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(54) Title: EDIBLE WATER-IN-OIL EMULSION AND PROCESS FOR THE MANUFACTURE THEREOF

(57) Abstract: The present invention relates to edible water-in-oil emulsion comprising 15-65wt.% of a continuous fat phase and 35-85 wt.% of a dispersed aqueous phase, said emulsion containing by weight of the dispersed aqueous phase: 0.2-7 wt.% of gelatinized starch; and 0.1-4 wt.% of pulse seed globulin; wherein the mean Sauter diameter ($D_{3,2}$) of the particles contained in the aqueous phase is less than 60 μm. Examples of water-in-oil emulsions according to this invention include spreads, kitchen margarines and bakery margarines. The invention further provides a process of preparing such a water-in-oil emulsion.

EDIBLE WATER-IN-OIL EMULSION AND PROCESS FOR THE MANUFACTURE THEREOF

TECHNICAL FIELD OF THE INVENTION

The present invention relates to an edible water-in-oil emulsion such as a spread, a kitchen margarine or a bakery margarine. The edible water-in-oil emulsion according to the invention contains gelatinized starch and pulse seed globulin. The invention also provides a process for the manufacture of the aforementioned water-in-oil emulsion.

BACKGROUND OF THE INVENTION

Food products in the form of edible water-in-oil emulsions are well known in the art. These food products comprise a continuous fat phase and a dispersed aqueous phase. The continuous fat phase typically constitutes 15-85 wt.% of these food products, and the aqueous phase the remainder. Usually, the fat phase comprises a liquid oil and a structuring fat (also known as hard stock). The structuring fat is solid at room temperature and serves to structure the fat phase and to impart plasticity to the product. The liquid oil typically comprises unmodified vegetable oils such as soybean oil, sunflower oil, low erucic rapeseed oil (Canola), corn oil and blends of vegetable oils. Also marine type oils such as fish oil and algae oil may be used.

Butter and margarine are examples of water-in-oil emulsions that typically contain around 80 wt.% of a continuous fat phase. Due to the high oil-to-water ratio butter and margarine products usually exhibit very high emulsion stability. However, when the fat content is reduced from 80 wt.% to, for instance, 60 wt.%, it becomes increasingly more difficult to produce a stable water-in-oil emulsion. Nonetheless, stable water-in-oil emulsions having a fat content as low as 15 wt.% have been produced by employing ingredients that help to stabilize these emulsions, notably water-in-oil emulsifiers, water phase structurants (e.g. gelling agents) and fat phase structurants (e.g. hard stock).

In the food industry there is an ongoing effort to reduce the number of additives that are being used in commercial food products. In addition, food manufacturers are looking for ways to replace ‘artificial’ ingredients by natural ingredients from renewable, sustainable sources. In

the field of edible water-in-oil emulsions emulsifiers and water structurants are examples of ingredients for which sustainable alternatives are required.

WO 99/51106 describes lupin protein compositions and the use of these protein compositions in oil-and-water emulsions. Example 3 describes the preparation of a non-dairy spread from a mixture of fat blend (7 kg), flavour (0.1 kg), lupin isolate having a lupine protein content of ca. 90% (0.8 kg) and water (2.1 kg).

Okaka J C et al. (*Physico-chemical and functional properties of cowpea powders processed to reduce beany flavor*, J. of Food Science vol. 44. no. 4. 1979. pages 1235-1240) describes a method for preparing a cowpea powder with decreased beany flavor. This is done by soaking cowpeas in acidified water, dehulling, blanching in 100 °C steam, grinding and drum-drying. In order to test the emulsifying activity and stability of the cowpea powder, samples weighing 2.0g were dispersed in 50 ml cold distilled water (4°C) with pH adjusted to 6.8 and were blended with 50 ml red-dyed peanut oil for 2 min at 20,500 rpn) using a Waring semi-micro Blendor.

Aluko et al. (*Emulsifying and Foaming Properties of Commercial Yellow Pea (Pisum sativum L.) Seed Flours*, J. Agric. Food Chern. 2009, 57, 9793-9800) describes the emulsifying and foaming properties of flours and protein isolates derived from yellow pea seed. Oil-in-water emulsions were prepared in 5 mL of 0.1 M phosphate buffer pH 3.0, 5.0, or 7.0 followed by addition of 0.5 mL of pure canola oil (10%, v/v). The oil/water mixture was homogenized at 20,000 rpm for I min, stopped for 5 s and then homogenized again for another I min using the 20 mm nonfoaming shaft on a Polytron PT 3100 homogenizer (Kinematica AG, Lucerne, Switzerland).

WO 01/56270 describes food products comprising a starch and protein containing pea or lentil flour wherein the flour starch has been at least partially gelatinized and the flour protein has been at least partially denatured and coagulated. Example 3 describes the preparation of a pea flour-snack spread by mixing 200 ml water with 25 g yellow pea flour, followed by heating to 60°C with continuous mixing. The heated mixture was then poured into a blender containing 40 ml canola oil and whizzed on high for 3 minutes. The mixture was then heated to 85°C, calcium sulfate (3.0 g) was added and mixed uniformly throughout the liquid. It was then immediately poured into a cup, allowed to stand at room temperature for 15 minutes and then placed in a refrigerator overnight. The creamy gel was sliced and spread on water crackers as a gel. It is observed in the example that after addition of the calcium salt the

emulsion droplets aggregate and segregate into a discontinuous phase without disruption of the stable emulsion.

WO 2010/127415 describes a composition useful as an emulsifier for stabilising a water-in-oil emulsion to form a food product, the composition including: lupin protein in an amount of 75 to 98 % by dry weight of the composition, wherein 60 to 70 % of the lupin proteins have a molecular weight of no more than about 30 kDa. Example 4 describes a non-dairy creamer comprising a lupine derived water-in-oil emulsifier composition, hydrogenated vegetable fat, monglycerides, maltodextrin and water.

WO 2011/029725 describes an edible water-in-oil emulsion spread comprising

- 20 to 80 wt% of a fat phase,
- a dispersed water phase,
- poly unsaturated fatty acid (PUFA),
- trace mineral, and
- an evenly dispersed edible seed mixture,

wherein the amount of seed mixture is 0.1 to 20 wt%, the seed mixture comprises two or more different edible seed and/or seed fragments, 60 to 100 % (w/w) of the seed mixture is bigger than 0.5 mm and 50 to 100 % (w/w) is smaller than 4 mm; and wherein at least part of the trace mineral is present in the seed mixture.

Kaur, M. and Singh, N. (*Studies on functional, thermal and pasting properties of flours from different chickpea (Cicer arietinum L.) cultivars*. Food Chem. 91 (2005), pp. 403–411) describe the variability in physicochemical, functional, thermal, and pasting properties of flours from different chickpea cultivars. Emulsion activity and stability of emulsions containing 3.5 g flour, 50 ml water and 50 ml groundnut oil were studied.

SUMMARY OF THE INVENTION

The inventors have found that stable water-in-oil emulsions having a reduced fat content can be produced by employing a combination of gelatinized starch and pulse seed globulin in the aqueous phase of these emulsions and by ensuring that the bulk of particular matter that is contained in the aqueous phase – including the gelatinized starch – has a diameter of less than 80 µm. More particularly, the inventors have developed an edible water-in-oil emulsion

comprising 15-65 wt.% of a continuous fat phase and 35-85 wt.% of a dispersed aqueous phase, said emulsion containing by weight of the dispersed aqueous phase:

- 0.2-7 wt.% of gelatinized starch; and
- 0.1-4 wt.% of pulse seed globulin;

wherein the mean Sauter diameter ($D_{3,2}$) of the particles contained in the aqueous phase is less than 60μm.

Although the inventors do not wish to be bound by theory, it is believed that the gelatinized starch provides water structuring properties that help to stabilize the emulsion. In addition, the pulse seed globulin contributes to emulsion stability by acting as a water-in-oil emulsifier. The inventors have further found that in order to obtain an emulsion of high stability using gelatinized starch and pulse seed globulin it must be ensured that the aqueous phase does not contain significant quantities of particulate material of large dimensions, e.g. of a diameter of more than 80 μm. Thus, the size of the gelatinized starch and other particulate components (e.g. fibres) should be reduced, e.g. by subjecting the aqueous phase to microfluidization.

The invention further provides a process of preparing the aforementioned water-in-oil emulsion, said process comprising:

- preparing an aqueous composition by combining finely ground pulse seed, water and optionally other ingredients;
- combining the aqueous composition with a fat composition; and
- emulsifying the combination of the aqueous composition and the fat composition.

DETAILED DESCRIPTON OF THE INVENTION

A first aspect of the present invention relates to edible water-in-oil emulsion comprising 15-65 wt.% of a continuous fat phase and 35-85 wt.% of a dispersed aqueous phase, said emulsion containing by weight of the dispersed aqueous phase:

- 0.2-7 wt.% of gelatinized starch; and
- 0.1-4 wt.% of pulse seed globulin;

wherein the mean Sauter diameter ($D_{3,2}$) of the particles contained in the aqueous phase is less than 60 μm.

The term “starch” as used herein, unless indicated otherwise, refers to native, non-modified, starch. Starch consists of two types of molecules: the linear and helical amylose and the branched amylopectin.

The term “gelatinized starch” as used herein refers to starch that has undergone gelatinization. Starch gelatinization is a process that breaks down the intermolecular bonds of starch molecules in the presence of water and heat, allowing the hydrogen bonding sites to engage more water. This irreversibly dissolves the starch granule. Penetration of water increases randomness in the general starch granule structure and decreases the number and size of crystalline regions. Under the microscope in polarized light starch loses its birefringence and its extinction cross during gelatinization. Some types of unmodified native starches start swelling at 55 °C, other types at 85 °C. The gelatinization temperature depends on the degree of cross-linking of the amylopectin.

The term “protein” as used herein refers to a linear polypeptide comprising at least 10 amino acid residues. Preferably, said protein contains more than 20 amino acid residues. Typically, the protein contains not more than 35,000 amino acid residues.

The term “globulin” as used herein refers to a protein that is insoluble in water, but soluble in saline solutions (e.g. 0.6 g/l NaCl at pH=8).

The term “albumin” as used herein refers to a protein that is soluble in water and in moderately concentrated salt solutions and that experiences heat coagulation. Reference is made to the Osborne protein classification system (T.B. Osborne, *The Vegetable Proteins, Monographs in Biochemistry* (London; Longmans, Green and Co., 1924.

The term “pulse” as used herein refers to an annual leguminous crop yielding from one to twelve seeds of variable size, shape, and colour within a pod and is reserved for crops harvested solely for the dry seed. This excludes fresh beans and fresh peas, which are considered vegetable crops. Also excluded are crops that are mainly grown for oil extraction (oilseeds like soybeans and peanuts), and crops which are used exclusively for sowing (clovers, alfalfa). Just like words such as "bean" and "lentil", the word "pulse" may also refer to just the seed, rather than the entire plant.

The term “oil” as used herein refers to lipids selected from the group of triglycerides, diglycerides, monoglycerides, phospholipids and free fatty acids. The term “oil” encompasses

lipids that are liquid at ambient temperature as well as lipids that are partially or wholly solid at ambient temperature.

The term “dietary fiber” as used herein refers to indigestible non-starch polysaccharides such as arabinoxylans, cellulose, lignin, pectins and beta-glucans.

The term “sugars” as used herein refers to mono- and disaccharides.

The term “biopolymer material” as used herein refers to a polymeric material of natural origin that comprises at least 10 covalently bound monomeric units.

The (finely) “ground pulse seed” of the present invention is suitably produced by milling or grinding dehulled or non-dehulled pulse seeds. The pulse seeds may be milled or ground as such, or they may be milled or ground in the presence of water, e.g. to produce an aqueous slurry or paste.

The the mean Sauter diameter ($D_{3,2}$) and the volume weighted mean diameter ($D_{4,3}$)of the particles contained in the aqueous phase (T=20°C) are suitably determined by means of a static light scattering (SLS) technique (e.g. by using a Malvern Mastersizer 2000). About 1-2 ml of aqueous phase is added to water whilst stirring in the Mastersizer cell. The Malvern software applying the Mie scattering model does all the calculation, giving the mean Sauter diameter ($D_{3,2}$) and the volume weighted mean diameter ($D_{4,3}$). Three measurements are carried out on a sample and the measurement result equates the average of these three measurements.

The Malvern Mastersizer 2000 works on the principle of static light scattering (SLS) using two detection systems consisting of red and blue polarised lights. The red light has a wavelength of about 750 nm and measures forward scattering, side scattering and back scattering. The blue light with a wavelength of about 400 nm, measures wide angle forward and back scattering. This instrument is able to size particles from 0.2 µm to 2000 µm by laser diffraction. The Mastersizer is controlled and managed by the software MTS version 5.53 and particle size calculation can be done using Mie scattering model.

The gelatinized starch is, without wishing to be bound by any theory, believed to enhance the emulsion stability by structuring the aqueous phase of the emulsion of the present invention.

The extent to which the starch present in the emulsion is gelatinized can suitably be determined by cross polarised light microscopy.

As explained herein before, an emulsion of high stability can be obtained by ensuring that the particulate material contained in the aqueous phase has a relatively small particle size. According to a particularly preferred embodiment, volume weighted mean diameter ($D_{4,3}$) of the particles contained in the aqueous phase is less than 100 µm, more preferably less than 90 µm.

According to another preferred embodiment, the mean Sauter diameter ($D_{3,2}$) of the particles contained in the aqueous phase is less than 50 µm, more preferably less than 40 µm and most preferably less than 30 µm.

In accordance with a preferred embodiment of the present invention, the gelatinized starch and the pulse seed globulin together represent at least 33 wt.%, more preferably at least 45 wt.% and most preferably at least 55 wt.% of the biopolymer material that is contained in the aqueous phase.

Gelatinized starch is preferably contained in the aqueous phase of the present emulsion in a concentration of 0.5-10 wt.%, more preferably of 1-8 wt.% and most preferably of 1.5-6 wt.%.

The water-in-oil emulsion typically contains 0.15-3.75%, more preferably 0.3-3.0% and most preferably 0.45-1.5% pulse seed globulin by weight of aqueous phase.

Besides pulse seed globulin the present emulsion may suitably contain other pulse seed proteins. Typically, the emulsion contains 0.15-3.5%, more preferably 0.45-2.5% and most preferably 0.6-2.1% of pulse seed proteins by weight of aqueous phase.

The emulsion of the present invention advantageously contains pulse seed albumin as this protein. promotes in-mouth emulsion destabilization. In-mouth destabilization of the water-in-oil emulsion is essential for the perception of flavour components, such as salt, that are contained in the dispersed aqueous phase. Furthermore, in-mouth destabilization contributes to the perceived ‘creaminess’ of the emulsion. Thus, in accordance with another preferred embodiment, the emulsion contains 0.05-2%, more preferably 0.1-1.5% and most preferably 0.15-1% of pulse seed albumin by weight of the aqueous phase.

The pulse seed protein, such as the pulse seed globulin, and the starch originate preferably from a pulse selected from lentils, chickpeas, beans and combinations thereof. It is particularly preferred when the gelatinized starch and the pulse seed globulin originate from the same pulse seed.

In accordance with a particularly preferred embodiment, the gelatinized starch and pulse seed protein together represent at least 45 wt.%, more preferably at least 60 wt.% and most preferably at least 80 wt.% of the biopolymer material that is contained in the aqueous phase.

In another preferred embodiment of the present invention the emulsion comprises the starch and the pulse seed globulin in a weight ratio of 1:1 to 15:1, more preferably in a weight ratio of 2:1 to 10:1 and most preferably in a weight ratio of 3:1 to 8:1.

The aqueous phase of the emulsion typically has a pH in the range of 3 to 8, more preferably of 4 to 6.

Besides the gelatinized starch, the aqueous phase of the present emulsion may also contain non-gelatinized starch. Typically, at least 50 wt.% of the starch contained in the emulsion is gelatinized starch. Even more preferably at least 70 wt.% and most preferably at least 90 wt.% of said starch is gelatinized starch.

The present emulsion typically has a Stevens value at 5 °C of 10-300 g, more preferably of 20-200 g and most preferably of 25-250 g. The Stevens value is indicative of a product's hardness or firmness. The Stevens value was measured with a Stevens penetrometer (Brookfield LFRA Texture Analyser (LFRA 1500), ex Brookfield Engineering Labs, UK) equipped with a stainless steel 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.

The aqueous phase of the emulsion according to the present invention preferably contains 0-3 g/l, more preferably 0.1-1 g/l of metal ions selected from Na^+, K^+, Ca^{2+}, Mg^{2+} and combinations thereof. The Ca^{2+} concentration of the aqueous phase preferably does not exceed 400 mg/l, more preferably it does not exceed 130 mg/l and most preferably it does not exceed 90 mg/l.

The aqueous phase of the water-in-oil emulsion typically has an elastic modulus G', measured at 20°C, within the range of 0.05-600 Pa, most preferably in the range of 0.1-500 Pa.

It is further preferred that the aqueous phase has a viscosity at 20 °C and 50 s^{-1} in the range of 3-3,000 mPa.s, more preferably of 10-2,500 mPa.s.

The G' and viscosity of the aqueous phase of the present emulsion are measured using a standard protocol with the following 3 consecutive steps:

- The sample is rested for 3 minutes after the introduction into the rheometer to allow relaxation of the stresses accumulated due to the loading of the sample.
- A stress sweep as applied in which the oscillatory stress is increased from 0.1 to 1768 Pa in logarithmic steps (15 per decade). This step is terminated when the phase angle exceeds 80°. From this step the G' (shear storage modulus) is taken as described below.
- A viscosity measurement is done at a shear rate of 50 s^{-1} for a total of 1 minute. A viscosity point is measured every 10 seconds. Typically the last point is reported. The test is carried out at 20°C using a cone and plate rheometer. The cone used has a diameter of 4 cm and a cone angle of 2° degrees.

The shear storage modulus G' is the mathematical description of an object's or substance's tendency to be deformed elastically (i.e., non-permanently) when a force is applied to it. The term "storage" in shear storage modulus refers to the storage of the energy applied to the sample. The stored energy is recovered upon the release of the stress. The shear storage modulus of an aqueous phase is suitably determined by a dynamic oscillatory measurement, where the shear stress is varied (from low to high stress) in a sinusoidal manner. The resulting strain and the phase shift between the stress and strain is measured. From the amplitude of the stress and the strain and the phase angle (phase shift) the shear storage modulus is calculated. Herein, the G' (Pa) is taken at the plateau value at low stress (linear viscoelastic region). For these measurement a suitable state of the art rheometer is used (e.g. a TA AR2000EX, United Kingdom).

The emulsion of the present invention preferably contains no modified starch. The term "modified starch" as used herein refers to an enzymatically or chemically modified starch.

Unlike the emulsions described in WO 2011/029725, the present emulsion does not contain 0.1-20 wt.% of an evenly dispersed edible seed mixture, 60 to 100% (w/w) of the seed mixture being bigger than 0.5 mm.

The continuous fat phase typically contains 50-100 wt.%, more preferably 70-100 wt.% and most preferably 90-100 wt.% of triglycerides. The fat phase advantageously contains a high level of unsaturated fatty acids. Typically, 40-96 wt.%, more preferably 60-90 wt.% of the fatty acids contained in the continuous fat phase are unsaturated fatty acids.

According to a preferred embodiment of the invention the fat phase of the emulsion has a N_{20} in the range of 3% to 50% and a N_{35} in the range of 0% to 20%. Even more preferably, the emulsion has a N_{20} in the range of 3 to 40%, preferably of 4 to 25% and a N_{35} in the range of 0 to 15%, preferably of 0.5 to 8%. Here N_x refers to the solid fat content at x °C as determined by the methodology described in Fette, Seifen Anstrichmittel 80 180-186 (1978). The stabilization profile applied is heating to a temperature of 80°C, keeping the oil for at least 10 minutes at 60°C or higher, keeping the oil for 1 hour at 0°C and then 30 minutes at the measuring temperature.

The oil phase of the present water-in-oil phase generally comprises conventional oils and fats which may be of both animal and vegetable origin. Examples of sources of conventional oils and fats include, optionally fractions of, coconut oil, palmkernel oil, palm oil, marine oils, lard, tallow fat, butter fat, soybean oil, safflower oil, cotton seed oil, rapeseed oil, poppy seed oil, corn oil, sunflower oil, olive oil, algae oil and blends thereof. Hydrogenation may be used to alter the degree of unsaturation of the fatty acids and as such to alter the fatty acid composition. A drawback of hydrogenation, especially of partial hydrogenation, is the formation of by products like e.g. trans-unsaturated fatty acids. Interesterification retains the fatty acid composition but alters the distribution of the fatty acids over the glycerol backbones. Preferably, the water-in-oil emulsion of the invention comprises hardstock which does not contain fully hydrogenated fats, partially hydrogenated fats or interesterified fats.

The continuous fat phase comprised in the present emulsion preferably represents at least 20 wt.%, more preferably at least 30 wt.% and most preferably at least 35 wt.% of the emulsion. The dispersed aqueous phase preferably represents not more than 62 wt.%, more preferably not more than 58 wt.% and most preferably not more than 55 wt.% of the emulsion.

Typically, at least 95 vol% of the water droplets contained in the present emulsion have a diameter within the range of 1-40 µm, more preferably within the range of 1.5-35 µm.

The droplet size and its distribution of the dispersed aqueous phase is suitably determined by Nuclear Magnetic Resonance (NMR). On the basis of this method the parameters $D_{3,3}$ and exp(σ) of a lognormal water droplet size distribution can be determined. 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 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 parameters of the water droplet size distribution $D_{3,3}$ (volume weighed geometric mean diameter) and σ (distribution width) are calculated. 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).

Preferably, the $D_{3,3}$ of the dispersed aqueous phase lies in the range of 1-15 µm. More preferably said $D_{3,3}$ is in the range of 3-11 µm, most preferably in the range of 5-9 µm.

The edible emulsion may suitably contain one or more additional ingredients besides water, oil, gelatinized starch and pulse seed globulin. Examples of such optional ingredients include acidulant, salt, vitamins, minerals, emulsifier, gelling agents, thickening agents, flavouring, colouring, preservatives and antioxidants. Such optional additives, when used, collectively, do not make up more than 20%, more preferably not more than 10% by weight of the emulsion.

Examples of edible water-in-oil emulsions according to the present invention include spreads and margarines, such as kitchen margarines and bakery margarines. Most preferably, the emulsion is a spread.

Another aspect of the present invention relates to a process of preparing a water-in-oil emulsion as described above. The process comprises the following steps:

- preparing an aqueous composition by combining finely ground pulse seed, water and optionally other ingredients;

- combining the aqueous composition with a fat composition; and
- emulsifying the combination of the aqueous composition and the fat composition.

In accordance with a particularly preferred embodiment, the aqueous is subjected to high shear to reduce the diameter of the particulate matter that is contained therein. Examples of high shear techniques that may be employed include rotor stator systems, high pressure homogenizers, colloid mills, sonolators, utrasonication and combinations thereof. Most preferably, the high shear technique employed include high pressure homogenisation.

The finely ground pulse seed is preferably obtained from a pulse seed selected from lentils, chickpeas, beans and combinations thereof. Even more preferably, the ground pulse seed is obtained from a pulse seed selected from lentils, chickpeas, mung beans and combinations thereof. Most preferably, the ground pulse seed is ground lentil.

The finely ground pulse that is employed in accordance with the present invention may be obtained from dehulled and/or non-dehulled pulse seed. The water-structuring and emulsifying properties of the finely ground pulse seed are believed to be largely attributable to the starch and protein components. Preferably, the finely ground pulse seed employed is obtained from dehulled pulse seed.

It was found that a particularly stable emulsion can be produced by combining the finely ground pulse seed and water and heating the resulting combination to gelatinize the starch before adding the oil. Thus, in accordance with a particularly preferred embodiment, prior to the addition of oil, the combination of the finely ground pulse seed and water is heated to a temperature of more than 80°C for at least 30 seconds. Preferably the heating conditions employed are sufficient to gelatinize at least 50 wt.%, more preferably at least 70 wt.% and most preferably at least 90 wt.% of the starch contained therein. Furthermore, due to the heat treatment preferably, 60-100 wt.%, more preferably at least 90-100 wt.% of the protein comprised in the finely ground pulse seed is denatured.

Thus, the present process comprises preferably the step of heating the aqueous composition containing the finely ground pulse seed to gelatinize the starch contained therein. Depending on the heating temperature, the preferred times are as follows:

80-100°C: 1-70 minutes

100-120°C: 30-1200 seconds

120-150°C: 10-480 seconds

Typically, the (ground) pulse seed employed in accordance with the present invention contains less than 25%, most preferably less than 20% of dietary fibre by weight of dry matter. The oil content of the pulse seed preferably lies in the range of 0.8-8 wt%.

The aforementioned pulse seed typically has the following composition, calculated on dry matter:
30-60 wt.% of starch;
1-40 wt.% of dietary fiber;
0.5-12 wt.% of sugars;
15-35 wt.% of protein;
0.8-12 wt.% of oil;
wherein the pulse seed contains starch and protein in a weight ratio of 2:3 to 3:1. Typically, starch, dietary fiber, sugars, protein and oil together make up 90-100 wt.%, more preferably 95-100 wt.% of the dry matter contained in the pulse seed.

The pulse seed from which the globulin and starch originate typically contains starch and protein in a weight ratio of 1:1 to 15:1, more preferably of 2:1 to 10:1 and most preferably of 3:1 to 8:1.

Globulins and albumins typically represent a major part of the protein contained in the pulse seed. Accordingly, in a preferred embodiment, globulins and albumins together represent at least 50 wt.%, more preferably 55-95 wt.% and most preferably 60-90 wt.% of the protein contained in the pulse seed.

Emulsions of particular good quality can be obtained if the pulse seed contains globulins and albumins in a weight ratio that lies within the range of 10:1 to 1:2, or even more preferably in a weight ratio of 7:1 to 1:1.

In accordance with another preferred embodiment the globulins legumin and vicilin together represent at least 35 wt.%, more preferably 40-75 wt.% and most preferably 45-70 wt.% of the protein comprised in the pulse seed.

The content of globulin, albumin, legumin, vicilin, and glutelin in the pulse seeds of the present invention is suitably determined by the method described by Gupta & Dhillon [Gupta,

R., & Dhillon, S. 1993. Characterization of seed storage proteins of Lentil (Lens culinaris M.). Annals of Biology, 9, 71-78].

The protein provided by the finely ground pulse seed preferably comprises not more than a minor amount of sizeable coagulated protein aggregates. Typically, the finely ground pulse seed comprises 0-1 wt.% of coagulated protein aggregates having a hydrated diameter of at least 1.0 µm. The hydrated diameter can suitably be determined by Confocal Scanning Laser Microscopy with Nile Blue as fluorescent dye.

It is important that the pulse seed employed in the present emulsion is finely ground in order to release starch and protein from the seed material. Advantageously, the finely ground pulse seed contains less than 10 wt.%, more preferably less than 5 wt.% and most preferably less than 1 wt.% of particles having a hydrated diameter of 200 µm or more. The hydrated diameter of the finely ground pulse seed is suitably determined by means of Confocal Scanning Laser Microscopy, using the fluorescent dye Acridine Orange.

Even when used in relatively low concentrations, the finely ground pulse seed used in the present invention is capable of substantially improving the stability of the water-in-oil emulsion. Accordingly, the finely ground pulse seed preferably represents not more than 20%, more preferably not more than 14%, most preferably not more than 10% of the water-in-oil emulsion, calculated as dry matter by weight of aqueous phase. Typically, the finely ground pulse seed is employed in a concentration of at least 1%, even more preferably of at least 2% and most preferably of at least 2.5%, where the percentages are again calculated as dry matter by weight of the aqueous phase.

In accordance with one embodiment of the present process the emulsion obtained by emulsification of the combination of the aqueous composition and the fat composition is subjected to cooling in a scraped surface heat exchanger, such as a Votator, or in a C-unit to produce an emulsion of plastic consistency.

In accordance with another embodiment, the process comprising the formation of a dispersion by mixing oil, solid structuring agent particles and aqueous phase, wherein the solid structuring agent particles have a microporous structure and have a mean Sauter diameter $D_{3,2}$ of not more than 60 µm, preferably of not more than 30 µm. Typically, the solid structuring agent particles are employed in a concentration of 5-20%, more preferably of 7-15% and most preferably of 8-13% by weight of the fat phase. A description of a process for

the preparation of such solid structuring agent particles can be found in '*Particle formation of ductile materials using the PGSS technology with supercritical carbon dioxide*', P.Münüklü, Ph.D.Thesis, Delft University of Technology, 16-12-2005, Chapter 4, pp. 41-51

The invention is further illustrated by means of the following non-limiting examples.

EXAMPLES

Example 1

A spread according to the present invention having a fat content of 39 wt.% was prepared on the basis of the recipes shown in Table 1.

Table 1

	Wt.%
Aqueous phase	
• demi water	94.7
• NaCl	0.13
• Potassium Sorbate	0.13
• lentil flour	5
• citric acid (20%)*	
Fat phase	
• Fat powder	12
• Sunflower oil SF	87
• Dimodan HP**	0.77
• B-carotene (1% solution)	0.26

* pH was adjusted to 4.6 with 20 wt% citric acid solution.

- Milled dehulled red lentil (Turkey) having a particle size of less than 200 µm
- Solid structuring agent particles, consisting of an interesterified blend of 65 parts of a multi-fractionated palm oil stearin (iodine value =14) and 35 parts of palm kernel oil.
- Dimodan HP (monoglyceride) obtained from Danisco

The preparation process employed was as follows:

- Lentil flour (150 g) was dispersed in 1.5 L water with a magnetic stirrer
- The mixture was sheared with an UltraTurax at room temperature for 60 minutes
- Water was added to produce 3 L of an aqueous mixture

- The aqueous mixture was heated up to and held at 90°C for 60 minutes, and was cooled to ambient temperature
- Salt, and potassium sorbate were added and pH was adjusted to 4.6 with citric acid
- The aqueous phase was high pressure homogenized by giving it 2 passes at 320 bar through a Niro Soavi (type Panda). Next, it was high pressure homogenized in 2 passes in a microfluidizer (type 110S from Microfluidics). The preset air pressure used was 5.5 bar, which was amplified via a hydraulic assembly by a factor of 243 to approximately 1300-1400 bar of hydrostatic pressure in the fluid circuit.
- The fat phase was prepared by blending the sunflower oil with the fat powder. The mixing temperature of the sunflower oil and the fat powder is 14°C. The mixing conditions are 90 seconds at 14,500 rpm speed in a Fryma Delmix , type MZM / VK-7
- Immediately after homogenisation, the aqueous phase (12 °C) was combined with the fat phase (20°C) and the mixture was emulsified in a C-unit (pin stirrer) at15 kg/h.

Samples of the homogenized aqueous phase were subjected to rheological analyses. The results of these analyses are summarized in Table 2.

Table 2

Aqueous phase	
G' *	31 Pa
Viscosity *	303 mPa.s
$D_{3,2}$ *	32 µm
$D_{4,3}$ *	61 µm

* Using the measurement procedures described herein before

The spread so produced was stored at 5°C. Samples of the spread were subjected to a number of analyses after 1 day and 1 week storage. The results of these analyses are depicted in Table 3.

Table 3

W/O emulsion		
	1 day	**1 week**
Droplet size distribution • $D_{3,3}$ • d(0.025) • d(0.975)	 9.4 µm 3.4 µm 26 µm	 9.5 µm 3.3 µm 27 µm
Stevens hardness	43 g	75 g
WATOR score *	0	0
Spreading score **	1	2

* This score was obtained by spreading a sample. 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 any water and the paper does not change. Very unstable products release considerable amounts of free water that show up as dark spots on the paper.
A six point scale (0 to 5) is used to quantify the water loss (DIN 10 311):

** This score was obtained by carrying out the following test: Use a broad palette knife (25 to 30 mm in width) to spread about 30 g of the sample backwards and forwards on greaseproof paper. During four to six spreading actions in each direction, the thickness of the spread should be reduced to about 2 to 3 mm. The appearance of the product after spreading is scored between 1 (good/smooth) and 5 (very poor/very rough and "broken") by a comparison with photographic references.

Comparative Example A

Example 1 was repeated except that this time no homogenization step was employed. The results obtained are depicted in Tables 4 and 5.

Table 4

Aqueous phase	
G'	174 Pa
Viscosity	1010 mPa.s
$D_{3,2}$	88 µm
$D_{4,3}$	204 µm

Table 5

W/O emulsion		
	1 day	**1 week**
Droplet size distribution • $D_{3,3}$ • d(0.025) • d(0.975)	 11.6 µm 2.9 µm 47 µm	 11.3 µm 2.8 µm 46 µm
Stevens hardness	25 g	45 g
WATOR score	2	3
Spreading score	3	4

Example 2

A spread according to the present invention having a fat content of 39 wt.% using the same aqueous phase as described in Table 1 of Example 1. The composition of the fat phase is shown Table 6.

Table 6

	Wt.%
• Fat powder	12
• Sunflower oil SF	86.6
• Dimodan RT/B*	1.1
• β-carotene (1% solution)	0.36

- Fat powder was identical to the powder used in Example 1
- Dimodan RT/B (monoglyceride) obtained from Danisco

The aqueous phase was prepared as follows:

- Lentil flour was dispersed by paddle mixing in luke warm tap water to create a 3.0 kg batch of 5 wt% lentil flour slurry
- The slurry was heated for 30 minutes (UltraTurrax, IKA T25 digital with probe S25N-18G, at 16.4 krpm) at a temperature in excess of 90 °C au bain marie in a metal can using continuously a higher shear Ultraturrax (T25 basic with 10 mm diameter probe at 17.5 krpm) mixer (high shear cooking).
- The slurry was cooled by immersion of the can in a sink with cold water.
- The cooked slurry was homogenized at 320 bar pressure with a Niro Soavi (type Panda). The obtained “cooked” suspension was passed two times through a Microfluidiser®-110S. The preset air pressure used was 5.5 bar, which was amplified via a hydraulic assembly

by a factor of 243 to approximately 1300 -1400 bar of hydrostatic pressure in the fluid circuit.

- Immediately after homogenisation, the aqueous phase (12 °C) was combined with the fat phase (20°C) and the mixture was emulsified in a C-unit (pin stirrer)

Sample of the homogenized aqueous phase were subjected to rheological analyses. The results of these analyses are summarized in Table 7.

Table 7

Aqueous phase	
G'	14.5 Pa
Viscosity	85 mPa.s
$D_{3,2}$	20 µm
$D_{4,3}$	71 µm

The fat phase was kept at 20 °C, the water phase at 12 °C before they were used in the preparation of a fat-continuous spread in a pilot plant at a throughput of 15 kg/h with 39% fat.

The spread so produced was stored at 5°C for 16 weeks. Analyses were conducted during this storage period. The results of these analyses are depicted in Table 8.

Table 8

W/O emulsion				
	4 days	**5 weeks**	**10 weeks**	**16 weeks**
Droplet size distribution • $D_{3,3}$ • d(0.025) • d(0.975)	 7.9 µm 2.9 µm 29 µm	 8.3 µm 2.0 µm 37 µm	 8.1 µm 2.1 µm 34 µm	 8.1 µm 2.0 µm 31 µm
Stevens hardness	43 g	61 g	65 g	70 g
WATOR score	0	0	0	0
Spreading score	2	1	2	3

<u>Comparative Example B</u>

Example 2 was repeated except that this time no homogenization step was employed. Samples of the freshly prepared homogenized aqueous and of the emulsion after 1 week storage at 5°C were analysed. The results obtained are depicted in Tables 9 and 10.

Table 9

Aqueous phase	
G'	316 Pa
Viscosity	1,120 mPa.s
$D_{3,2}$	97 µm
$D_{4,3}$	252 µm

Table 10

W/O emulsion		
	1 day	**1 week**
Droplet size distribution • $D_{3,3}$ • d(0.025) • d(0.975)	 7.7 µm 1.7 µm 36 µm	 7.5 µm 1.7 µm 33 µm
Stevens hardness	49 g	61 g
WATOR score	2	3
Spreading score	3	3

Example 3

A fraction containing starch and globulin, but virtually no fibres and albumin, was isolated from lentil flour using the following procedure.

1.5 L lentil slurry (10wt.% lentil flour) was high shear mixed for 60 minutes at room temperature, using UltraTurrax mixing at 17,500 rpm. A precipitate formed at rest containing insoluble fibres, starch granules and globulins. The solution contains albumin and some soluble fibre. The suspension was sieved through a polyester cheese cloth of 97 micron mesh size to remove (insoluble) fibre. The cloth was placed on a Büchner funnel that was placed on a glass bottle that was sucked to low pressure by a water jet. Albumin and a precipitate of globulin and starch were separated by centrifuge (30 min 4500 rpm). To the precipitate 3.9 g NaCl, 3.9 g Potassium Sorbate was added and diluted to 3L to create a 5 wt% lentil flour equivalent of starch and globulin.

The fraction so obtained was heat treated (UltraTurrax, IKA T25 digital with probe S25N-18G, at 16,400 rpm) for 60 minutes in excess of 90 °C au bain marie in metal cans, following which salt and potassium sorbate were added and pH was set to 4.6 with 20% citrate as described in Example 1. The aqueous phase was used after 2 passes of homogenization (Niro Soavi, Panda) at 360 bar and 2 passes of microfluidation in Microfluidiser®-110S at 1300 bar.

A spread was made in the same way as described in Example 1, except that this time the starch and globulin containing aqueous phase was employed instead of the homogenized lentil flour slurry.

Samples of the freshly prepared homogenized aqueous and of the emulsion after 1 day and 1 week storage at 5°C were analysed. The results obtained are depicted in Tables 11 and 12.

Table 11

Aqueous phase	
G'	0.78 Pa
Viscosity	16 mPa.s
$D_{3,2}$	22 µm
$D_{4,3}$	34 µm

Table 12

W/O emulsion		
	1 day	1 week
Droplet size distribution • $D_{3,3}$ • d(0.025) • d(0.975)	 9.3 µm 3.6 µm 24 µm	 9.2 µm 3.7 µm 23 µm
Stevens hardness	38 g	101 g
WATOR score	0	0
Spreading score	1	1

Claims

1. An edible water-in-oil emulsion comprising 15-65 wt.% of a continuous fat phase and 35-85 wt.% of a dispersed aqueous phase, said emulsion containing by weight of the dispersed aqueous phase:
   - 0.2-7 wt.% of gelatinized starch; and
   - 0.1-4 wt.% of pulse seed globulin;

   wherein the mean Sauter diameter ($D_{3,2}$) of the particles contained in the aqueous phase is less than 60 µm.

2. Emulsion according to claim 1, wherein the gelatinized starch and the pulse seed globulin together represent at least 33 wt.% of the biopolymer material that is contained in the aqueous phase.

3. Emulsion according to claim 1 or 2, wherein the pulse seed protein and the starch originate from a pulse selected from lentils, chickpeas, beans and combinations thereof

4. Emulsion according to claim 3, wherein the gelatinized starch and the pulse seed globulin originate from the same pulse seed.

5. Emulsion according to any one of the preceding claims, wherein the emulsion contains 0.05-2% of pulse seed albumin by weight of the aqueous phase.

6. Emulsion according to any one of the preceding claims, wherein starch and the pulse seed globulin are contained in the emulsion in a weight ratio of 1:1 to 8:1.

7. Emulsion according to any one of the preceding claims wherein the aqueous phase has a pH in the range of 3 to 8

8. Emulsion according to any one of the preceding claims, wherein the emulsion contains 0.15-3.5% pulse seed proteins by weight of aqueous phase.

9. Emulsion according to any one of the preceding claims, wherein the aqueous phase contains 0-3 g/l of metal ions selected from Na^+, K^+, Ca^{2+}, Mg^{2+} and combinations thereof.

10. Emulsion according to any one of the preceding claims, wherein the aqueous phase has a viscosity at 20°C and 50 s^{-1} in the range of 3-3,000 mPa.s.

11. Emulsion according to any one of the preceding claims, wherein the aqueous phase does not contain modified starch.

12. Emulsion according to any one of the preceding claims, wherein the fat phase has a N_{20} in the range of 3% to 50% and a N_{35} in the range of 0% to 20%.

13. Emulsion according to any one of the preceding claims, wherein the emulsion does not contain 0.1-20 wt.% of an evenly dispersed edible seed mixture, 60 to 100% (w/w) of the seed mixture being bigger than 0.5 mm.

14. A process of preparing an emulsion according to any one of the preceding claims, said process comprising:
    - preparing an aqueous composition by combining ground pulse seed, water and optionally other ingredients;
    - combining the aqueous composition with a fat composition; and
    - emulsifying the combination of the aqueous composition and the fat composition.

15. Process according to claim 14, wherein the ground pulse seed is obtained from a pulse seed selected from lentils, chickpeas, beans and combinations thereof.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/061454

A. CLASSIFICATION OF SUBJECT MATTER
INV. A23D7/00 A23L1/20 A23D7/005
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 A23L

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, FSTA, WPI Data, BIOSIS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	OKAKA J C ET AL: "PHYSICOCHEMICAL AND FUNCTIONAL PROPERTIES OF COWPEA POWDERS PROCESSED TO REDUCE BEANY FLAVOR", JOURNAL OF FOOD SCIENCE, vol. 44, no. 4, 1979, pages 1235-1240, XP002688417, ISSN: 0022-1147 abstract page 1235, column 1, paragraph 4 - column 2, paragraph 1 page 1236, column 2, paragraph 3 ----- -/--	1-15

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
24 September 2013	09/10/2013

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/061454

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

International application No
PCT/EP2013/061454

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