between structuring fat and liquid oil ranges from 1:100 to 50:100, preferably from 5:100 to 25:100. This means that the total fat phase of the emulsion preferably comprises from 1% by weight to 50% by weight of structuring fat, and from 50% by weight to 99% by weight of liquid oil. More preferably the total fat phase of the emulsion preferably comprises from 5% by weight to 25% by weight of structuring fat, and consequently from 75% by weight to 95% by weight of liquid oil. With these ratios a fat-continuous emulsion can be produced which has the correct hardness and consistency. An emulsifier may be comprised in the liquid oil fraction, any numbers referring to the concentration of liquid oil includes a possible emulsifier.

In order to create a good dispersion of aqueous phase in the fat phase, the fat phase preferably contains an emulsifier having a HLB value of lower than 7. The HLB value is the hydrophilic-lipophilic balance, and is a measure for the degree of hydrophilicity or lipophilicity. An emulsifier with a HLB value lower than 10 generally is oil soluble, while

- an emulsifier with a HLB value higher than 10 generally is water-soluble. Hence preferably an emulsifier having a HLB value of 7 or lower, is mixed with the liquid oil prior to introducing the liquid oil into the mixer. Preferably, the concentration of the emulsifier is maximally 5% based on the weight of the mixture of liquid oil and
- 5 emulsifier, preferably maximally 1%, preferably maximally 0.1%, preferably maximally 0.01%. A relatively high emulsifier content may lead to the ability to produce low fat or very low fat water-in-oil emulsions, although a relatively high emulsifier content is not necessary to produce low-fat water-in-oil emulsions. Preferably the emulsifier comprises one or more of emulsifiers chosen from the group consisting of saturated
- 10 monoglycerides, unsaturated monoglycerides, and sugar-fatty acid esters (also known as the 'Spans', e.g. sorbitan monostearate). Preferably the HLB value of the emulsifier is lower than 5, preferably lower than 3, preferably 1. Another preferred emulsifier is lecithin from soyabean, or egg.
- 15 The amount of aqueous phase and fat phase in the emulsion that is produced in the method according to the invention may range widely. The fat phase includes the structuring fat and the liquid oil, and preferably an emulsifier as defined before. Preferably the concentration of the fat phase ranges from 5% to 95% based on the weight of the emulsion, preferably from 15% to 50%. The emulsion may comprise a fat
- 20 phase as a majority phase (for example, a margarine containing about 70 to 80% by weight of fat phase), preferably the emulsion comprises from 10% to 80%, preferably from 15% to 60% fat phase based on the weight of the emulsion. Most preferably though the produced emulsion is a low fat emulsion with a fat content ranging from 15% to 50% by weight, preferably ranging from 18% to 45% by weight of fat phase,
- 25 preferably ranging from 25% to 45% by weight of fat phase, preferably ranging from 30% to 45% by weight of fat phase. An advantage of the method of the present invention is that low fat spreads (maximally 50% by weight of fat phase) can be produced in a single step.
- 30 The aqueous phase is dispersed in small droplets in the continuous fat phase during the mixing in the mixing apparatus. Preferably the $d_{3,3}$ value of the dispersed aqueous phase droplets is less than 10 micrometer, preferably less than 8 micrometer, preferably less than 6 micrometer. Preferably the $d_{3,3}$ value of the dispersed aqueous phase droplets is less than 3 micrometer, or even less than 2 micrometer. The

distribution of the aqueous phase droplets preferably is narrow, meaning that the $\exp(\sigma)$ is maximally 2.5.

The three phases (aqueous phase, structuring fat phase, and liquid oil phase) may be added as three separate streams to the mixing apparatus. Alternatively, the molten structuring fat is brought into contact with liquid oil prior to introduction into the mixer. The mixing of the two phases prior to introduction into the mixing apparatus can be done in line in a static mixing operation, or can be actively mixed in batch using any mixing device commonly known.

The Controlled Deformation Dynamic Mixer

In step b) of the method of the invention the fat-continuous emulsion is prepared by introducing the molten structuring fat from step a), a liquid oil and an aqueous phase into a distributive and dispersive mixing apparatus of the Controlled Deformation Dynamic Mixer type to create a water-in-oil emulsion, and wherein the mixer is suitable for inducing extensional flow in a liquid composition, and wherein the mixer comprises closely spaced relatively moveable confronting surfaces at least one having a series of cavities therein in which the cavities on each surface are arranged such that, in use, the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus.

For the purposes of understanding the operation of the CDDM in general, the disclosure of WO 96/20270 is incorporated herein by reference. Regions of distributive mixing (where the flow path is wide) comprises CTM-like cavities moving across each other in a direction perpendicular to the bulk flow of liquid. Between these regions of distributive mixing are regions in which the flow path is narrower and the flow is more extensional. It is possible for a mixer used in the method according to the invention to be provided with one or more regions in which the juxtaposition is such that the arrangement is CTM-like and one or more regions in which the arrangement is CDDM-like.

In a preferred embodiment the CDDM apparatus can be described by the following. With reference to Figure 3 and Figure 4, preferably the Controlled Deformation Dynamic Mixer comprises two confronting surfaces (1, 2), spaced by a distance (7),

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wherein the first surface (1) contains at least three cavities (3), wherein at least one of the cavities has a depth (9) relative to the surface (1),
wherein the second surface (2) contains at least three cavities (4) wherein at least one of the cavities has a depth (10) relative to the surface (2),
5 wherein the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases and decreases at least 3 times, and
wherein the surface (1) has a length (5) between two cavities, and
wherein the surface (2) has a length (6) between two cavities, and
wherein the surfaces (1, 2) are positioned such that the corresponding lengths (5, 6)
10 overlap to create a slit having an offset distance (8) or do not overlap creating an offset distance (81),
wherein the cavities are arranged such that the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases in the cavities and decreases in the slits by a factor of at least 5 and
15 wherein the distance (7) between the two surfaces (1,2) is between 2 micrometer and 300 micrometer, and wherein
either the ratio between the offset distance (8) and the distance (7) between the two surfaces (1, 2) ranges from 0 to 250,
or wherein the ratio between the offset distance (81) and the distance (7) between the
20 two surfaces (1, 2) ranges from 0 to 60.

With reference to Figure 3 and Figure 4: there are several possible configurations for the mixing apparatus. In one preferred combination the confronting surfaces 1, 2 are cylindrical. In such a configuration the apparatus will generally comprise a cylindrical
25 drum and co-axial sleeve. The confronting surfaces 1, 2 will be defined by the outer surface of the drum and the inner surface of the sleeve. However, there are alternative configurations in which the confronting surfaces are circular or disk-shaped. Between these two extremes of configuration are those in which the confronting surfaces are conical or frusto-conical. Non-cylindrical embodiments allow for further variation in the
30 shear in different parts of the flow through the mixer.

The regions where the confronting surfaces 1, 2 are most closely spaced are those where the shear rate within the mixer tends to be the highest. The slit 7 between the surfaces between the confronting surfaces 1, 2 forms this region, combined with offset
35 distance 8 or offset distance 81. High shear contributes to power consumption and

heating. This is especially true where the confronting surfaces of the mixer are spaced by a gap of less than around 50 micrometer. Advantageously, confining the regions of high shear to relatively short regions means that the power consumption and the heating effect can be reduced, especially where in the CTM-like regions the confronting
5 surfaces are spaced apart relatively widely.

Hence the apparatus can be designed such that good mixing is obtained, while keeping the pressure drop over the apparatus as small as possible. The design can be modified by adjusting the dimensions of the various parts of the apparatus, as explained in the
10 following.

The distance 7 between the corresponding surfaces preferably is from 2 micrometers to 300 micrometers, which corresponds to the height of the slit. Preferably the distance 7 is between 3 micrometer and 200 micrometer, preferably between 5 micrometer and
15 150 micrometer, preferably between 5 micrometer and 100 micrometer, preferably between 5 micrometer and 80 micrometer, preferably between 5 and 60 micrometer, preferably between 5 micrometer and 40 micrometer. More preferably the distance 7 is between 8 micrometer and 40 micrometer, more preferably between 8 micrometer and 30 micrometer, more preferably between 10 micrometer and 30 micrometer, more
20 preferably between 10 micrometer and 25 micrometer, more preferably between 15 micrometer and 25 micrometer.

The actual height of the slit 7 depends on the dimensions of the apparatus and the required flow rate, and the skilled person will know how to design the apparatus such
25 that the shear rates within the apparatus remain relatively constant irrespective of the size of the apparatus.

The surfaces 1 and 2 that each contain at least three cavities 3, 4 create a volume between the surfaces for flow of the two fluids which are mixed. The cavities in the
30 surface effectively increase the surface area available for flow. Due to the presence of the cavities, the small area for flow between the surfaces 1 and 2 can be considered to be a slit having a height 7. The spacing 5 between two cavities in surface 1 and spacing 6 between two cavities in surface 2 and the relative position of these corresponding parts (the offset) determine the maximum length or offset distance 8 of

the slit (in the direction of bulk liquid flow). The maximum length of the slit is equal to the smallest of the spacings 5, 6.

Preferably, the two surfaces 1, 2 with cavities 3, 4, that together form the volume for the mixing of the three phases (aqueous phase, liquid oil, and structuring fat), are positioned such that the corresponding spacings 5, 6 of the surfaces (that create the length of the slit) create an offset distance 8 of the slit (in the direction of the bulk flow) which is maximally 250 times as large as the distance 7 between the surfaces. The two surfaces 1, 2 can be positioned such that offset distance 8 can be adjusted. Preferably the ratio between the offset distance 8 and the distance 7 between the two surfaces 1, 2 ranges from 0 to 100, preferably 0 to 10, preferably 0 to 5. Most preferably the ratio between the offset distance 8 and the distance 7 ranges from 0 to 1. As an example, when the ratio between offset distance 8 and distance 7 is 5, and the distance 7 between the two surfaces 1, 2 is 15 micrometer, then the offset distance 8 of the slit is 75 micrometer.

Preferably and alternatively the surfaces 1, 2 are positioned such that no overlap is created, however in that case an offset distance 81 is created. In that case there is no overlap between the corresponding parts of the surfaces 1, 2, and the slit is created with what could be called a 'negative overlap'. The two surfaces 1, 2 can be positioned such that offset distance 81 can be adjusted. The ratio between the offset distance 81 and the distance 7 between the two surfaces 1, 2 preferably ranges from 0 to 30. This 'negative overlap' accommodates the possibility of near zero distance 7 between the two corresponding surfaces 1 and 2. Preferably the offset distance 81 is such, that the ratio between the offset distance 81 and the distance 7 between the two surfaces 1, 2 ranges from 0 to 15, more preferred from 0 to 10, preferably from 0 to 5, preferably from 0 to 2 and more preferably from 0 to 1. Alternatively and preferably the offset distance 81 is maximally 600 micrometer, more preferably maximally 300 micrometer. As an example, when the ratio between length 81 and distance 7 is 2, and the distance 7 between the two surfaces 1, 2 is 15 micrometer, then length 81 (or what could be called negative overlap) is 30 micrometer.

A further benefit of this variation in the normal separation of the confronting surfaces in the direction of bulk flow, is that by having relatively small regions of high shear,

especially with a low residence time is that the pressure drop along the mixer can be reduced without a compromise in mixing performance.

The little overlap (meaning that offset distance 8 approaches zero, or that the mixing
5 apparatus has a 'negative overlap' or offset distance 81) between the corresponding
parts of the surfaces 1, 2 leads to a relatively small pressure that is required in order to
create a fine dispersion, as compared to apparatuses which have a longer overlap and
consequently also need a higher pressure. Usually a longer distance of a slit (or longer
capillary) leads to smaller droplets of the dispersed phase. Now we found that with a
10 short capillary or even without capillary the droplets of the dispersed phase remains
small, while the pressure required is relative low, as compared to a longer overlap. For
example high pressure homogenisers may operate at pressures up to 1,600 bar or
even higher. Hence preferably in the method of the invention, the mixing apparatus is
operated at a pressure less than 200 bar, preferably less than 80 bar, preferably less
15 than 60 bar, preferably less than 40 bar, most preferred less than 30 bar. With these
relatively low pressures a good mixing process is obtained.

An additional advantage of the relatively low pressure is that the energy consumption
for applying the pressure is much lower than in conventional devices which use
20 pressures of 1,000 bar or higher to achieve dispersed phases having a size less than
1 micrometer. Moreover less stringent material specifications for design of an
apparatus to withstand high pressures is required, such that raw materials can be
saved.

25 With reference to Figure 3 and Figure 4, the fluids preferably flow from left to right
through the apparatus. The slits create an acceleration of the flow, while at the exit of
the slit the fluids decelerate due to the increase of the surface area for flow and the
expansion which occurs. The acceleration and deceleration leads to the break up of the
large droplets of the dispersed phase, to create finely dispersed droplets in a
30 continuous phase. Droplets that are already small, remain relatively untouched. The
flow in the cavities is such that the droplets of the dispersed phase eventually become
evenly distributed in the continuous phase.

The cross-sectional area for flow of the liquid available during passage through the
35 apparatus successively increases and decreases at least 5 times, and these passages

lead to effective mixing of the two fluids. This means that the cross-sectional area for flow of liquid in the cavities is at least 5 times larger than the cross-sectional area for flow of liquid in the slits. This relates to the ratio between distance 11 and distance 7. Preferably the cross-sectional area for flow is designed such that the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases and decreases by a factor of at least 7, preferably at least 10, preferably at least 25, preferably at least 50, up to preferred values of 100 to 400. The cross-sectional surface area for flow of the fluids is determined by the depth 9 of the cavities 3 in the first surface 1 and by the depth 10 of the cavities 4 in the second surface 2.

10 The total cross-sectional area is determined by the distance 11 between the bottoms of two corresponding cavities in the opposite surfaces.

The surfaces 1, 2 each contain at least three cavities 3, 4. In that case the flow expands at least 3 times during passage, and the flow passes through at least 3 slits during the passage. Preferably the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases and decreases between 4 and 8 times. This means that the flow during passage experiences the presence of between 4 and 8 slits and cavities.

20 The shape of the cavities 3 may take any suitable form, for example the cross-section may not be rectangular, but may take the shape of for example a trapezoid, or a parallelogram, or a rectangle where the corners are rounded. Seen from above, the cavities may be rectangular, square, or circular, or any other suitable shape. Any arrangement of the cavities and the number of cavities and size of the cavities may be within the scope of the present invention.

The mixing apparatus preferably is operated dynamically, meaning that one of the surfaces rotates relative to the other. In that case preferably one of the surfaces is able to rotate relative to the other surface at a frequency between 1,000 and 25,000 rotations per minute, preferably between 3,000 and 12,000 rotations per minute. In general rotation may lead to improved mixing process and creation of smaller dispersed phase droplets. In case the emulsion contains less fat (amount of fat phase is lower), then the required rotation speed may be higher in order to create a finely dispersed aqueous phase in the continuous fat phase.

Additional features of the known CTM and CDDM may be incorporated in the mixer described herein. For example, one or both of the confronting surfaces may be provided with means to heat or cool it. Where cavities are provided in the confronting surfaces these may have a different geometry in different parts of the mixer to as to
5 further vary the shear conditions.

In a preferred example, the dimensions of such a CDDM apparatus used in the invention are such that the distance between the two surfaces 7 is between 10 and 20 micrometer; and/or wherein the length of the slit 8 is maximally 2 millimeter, for
10 example 80 micrometer, or 20 micrometer, or even 0 micrometer. The length of the slit 8 plus the length of the cavity 17, 18 combined is maximally 10 millimeter; and/or wherein the depth of the cavities 9, 10 is maximally 2 millimeter. In that case preferably the internal diameter of the outer surface is between 20 and 30 millimeter, preferably about 25 millimeter. The total length of the apparatus in that case is between 7 and 13
15 centimeter, preferably about 10 centimeter. The length means that this is the zone where the fluids are mixed. The rotational speed of such a preferred apparatus is preferably 0 (static), or more preferred alternatively between 5,000 and 25,000 rotations per minute.

20 The shape of the area for liquid flow may take different forms, and naturally depends on the shape of the confronting surfaces. If the surfaces are flat, then the cross-sectional area for flow may be rectangular. The two confronting surfaces may also be in a circular shape, for example a cylindrical rotor which is positioned in the centre of a cylindrical pipe, wherein the outside of the cylindrical rotor forms a surface, and the
25 inner surface of the cylindrical pipe forms the other surface. The circular annulus between the two confronting surface is available for liquid flow.

EXAMPLE

30 The following non-limiting example illustrates the present invention.

CDDM Apparatus

Experiments were carried out in a CDDM apparatus as schematically depicted in Figure 2 and Figure 3, wherein the apparatus comprises a stainless steel cylindrical
35 drum and co-axial sleeve (the confronting surfaces 1, 2 are cylindrical). The confronting

surfaces 1, 2 are defined by the outer surface of the drum and the inner surface of the sleeve, respectively. The CDDM can be described by the following parameters:

- slit height (distance 7) is 35-40 micrometer;
- offset distance 8 is 20 micrometer;
- 5 - total length of the apparatus is 10 centimeter (length means the zone where the fluids are mixed);
- across the length of the CDDM in axial direction (in flow direction) the flow experiences six slits with height 7, the flow is contracted 6 times;
- depth 9, 10 of cavities 3, 4 is maximally 2 millimeter;
- 10 - internal diameter of the stator is 25 millimeter;
- rotational speed of the apparatus is up to 25,000 rotations per minute;

Droplet Size and Droplet Size Distribution

Droplet size and droplet size distribution are determined using standardised NMR
 15 equipment. A Bruker magnet with a field of 0.47 Tesla (20 MHz proton frequency) with an air gap of 25 mm is used (NMR Spectrometer Bruker Minispec MQ20 Grad, ex Bruker Optik GmbH, Germany) The NMR signal (echo height) of the protons of the water in a water-in-oil emulsion are measured using a sequence of 4 radio frequency pulses in the presence (echo height E) and absence (echo height E*) of two magnetic
 20 field gradient pulses as a function of the gradient power. The oil protons are suppressed in the first part of the sequence by a relaxation filter. The ratio ($R=E/E^*$) reflects the extent of restriction of the translational mobility of the water molecules in the water droplets and thereby is a measure of the water droplet size. By a mathematical procedure (which uses the log-normal droplet size distribution) the
 25 parameters of the water droplet size distribution $d_{3,3}$ (volume weighted geometric mean diameter) and sigma or $\exp(\sigma)$ (measures for distribution width) are calculated.

The droplet size of the spread is measured, according to the above described
 30 procedure, of a spread stabilized at 5°C right after production for one week. This gives the $d_{3,3}$ after stabilization at 5°C.

Hardness and spreadability

Stevens values give an indication about the hardness (also called firmness) of a
 35 product. The Stevens value is determined according to the following protocol.

Freshly prepared products are stabilized at 5 degrees Celsius. The hardness of the product is measured with a Stevens penetrometer (Brookfield LFRA Texture Analyser (LFRA 1500), ex Brookfield Engineering Labs, UK) equipped with a stainless steel
5 probe with a diameter of 6.35 mm and operated in "normal" mode. The probe is pushed into the product at a speed of 2 mm/s, a trigger force of 5 gram from a distance of 10 mm. The force required is read from the digital display and is expressed in grams.

Spreadability is determined according to the following protocol. A flexible palette knife is
10 used to spread a small amount of the spread onto fat free paper. The spreading screen is evaluated according to standardized scaling. A score of 1 represents a homogeneous and smooth product without any defects, a 2 refers to the same product but then with small remarks as slightly inhomogeneous or some vacuoles, a 3 refers to the level where defects become almost unacceptable, like loose moisture or
15 coarseness during spreading. A score of 4 or 5 refers to unacceptable products, where the 4 refers to a product still having some spreading properties, but an unacceptable level of defects.

Solid Fat Content (SFC) measurements

20 The solid fat content (SFC) in this description and claims is expressed as N-value, as defined in Fette, Seifen Anstrichmittel 80 180-186 (1978). The stabilization profile applied is heating to a temperature of 80 degrees Celsius, keeping the oil for at least 10 minutes at 60 degrees Celsius or higher, keeping the oil for 1 hour at 0 degrees Celsius and then 30 minutes at the measuring temperature.

25

Free Water in Spreads

After spreading a sample of a fat spread, the stability of the emulsion after spreading is determined by using indicator paper (Wator, ref 906 10, ex Machery-Nagel, DE) which develops dark spots where free water is adsorbed. A stable product does not release
30 any water and the paper does not change. Very unstable products release free water easily and this is indicated by dark spots on the paper.

A six point scale is used to quantify the quality of fat spread (DIN 10 311):

- 0 (zero) is a very stable and good product;

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- 1 (one) is showing some loose moisture (one or two spots, or the paper changes a little in color as a total);
- 2 (two) as one but more pronounced;
- 3 (three) as one but to an almost unacceptable level;
- 5 - 4 (four) indicator paper is almost fully changing into a darker color;
- 5 (five) the paper changes completely and very fast into the maximum level of color intensity.

Spreads with a score of 4 or 5 are rejected for their stability. Spreads with a score of 0 or 1 show an acceptable quality with respect to free water.

10

Materials

- Sunflower oil (100%), supplier Cargill, Inc. (Minneapolis, MN, USA);
- Emulsifier Dimodan RT (monoglyceride, ex Danisco A/S (Copenhagen, Denmark);
- 15 ▪ Demineralised water was obtained from a Millipore filter system;
- Structuring fat inES48: inES48 is an interesterified mixture of 65% dry fractionated palm oil stearin with an iodine value of 14 and 35% palm kernel oil. inES48 contains about 86.2% saturated fatty acids, about 11.5% mono-unsaturated fatty acids, and about 2.3% poly-unsaturated fatty acids, and is free
- 20 from trans fatty acids. Obtained from Unimills (Zwijndrecht, Netherlands);
- Beta-carotene (30% in sunflower oil), ex BASF (Ludwigshafen, Germany);
- Kitchen salt (NaCl);
- Citric acid (20% solution in water); ex Jungbunzlauer AG (Basel, Switzerland);
- Flavour mix ex Givaudan SA (Vernier, Switzerland).

25

Production of Spreads

Spreads (water-in-oil emulsions) were produced in the CDDM apparatus according to the following general procedure. The emulsions had one of the following general

30 compositions:

Table 1 Overall composition of water-in-oil emulsions produced.

Ingredients	#1	#2
<i>Total Fat phase [wt%]</i>	45	35
Sunflower oil [wt%]	37.3	29.7
Emulsifier Dimodan RT [wt%]	0.3	0.3
Structuring fat inES48 [wt%]	7.2	4.8
Beta-carotene [wt%]	0.15	0.15
Flavour [wt%]	0.04	0.04
<i>Total Aqueous Phase [wt%]</i>	55	65
Water [wt%]	54.8	64.8
NaCl [wt%]	0.2	0.2
citric acid	to pH 5.2	to pH 5.2

The following steps are applied in the production of the spreads:

- 5 1. Structuring fat inES48 is made liquid by bringing it to a temperature of 90°C.
2. The temperature of the structuring fat is reduced to 55°C.
3. Emulsifier Dimodan RT is mixed with sunflower oil, at a concentration varying from about 0.3% to 1.2% by weight, based on the weight of sunflower oil and emulsifier. This mixture is kept at a temperature of 8°C.
- 10 4. The structuring fat (at a temperature of 55°C) is mixed with the sunflower oil-emulsifier mixture (at a temperature of 8°C) at a weight ratio of 16 to 84, resulting in a temperature of the fat phase of 15°C. This is the fat phase of the emulsion.
5. The aqueous phase is prepared by mixing water and salt, and adjusting the pH to 5.2 using citric acid, and kept at a temperature of 10°C.
- 15 6. The fat phase and aqueous phase both are fed into the CDDM apparatus using two separate inlets, opposite each other with the stator as the reference (both at the same distance from the outlet of the CDDM apparatus). The rotor rotated at rate of 5,000 rpm. The pressure was 30 bar, this pressure is the line pressure between the intensifier pump supplying the raw materials and the mixer itself.
- 20 7. The resulting product is a water-in-oil emulsion that can be used as a spread.

The following spreads were produced, and they had the following production parameters and product properties (shortly after production and after 18 weeks storage at 5°C):

- 5 **Table 2** Production parameters and emulsion properties of emulsions that have been produced (short after production and after 18 weeks storage).

Emulsion composition	Flow rate of emulsion [mL/s]	T outlet emulsion [°C]	d_{3,3} [micrometer]	exp (sigma) [-]	Stevens hardness [g]	Spreadability (1 to 5)	Free water (water paper)
#1	20	22	7.35	1.7	33	1	0
after 18 wks storage			9	1.6	25	1	0
#1	20	21	6.8	1.6	34	1	0
after 18 wks storage			7	1.5	36	1	0
#2	20	19	7.7	1.77	27	1	0
after 18 wks storage			9	1.8	27	1	0

- 10 All emulsions had good properties with regard to spreadability and hardness and water droplet size. All samples showed good structural stability during the storage of 18 weeks, average droplet size (d_{3,3}) remained below 9 micrometer and exp(sigma) below 1.8 over a period of 18 weeks. Also spreadability and free water values were good after 18 weeks.

CLAIMS

1. A method for production of an edible water-in-oil emulsion, wherein the emulsion comprises an aqueous phase, a structuring fat and a liquid oil, comprising the steps:
 - (a) melting a structuring fat; and
 - (b) introducing the molten structuring fat from step a), a liquid oil and an aqueous phase into a distributive and dispersive mixing apparatus of the Controlled Deformation Dynamic Mixer type to create a water-in-oil emulsion, and wherein the mixer is suitable for inducing extensional flow in a liquid composition, and wherein the mixer comprises closely spaced relatively moveable confronting surfaces at least one having a series of cavities therein in which the cavities on each surface are arranged such that, in use, the cross-sectional area for flow of the liquid successively increases and decreases by a factor of at least 5 through the apparatus.
2. A method according to claim 1, wherein in step a) the melting temperature is at least 50°C, preferably at least 60°C, preferably at least 70°C, and preferably maximally 90°C.
3. A method according to claim 1 or 2, wherein the temperature of the structuring fat when introduced into the mixer in step b) is maximally 70°C, preferably maximally 60°C.
4. A method according to any of claims 1 to 3, wherein the temperature of the liquid oil when introduced into the mixer in step b) is lower than 20°C, preferably lower than 15°C, preferably lower than 10°C.
5. A method according to any of claims 1 to 4, wherein the temperature of the aqueous phase when introduced into the mixer in step b) is lower than 20°C, preferably lower than 15°C, preferably lower than 10°C.

6. A method according to any of claims 1 to 5, wherein the weight ratio between structuring fat and liquid oil ranges from 1:100 to 50:100, preferably from 5:100 to 25:100.
7. A method according to any of claims 1 to 6, wherein an emulsifier having a HLB value of 7 or lower, is mixed with the liquid oil prior to introducing the liquid oil into the mixer.
8. A method according to claim 7, wherein the concentration of the emulsifier is maximally 5% based on the weight of the mixture of liquid oil and emulsifier, preferably maximally 1%, preferably maximally 0.1%, preferably maximally 0.01%.
9. A method according to any of claims 1 to 8, wherein the concentration of the fat phase ranges from 5% to 95% based on the weight of the emulsion, preferably from 15% to 50%.
10. A method according to any of claims 1 to 9, wherein the $d_{3,3}$ value of the dispersed aqueous phase droplets is less than 10 micrometer, preferably less than 8 micrometer, preferably less than 6 micrometer.
11. A method according to any of claims 1 to 10, wherein the molten structuring fat is brought into contact with liquid oil prior to introduction into the mixer.
12. A method according to any of claims 1 to 11, wherein the Controlled Deformation Dynamic Mixer comprises two confronting surfaces (1, 2), spaced by a distance (7), wherein the first surface (1) contains at least three cavities (3), wherein at least one of the cavities has a depth (9) relative to the surface (1), wherein the second surface (2) contains at least three cavities (4) wherein at least one of the cavities has a depth (10) relative to the surface (2), wherein the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases and decreases at least 3 times, and wherein the surface (1) has a length (5) between two cavities, and wherein the surface (2) has a length (6) between two cavities, and wherein the surfaces (1, 2) are positioned such that the corresponding lengths (5, 6) overlap to create a slit having an offset distance (8) or do not overlap creating an offset

distance (81),

wherein the cavities are arranged such that the cross-sectional area for flow of the liquid available during passage through the apparatus successively increases in the cavities and decreases in the slits by a factor of at least 5 and

wherein the distance (7) between the two surfaces (1,2) is between 2 micrometer and 300 micrometer, and wherein

either the ratio between the offset distance (8) and the distance (7) between the two surfaces (1, 2) ranges from 0 to 250,

or wherein the ratio between the offset distance (81) and the distance (7) between the two surfaces (1, 2) ranges from 0 to 60.

13. A method according to any of claims 1 to 12, wherein the mixing apparatus is operated at a pressure less than 200 bar.

14. A method according to any of claims 1 to 13, wherein one of the surfaces rotates relative to the other surface at a frequency between 1,000 and 25,000 rotations per minute, preferably between 3,000 and 12,000 rotations per minute.

Figure 1

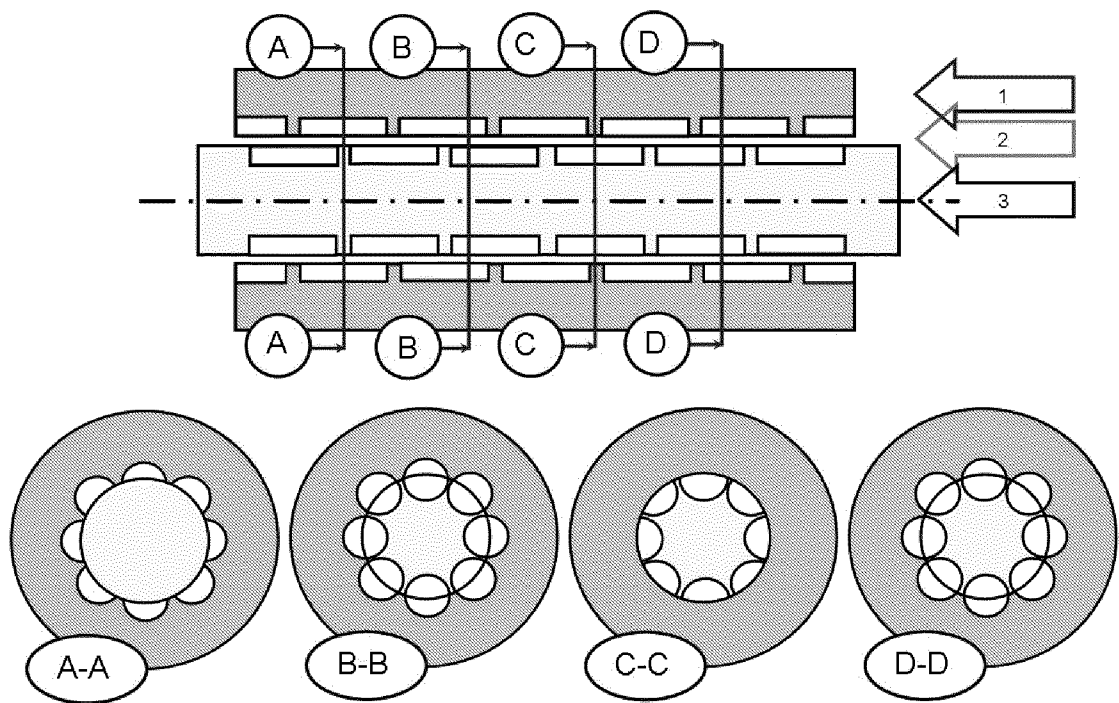


Figure 2

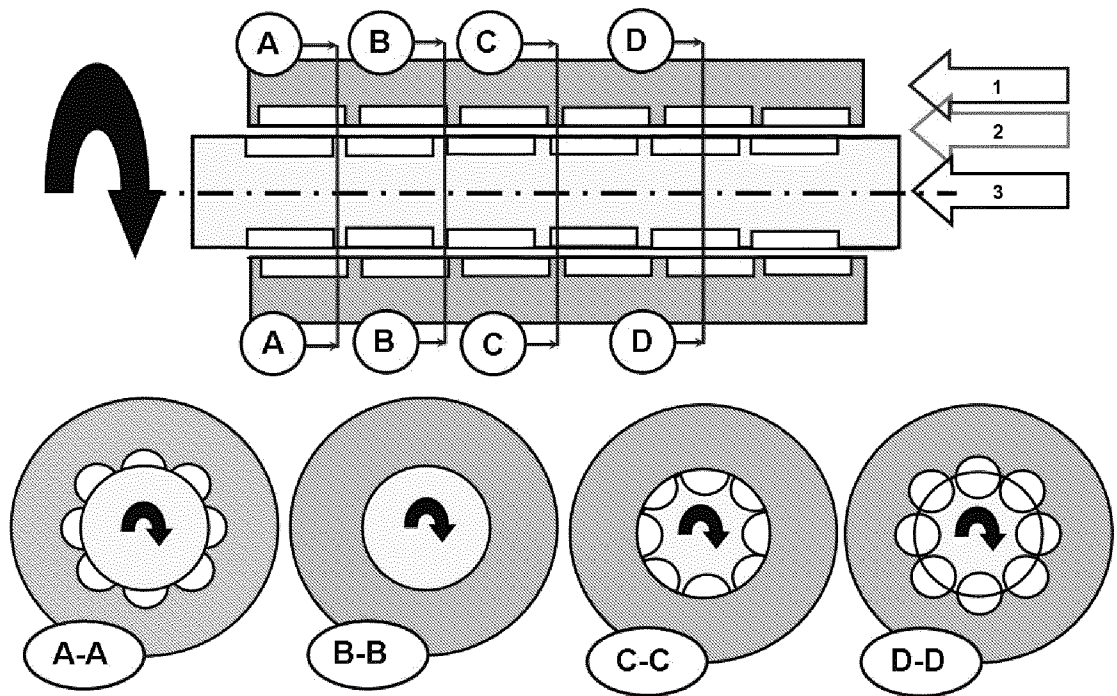


Figure 3

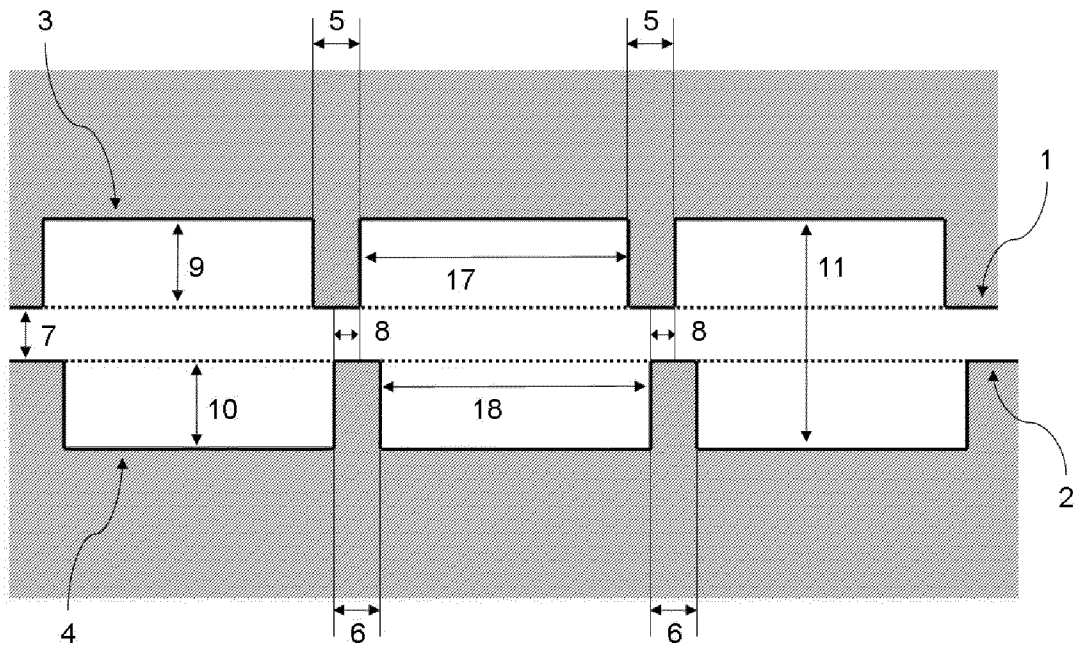
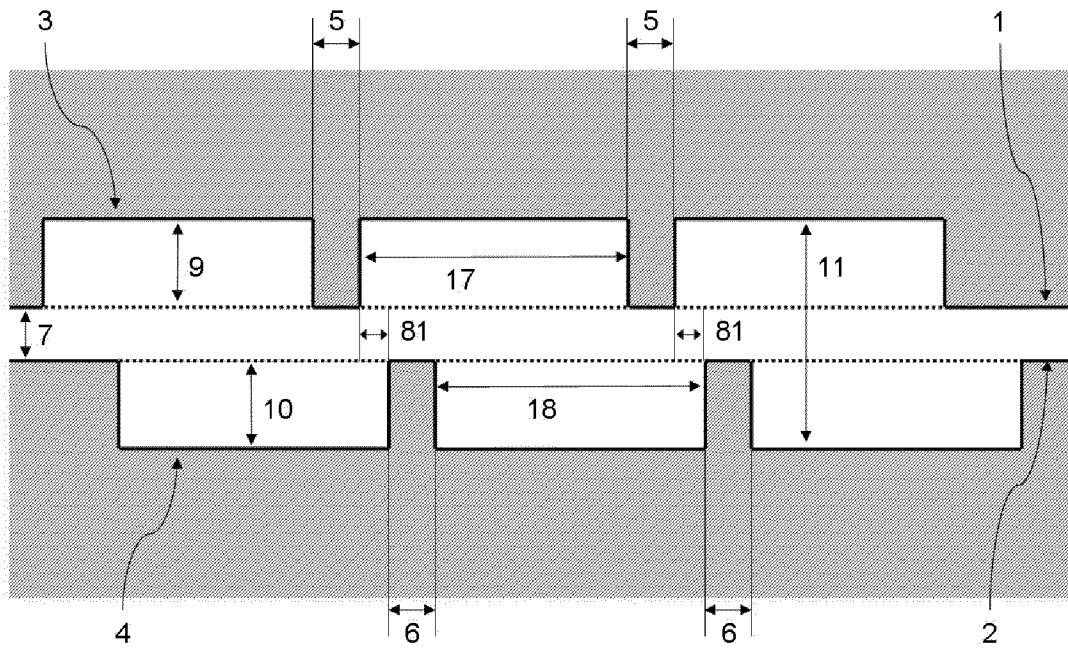


Figure 4



INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2012/069036

A. CLASSIFICATION OF SUBJECT MATTER INV. A23D7/04 B01F7/00 ADD.		
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) A23D B01F		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, BIOSIS, FSTA, PAJ, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2010/089322 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]; BROWN CH) 12 August 2010 (2010-08-12) cited in the application page 5, lines 9-24 page 7, line 24 - page 8, line 10 page 8, lines 6-10 page 11, lines 10-16	1-14
A	US 6 468 578 B1 (BODOR JANOS [NL] ET AL) 22 October 2002 (2002-10-22) cited in the application column 1, lines 5-10 column 2, lines 31-54 column 3, lines 14-27 column 5, lines 49-59 page 1; example 1 ----- -/-	1-14
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 10 January 2013		Date of mailing of the international search report 18/01/2013
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Couzy, François

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2012/069036

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	WO 2010/091983 A1 (UNILEVER PLC [GB]; UNILEVER NV [NL]; UNILEVER HINDUSTAN [IN]; BROWN CH) 19 August 2010 (2010-08-19) cited in the application page 5, line 6 - page 6, line 10 page 6, lines 19-21 page 8, lines 3-9 claims 1,5,8 -----	1-14
A,P	WO 2012/065824 A1 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; ALMEIDA) 24 May 2012 (2012-05-24) page 1, lines 3-5 page 5, line 10 - page 6, line 29 page 10, lines 12-14 page 15, line 33 - page 17, line 17 -----	1-14
A,P	WO 2012/089474 A1 (UNILEVER NV [NL]; UNILEVER PLC [GB]; UNILEVER HINDUSTAN [IN]; BONGERS) 5 July 2012 (2012-07-05) page 3, line 33 - page 4, line 35 -----	1-14

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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WO 2012089474 A1	05-07-2012	NONE	
