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(54) Title: FORTIFICATION OF EDIBLE OILS WITH HYDROXYTYROSOL

(57) Abstract: The present invention relates to a process of fortifying edible oil with hydroxytyrosol. By this process high fortification levels can be achieved. Particularly clear and storage stable fortified oils can be provided by the mentioned process. The fortified oils can be used for several purposes. The edible oils fortified with hydroxytyrosol can be particularly used in the field of nutrition for humans and animals. The use of edible oil fortified with hydroxytyrosol offers to use the established health benefit of hydroxytyrosol in all fields of nutrition.



FORTIFICATION OF EDIBLE OILS WITH HYRDOXYTYROSOL

Technical Field

The present invention relates to fortification of edible oils.

5

Background of the invention

Hydroxytyrosol (3,4-dihydroxyphenylethanol) is the key polyphenol which is present in the olive fruit. Hydroxytyrosol has been recognized as being a highly effective ingredient and shows a large number of health benefits. It has been
10 shown that it can be used for the prevention or treatment of cardiovascular diseases (CVD) and is highly active as anti-inflammatory ingredient. Hydroxytyrosol is regarded to be as one of the most potent natural antioxidant.

The European Food Safety Authority (EFSA) has acknowledged the cause and effect relationship between the consumption of olive oil polyphenols
15 (standardized by the content of hydroxytyrosol and its derivatives) and protection of LDL particles from oxidative damage

WO 2011/041937 A1 discloses that hydroxytyrosol gives improvement of muscle differentiation. WO 2010/118789 A1 and WO 2009/144093 A1 disclose that hydroxytyrosol increases mitochondria biogenesis which helps to boost the
20 body's energy production and increase the muscle/fat ratio. WO 2008/128629 A1 discloses that hydroxytyrosol induces and/or enhances the cartilage repair/regeneration.

The content of hydroxytyrosol in the olive fruit is much higher than in the olive oil. As hydroxytyrosol is highly water soluble, during the oil production
25 process, the majority of hydroxytyrosol is removed as part of water phase and of the pomace. The pomace and the water phase (vegetation water), have been regarded for a long time as a waste product. The amount of hydroxytyrosol in vegetation water is about 0.15 % by weight and represents an important source for hydroxytyrosol.

Due to the high health benefits of hydroxytyrosol, there exist a strong desire to fortify any edible oil, particularly olive oil, by hydroxytyrosol, which has been obtained from natural grown olive fruits. However, due to the polar character of hydroxytyrosol, only minor amounts can be expected to be added to the oils.

5

WO 2009/013596 A1 discloses a process of fortification of edible oil by hydroxytyrosol. According to this procedure the edible oil is fortified by mixing intensively an aqueous solution of hydroxytyrosol with the edible oil. This, however, leads to an oil/water mixture which results in turbid oils and storage problems when the fortified oil is encapsulated in gel capsules. Furthermore, the majority of gel-capsules is transparent. The presence of turbid or phase separating liquids in transparent capsules, however, are not desired as the consumer tends to correlate such visual effects with instability of the product. Therefore, there is high market demand for non-turbid oils fortified with hydroxytyrosol.

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15

Summary of the invention

Therefore, the problem to be solved by the present invention is to provide a method for manufacturing edible oils being highly fortified with hydroxytyrosol without leading to any negative properties.

20

Surprisingly, it has been shown, that the process of manufacturing according to claim 1 solves the above mentioned problem. Furthermore, this process allows to form particularly highly fortified edible oils which are clear in aspect. They are very advantageous in that they are and remain homogenous and do not show any negative stability.

25

These highly fortified oils obtained by the invention are suitable for preparing capsules which can be used as food supplements for humans and animals.

The process of fortification enables also to fortify edible oils which are heat-sensitive by a specific mild process.

30

Further aspects of the invention are subject of further independent claims. Particularly preferred embodiments are subject of dependent claims.

Detailed description of the invention

In a first aspect, the invention relates to a process of manufacturing edible oils fortified with hydroxytyrosol comprising the steps

- a) providing an edible oil;
- 5 b) providing an aqueous solution of hydroxytyrosol in which the amount of hydroxytyrosol is at least 5%, particularly at least 20 %, preferably at least 30 %, by weight;
- c) heating up to a temperature of between 25 and 95 °C;
- d) mixing said edible oil with said aqueous solution of hydroxytyrosol to
10 form a two phase mixture; followed by
- e) separating the fortified edible oil as oil phase from the two-phase mixture.

The term "polyunsaturated" is used in the present document as to indicate
15 that the molecule, particularly the fatty acid, has two or more carbon-carbon double bonds in its chemical structure.

The term "polyunsaturated fatty acid ester" is used in the present document as to indicate an ester of a polyunsaturated fatty acid with an alcohol.

The term "fatty acid" represents a carboxylic acid having at least 6,
20 preferably at least 12, particularly at least 16, carbon atoms.

The term "acid part" in the context of edible oils resp. esters is used to indicate the residue in the ester stemming from the carboxylic acid when condensing a carboxylic acid with an alcohol to form an ester.

25 Substance names starting with "poly" such as polyol as used in the present document refer to substances formally containing two or more functional groups as indicated in their name per molecule.

The term "fortified oil" as used in this documents is a short form of "edible
30 oil fortified with hydroxytyrosol", which means that the edible oil has a higher content of hydroxytyrosol as compared to the untreated oil.

"Room temperature" is used in the present document as to mean a temperature of 25°C.

"HT" is used in the present document as an abbreviation of hydroxytyrosol.

Edible oil

5 The edible oil which is provided in step a) is liquid at room temperature. Preferably the edible oil is selected from the group consisting of olive oil, sunflower oil, polyunsaturated fatty acid ester, corn oil, safflower oil, almond oil, palm oil, soybean oil and rapeseed oil. Particularly preferred is the edible oil selected from the group consisting of olive oil, sunflower oil and polyunsaturated
10 fatty acid ester. The edible oil provided in step a) is non-fortified.

In one embodiment mixtures of the above mentioned edible oils are used.

A particular suitable edible oil is a polyunsaturated fatty acid ester (PUFA).
15 PUFAs are classified according to the position of the double bonds in the carbon chain of the molecule as n-9, n-6 or n-3 PUFAs. Examples of n-6 PUFAs are esters of linoleic acid (C18 : 2), arachidonic acid (C20 : 4), γ -linolenic acid (GLA, C18 : 13) and dihomo- γ -linolenic acid (DGLA, C20 : 3). Examples of n-3 PUFAs are esters of α -linolenic acid (C18 : 13), eicosapentaenoic acid (EPA, C20 : 5),
20 and docosahexaenoic acid (DHA, C22 : 6). Especially EPA and DHA have attracted interest of the food industry in recent years. The most available sources of these two fatty acids are fish and the marine oils extracted thereof.

The polyunsaturated fatty acid has preferably a chemical structure
25 comprising 16 or more carbon atoms, (for example, 16, 18, 20 or 22 carbon atoms ("C16," "C18," "C20," or "C22," respectively)) and two or more carbon-carbon double bonds in the chemical structure.

When the notation CA:Bn-X is used for a methylene-interrupted PUFA, the "CA" is the number of carbons (for example C18, C20 or C22), B is the number of
30 double bonds and X is the position of the first double bond counted from the methyl end of the fatty acid chain.

Particularly, the polyunsaturated fatty acid are omega-3, omega-6, and omega-9 fatty acids, particularly α -linolenic acid (C18:3n-3), C18:4n-4, ω -3

eicosapentaenoic acid (20:5n-3) (eicosapentaenoic acid), ω -3 docosapentaenoic acid (docosapentaenoic acid), ω -3 docosahexaenoic acid (22:6n-3) or docosatetraenoic acid (22:4n-6).

A preferred class of ester of polyunsaturated fatty acid are the esters of
5 docosahexaenoic acid (DHA), also known by its chemical name (all-Z)-
4,7,10,13,16,19-docosahexaenoic acid, as well as any salts or derivatives
thereof.

A further preferred class of esters of polyunsaturated fatty acid are the
esters of eicosapentaenoic acid (EPA), known by its chemical name (all-Z)-
10 5,8,11,14,17-eicosapentaenoic acid

Typical examples of esters include methyl, ethyl, trichloroethyl, propyl,
butyl, pentyl, *tert*-butyl, benzyl, nitrobenzyl, methoxybenzyl and benzhydryl ester
of the above mentioned polyunsaturated fatty acids. Other esters of PUFAs are
15 described in U.S. Patent Application Publication No. US 2010-0130608 A1, which
is incorporated herein by reference in its entirety.

Particularly suitable are methyl or ethyl esters of polyunsaturated fatty
acid ester, of particularly of DHA and/or EPA.

In case that the ester is an ester of a polyol, the ester can be an ester of
20 one or several of different carboxylic acids. In order to qualify as a
polyunsaturated fatty acid ester not all hydroxyl groups of the polyol need to be
esterified. Furthermore, it is not needed that all carboxylic acids are
polyunsaturated to qualify as a polyunsaturated fatty acid ester.

In other words, an ester of glycerol with α -linolenic acid and acetic acid,
25 e.g. (9Z,12Z,15Z)-3-acetoxy-2-hydroxypropyl octadeca-9,12,15-trienoate would be
regarded as being qualified as a polyunsaturated fatty acid ester.

In one embodiment triglycerides are preferably used, particularly a
polyunsaturated fatty acid ester whereby 30% of the fatty acid part are n-3 fatty
acids and of these 25% are long-chain polyunsaturated fatty acids. In a further
30 embodiment commercially available MEG-3 '30' N-3 FOOD OIL (DSM Nutritional
Products Ltd, Kaiseraugst, Switzerland) is used.

Particularly preferred polyunsaturated fatty acid ester are monoglycerides, diglycerides, and triglycerides of the respective polyunsaturated fatty acid, particularly of DHA and/or EPA.

- 5 Polyunsaturated fatty acid ester can be obtained particularly from fish oil or from algae.

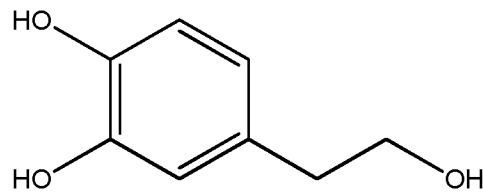
- 10 Examples for olive oil for being used as edible oil are particularly extra virgin olive oil (evoo), virgin olive oil, olive oil, refined olive oil, crude olive-pomace oil or refined olive-pomace oil, preferably extra virgin olive oil (evoo) or virgin olive oil.

- 15 As sunflower oil particularly high oleic sunflower oil is preferred.

- 20 The thermal stability differs significantly among the edible oils mentioned above. Particularly, the polyunsaturated fatty acid ester are prone to thermal degradation. Therefore, temperature exposure is a factor which needs to be taken into account when the edible oil is fortified.

Aqueous solution of hydroxytyrosol

Hydroxytyrosol (3,4-dihydroxyphenylethanol) is the key polyphenol which is present in the olive fruit and has the following formula



5

Hydroxytyrosol can be principally of synthetic or natural origin. It is preferred the hydroxytyrosol is obtained from pomace, water phase resulting from the olive oil production process, particularly from vegetation water.

It can be used as solution, i.e. in liquid form, or in powder form and is preferably obtained by extraction.

There exist different suitable extraction method using water or organic solvents.

It is most preferred that the hydroxytyrosol obtained by extraction has only traces of oleuropein and/or tyrosol. Oleuropein and tyrosol are ingredients which also occur naturally in olive fruits. Particularly, oleuropein is known as being responsible for the bitter taste of olive. WO 2009/013596 discloses that high content of aldehydic form of oleuropein aglycon are a source of bitterness. Hence, it is preferred that also the amount of aldehydic form of the oleuropein aglycon is strongly reduced by the extraction method.

For the acceptance of an edible oil being fortified with hydroxytyrosol, it is advantageous, that any bitter ingredient is absent or only present in negligible amounts.

Therefore, any extraction procedures for hydroxytyrosol leading to a relative high amount in oleuropein (oleuropein : hydroxytyrosol > 1:200) and/or tyrosol (tyrosol : hydroxytyrosol > 1: 50) are principally suitable, but not preferred.

Very suitable extraction processes involve the use of chromatographic separation, particular by means of organic resins, particularly of ion exchange resins, such as disclosed by WO 2002/064537 A1, WO 2008/142178 A1,

WO 2001/045514 A1 or WO 2008/090460 A1, the entire content of all these documents is hereby incorporated by reference.

5 In a preferred embodiment, the aqueous solution of hydroxytyrosol used in step b) is produced by extraction of olive fruit pomace or of the water phase obtained during the manufacturing of olive oil and followed by further purification of the aqueous extract by means of chromatographic columns, particularly as described in WO 2002/064537 A1 and WO 2008/090460 A1 the entire content of both documents is hereby incorporated by reference.

10

Hydroxytyrosol in a powder form can be obtained by spray drying of an aqueous or solvent based, preferred from an aqueous, solution of hydroxytyrosol. It is preferred that hydroxytyrosol in a powder form is adsorbed in or absorbed on a food grade carrier such as starch, maltodextrins, calcium carbonate or silica.

15

In one embodiment, the aqueous solution of hydroxytyrosol is prepared from dissolving hydroxytyrosol in form of a powder, particularly in a powder form as described above, in water. This procedure allows producing an aqueous solution of hydroxytyrosol in a high and accurate concentration of hydroxytyrosol.

20

In another embodiment, the aqueous solution is taken as obtained from the extraction process as described above. This is advantageous as spray drying can be skipped which leads to lower production cost. It is, however, for this embodiment important to select the extraction process such that it is capable to
25 deliver an aqueous solution of hydroxytyrosol in which the amount of hydroxytyrosol is at least 5% by weight.

For the purpose of the invention, it is important that the amount of hydroxytyrosol is at least 5%, particularly at least 20 %, preferably at least 30 %, by weight in the aqueous solution. It is more preferred, that the amount of
30 hydroxytyrosol is in the range between 30 % and 90 %, particularly between 35 and 80%, especially between 35 and 60 %, by weight in the aqueous solution. It has been found that only by using such highly concentrated aqueous solutions of

hydroxytyrosol a sufficiently high degree of fortifications of edible oils can be achieved.

Particularly, it is preferred that the aqueous solution of hydroxytyrosol
5 comprises less than 0.18 %, preferably less than 0.1 %, particularly less than 0.01 %, by weight of oleuropein.

Particularly, it is furthermore preferred that the aqueous solution of hydroxytyrosol comprises less than 0.5 %, preferably less than 0.05 %, by weight of tyrosol.

10

A very suitable aqueous solution of hydroxytyrosol is the product elaVida™ 40 or elaVida™ 15. elaVida™ 40, commercially available from DSM Nutritional Products Ltd, is an aqueous solution of hydroxytyrosol (HT content: 40% by weight), which has been extracted from olive water phase resulting from
15 production of olive oil from olive oil fruits, and purified by chromatography. It has an oleuropein / hydroxytyrosol ratio of max. 1:230 w/w% and an tyrosol / hydroxytyrosol ratio of max. 1:58 w/w%

Process of fortification

20 The process of manufacturing edible oils fortified with hydroxytyrosol comprises the steps

- a) providing an edible oil;
- b) providing an aqueous solution of hydroxytyrosol in which the amount of hydroxytyrosol is at least 5%, particularly at least 20 %, preferably at
25 least 30 %, by weight;
- c) heating up to a temperature of between 25 and 95 °C;
- d) mixing said edible oil with said aqueous solution of hydroxytyrosol to form a two phase mixture; followed by
- e) separating the fortified edible oil as oil phase from the two-phase
30 mixture.

In step d), the edible oil is mixed with the aqueous solution of hydroxytyrosol to form a two phase mixture. This mixing step can be performed principally

by any conventional mixing equipment. For the purpose of the invention, it is beneficial if the mixing step is performed in a very efficient manner. Particularly, it is beneficial to perform the mixing step b) by means of a conventional mixing equipment such a blade stirrer or paddle mixers.

5 It is preferred that the mixing takes place for at least 10, particularly at least 15 minutes.

By mixing the edible oil and aqueous solution of hydroxytyrosol a two-phase mixture (oil and water phase) is obtained. During mixing hydroxytyrosol is transferred from the water into the oil phase. The oil, now being fortified with hydroxytyrosol, is subsequently separated in a subsequent step e) from the water phase. The separation can be performed by conventional separation techniques, particularly using a separating funnel or a centrifuge. It is preferred that the separation of fortified edible oil as oil phase from the two-phase mixture is
10 performed in step d) by centrifugation.
15

In separating the oil phase, it is very advantageous that the water phase is completely removed. This is best achieved by stopping the separation of the oil phase from the two-phase mixture before the phase boundary is reached. In other words, it is preferred, that after separation of the fortified oil phase, there is still
20 some oil phase remaining on top of the remaining water phase.

By doing so it can be guaranteed that the edible oil being fortified with hydroxytyrosol comprises only traces of water.

It is very advantageous to obtain edible oils fortified with hydroxytyrosol with no or at least only traces of water as water leads to stability problems, particularly stability problems at high temperatures (i.e. larger than 50°C), of the fortified oil or gel capsules comprising the edible oil fortified with hydroxytyrosol. For example, the presence of water can leave to a saponification of the fatty acid esters leading to an increase of free fatty acids in the oil and can also increase the
25 poly-unsaturated fatty acids oxidation rate. By this degradation process, the properties of the fortified edible oil are changed in an undesired manner.
30

Step c) of the present process comprises heating up to a temperature of between 25 and 95 °C.

This heating step c) can be realized either by heating the oil and/or the aqueous solution of hydroxytyrosol before the mixing process (i.e. before step d)) or by heating during the mixing. However, in view of reducing the thermal exposure for the edible oil, it is preferred, that the heating step c) takes place in
5 the mixing step d).

The mixing is typically performed at normal pressure.

As mentioned before, certain edible oils show a tendency to degrade on prolonged heat exposure, particular in contact with air. Hence, in case of heat
10 sensitive oil, particularly polyunsaturated fatty acid ester, it is preferred to perform step c) at significantly lower temperatures than for the less sensitive edible oils.

The heat sensitivity is heavily linked to the degree of unsaturation of the fatty acids (resp. esters thereof) existing in the individual oils. The higher the amounts of double or polyunsaturated acid parts (stemming from double
15 unsaturated or polyunsaturated fatty acids) in the acid part of the oil is, the higher the heat sensitivity is.

It is, therefore, preferred that oils having a high amount of double or polyunsaturated acid parts of the oils, particularly safflower oil, corn oil, rapeseed oil and polyunsaturated fatty acid ester are heated only to moderate temperatures,
20 i.e. at a temperature in the range of between 25 and 55 °C, particularly of between 30 and 45 °C.

Oils having a lower amount of double or polyunsaturated acid parts, particularly olive oil, palm oil, almond oil and sunflower oil, particularly high oleic sunflower oil, are suitable to be heated up at higher temperatures, i.e. to a
25 temperature in the range of between 60 and 95°C, particularly between 70 and 90°C.

It has been observed that the higher the temperature is, the higher is the fortification level of hydroxytyrosol in the edible oil which can be achieved.

30

The amount of hydroxytyrosol in the fortified edible oil can be varied in broad ranges using the described process.

In one embodiment, the amount of hydroxytyrosol in the fortified edible oil is more than 250 mg / kg fortified oil, particularly more than 400 mg / kg fortified oil, particularly more than 1000 mg / kg fortified oil, preferably more than 2500 mg / kg fortified oil. In certain cases fortification levels of 3700 or more mg / kg fortified oil have been achieved.

It has been shown that it is preferable to select a specific time of mixing t_{mix} , measured in minutes, and to select a specific mixing temperature T_{mix} , measured in Celsius ($^{\circ}\text{C}$), so that the value v_{mix} calculated according to the following formula

$$v_{mix} = t_{mix} * (T_{mix})^2$$

is in the range of between 70'000 and 250'000 $\text{min}(\text{^{\circ}C})^2$, particularly between 80'000 and 120'000 $\text{min}(\text{^{\circ}C})^2$.

15

The described process allows on the one hand to form edible oil having a very high fortification by hydroxytyrosol.

On the other hand the desired process allows the preparation of an edible oil fortified with hydroxytyrosol which remain clear and homogenous. Clear oils are not turbid. The turbidity can be quantified by means nephelometry. Turbidity values were given in NTU (nephelometric turbidity units). Nephelometer measures the light scattered by a sample in 90° from the incident light path.

25

Hence, in one embodiment, it is preferred that the edible oils fortified with hydroxytyrosol have a turbidity of less than 15 NTU, particularly less than 10 NTU, preferably less than 3 NTU, measured by using a nephelometer (Hach 2100N IS®, USA, angle of measurement: 90°) at room temperature.

30

In a further aspect, the invention relates to an edible oil fortified with hydroxytyrosol having an amount of more than 250 mg hydroxytyrosol / kg fortified oil, particularly more than 1000 mg hydroxytyrosol / kg fortified oil and has a

turbidity of less than 15 NTU, particularly less than 10 NTU, preferably less than 3 NTU, measured by using a nephelometer (Hach 2100N IS®, USA, angle of measurement: 90°) at room temperature.

5 Principally, it is preferred to obtain a fortified oil which contains as much hydroxytyrosol / kg fortified oil, as possible, however, typically clear fortified edible oils have an amount of between 250 mg and 5000 mg hydroxytyrosol / kg fortified oil, particularly between 500 mg and 4500 mg hydroxytyrosol / kg fortified oil, more particularly between 1000 mg and 4000 mg hydroxytyrosol / kg fortified oil. Above
10 this concentration it is difficult to obtain clear fortified oils.

 In a further aspect, the invention relates to a capsule for oral consumption which comprises between 0.2 mL and 2.5 mL of an edible oil, which is fortified with hydroxytyrosol in which the amount of hydroxytyrosol is more than 1000 mg
15 /kg fortified oil, particularly between 1000 and 4000 mg / kg fortified oil, preferably between 2000 and 4000 mg / kg fortified oil. The edible oil fortified with hydroxytyrosol comprises less than 1 g water / kg fortified oil.

 The capsule is particularly a gel capsule. Gel capsules have the property
20 that they dissolve in contact with water in the body (i.e. mouth, stomach) to release the content. Therefore, the composition of the of gel capsules are normally based on gelatin or on modified starches or cellulose (such as hydroxypropyl methylcellulose). There exist softgel and hardgel capsules. Preferred are hard gel capsules.

25 Due to the water solubility of the material forming the shell any undesired exposure of the gel capsule is to be avoided to guarantee long shelf life of the capsule particularly at higher temperatures. Therefore, it is advantageous that the edible oil fortified with hydroxytyrosol inside the capsule shell comprises as little water as possible. The content of water is typically determined by the Karl Fischer
30 titration method. With the process of manufacturing edible oils fortified with hydroxytyrosol, as above described in great detail, it is possible to manufacture edible oil having significantly less than 1 g, particularly less than 0.1 g, water per kg of fortified oil.

The capsule comprises between 0.2 mL and 2.5 mL of the fortified edible oil. This allows that the size of the capsule is an reasonable size to be swallowed. The capsules can be rather different in shape, particularly spherical, oblong or tubular. Preferably, the capsules are oblong.

5 It is preferred, that the desired dosage of hydroxytyrosol, particular in the range of between 3 mg and 60 mg hydroxytyrosol per day for a 70 kg adult, can be administered by swallowing of maximal 9 capsules per day, preferably by a single capsule.

10

In a further aspect, the invention relates to a process of fortification of an edible oil with hydroxytyrosol comprising the steps

- i) providing a non-fortified edible oil;
- ii) providing an edible oil fortified with hydroxytyrosol according to a process
15 of fortification as disclosed above in great detail;
- iii) blending said non-fortified edible oil and said edible oil fortified with hydroxytyrosol.

As mentioned above certain edible oils have a limited thermal stability. It
20 is, hence, not advisable to expose these edible oils to higher temperatures particularly during extended time periods. In order to obtain also high fortification levels, i.e. high amount of hydroxytyrosol, particular to obtain clear fortified oils having high fortification levels, the just above mention process of fortification is especially suited.

25 It is, hence, preferred that the fortified edible oil in step ii) is an oil which has a low amount of double or polyunsaturated acid parts, particularly selected from the group consisting of olive oil, palm oil, almond oil and sunflower oil, particularly high oleic sunflower oil. It is, furthermore, preferred that the fortification of the edible oil of step ii) is performed at a temperature of in the range of between
30 60 and 95°C, particularly between 70 and 90°C. The objective of this fortification process is to achieve a fortified oil having a high (as high as possible) concentration of hydroxytyrosol. It is not necessary that the fortified oil used in step ii) is clear.

The non-fortified edible oil of step i) is preferably an oil which has a high amount of double or polyunsaturated acid parts, particularly selected from the group consisting of safflower oil, corn oil, rapeseed oil and polyunsaturated fatty acid ester.

5

The non-fortified edible oil of step i) is preferably a polyunsaturated fatty acid ester.

The non-fortified edible oil of step i) and the edible oil fortified with hydroxytyrosol of step ii) are blended in step iii).

10

This blending is done preferably at low temperatures, typically at temperatures of less than 60°C, particularly at a temperature of between 25°C and 50°C.

The blending is done in such a ratio of fortified oil of step ii) / non fortified oil of step i) that the concentration of the fortified oil mixture is in the desired fortification level.

When -on the one hand- high concentrations of hydroxytyrosol in a heat-sensitive edible oil are desired and -on the other hand- only minor amounts of heat-stable oil are accepted, it is mandatory that the fortified oil of step ii) needs to have a very high concentration in hydroxytyrosol.

By this process, also fortification of heat-sensitive oils with a very high amount of hydroxytyrosol can be achieved under very mild conditions. Particularly, it is now also possible to achieve clear and highly fortified heat-sensitive oils.

The edible oils fortified with hydroxytyrosol, being produced by the procedures as shown above in great details, can be used in any fields of application of edible oil. They find particularly application where the beneficial effects of hydroxytyrosol for humans and animals are desired.

Hence, in a further aspect, the invention relates to a nutritional product comprising an edible oil fortified or prepared by the processes of the present invention shown in detail above.

A nutritional product is any product which can be used in food or feed application, i.e. in the field of nutrition for animals or humans. Preferably the nutritional product is intended for human use.

In one of the preferred embodiments, the nutritional product is a feed or
5 food supplement. The terms nutritional product or food supplement are particularly defined as being purely for the field of cosmetic or a non-therapeutic use.

Particular the nutritional product is a water-in-oil or an oil-in-water food
10 emulsion. Preferred examples of such water-in-oil or an oil-in-water food emulsion are margarines or a mayonnaise or a sauce or a dressing.

As a further aspect, the edible oil fortified with hydroxytyrosol can be used in the field of pharmaceuticals when the fortified edible oil or any composition
15 thereof is administered to treat a disease or medical disorder. The different diseases and disorders where hydroxytyrosol can be used in the treatment are known to the person skilled in the art.

Examples

The present invention is further illustrated by the following experiments.

Turbidity measurements

- 5 Turbidity in the following examples have been quantified by measuring the nephelometric turbidity units, or in short NTU, using a commercial nephelometer (Hach 2100N IS®, USA). The measurement involves the measurement of the light scattered by a sample in 90° from the incident light path and is performed at room temperature.

10

Determination of amount of hydroxytyrosol

- The amount of hydroxytyrosol in oil was determined after extraction from the sample by water by reversed phase HPLC-UV at 280 nm with hydroxytyrosol as external standard (Column: Thermo Aquasit C18, 150 x 3 mm, 3 m, pressure
15 approx.. 200 bar, 30°C, injection volume 20 µml, mobile Phase: water + 0.05 % methanesulfonic acid / methanol + 0.05 % methanesulfonic acid used in gradient, detection: 280 nm)

20 Oils used

Edible oil	Hydroxytyrosol ¹
Olive oil : extra virgin olive oil (evoo),	41.6 mg/kg
Sunflower oil	n.d. ²
<i>PUFA1</i> : PUFA from fish oil source: MEG-3 '30' N-3 FOOD OIL, DSM Nutritional Products Ltd.	n.d. ²
<i>PUFA2</i> : PUFA from algae source: life's DHA algal vegetable oil, DSM Nutritional Products Ltd.	n.d. ²

Table 1: Edible oils used for the examples.

¹amount of hydroxytyrosol measured in the non-fortified oil, i.e. as bought, indicated as mg hydroxytyrosol per kg oil.

² n.d. = non detectable

Experimental Series 1: Olive oil

100 g of the mentioned olive oil have been mixed using a heating plate equipped with a magnetic stirrer (stirring bar 4.5 cm length and a diameter of 0.6 cm), 600 rpm) with the amount of elaVida™ 40 during the time of mixing t_{mix} at the temperature during mixing T_{mix} as indicated in table 2.

After the mixing, the fortified olive oil phase has been separated by centrifugation (3000 rpm, 15 minutes). The concentration of hydroxytyrosol in the fortified olive oil and the turbidity have been measured and are given in table 2.

Exp.	T_{mix} [°C]	t_{mix} [min]	elaVida™ 40 [mg]	HT _{add} ³ [mg]	Fortification ⁴ [mg HT/kg]	Transfer yield ⁵ [%]	turbidity [NTU]
1	70	45	431	1725	1410	81.7	0.54
2	50	45	250	1000	746	74.6	0.47
3	60	10	625	2500	1400	56.0	5.01
4	75	15	62.5	250	222	88.8	0.48
5	55	40	5625	2500	1900	76.0	1.87
6	85	10	100	400	316	79.0	0.49

Table 2. Fortification of olive oil.

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.

⁵Transfer yield = HT_{add}/Fortification

10

All examples provide clear fortified oils. After 1 month of storage in a closed bottle at room temperature the turbidity was measured to remained unchanged.

15

Experimental Series 2: Sunflower oil

In this series, the experimental series 1 has been repeated with the difference that the mentioned sunflower oil is used instead of the olive oil. Results are compiled in table 3:

Exp.	T_{mix} [°C]	t_{mix} [min]	elaVida™40 [mg]	HT _{add} ³ [mg]	Fortification ⁴ [mg HT/kg]	Transfer yield ⁵ [%]	turbidity [NTU]
7	70	60	550	2200	1811	82.3	0.08
8	80	60	225	900	685	76.1	0.07
9	60	10	625	2500	1180	47.2	0.07
10	75	15	62.5	250	212	84.8	0.10
11	55	40	625	2500	1400	56.0	0.08
12	85	10	100	400	322	80.5	0.08

Table 3. Fortification of sunflower oil.

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil. ⁵Transfer yield = HT_{add}/Fortification

5

All examples provide clear fortified oils. After 1 month of storage in a closed bottle at room temperature the turbidity was measured to remained unchanged. Furthermore, also the amount of hydroxytyrosol remained constant.

10 Experimental Series 3: Olive Oil: Fortification

100 g of the mentioned olive oil have been mixed with an increasing amount of hydroxytyrosol as indicated in table 4 at temperature during mixing T_{mix} of 80°C during time of mixing t_{mix} of 15 minutes.

Exp.	elaVida™40 [mg]	HT _{add} ³ [mg]	Fortification ⁴ [mg HT/kg]	Transfer yield ⁵ [%]	turbidity
13	67	268	238	89%	Clear ⁶
14	348.3	1393	1166	84%	Clear ⁶
15	654.1	2616	2248	86%	Clear ⁶
16	1319.9	5279	3977	75%	Clear ⁶

Table 4. Fortification of olive oil.

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.

⁵Transfer yield = HT_{add}/Fortification ⁶clear = < 15 NTU

Experimental Series 4: concentration effect of hydroxytyrosol in the aqueous solution used

- Aqueous solutions with different concentrations (20%, 10%, 5%, 1%, 0.5% and 0.15 % by weight) of hydroxytyrosol have been prepared by diluting
- 5 elavida™ 40 with the respective amount of water. To 100 g of the above olive oil 25 mg of hydroxytyrosol in form of a respective amount of the aqueous solution of hydroxytyrosol of the indicated concentration as indicated in table 5 at temperature during mixing T_{mix} of 80°C and time of mixing t_{mix} of 15 minutes have been added and mixed, separation by centrifugation.

Exp.	c(HT)² [% by weight]	Solution added [mg]	HT_{add}³ [mg]	Fortification⁴ [mg HT/kg]	Transfer yield⁵ [%]	turbidity [NTU]
17	40	62.5	250	200	80%	0.16
18	20	125	250	222	89%	0.16
19	10	250	250	215	86%	0.15
20	5	500	250	235	94%	0.14
Ref.1	1	2500	250	143	57%	>1000
Ref.2	0.5	5000	250	101	40%	>1000
Ref. 3	0.15	16'667	250	55	22%	830
Ref. 0	-	-	-	41.6 ⁶	-	0.14

Table 5. Fortification of olive oil with solutions of different concentration in HT.

²c(HT) corresponds to the concentration of hydroxytyrosol used as an aqueous solution of hydroxytyrosol

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.

⁵Transfer yield = HT_{add}/Fortification

⁶non-fortified olive oil has a concentration of 39.1 mg HT per 1000 g oil.

10

The experiments in table 5 show that the concentration of hydroxytyrosol in the aqueous solution of hydroxytyrosol is very critical for the suitability for fortification. Below a concentration of 5 % by weight the fortified oil gets very turbid and has a very bad fortification efficiency.

Natural vegetation water resulting from the oil production process has typically a concentration of about 0.15 % by weight in hydroxytyrosol. Reference example **Ref. 3** which corresponds to this concentration of vegetation water shows that such low concentrated aqueous solutions have almost no fortification effect.

5 The same effect has been shown for sunflower oil in table 6 (temperature during mixing T_{mix} of 80°C, time of mixing t_{mix} of 15 minutes, centrifugation).

Exp.	c(HT)² [% by weight]	Solution added [mg]	HT_{add}³ [mg]	Fortification⁴ [mg HT/kg]	Transfer yield⁵ [%]	turbidity [NTU]
21	40	62.5	250	239	96	Clear ⁶
22	20	125	250	248	99	Clear ⁶
23	10	250	250	267	98	Clear ⁶
24	5	500	250	204	82	Clear ⁶
Ref. 4	1	2500	250	151	60	Very turbid ⁷

Table 6. Fortification of sunflower oil with solutions of different concentration in HT.

²c(HT) corresponds to the concentration of hydroxytyrosol used as an aqueous solution of hydroxytyrosol

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.

⁵Transfer yield = HT_{add}/Fortification

⁶clear = < 15 NTU; ⁷very turbid = > 800 NTU

Experimental Series 5: concentration effect of hydroxytyrosol in the aqueous

10 solution used

In a further series the aqueous solution of 0.15 % by weight hydroxytyrosol of experimental series 5 has been added to 100 g of the above mentioned olive oil, respectively sunflower oil, in the amount mentioned in table 7. After mixing at 85 °C and 15 minutes, the fortified oil phase has been separated.

15 In example **Ref. 5** and **Ref. 8** this process has been repeated further 4 times.

Exp.	Solution added ⁱ [g]	HT _{add} ⁱⁱ [mg]	cycles ⁱⁱⁱ	Total HT _{add} ^{iv} [mg]	Fortification ^v [mg HT/kg]	Transfer yield ^{vi} [%]	turbidity
Olive oil							
Ref. 5	16.67	250	5	1250	60	5	Very turbid ^{ix}
Ref. 6	83.36	1250	1	1250	59	5	Very turbid ^{ix}
Ref. 7	100	1500	1	1500	60	4	Very turbid ^{ix}
Ref. 0	-	-	-	-	41.6 ^{viii}	-	Clear
Sunflower oil							
Ref. 8	16.67	250	5	1250	61	5	Very turbid ^{ix}
Ref. 9	83.36	1250	1	1250	59	5	Very turbid ^{ix}
Ref. 10	100	1500	1	1500	66	4	Very turbid ^{ix}
Ref. 00	-	-	-	-	0 ^{viii}	-	Clear

Table 7. Effect of concentration of hydroxytyrosol in the aqueous solution.

ⁱaqueous solution has a concentration of 0.15 % by weight of hydroxytyrosolⁱⁱHT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.ⁱⁱⁱnumber of fortification cycles^{iv}Total of the amount of hydroxytyrosol being added to 1000 g oil = HT_{add}ⁱⁱ*cyclesⁱⁱⁱ^vFortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.^{vi}Transfer yield = HT_{add}/Fortification^{viii}non-fortified oil^{ix}very turbid = > 800 NTU

Starting from comparison example **Ref. 3** in table 5, the results of table 7 confirm that aqueous solutions having low concentration in hydroxytyrosol are not suitable for oil fortification. It is showed that even when large amounts of aqueous solutions of low concentration in hydroxytyrosol are used, they do not yield to significant fortification levels neither when fortification is performed in a single fortification nor in repetitive fortification process.

Fortification of PUFA:

To 100 g of the polyunsaturated fatty acid ester *PUFA1* resp. *PUFA2* have been fortified by mixing with the amount of *elaVida*TM 40 at 30°C during 2 hours to obtain 250mg , 500 mg or 1000 mg hydroxytyrosol per kg fortified oil according to

5 table 8.

Exp.	Oil	<i>elaVida</i>TM40 [mg]	HT_{add}³ [mg]	Fortification⁴ [mg HT/kg]	Transfer yield⁵ [%]	turbidity
25	<i>PUFA1</i>	125	500	413	83%	Clear ⁶
26	<i>PUFA2</i>	62.5	250	249	100%	Clear ⁶
27	<i>PUFA2</i>	125	500	486	97%	Clear ⁶
28	<i>PUFA2</i>	250	1000	832	83%	Clear ⁶

Table 8. Fortification of PUFA.

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

⁴Fortification corresponds to the amount of hydroxytyrosol analyzed in the fortified oil in mg hydroxytyrosol / 1000 g fortified oil.

⁵Transfer yield = HT_{add}/Fortification

⁶clear = < 15 NTU

Mild fortification of PUFA:

In a first step a fortified sunflower oil has been produced by mixing the mentioned sunflower oil and *elaVida*TM 40 at 30°C during 120 minutes as

10 described above. The resulting sunflower oil fortified with hydroxytyrosol comprised 2533 mg hydroxytyrosol per kg fortified oil after separation by centrifugation.

9.8 g of this fortified sunflower oil was mixed with 40.2 g polyunsaturated fatty acid ester *PUFA1* at room temperature. The resulting fortified oil having a
15 ratio of sunflower oil/PUFA of 20/80 showed a content of 495 mg hydroxytyrosol per kg fortified oil.

Increasing hydroxytyrosol levels:

Fortified sunflower oil has been produced by mixing 100 g of the sunflower oil and elaVida™ 40 in amount as indicated in table 9 at 80°C during 15 minutes.

Exp.	elaVida™ 40 [mg]	HT _{add} ³ [mg]	Turbidity [NTU]
29	650	2600	0.64
30	675	2700	1.9
31	700	2800	1.4
32	725	2900	1.7
33	750	3000	1.6
34	775	3100	6.2
35	800	3200	11.9
36	825	3300	11.5
37	850	3400	17.9
38	875	3500	12.5
39	900	3600	17.4
40	925	3700	27

Table 9. Fortification of PUFA.

³HT_{add} corresponds to the amount of hydroxytyrosol being added to 1000 g oil.

5 Mayonnaise (manufactured by employing an elaVida fortified oil):

375 grams of sunflower oil fortified by hydroxytyrosol, example **8**, the HT fortified oil produced as indicated in the experimental series 2 respectively the unfortified sunflower oil, was employed in order to produce 500 g of mayonnaise (ingredients listed in table 10).

Ingredients	Amount [wt.%]	Amount [wt.%]
	Ref.11	41
Water	12.5	12.5
NaCl	0.6	0.6
Liquid egg yolk (unsalted)	6.9	6.9
Sunflower oil (non fortified)	75	
8 (fortified sunflower oil)		75
Vinegar (4.5% acid)	5	5

Table 10. Ingredients for mayonnaise.

Both mayonnaises (**Ref.11, 41**) were produced the same way by employing the here below described procedure:

- The salt was dissolved into the water (warmed up at 50°C)
- The liquid pasteurized unsalted egg yolk was weighted directly into a 500 ml plastic vessel and the salted water was added. The water phase was manually stirred by employing a whip.
- Half of the oil amount was added slowly under stirring (Kenwood hand blender triblade HBM710 700W / turbo power) condition.
- Then, always under stirring conditions, the vinegar was added.
- Subsequently the rest of the oil was gradually added, always under stirring conditions.

The mayonnaises were eventually poured into glass vials and stored at 25°C, respectively at 4°C. Over time (up to 6 months) the color (values L^* , a^* and b^* have been measured on a LICO 200 (Dr. Lange) according to the CIE- $L^*a^*b^*$ method). The values measured after preparation are represented in tables 11 and 12 as L_o^* , resp. a_o^* , resp. b_o^* .

After the time t indicated in tables 11 and 12 the respective values of L_t^* , resp. a_t^* , resp. b_t^* , have been measured accordingly. The values measured are represented in tables 11 and 12.

The color difference ΔE_t^* was calculated from the corresponding values using the formula

$$\Delta E_t^* = \sqrt{(L_o^* - L_t^*)^2 + (a_o^* - a_t^*)^2 + (b_o^* - b_t^*)^2}$$

	Ref.11				41			
Time of storage t [months]	L_t^*	a_t^*	b_t^*	ΔE_t^*	L_t^*	a_t^*	b_t^*	ΔE_t^*
0	96.97	-2.18	10.73		95.75	-2.10	10.54	
1	95.25	-1.81	10.82	1.76	96.65	-1.85	10.81	0.97
3	93.97	-1.69	10.73	3.04	95.92	-1.51	10.31	0.66
6	96.41	-1.57	10.10	1.04	95.59	-1.24	9.32	1.50

Table 11 Color change of **Ref.11** and **41** stored at 4°C measured by CIE- $L^*a^*b^*$ method .

	Ref.11				41			
Time of storage t [months]	L_t^*	a_t^*	b_t^*	ΔE_t^*	L_t^*	a_t^*	b_t^*	ΔE_t^*
0	96.97	-2.18	10.73		95.75	-2.10	10.54	
1	95.08	-1.76	10.39	1.97	96.65	-1.56	10.12	1.24
3	95.72	-1.50	10.50	1.80	95.92	-1.21	9.81	2.26
6	95.59	-1.86	96.	1.46	95.59	-1.65	10.59	1.20

Table 12 Color change of **Ref.11** and **41** stored at 25°C measured by CIE-L*a*b* method.

From the values of tables 11 and 12 can be clearly observed that the mayonnaises color does not change significantly over time (ΔE values over 10 indicates a visible color difference, below that thresholds differences are almost imperceptible) and for both the storage temperatures studied. Moreover, the hydroxytyrosol content does not significantly decrease over time, showing an excellent stability, even when employed as a mayonnaise fortification ingredient.

Furthermore, the content of hydroxytyrosol c_t in the samples of which the color has been measured, has been determined by HPLC analysis and compared to the initial amount c_0 (i.e. before storage). The recovery ($=c_t/c_0$) found has been indicated in table 13.

Time of storage t [months]	41	41
Temperature of storage	4°C	25°C
	Recovery [%]	Recovery [%]
0		
1	99.5	97.0
3	99.5	95.9
6	98.1	95.1

Table 13 Recovery of hydroxytyrosol after storage at 4 °C or 25°C.

Claims

1. Process of manufacturing edible oils fortified with hydroxytyrosol comprising the steps
 - a) providing an edible oil;
 - 5 b) providing an aqueous solution of hydroxytyrosol in which the amount of hydroxytyrosol is at least 5%, particularly at least 20 %, preferably at least 30 %, by weight;
 - c) heating up to a temperature of between 25 and 95 °C;
 - d) mixing said edible oil with said aqueous solution of hydroxytyrosol to
10 form a two phase mixture; followed by
 - e) separating the fortified edible oil as oil phase from the two-phase mixture.
2. Process according to claim 1, characterized in that the edible oil is selected
15 from the group consisting of olive oil, sunflower oil, polyunsaturated fatty acid ester, corn oil, safflower oil, almond oil, palm oil, soybean oil and rapeseed oil.
3. Process according to anyone of the preceding claims, characterized in that
20 the amount of hydroxytyrosol in the fortified edible oil is more than 250 mg /kg fortified oil, particularly more than 400 mg / kg fortified oil, particularly more than 1000 mg/kg fortified oil, preferably more than 2500 mg/kg fortified oil.
- 25 4. Process according to anyone of the preceding claims, characterized in that the aqueous solution of hydroxytyrosol comprises less than 0.18 %, preferably less than 0.1 %, particularly less than 0.01 %, by weight of oleuropein.
- 30 5. Process according to anyone of the preceding claims, characterized in that the mixing of the edible oil with the aqueous solution of hydroxytyrosol is so that the value v_{mix} calculated according to the following formula

$$v_{mix} = t_{mix} * (T_{mix})^2$$

is in the range of between 70'000 and 250'000 $\text{min}(\text{°C})^2$, wherein t_{mix} represents the time of mixing in minutes and wherein T_{mix} represents the temperature during mixing in Celsius (°C).

- 5 6. Process according to anyone of the preceding claim, characterized in that the edible oil fortified with hydroxytyrosol has a turbidity of less than 15 NTU, particularly less than 10 NTU, preferably less than 3 NTU, measured by using a nephelometer (Hach 2100N IS®, USA, angle of measurement: 90°) at room temperature
- 10
7. Edible oil fortified with hydroxytyrosol having an amount of more than 250 mg hydroxytyrosol / kg fortified oil, particularly more than 1000 mg hydroxytyrosol / kg fortified oil, characterized in that the turbidity of the fortified oil is less than 15 NTU, particularly less than 10 NTU, preferably less than 3 NTU, measured by using a nephelometer (Hach 2100N IS®, USA, angle of measurement: 90°) at room temperature.
- 15
8. Capsule for oral consumption, comprising between 0.2 mL and 2.5 mL of an edible oil which is fortified with hydroxytyrosol, characterized in that the amount of hydroxytyrosol in the fortified edible oil is more than 1000 mg /kg fortified oil, particularly between 1000 and 4000 mg / kg fortified oil, preferably between 2000 and 4000 mg / kg fortified oil; and that the edible oil fortified with hydroxytyrosol comprises less than 1 g water / kg fortified oil.
- 20
9. Capsule according to claim 8, characterized in that the capsule is a gel capsule, particular a hard gel capsule.
- 25
10. Process of fortification of an edible oil with hydroxytyrosol comprising the steps
- 30
- i) providing a non-fortified edible oil;
 - ii) providing an edible oil fortified with hydroxytyrosol according to a process of fortification according to anyone of the claims 1 to 6;

iii) blending said non-fortified edible oil and said edible oil fortified with hydroxytyrosol.

- 5 11. Process according to claim 10, characterized in that the non-fortified edible oil is a polyunsaturated fatty acid ester.
12. Process according to claim 10 or 11, characterized in that the step iii) of blending is done at temperatures of less than 60°C, particularly at a temperature of between 25°C and 50°C.
- 10 13. A nutritional product comprising an edible oil fortified according to claim 7 or prepared by a process according to anyone of the claims 1 to 6 or by a process according to anyone of the claims 10 to 12.
- 15 14. Nutritional product according to claim 13, characterized in that the nutritional product is a water-in-oil or an oil-in-water food emulsion, particularly a margarine or a mayonnaise or a sauce or a dressing.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2015/078179

A. CLASSIFICATION OF SUBJECT MATTER

INV. A23D7/005 A23D9/007 C11B5/00
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 C11B

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, BIOSIS, COMPENDEX, WPI Data

C. 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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X	WO 2010/000373 A1 (LACHIFARMA S R L LAB CHIMICO F [IT]; CISALE FELICIA [IT]; VILLANOVA LU) 7 January 2010 (2010-01-07) claims 1-6; examples 1-2	7,13,14
X	US 2013/005682 A1 (RAEDERSTORFF DANIEL [FR] ET AL) 3 January 2013 (2013-01-03) examples 1-7	1-9,13, 14
	----- -/-	



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

1 March 2016

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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2015/078179

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	CONCEPCIÓN ROMERO ET AL: "Analysis of Total Contents of Hydroxytyrosol and Tyrosol in Olive Oils", JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY, vol. 60, no. 36, 12 September 2012 (2012-09-12), pages 9017-9022, XP055187664, ISSN: 0021-8561, DOI: 10.1021/jf3026666 page 9020, column 2 -----	1-14
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2015/078179

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