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**(54) MICROEMULSIONS FOR USE IN FOOD AND BEVERAGE PRODUCTS**

MIKROEMULSIONEN FÜR DIE VERWENDUNG IN NAHRUNGSMITTEL- UND GETRÄNKEPRODUKTEN

MICRO-EMULSIONS DESTINEES À UN USAGE DANS DES PRODUITS ALIMENTAIRES ET DES BOISSONS

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(72) Inventor: **CHANAMAI, Ratjika Erlanger, KY 41017 (US)**

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(74) Representative: **Samson & Partner Widenmayerstraße 5 80538 München (DE)**

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(73) Proprietor: **Wild Flavors, Inc. Erlanger KY 41018 (US)**

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**Description**

## CROSS-REFERENCE TO RELATED APPLICATION

5 **[0001]** The present application relates to and claims priority from US Provisional Application No. 60/727,122, filed October 14, 2005.

## TECHNICAL FIELD

10 **[0002]** The present application relates to microemulsions which are effective for incorporating water-insoluble components into aqueous-based food and beverage compositions or water-soluble components into lipid-based food compositions.

## BACKGROUND OF THE INVENTION

15 **[0003]** The formulation of food and beverage products, particularly aqueous-based food and beverage products, can be difficult. For example, it is frequently necessary to incorporate water immiscible components, such as colors, flavors, nutrients, nutraceuticals, therapeutic agents, or antioxidants, into compositions which are primarily aqueous based. The difficulty of this task is increased by the fact that the compositions need to be formulated such that they are esthetically  
20 pleasing to the consumer. For example, it is frequently necessary to incorporate a water-insoluble material into an aqueous beverage while still maintaining the optical clarity of the beverage. These compositions also need to exhibit long-term shelf stability under typical food and beverage shipping, storage and use conditions.

**[0004]** One way that the industry has attempted to satisfy these conflicting requirements is to incorporate the water immiscible materials using microemulsions. A microemulsion is a dispersion of two immiscible liquids (one liquid phase  
25 being "dispersed" and the other being "continuous") in which the individual droplets of the dispersed phase have an average radius less than about one-quarter the wavelength of light. Such microemulsions have also been termed "nanoemulsions". Typically, in a microemulsion, the dispersed phase droplets have a radius of less than about 1400 Å, and preferably on the order of about 100 to about 500 Å. The basic theory of microemulsions is more fully described in Rosano, Journal of the Society of Cosmetic Chemists, 25: 609-619 (November, 1974).

30 **[0005]** Microemulsions can be difficult to formulate, frequently requiring the use of co-solvents, such as ethanol or propylene glycol. These co-solvents can lead to off-flavors in the final product. Further, the formation of microemulsions frequently requires some rather stressful processing conditions, such as high pressure homogenization, which require specialized equipment and can increase the cost of the final product.

35 **[0006]** It therefore would be useful to have a procedure for formulating microemulsions, using relatively low levels of food grade emulsifiers, which allow the incorporation of water-immiscible components into aqueous-based food and beverage compositions without requiring the use of such co-solvents and relatively extreme processing conditions.

**[0007]** The prior art describes the formation of microemulsions, as well as the use of microemulsions formed by conventional processes for the incorporation of materials into food and beverage products.

40 **[0008]** -U.S. Patent 4,146,499, Rosano, issued March 27, 1979, describes an oil-in-water microemulsion which utilizes a high/low HLB surfactant mixture for forming the emulsion. The patent does not teach or suggest use of a ternary (high/low/medium HLB) surfactant system in forming the emulsion.

45 **[0009]** U.S. Patent 4,752,481, Dokuzovic, issued June 21, 1988, describes a flavored chewing gum product which includes a chewing gum base, a sweetener, and a flavor-containing emulsion. The emulsion comprises 19 to 59% of a flavoring oil, 1 to 5% of an emulsifier having an HLB of from about 1.6 to about 7.0, and an alkyl polyol (for example, glycerin or polyethylene glycol).

50 **[0010]** U.S. Patent 4,835,002, Wolf et al., issued May 30, 1989, describes a microemulsion of an edible essential oil (such as citrus oil) in a water/alcohol matrix. The composition comprises water, the essential oil, alcohol and a surfactant. The surfactant component utilized must include a high HLB surfactant, although a mixture of high HLB and low HLB surfactants can also included. There is no disclosure of a ternary surfactant emulsifier system for use in forming the emulsion.

55 **[0011]** U.S. Patent 5,320,863, Chung et al, issued June 14, 1994, describes microemulsions used to deliver high concentrations of flavor or fragrance oils. The compositions are said to exhibit high stability even in the absence of lower alcohols. The compositions include a nonionic surfactant (generally not edible or food grade); no discussion of HLB criticality is provided. There is no disclosure or suggestion to combine high, low and medium HLB surfactants into a ternary emulsifying system.

**[0012]** U.S. Patent 5,447,729, Belenduik et al, issued September 5, 1995, describes a particulate pharmaceutical composition wherein a pharmaceutical active material may be incorporated into particles in the form of a microemulsion. The outer layers of the particles have hydrophobic/lipophilic interfaces between them. The disclosed compositions can

include polysorbate 80 or glycerol monooleate as an emulsifier. There is no teaching in the patent of a ternary surfactant emulsifier system.

5 [0013] U.S. Patent 5,948,825, Takahashi et al., issued September 7, 1999, describes water-in-oil emulsions of hard-to-absorb pharmaceutical agents for use in topical or oral administration. There is no disclosure or suggestion of a ternary surfactant emulsifier system. The emulsifiers disclosed in the '825 patent can include a mixture of two types of nonionic surfactants, one having an HLB of from 10 to 20, and the other having an HLB from 3 to 7.

[0014] U.S. Patent 6,048,566, Behnam et al., issued April 11, 2000, describes a nonalcoholic, clear beverage which incorporates from 10 to 500 mg/l of ubiquinone Q10, together with a polysorbate stabilizer.

10 [0015] U.S. Patent 6,077,559, Logan et al., issued June 20, 2000, relates to flavored vinegars which are based on the inclusion of specifically defined microemulsions. The oil-in-vinegar microemulsions comprise from 20% to 70% vinegar, 5% to 35% ethanol, 0.1% to 5% of a flavor material, and 0.5% to 5% of a surfactant. The surfactants utilized are high HLB surfactants; they can also include a small amount of low HLB (4 to 9) surfactant. There is no disclosure of a ternary surfactant emulsifier system in the '559 patent.

15 [0016] U.S. Patent 6,146,672, Gonzalez et al., issued November 14, 2000, relates to spreadable water-in-oil emulsions which are used as fillings in pastry products, particularly frozen pastries. The fillings are said to exhibit enhanced shelf-life and stability. The described emulsions include a mixture of high and low HLB emulsifiers. Although the '672 patent describes a mixture of high and low HLB surfactants, it does not disclose or suggest the ternary surfactant emulsifier system which is utilized in the present invention. Further, the '672 patent does not teach microemulsions or the use of an emulsion to incorporate water-insoluble materials into food products.

20 [0017] U.S. Patent 6,303,662, Nagahama et al., issued October 16, 2001, describes microemulsions used in the delivery of fat-soluble drugs. The disclosed compositions require a high polarity oil, a low polarity oil, a polyglycerol mono fatty acid ester, and a water-soluble polyhydric alcohol. There is no disclosure of a ternary surfactant emulsifier system.

25 [0018] U.S. Patent 6,376,482, Akashe et al., issued April 23, 2002, describes mesophase-stabilized compositions which incorporate plant sterols as cholesterol-lowering agents. The compositions can include a mixture of a surfactant having an HLB of from 6 to 9, a surfactant having an HLB of from 2 to 6, and a surfactant having an HLB of from 9 to 22. Although this patent does teach a ternary emulsifier system, the product formed is not a microemulsion, but rather a mesophase-stabilized emulsion (the mesophase does not have separate oil and water phases). The disclosed compositions are said to provide mouth feel and texture benefits to food products. The emulsion particles formed in the '482 patent are relatively large (i.e., from about 2 to about 10  $\mu\text{m}$ ).

30 [0019] U.S. Patent 6,426,078, Bauer et al., issued July 30, 2002, describes oil-in-water microemulsions which comprise from 10% to 99% of a triglycerol mono fatty acid emulsifier (for example, triglycerol monolaurate, triglycerol monocaproate or triglycerol monocaprylate), 1% to 20% of a lipophilic substance (for example, beta-carotene, vitamin A or vitamin E), and water. These compositions are said to be useful in foods, cosmetics or pharmaceuticals for incorporating non-water-soluble (lipophilic) substances. There is no disclosure of a ternary surfactant emulsifier system for forming the microemulsion.

35 [0020] -U.S. Patent 6,444,253, Conklin et al, issued September 3, 2002, describes a microemulsion flavor delivery system in the form of an oil-in-alcohol composition. These compositions require the use of alcohols which generally are not included in typical food or beverage formulations. Further, the '253 patent does not teach or suggest a ternary surfactant emulsifier system.

40 [0021] U.S. Patent 6,509,044, Van Den Braak et al., issued January 21, 2003, describes microemulsions of beta-carotene. These microemulsions are said to be based on an emulsifier system which preferably is a binary surfactant system, but can also be a ternary system (although there are no examples of a ternary system provided). It is taught that the fatty acid profiles of the emulsifiers are matched with the fatty acid profiles of the oily ingredient to be incorporated into the composition. There is no teaching in the '044 patent of a ternary high/low/medium HLB surfactant emulsifier system for use in forming the microemulsion.

45 [0022] U.S. Patent 6,774,247, Behnam, issued August 10, 2004, relates to aqueous ascorbic acid solutions. These solutions are said to contain an excess of an emulsifier having an HLB of from about 9 to about 18, such as polysorbate 80. There is no suggestion in the '247 patent to utilize a ternary surfactant emulsifier system.

50 [0023] U.S. Published Patent Application 2002/0187238, Vlad, published December 12, 2002, relates to clear, stable oil-loaded microemulsions used as flavoring components in clear beverage compositions. These compositions utilize a co-solvent at a co-solvent:surfactant ratio of about 1:1. Further, the surfactant component comprises a mixture of at least two surfactants having an average HLB of from about 9 to about 18, preferably from about 12 to about 15. There is no disclosure in the '238 application of a ternary surfactant emulsifier composition comprising a mixture of low/medium/high HLB surfactants. The microemulsions defined in the '238 application comprise at least 30% oil, 1% to 30% of a surfactant mixture having an HLB of from 9 to 18, less than 20% co-solvent, and at least 35% water.

55 [0024] PCT Published Patent Application WO 94/06310, Ford et al., published March 31, 1994, describes a colorant composition in the form of a microemulsion. Compositions disclosed in the '310 application include beta-carotene, alpha-tocopherol and ascorbic acid. Polysorbates are preferred emulsifiers in the '310 application. There is no teaching of a

ternary surfactant emulsifier system in the formation of the microemulsion.

5 [0025] US 2003/087967 A discloses a nanoemulsion comprising an oily phase dispersed in an aqueous phase which contains a ternary surfactant system that comprises ionic surfactants chosen from alkali metal salts of cetyl sulfate and alkali metal salts of palmitoyl sarcosinate. US 6,068,876 A describes mesophase-stabilized emulsions that are prepared using three different emulsifiers. The emulsions have an oil droplet size in the micron range. The abstract of JP 2001 200069 discloses meso-phase-stabilized emulsions and dispersions the droplet size of which is in the micron range.

#### SUMMARY OF THE INVENTION

10 [0026] The present invention relates to microemulsions used to incorporate lipophilic water-insoluble materials into food and beverage compositions, comprising:

- 15 (a) an oil phase as defined in claim 1;
- (b) an aqueous phase; and
- (c) a food grade emulsifier system as defined in claim 1;

wherein said oil phase is dispersed as particles having an average diameter of from 1 to 300 nm, within said aqueous phase, the micro emulsion having the further features as defined in claim 1.

20 [0027] The present invention also encompasses food compositions and beverage compositions which incorporate the microemulsions defined above.

[0028] The present invention also relates to a method for preparing the microemulsions defined above, comprising the steps of:

- 25 (a) mixing the lipophilic water-insoluble components with the low HLB emulsifier to form the oil phase;
- (b) mixing the emulsifier system into the oil phase; and
- (c) adding the aqueous phase into the product of step (b) and mixing to form the microemulsion.

30 [0029] Finally, the present invention relates to water-in-oil microemulsions using the ternary emulsifier system as defined in claim 7 and concentrates used for making oil-in-water and water-in-oil microemulsions.

[0030] The microemulsions of the present invention provide several advantages over conventional compositions. Specifically, the microemulsions of the present invention can carry effective levels of difficult-to-disperse components, such as carotenoids, in optically transparent beverages. The compositions of the present invention are sufficiently stable under normal soft drink transport and storage conditions. The taste of the food and beverage products of the present invention is very acceptable.

35 [0031] The physical and optical characteristics of the emulsions are controllable by selection of appropriate emulsifiers and the heating temperature used, as well as the order of addition of the components. Importantly, the microemulsions of the present invention form essentially spontaneously under normal stirring, without requiring extreme processing conditions, such as high-pressure homogenization. Finally, the microemulsions of the present invention can demonstrate improved bioavailability of the dispersed elements, such as carotenoids.

40 [0032] With the present invention it is also possible to prepare oil-in-water microemulsions containing omega-3 fatty acids or their esters that are highly susceptible to oxidation (or other acids/esters which are highly susceptible to oxidation). It is observed that such components exhibit higher oxidative stability in microemulsions of the present invention than in conventional emulsions.

45 [0033] All ratios and proportions described in this application are intended to be "by weight," unless otherwise specified.

#### DETAILED DESCRIPTION OF THE INVENTION

50 [0034] The present invention provides for microemulsions which are easily formed and which allow for the incorporation of water immiscible components into aqueous-based food and beverage compositions. Similarly, the microemulsions can be used to incorporate water-soluble materials into fat-based products. For example, water-soluble natural colorants, flavors, vitamins, salts or antioxidants can be incorporated into fat-based products like coating layers on a snack bar, frosting, chocolate, margarine, fat spread or confectionary products.

55 [0035] The water-insoluble components which may be incorporated into the food and beverage compositions of the present invention encompass any materials which are desirably incorporated into a food or beverage product, but which are insoluble in or immiscible with an aqueous-based composition. Such materials generally are lipophilic. Examples of such materials include certain colorants, flavorants, nutrients, nutraceuticals, therapeutic agents, antioxidants, extracts of natural components (such as plants, roots, leaves, flowers, etc.), medicaments, preservatives, and mixtures of these

materials. Specific examples of such materials which are frequently used in food and beverage compositions include the following: carotenoids and their derivatives (such as beta-carotene, apocarotenal, lutein, lutein ester, lycopene, zeaxanthin, crocetin, astaxanthin), essential oils, edible oils, fatty acids, proteins and peptides, polyunsaturated fatty acids and their esters, vitamin A and its derivatives, vitamin E and its derivatives, vitamin D and its derivatives, vitamin K and its derivatives, colorants, flavorants, nutrients, nutraceuticals, therapeutic agents, antioxidants, extracts of natural components (such as plants, roots, leaves, flowers, seeds, etc.), medicaments, preservatives, lipoic acid, phytosterins, quercetin, phytosterols and their esters, co-enzyme Q10 (ubidecarone), plant isoflavones (such as genistein, isogenistein or formononetine), and mixtures thereof.

**[0036]** Particularly preferred materials which can be incorporated using the present invention include, for example, oil-soluble, oil-insoluble or water-soluble food ingredients, such as food additives, food preservatives, food supplements, antioxidants, nutraceuticals, cosmoceuticals, plant extracts, medicaments, fatty acids, peptides, proteins, carbohydrates, natural flavors, synthetic flavors, colorants, vitamins, and combinations of those materials. The specific microemulsion systems of beta-carotene, vitamin E, vitamin A materials, such as vitamin A palmitate, vitamin E acetate, and mixtures of those components are given as examples of this invention.

**[0037]** A key element for forming the microemulsions of the present invention is the ternary surfactant emulsifier system. It is through the use of this ternary system that microemulsions which provide the benefits of the present invention are formed. This ternary emulsifier system is a mixture of at least three food grade emulsifiers in the form of nonionic or anionic surfactants. Nonionic surfactants are preferred. Nonionic surfactants are well known in the art and are described, for example, in *Nonionic Surfactants: Organic Chemistry*, Nico M. van Os (ed.), Marcel Dekker, 1998. At least one of the emulsifiers has a low HLB (i.e., from 1 to 5), at least one of the emulsifiers has a medium HLB (i.e., from 6 to 8), and at least one of the emulsifiers has a high HLB (i.e., from 9 to 17 in w/o microemulsions from 10 to 16 in o/w microemulsions).

**[0038]** The selection of the particular surfactants used in the ternary emulsifier system depends on the HLB (hydrophilic-lipophilic balance) value of such surfactants. The surfactants are selected such that they have the HLB values described above. The HLB value, and the determination thereof, for surfactants is well known in the art and is disclosed, for example, by Milton J. Rosen in *Surfactants and Interfacial Phenomena*, J. Wiley and Sons, New York, New York, 1978, pages 242-245, or in the *Kirk-Othmer Encyclopedia of Chemical Technology*, 3rd edition, volume 8, 1979, at pages 910-915.

**[0039]** The following table sets forth the HLB values for a variety of anionic and nonionic surfactants which can, as examples, be used in the ternary system of the present invention. The HLB of other non-listed surfactants can be calculated using procedures well known in the art.

Anionic Surfactant	HLB Value*
myristic acid	22
palmitic acid	21
stearic acid	20
oleic acid	20
monoglyceride ester of diacetyltartaric acid	9.2
diglyceride ester of diacetyltartaric acid	3.2
monoglyceride ester of citric acid + and salts thereof	27
diglyceride ester of citric acid	20
monoglyceride ester of lactic acid	21
diglyceride ester of lactic acid	14
dioctyl sodium sulfosuccinate	18
monoglyceride ester of phosphoric acid	14
diglyceride ester of phosphoric acid	8
lecithin	7 to 9

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(continued)

	<u>Anionic Surfactant</u>	<u>HLB Value*</u>
5	hydroxylated lecithin**	8 to 9
	<u>Nonionic Surfactants</u>	
	polysorbates	10 to 18
	sorbitan ester of myristic acid	6.7
10	sorbitan ester of palmitic acid	5.7
	sorbitan ester of stearic acid	4.7
	sorbitan ester of oleic acid	4.7
15	polyglycerol esters of myristic acid	3 to 16 depending on the number of glycerol units and fatty acid side chains present therein
	polyglycerol esters of palmitic acid	
	polyglycerol esters of stearic acid	
	polyglycerol esters of oleic acid	
20	monoglyceride ester of myristic acid	4.8
	monoglyceride ester of palmitic acid	4.3
	monoglyceride ester of stearic acid	3.8
25	monoglyceride ester of oleic acid	3.1
	diglyceride ester of myristic acid	2.3
	diglyceride ester of palmitic acid	2.1
	diglyceride ester of stearic acid	1.8
30	diglyceride ester of oleic acid	1.8
	(ethoxy) <i>n</i> monoglyceride of myristic acid***	13 to 21
35	(ethoxy) <i>n</i> monoglyceride of palmitic acid***	
	(ethoxy) <i>n</i> monoglyceride of stearic acid***	
40	(ethoxy) <i>n</i> monoglyceride of oleic acid***	
	(ethoxy) <i>n</i> diglyceride of myristic acid***	7 to 15
45	(ethoxy) <i>n</i> diglyceride of palmitic acid***	
	(ethoxy) <i>n</i> diglyceride of stearic acid***	
	(ethoxy) <i>n</i> diglyceride of oleic acid***	
50	sucrose ester of myristic acid	18
	ester of palmitic acid	17
	ester of stearic acid	16
	ester of oleic acid	16
55	propylene glycol ester of myristic acid	4.4
	ester of palmitic acid	3.9

(continued)

Nonionic Surfactants	
ester of stearic acid	3.4
ester of oleic acid	4.3
*in fully ionized form in water at 20-25°C. **amphoteric depending on pH of matrix ***wherein n is a whole number from 10 to 30	

**[0040]** Any edible oil may be used as the oil phase in the present compositions. Specifically, the oil phase can be selected from edible fat/oil sources, such as the oil extracts from natural components (e.g., plants, flowers, roots, leaves, seeds). For example, these materials can include carrot seed oil, sesame seed oil, vegetable oil, soybean oil, corn oil, canola oil, olive oil, sunflower oil, safflower oil, peanut oil, or algae oil. Also included are flavor oils, animal oils (such as fish oils), and dairy products (such as butterfat). The oil phase can be made from pure oil, mixtures of different oils, or a mixture of different oil-soluble materials, or mixtures thereof.

**[0041]** In the oil-in-water microemulsions of the present invention the low HLB surfactant is present at from 0.1 % to 5%, particularly 0.7% to 1%, of the microemulsion. The high HLB surfactant is present at from 5% to 25%, particularly from 12% to 18%, of the microemulsion. The medium HLB surfactant is present at from 0.1% to 5%, particularly from 0.5% to 1.5% of the microemulsion. Particularly preferred low HLB surfactants include glycerol monooleate, polyglycerol riconoleate, decaglycerol decaoleate, sucrose erucate and sucrose oleate. Particularly preferred medium HLB surfactants are polyglycerol esters, such as decaglycerol hexaoleate, and triglycerol monofatty acids, such as triglycerol monooleate, and sucrose stearate. Particularly preferred high HLB surfactants include polysorbate 80 or polyoxysorbitan monolaurate (commercially available as the TWEEN® series of surfactants), polyglycerol-6 laurate, decaglycerol lauric acid esters, decaglycerol oleic acid esters and sucrose esters.

**[0042]** In one embodiment of the microemulsions of the present invention, the oil phase is dispersed within the aqueous phase (i.e., an oil-in-water (o/w) microemulsion). The oil phase is present in particulate form, having a particle size mean diameter of from 1 to 300 nm, preferably from 1 to 200 nm. The aqueous phase typically comprises water and the water-soluble ingredients of the composition, and is present at from 50% to 90%, preferably from 70% to 85%, of the microemulsion. The oil phase generally comprises from 1% to 15%, preferably from 2% to 6%, of the microemulsion. Typically, the oil phase includes the water-insoluble components, as they have been defined above, together with the low HLB emulsifier component.

**[0043]** This oil-in-water microemulsion of the present invention, described above, can be formulated in a relatively simple manner as follows. The lipophilic water-insoluble components are mixed with the low HLB emulsifier to form the oil phase. Heat may be applied, if necessary, to melt the insoluble components and/or the surfactant to form the oil phase. The emulsifier system, which comprises the high HLB and the medium HLB emulsifiers is then formed and mixed into the oil phase. The aqueous phase is then added into the previously made (oil phase/emulsifier) mixture and further mixed to form the microemulsion. The mixing which is required to form the microemulsion is relatively easy mixing. Typical equipment which can be used to mix the components to form the microemulsion include, for example, a magnetic stirrer or an overhead mixer.

**[0044]** In selecting the emulsifiers utilized in the microemulsions of the present invention, the following criteria may also be important. In w/o microemulsions, the high HLB emulsifier has an HLB value between 9 and 17, in o/w microemulsions between 10 and 16. Without wishing to specify a particular mechanism of action of the emulsifiers, it may be advantageous to use emulsifiers with relatively bulky head groups and non-bulky tails selected as to their length so they can form micelles readily. This is the major emulsifier which confers water-soluble characteristics to the system. The hydrophilic portions of the molecule repel each other sideways to curve the interface around the oil side and promote the formation of the oil-in-water microemulsions. The low HLB emulsifier must be lipophilic and have an HLB value between 1 and 5. This minor emulsifier stays within the oil phase and acts as a co-surfactant. The emulsifier molecules align their heads and tails in nearly a perfect way with the oil and the first hydrophilic surfactant to promote formation of micelles as small as possible. The third emulsifier has a medium HLB between about 6 and about 8. This minor emulsifier can stay in either the water or oil phase and also acts as a co-surfactant. It is believed that this emulsifier not only further reduces the interfacial tension between droplets, but also tends to bend the interface to make the droplets smaller. The combination of the very low interfacial tension, long hydrophobic tails of the first emulsifier and close packing, and the effect of the co-surfactants on the curvature of the interface provides a dispersed and stable system of small droplet size. Examples of food grade surfactants which can be used in the microemulsions of the present invention include polysorbates (ethoxylated sorbitan esters), such as polysorbate 80; sorbitan esters, such as sorbitan monostearate; sugar esters, such as sucrose laurate; polyglycerol esters of fatty acids (from mono-, di-, tri-, and up to deca-, glycerol

esters of fatty acids), mono and diglycerides, combinations of fatty acids and ethoxylated monodiglycerides, and mixtures thereof.

5 [0045] In addition to the oil-in-water microemulsions described above, the present invention also encompasses water-in-oil (w/o) microemulsions. These are particularly useful for incorporating water-soluble materials into oil- or fat-based compositions. In these water-in-oil microemulsions, the aqueous phase is dispersed in the oil phase. The aqueous phase is present in particulate form, having a particle size mean diameter of from 1 to 300 nm, preferably from 1 to 200 nm. The aqueous phase typically comprises water and the water-soluble ingredients of the composition, and is present at from 1% to 15%, preferably from 2% to 6%, of the microemulsion. The oil phase includes the water-insoluble components and the oily/fatty base, and is present at from 50% to 90%, preferably from 70% to 85%, of the microemulsion. In forming these water-in-oil microemulsions, the water-soluble components are mixed with the high HLB emulsifier to form the aqueous phase. The low HLB and medium HLB emulsifiers are then mixed together and added to the aqueous phase. The oil phase is then added to the aqueous phase with mixing, for example, with an overhead mixer to form the water-in-oil microemulsion. Typically, in water-in-oil microemulsions, the high HLB surfactant is present at from about 0.1% to about 5%, the medium HLB surfactant is present at from 0.1% to 5%, and the low HLB surfactant is present at from 5% to 30%, of the final composition.

15 [0046] Physical properties of the microemulsion composition, and the final product, can be adjusted by increasing or decreasing the amount of oil or water in the dispersed phase of the microemulsion.

20 [0047] Finally, the present invention encompasses concentrate microemulsion systems which comprise the dispersed phase (including the component(s) which is (are) to be incorporated into the final composition) and the three emulsifiers defined herein; the concentrate does not include the continuous phase. The concentrate is added to the continuous phase, with stirring, and the microemulsion is formed. Thus, in a concentrate to form an oil-in-water microemulsion, there will be included an oilbased phase of selected lipid-soluble ingredients, together with the ternary emulsifier system, with no aqueous phase. This concentrate is added to an aqueous phase, with mixing, to form the oil-in-water microemulsion. On the other hand, for a concentrate to form a water-in-oil microemulsion, there will be an aqueous phase of particular water-soluble ingredients, together with the ternary emulsifier system, with no oil phase. Examples of such concentrates are described in this application. These concentrates that will form oil-in-water microemulsions comprise from 1% to 40% of the disperse phase, and from 1% to 10% of the low HLB emulsifier, from 1% to 10% of the medium HLB emulsifier, and from 65% to 95% of the high HLB emulsifier. These concentrates that will form water-in-oil microemulsions comprise from 1% to 40% of the dispersed phase, and from 65% to 95% of the low HLB emulsifier, from 1% to 10% of the medium HLB emulsifier, and from 1% to 10% of the high HLB emulsifier. The concentrate is added, with mixing, to the continuous phase such that the final microemulsion composition comprises from 1% to 15% (preferably from 2% to 6%) of the dispersed phase, and from 50% to 99% (preferably from 70% to 85%) of the continuous phase.

25 [0048] The microemulsions of the present invention may be incorporated into aqueous-based or lipid-based food and beverage products. These products are conventional and are well known in the art. Examples and information about the formulation of such products may be found in the Encyclopedia of Food Sciences and Nutrition, by Benjamin Caballero, Luis C. Trugo and Paul M. Finglas (editors), 2nd Edition, London: Academic, 2003, or in the Dictionary of Food Compounds with CD-ROM: Additives, Flavors and Ingredients, edited by Shmuel Yannai, Boca Raton, Fla., CRC Press, 2004, or in The Soft Drinks Companion; A Technical Handbook for the Beverage Industry, by Maurice Shachman, Boca Raton, Fla., CRC Press, 2005, all of which are incorporated herein by reference.

30 [0049] The microemulsions of the present invention may be incorporated into those products using the following conventional techniques. The microemulsions can be incorporated into those products as color, flavor or other types of food ingredients. The microemulsions can simply be added and mixed or diluted directly into aqueous-based or lipid-based food and beverage compositions using typical mixers or stirrers. The speed with which the microemulsion systems are incorporated into food and beverage products depends on the velocity at which individual components in the microemulsions dissolve into the specific food and beverage systems; typically the products can be homogeneous within 5 minutes. The speed of incorporation of microemulsions into various systems could be accelerated by increasing the speed of mixing and/or possibly warming the food systems to about 40°C, if it is necessary.

35 [0050] - In addition to the components described above, the food and beverage compositions, as well as the microemulsions of the present invention, may include adjunct components conventionally used in food or beverage products at their artestablished levels. Examples of such components include preservatives, antioxidants, flavorants, colorants, nutrients, nutraceuticals, food supplements, antioxidants, plant extracts, therapeutic agents (for example, chondroitin or electrolytes), and combinations of those materials. To the extent such components are water-immiscible or lipid-immiscible, they may be incorporated into the food and beverage compositions using the microemulsions of the present invention.

40 [0051] By using the compositions and methods of the present invention, it is possible to form effective microemulsions without the use of co-solvents, such as ethanol and propylene glycol. These co-solvents can result in off-flavors in the food or beverage compositions. In addition, the microemulsions of the present invention are formed using lower levels of surfactants than are typically needed in microemulsion formation. Because of this, the microemulsions of the present

invention exhibit less off-flavor caused by surfactants, are able to carry high levels of difficult-to-disperse ingredients, and are more stable either in concentrated or dilute form. In addition, the present invention allows for the preparation of stable compositions containing difficult-to-disperse ingredients (such as beta-carotene). Beta-carotene is highly insoluble and tends to recrystallize, hence breaking a typical microemulsion system). The present invention allows for a stable composition of such materials, such as beta-carotene, formed in a way which does not require extreme processing conditions. Further, the microemulsions of the present invention, as well as the food and beverage products containing them, have a controllable appearance in that by adjusting the types and concentrations of surfactants and/or the oil phase, the optical properties, from crystal clear to cloudy, can be adjusted in the finished product.

[0052] The following examples are intended to be illustrative of various embodiments of the present invention.

Example 1 - Beta-carotene oil-in-water microemulsion

[0053] The following is an example of the preparation of a beta-carotene oil-in-water microemulsion of the present invention. The microemulsion has the following composition:

Component	Weight %
Water, deionized	79.335
Sodium benzoate agglomerate	0.075
Ascorbic acid	0.20
Polysorbate 80 (TWEEN®) - high HLB	15.00
Triglyceryl monostearate - medium HLB	1.00
Beta-carotene (30% suspension in vegetable oil)	3.36
Vitamin E (tocopherol alpha)	0.10
Vitamin A palmitate	0.10
Glyceryl monooleate - low HLB	0.83
Total	100.00

[0054] The above ingredients are prepared in three separate parts: (1) a water phase (water, sodium benzoate and ascorbic acid); (2) a mixture of emulsifiers containing the high and medium HLB materials (polysorbate and Triglyceryl monostearate); and (3) an oil phase which comprises the water-insoluble components and the low HLB emulsifier (beta-carotene 30%, vitamin E, vitamin A and glycerol monooleate). Heat is used to melt the beta-carotene and surfactant so that the components form a single liquid phase. These three parts are then added in the following order to form a concentrated microemulsion:

[0055] In the first vessel, prepare the aqueous phase by adding sodium benzoate to deionized water. Mix for 5 minutes with medium agitation until the powder is completely dissolved. Add ascorbic acid and mix for 5 minutes. In the second vessel, prepare the emulsifier phase by combining polysorbate 80 (TWEEN®) and polyglycerol ester (tryglyceryl monostearate). Mix well until it is homogeneous. In the heating kettle, prepare the oil phase by combining beta-carotene 30% oil, glyceryl monooleate, vitamin A palmitate and alpha tocopherol. After the oil phase is completely mixed, heat the kettle containing beta-carotene, vitamin E, vitamin A and glyceryl monooleate to 280-285°F with medium agitation until beta-carotene crystals are completely dissolved. Immediately add the oil phase from the kettle to the emulsifier phase in the second vessel, then mix for an additional 5 minutes or until homogeneous. Then add the aqueous phase (water/sodium benzoate/ascorbic acid) from the first vessel to the mixture of the oil phase and emulsifier in the second vessel. Mix at high speed for 15 minutes or until the microemulsion is uniform. The microemulsion can then be diluted to the desired concentration and added to a food or beverage product.

[0056] Examples of commercial sources of emulsifiers suitable for use in the present invention, include Abitec ADM, BASF, Danisco, ICI, Lambent Technologies, Lonza, Mitsubishi Chemical, and Stepan.

Example 2 - Lemon oil-in-water microemulsion

[0057]

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Component	Weight %
Water, deionized	77.225
Sodium benzoate agglomerate	0.075
Ascorbic acid	0.20
Decaglycerol lauric acid ester - high HLB	16.67
Decaglycerol oleic acid ester - medium HLB	1.67
Lemon oil	3.33
Sucrose oleate - low HLB	0.83
Total	100.00

**[0058]** First, mix 16.67 g of decaglycerol lauric acid ester with 1.67 g of decaglycerol oleic acid ester. Second, mix 3.33 g of lemon oil with 0.83 g of sucrose oleate in a separate container, then add to the mixture obtained above. Third, mix sodium benzoate with deionized water before adding ascorbic acid. Then add the aqueous phase to the mixture from step two. Microemulsion is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of water.

Example 3 - Paprika oil-in-water microemulsion

**[0059]**

Component	Weight %
Water, deionized	71.00
Sodium benzoate agglomerate	0.075
Ascorbic acid	0.20
Decaglycerol lauric acid ester - high HLB	25.00
Decaglycerol tetraoleate - medium HLB	1.67
Paprika oleoresin	1.00
Decaglycerol decaoleate - low HLB	1.00
Total	100.00

**[0060]** First, mix 25 g of decaglycerol lauric acid ester with 1.67 g of decaglycerol tetraoleate. Second, mix 1 g of paprika oleoresin with 1 g of decaglycerol decaoleate in a separate container, then add to the mixture obtained above. Third, mix sodium benzoate with deionized water before adding ascorbic acid. Then add the aqueous phase to the mixture from step two. Microemulsion is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of water.

Example 4 - Beet juice water-in-oil microemulsion

**[0061]**

Component	Weight %
Cottonseed oil	74.64
Polysorbate 80 - high HLB	1.49
Triglycerol monooleate - medium HLB	1.49
Beet juice	7.46
Polyglycerol ricinoleate - low HLB	14.92
Total	100.00

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**[0062]** First, mix 7.46 g of beet juice and 1.49 g of polysorbate 80. Second, mix 1.49 g of triglycerol monooleate with 14.92 g of polyglycerol ricinoleate in a separate container, then add to the mixture obtained above. Third, cottonseed oil is added to the mixture from step two. Concentrate beet juice water-in-oil microemulsion system is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of edible vegetable or mineral oil or lipid-based systems provided the system does not contain substantial levels of emulsifier (s).

### Example 5 - Aronia extract water-in-oil microemulsion

#### **[0063]**

Component	Weight %
Canola oil	70.17
Decaglycerol monocaprylate - high HLB	1.75
Decaglycerol tetraoleate - medium HLB	1.75
Aronia extract	8.77
Polyglycerol ricinoleate - low HLB	17.54
Total	100.00

**[0064]** First, mix 8.77 g of aronia extract (natural water-soluble colorants) and 1.75 g of decaglycerol monocaprylate. Second, mix 1.75 g of decaglycerol tetraoleate with 17.54 g of polyglycerol ricinoleate in a separate container, then add to the mixture obtained above. Third, canola oil is added to the mixture from step two. Concentrate aronia extract water-in-oil microemulsion system is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of edible vegetable or mineral oil or lipid-based systems provided the system does not contain substantial levels of emulsifier (s).

### Example 6 - Elderberry extract water-in-oil microemulsion concentrate

#### **[0065]**

Component	Weight %
Polyethyleneglycol monooleate - high HLB	5.89
Decaglycerol hexaoleate - medium HLB	5.89
Elderberry extract	29.41
Decaglycerol decaoleate - low HLB	58.82
Total	100.00

**[0066]** First, mix 29.41 g of elderberry extract (natural water-soluble colorants) and 5.89 g of polyethyleneglycol monooleate. Second, mix 58.82 g of decaglycerol tetraoleate with 5.89 g of decaglycerol hexaoleate in a separate container, then add to the mixture obtained above to form the concentrate. Canola oil is added to the mixture from step two to form the microemulsion by mixing using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of edible vegetable or mineral oil or lipid-based systems provided the system does not contain substantial levels of emulsifier (s).

### Example 7 - Alpha-tocopherol oil-in-water microemulsion concentrate

#### **[0067]**

Component	Weight %
Polysorbate 20 (TWEEN®) - high HLB	85.00

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(continued)

Component	Weight %
Triglycerol monooleate - medium HLB	5.00
Alpha-tocopherol	6.67
Decaglycerol decaoleate - low HLB	3.33
Total	100.00

**[0068]** First, mix 6.67 g of alpha-tocopherol (vitamin E) and 3.33 g of decaglycerol decaoleate. Second, mix 5 g of triglycerol monooleate with 85 g of polysorbate 20 in a separate container, then add to the mixture obtained above. Concentrate alpha-tocopherol microemulsion (micellar) system is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of water.

Example 8 - Vitamin E acetate oil-in-water microemulsion concentrate

**[0069]**

Component	Weight %
Decaglycerol lauric acid ester - high HLB	89.26
Decaglycerol tetraoleate - medium HLB	1.65
Vitamin E acetate	7.44
Glyceryl monooleate - low HLB	1.65
Total	100.00

**[0070]** First, mix 7.44 g of vitamin E acetate and 1.65 g of glyceryl monooleate. Second, mix 1.65 g of decaglycerol tetraoleate with 89.26 g of decaglycerol lauric acid ester in a separate container, then add to the mixture obtained above. Concentrate vitamin E acetate microemulsion (micellar) system is obtained by mixing, using an overhead mixer. The entire process is done at room temperature. This system can be diluted with any amount of water.

Example 9 - Beverage with vitamin E microemulsion

**[0071]**

Component	Weight %
Water	86.67
Sucrose	6.00
Citric acid	1.00
Ascorbic acid	0.30
Apple juice	5.00
Pineapple juice	1.00
Vitamin E microemulsion (e.g., see Example 8)	0.03
Total	100.00

**[0072]** First, mix 6 g of sucrose, 1 g of citric acid and 0.3 g of ascorbic acid with 86.67 g of water. Second, add 5 g of apple juice, 1 g of pineapple juice and 0.3 g vitamin E microemulsion into the solution of step one, and mix until homogeneous using a stirrer or an overhead mixer. The entire process is done at room temperature. This system can then be passed through a thermal process, such as pasteurization or sterilization, to prevent microbial spoilage.

Example 10 - Beverage with beta-carotene microemulsion**[0073]**

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Component	Weight %
Water	86.44
Sucrose	12.00
Citric acid	1.30
Ascorbic acid	0.20
Orange flavor	0.05
Beta-carotene microemulsion (e.g., see Example 1)	0.01
Total	100.00

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**[0074]** First, mix 12 g of sucrose, 1.03 g of citric acid and 0.2 g of ascorbic acid with 86.44 g of water. Second, add 0.05 g of orange flavor and 0.01 g of beta-carotene emulsion into the solution formed in step one, and mix until homogeneous using a stirrer or an overhead mixer. The entire process is done at room temperature. This system can then be passed through a thermal process, such as pasteurization or sterilization, to prevent microbial spoilage.

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Example 11 - Icing with aronia extract natural color microemulsion**[0075]**

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Component	Weight %
Confectioners sugar	77.88
Canola oilL	11.88
Water	9.11
Cream of tartar	0.77
Salt	0.45
Aronia extract microemulsion (e.g., see Example 5)	0.17
Total	100.00

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**[0076]** First, mix 77.8 g of confectioners sugar, 0.77 g of cream of tartar and 0.45 g of salt with 9.11 g of water and 11.88 g of canola oil. Then add aronia extract microemulsion to the mixture formed in step one. Mix thoroughly until homogeneous. The entire process is done at room temperature.

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**[0077]** While this invention has been described with reference to certain specific embodiments, it will be recognized by those skilled in the art that many variations are possible without departing from the scope of the invention as claimed in the appended claims. Further, it will be understood that the present application is intended to cover all changes and modifications of the invention disclosed herein for the purposes of illustration which do not constitute departure from the scope of the invention, as claimed in the appended claims.

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**Claims**

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1. A microemulsion used to incorporate lipophilic water-insoluble materials into food and beverage compositions, comprising

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(a) an oil phase comprising said water-insoluble material and a low HLB emulsifier having an HLB of from 1 to 5 selected from glycerol ester of fatty acids, monoglycerides, diglycerides, ethoxylated monoglycerides, polyglycerol ester of fatty acids, lecithin, sorbitan esters of fatty acids, sucrose esters of fatty acids, and mixtures thereof;

- (b) an aqueous phase; and  
(c) a food grade emulsifier system comprising:

- (i) a high HLB emulsifier having an HLB of from 10 to 16 selected from acetic acid esters of mono and diglycerides, lactic acid esters of mono and diglycerides, succinic acid esters of mono and diglycerides, diacetyl tartaric esters of mono and diglycerides, polysorbitan esters, polyglycerol esters of fatty acids, lecithin, sucrose esters of fatty acids, and mixtures thereof; and  
(ii) a medium HLB emulsifier having an HLB of from 6 to 8 selected from monoglycerides, diglycerides, ethoxylated monoglycerides, sorbitan esters of fatty acids, phosphoric acid esters of mono and diglycerides, polyglycerol esters of fatty acids, lecithin, sucrose esters of fatty acids, and mixtures thereof;

wherein said oil phase is dispersed as particles having an average diameter of from 1 to 300 nm, preferably 1 to 200 nm, within said aqueous phase, which comprises aqueous phase at from 50% to 90%, preferably 70% to 85%, of the total, the balance being oil phase and food grade emulsifier system, of which the oil phase comprises from 1% to 15%, preferably 2% to 6% and which comprises from 0.1% to 5% of the low HLB emulsifier, from 5% to 25% of the high HLB emulsifier, and from 0.1% to 5% of the medium HLB emulsifier, and wherein the aqueous phase may contain dissolved materials selected from colorants, vitamins, juices, antioxidants, extracts of natural components (such as plant roots, leaves, seeds, flowers, etc.), medicaments, simple phenols, polyphenols, bioflavonoids, dairy products, proteins, peptides, salts, sugars, sweeteners, flavors, flavor precursors, nutrients, minerals, acids and seasonings, and mixtures thereof.

2. The microemulsion according to claim 1 wherein the low HLB emulsifier is selected from glycerol monooleate, polyglycerol ricinoleate, decaglycerol decaoleate, sucrose erucate, sucrose oleate, and the medium HLB emulsifier is selected from polyglycerol esters, such as decaglycerol hexaoleate, and triglycerol monofatty acids, such as triglycerol monooleate, and sucrose stearate, and the high HLB emulsifier is selected from polysorbate 80, polyoxysorbitan monolaurate, polyglycerol-6-laurate, decaglycerol lauric acid esters, decaglycerol oleic acid esters and sucrose esters.
3. The microemulsion according to claim 1 wherein the low HLB emulsifier is selected from glycerol monooleate, polyglycerol ricinoleate, decaglycerol decaoleate, sucrose erucate, sucrose oleate, and mixtures thereof, or the medium HLB emulsifier is selected from decaglycerol hexaoleate, triglycerol monooleate, sucrose stearate, and mixtures thereof, or the high HLB emulsifier is selected from polysorbate 80, polyoxysorbitan monolaurate, polyglycerol-6-laurate, decaglycerol lauric acid esters, decaglycerol oleic acid esters and mixtures thereof.
4. The microemulsion according to claim 1 wherein the water-insoluble material is selected from carotenoids and their derivatives, essential oils, edible oils, fatty acids, proteins and peptides, polyunsaturated fatty acids and their esters, vitamin A and its derivatives, vitamin E and its derivatives, vitamin D and its derivatives, vitamin K and its derivatives, colorants, flavorants, nutrients, nutraceuticals, therapeutic agents, antioxidants, extracts of natural components, medicaments, preservatives, lipoic acid, phytosterins, quercetin, phytosterols and their esters, co-enzyme Q10 (ubidecarone), plant isoflavones, and mixtures thereof, preferably beta-carotene, apocarotenal, vitamin E, vitamin A, vitamin A palmitate, vitamin E acetate, and mixtures thereof.
5. The microemulsion according to claim 1 wherein the emulsifiers used in the microemulsion comprise glycerol monooleate, polysorbate 80, and polyglycerol esters.
6. A method for preparing the microemulsion of claim 1, comprising the steps of:
  - (a) mixing the lipophilic water-insoluble components with the low HLB emulsifier to form the oil phase;
  - (b) mixing the emulsifier system into the oil phase; and
  - (c) adding the aqueous phase into the product of step (b) and mixing to form the microemulsion.
7. A microemulsion used to incorporate water-soluble materials into food and beverage compositions, comprising:
  - (a) an aqueous phase comprising said water-soluble material and a high HLB emulsifier having an HLB of from 9 to 17 selected from polysorbate 80, polyoxysorbitan monolaurate, polyglycerol-6-laurate, decaglycerol lauric acid esters, decaglycerol oleic acid esters and sucrose esters;
  - (b) an oil phase; and

(c) a food grade emulsifier system comprising:

(i) a low HLB emulsifier having an HLB of from 1 to 5 selected from glycerol monooleate, polyglycerol ricinoleate, decaglycerol decaoleate, sucrose erucate, sucrose oleate;

(ii) a medium HLB emulsifier having an HLB of from 6 to 8 selected from polyglycerol esters, such as decaglycerol hexaoleate, and triglycerol monofatty acids, such as triglycerol monooleate, and sucrose stearate;

wherein said aqueous phase is dispersed as particles having an average diameter of from 1 to 300 nm, within said oil phase,

which comprises oil phase at from 50% to 90% of the total, the balance being aqueous phase and food grade emulsifier system, of which the aqueous phase comprises from about 1% to about 15% of the total, the balance being oil phase and food grade emulsifier system and which includes from 0.1 % to 5% of the high HLB emulsifier, from 0.1 % to 5% of the medium HLB emulsifier, and from 5% to 30% of the low HLB emulsifier.

8. The microemulsion according to claim 7 wherein the oil phase includes dissolved materials selected from carotenoids and their derivatives, essential oils, edible oils, fatty acids, proteins and peptides, polyunsaturated fatty acids and their esters, vitamin A and its derivatives, vitamin E and its derivatives, vitamin D and its derivatives, vitamin K and its derivatives, colorants, flavorants, nutrients, nutraceuticals, therapeutic agents, antioxidants, extracts of natural components, medicaments, preservatives, lipoic acid, phytosterins, quercetin, phytosterols and their esters, coenzyme Q10 (ubidecarone), plant isoflavones, and mixtures thereon.

9. The microemulsion according to claim 7 wherein the water-soluble material is selected from food additives, food preservatives, food supplements, antioxidants, nutraceuticals, plant extracts, medicaments, fatty acids, proteins, peptides, carbohydrates, salt, natural flavors, synthetic flavors, colorants, vitamins, and mixtures thereof.

10. A method for preparing the microemulsion of claim 7, comprising the steps of:

(a) mixing the water-soluble material components with the high HLB emulsifier to form the aqueous phase;

(b) mixing the emulsifier system into the aqueous phase; and

(c) adding the oil phase into the product of step (b) and mixing to form the microemulsion.

11. A concentrate used to form an oil-in-water microemulsion for use in food or beverage products, comprising:

(a) from 1% to 40% of a dispersed oil phase containing a lipophilic component to be incorporated into said food or beverage composition;

(b) from 1% to 10% of a food grade low HLB emulsifier having an HLB of from 1 to 5 as defined in claim 1;

(c) from 1% to 10% of a food grade medium HLB emulsifier having an HLB of from 6 to 8 as defined in claim 1; and

(d) from 65% to 95% of a food grade high HLB emulsifier having an HLB of from 10 to 16 as defined in claim 1.

12. A concentrate used to form a water-in-oil microemulsion for use in oil- or fat-based products, comprising:

(a) from 1% to 40% of a dispersed aqueous phase containing a component to be incorporated into said food or beverage composition;

(b) from 65% to 95% of a food grade low HLB emulsifier having an HLB of from 1 to 5 as defined in claim 7;

(c) from 1% to 10% of a food grade medium HLB emulsifier having an HLB of from 6 to 8 as defined in claim 7; and

(d) from 1% to 10% of a food grade high HLB emulsifier having an HLB of from 9 to 17 as defined in claim 7.

13. A food or beverage composition comprising the microemulsion of any of claims 1 to 5 and 7 to 9.

14. A food or beverage composition prepared using the concentrate of claim 11 or 12.

## Patentansprüche

1. Mikroemulsion, die verwendet wird, um Nahrungsmittel- und Getränke-Zusammensetzungen lipophile in Wasser unlösliche Materialien einzuverleiben, umfassend

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(a) eine Ölphase, die das in Wasser unlösliche Material und einen Emulgator mit niedrigem HLB-Wert mit einem HLB-Wert von 1 bis 5 umfasst, der aus Glycerolestern von Fettsäuren, Monoglyceriden, Diglyceriden, ethoxylierten Monoglyceriden, Polyglycerolester von Fettsäuren, Lecithin, Sorbitanestern von Fettsäuren, Saccharoseestern von Fettsäuren und deren Mischungen ausgewählt ist;

(b) eine wässrige Phase; und

(c) ein für Nahrungsmittel geeignetes Emulgator-System, umfassend:

(i) einen Emulgator mit hohem HLB-Wert mit einem HLB-Wert von 10 bis 16, der aus Essigsäureestern von Mono- und Diglyceriden, Milchsäureestern von Mono- und Diglyceriden, Bernsteinsäureestern von Mono- und Diglyceriden, Diacetylweinsäureestern von Mono- und Diglyceriden, Polysorbitanestern, Polyglycerolestern von Fettsäuren, Lecithin, Saccharoseestern von Fettsäuren und deren Mischungen ausgewählt ist; und

(ii) einen Emulgator mit mittlerem HLB-Wert mit einem HLB-Wert von 6 bis 8, der aus Monoglyceriden, Diglyceriden, ethoxylierten Monoglyceriden, Sorbitanestern von Fettsäuren, Phosphorsäureestern von Mono- und Diglyceriden, Polyglycerolestern von Fettsäuren, Lecithin, Saccharoseestern von Fettsäuren und deren Mischungen ausgewählt ist;

wobei die Ölphase als Teilchen mit einem durchschnittlichen Durchmesser von 1 bis 300 nm, vorzugsweise 1 bis 200 nm, in der wässrigen Phase dispergiert ist,

welche wässrige Phase zu 50 % bis 90 %, vorzugsweise 70 % bis 85 %, der gesamten umfasst, wobei der Rest Ölphase und für Nahrungsmittel geeignetes Emulgatorsystem ist, von welcher die Ölphase 1 % bis 15 %, vorzugsweise 2 % bis 6 % umfasst und welche 0,1 % bis 5 % des Emulgators mit niedrigem HLB-Wert, 5 % bis 25 % des Emulgators mit hohem HLB-Wert und 0,1 % bis 5 % des Emulgators mit mittlerem HLB-Wert umfasst, und

wobei die wässrige Phase gelöste Materialien enthalten kann, die ausgewählt sind aus Färbemitteln, Vitaminen, Säften, Antioxidantien, Extrakten von natürlichen Komponenten (wie Pflanzenwurzeln, Blättern, Samen, Blüten usw.), Medikamenten, einfachen Phenolen, Polyphenolen, Bioflavonoiden, Milchprodukten, Proteinen, Peptiden, Salzen, Zuckern, Süßungsmitteln, Geschmacksstoffen, Geschmacksstoffvorläufern, Nährstoffen, Mineralien, Säuren und Gewürzen und deren Mischungen.

2. Mikroemulsion nach Anspruch 1, bei der der Emulgator mit niedrigem HLB-Wert ausgewählt ist aus Glycerolmonooleat, Polyglycerolricinoleat, Decaglyceroldecaoleat, Saccharoseerucat, Saccharoseoleat, und der Emulgator mit mittlerem HLB-Wert ausgewählt ist aus Polyglycerolestern, wie Decaglycerolhexaoleat, und Triglycerolmonofettsäuren, wie Triglycerolmonooleat, und Saccharosestearat und der Emulgator mit hohem HLB-Wert ausgewählt ist aus Polysorbat 80, Polyoxysorbitanmonolaurat, Polyglycerol-6-laurat, Decaglycerollaurinsäureestern, Decaglycerolölsäureestern und Saccharoseestern.
3. Mikroemulsion nach Anspruch 1, bei der der Emulgator mit niedrigem HLB-Wert ausgewählt ist aus Glycerolmonooleat, Polyglycerolricinoleat, Decaglyceroldecaoleat, Saccharoseerucat, Saccharoseoleat und deren Mischungen, oder der Emulgator mit mittlerem HLB-Wert ausgewählt ist aus Decaglycerolhexaoleat, Triglycerolmonooleat, Saccharosestearat und deren Mischungen oder der Emulgator mit hohem HLB-Wert ausgewählt ist aus Polysorbat 80, Polyoxysorbitanmonolaurat, Polyglycerol-6-laurat, Decaglycerollaurinsäureestern, Decaglycerolölsäureestern und deren Mischungen.
4. Mikroemulsion nach Anspruch 1, bei der das in Wasser unlösliche Material ausgewählt ist aus Carotinoiden und deren Derivaten, etherischen Ölen, essbaren Ölen, Fettsäuren, Proteinen und Peptiden, polyungesättigten Fettsäuren und deren Estern, Vitamin A und dessen Derivaten, Vitamin E und dessen Derivaten, Vitamin D und dessen Derivaten, Vitamin K und dessen Derivaten, Färbemitteln, Geschmacksstoffen, Nährstoffen, Nutrazeutika, therapeutischen Mitteln, Antioxidantien, Extrakten von natürlichen Komponenten, Medikamenten, Konservierungsmitteln, Liponsäure, Phytosterinen, Quercetin, Phytosterolen und deren Estern, Coenzym Q10 (Ubidecaron), Pflanzenisoflavonen und deren Mischungen, vorzugsweise beta-Carotin, Apocarotenal, Vitamin E, Vitamin A, Vitamin A-Palmitat, Vitamin E-Acetat und deren Mischungen.
5. Mikroemulsion nach Anspruch 1, bei der die in der Mikroemulsion verwendeten Emulgatoren Glycerolmonooleat, Polysorbat 80 und Polyglycerolester umfassen.
6. Verfahren zur Herstellung der Mikroemulsion nach Anspruch 1, umfassend die Schritte:

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- (a) Mischen der lipophilen, in Wasser unlöslichen Komponenten mit dem Emulgator mit niedrigem HLB-Wert, um die Ölphase zu bilden;
- (b) Einmischen des Emulgatorsystems in die Ölphase; und
- (c) Zugeben der wässrigen Phase in das Produkt von Schritt (b) und Mischen, um die Mikroemulsion zu bilden.

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7. Mikroemulsion, die verwendet wird, um Nahrungsmittel- und GetränkeZusammensetzungen wasserlösliche Materialien einzuverleiben, umfassend:

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- (a) eine wässrige Phase, die das wasserlösliche Material und einen Emulgator mit hohem HLB-Wert mit einem HLB-Wert von 9 bis 17 umfasst, der aus Polysorbat 80, Polyoxysorbitanmonolaurat, Polyglycerol-6-laurat, Decaglycerollaurinsäureestern, Decaglycerolölsäureestern und Saccharoseestern ausgewählt ist;
- (b) eine Ölphase; und
- (c) ein für Nahrungsmittel geeignetes Emulgatorsystem, umfassend:

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- (i) einen Emulgator mit niedrigem HLB-Wert mit einem HLB-Wert von 1 bis 5, der ausgewählt ist aus Glycerolmonooleat, Polyglycerolricinoleat, Decaglyceroldecaoleat, Saccharoseerucat, Saccharoseoleat;
- (ii) einen Emulgator mit mittlerem HLB-Wert mit einem HLB-Wert von 6 bis 8, der ausgewählt ist aus Polyglycerolestern, wie Decaglycerolhexaoleat, und Triglycerolmonofettsäuren, wie Triglycerolmonooleat, und Saccharosestearat; wobei die wässrige Phase als Teilchen mit einem durchschnittlichen Durchmesser von 1 bis 300 nm in der Ölphase dispergiert ist,

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welche Ölphase zu 50 % bis 90 % der gesamten umfasst, wobei der Rest wässrige Phase und für Nahrungsmittel geeignetes Emulgatorsystem ist, von welcher die wässrige Phase etwa 1 % bis etwa 15 % der gesamten umfasst, wobei der Rest Ölphase und für Nahrungsmittel geeignetes Emulgatorsystem ist, und welche 0,1 % bis 5 % des Emulgators mit hohem HLB-Wert, 0,1 % bis 5 % des Emulgators mit mittlerem HLB-Wert und 5 % bis 30 % des Emulgators mit niedrigem HLB-Wert einschließt.

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8. Mikroemulsion nach Anspruch 7, bei der die Ölphase gelöste Materialien einschließt, die ausgewählt sind aus Carotinoiden und deren Derivaten, etherischen Ölen, essbaren Ölen, Fettsäuren, Proteinen und Peptiden, polyungesättigten Fettsäuren und deren Estern, Vitamin A und dessen Derivaten, Vitamin E und dessen Derivaten, Vitamin D und dessen Derivaten, Vitamin K und dessen Derivaten, Färbemitteln, Geschmacksstoffen, Nährstoffen, Nutra-

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zutika, therapeutischen Mitteln, Antioxidantien, Extrakten von natürlichen Komponenten, Medikamenten, Konservierungsmitteln, Liponsäure, Phytosterinen, Quercetin, Phytosterolen und deren Estern, Coenzym Q10 (Ubidecarnon), Pflanzen-Isoflavonen und deren Mischungen.

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9. Mikroemulsion nach Anspruch 7, bei der das wasserlösliche Material aus Nahrungsmittelzusätzen, Nahrungsmittelkonservierungsmitteln, Nahrungsergänzungsmitteln, Antioxidantien, Nutra-

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zutika, Pflanzenextrakten, Medikamenten, Fettsäuren, Proteinen, Peptiden, Kohlenhydraten, Salz, natürlichen Geschmacksstoffen, synthetischen Geschmacksstoffen, Färbemitteln, Vitaminen und deren Mischungen ausgewählt ist.

10. Verfahren zur Herstellung der Mikroemulsion nach Anspruch 7, umfassend die Schritte:

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- (a) Mischen der Komponenten des wasserlöslichen Materials mit dem Emulgator mit hohem HLB-Wert, um die wässrige Phase zu bilden;
- (b) Einmischen des Emulgatorsystems in die wässrige Phase; und
- (c) Zugeben der Ölphase in das Produkt von Schritt (b) und Mischen, um die Mikroemulsion zu bilden.

11. Konzentrat, das verwendet wird, um eine Öl-in-Wasser-Mikroemulsion zur Verwendung in Nahrungsmittel- oder Getränke-Produkten zu bilden, umfassend:

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- (a) 1 % bis 40 % einer dispergierten Ölphase, die eine lipophile Komponente enthält, welche der Nahrungsmittel- oder Getränke-Zusammensetzung einzuverleiben ist;
- (b) 1 % bis 10 % eines für Nahrungsmittel geeigneten Emulgators mit niedrigem HLB-Wert mit einem HLB-Wert von 1 bis 5, wie in Anspruch 1 definiert;
- (c) 1 % bis 10 % eines für Nahrungsmittel geeigneten Emulgators mit mittlerem HLB-Wert mit einem HLB-Wert von 6 bis 8, wie in Anspruch 1 definiert; und
- (d) 65 % bis 95 % eines für Nahrungsmittel geeigneten Emulgators mit hohem HLB-Wert mit einem HLB-Wert von 10 bis 16, wie in Anspruch 1 definiert.

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12. Konzentrat, das verwendet wird, um eine Wasser-in-Öl-Mikroemulsion zur Verwendung in auf Öl oder Fett basierenden Produkten zu bilden, umfassend:

- 5 (a) 1 % bis 40 % einer dispergierten wässrigen Phase, die eine Komponente enthält, die der Nahrungsmittel- oder Getränke-Zusammensetzung einzuverleiben ist;  
 (b) 65 % bis 95 % eines für Nahrungsmittel geeigneten Emulgators mit niedrigem HLB-Wert mit einem HLB-Wert von 1 bis 5, wie in Anspruch 7 definiert,  
 (c) 1 % bis 10 % eines für Nahrungsmittel geeigneten Emulgators mit mittlerem HLB-Wert mit einem HLB-Wert von 6 bis 8, wie in Anspruch 7 definiert; und  
 10 (d) 1 % bis 10 % eines für Nahrungsmittel geeigneten Emulgators mit hohem HLB-Wert mit einem HLB-Wert von 9 bis 17, wie in Anspruch 7 definiert.

13. Nahrungsmittel- oder Getränke-Zusammensetzung, umfassend die Mikroemulsion nach einem der Ansprüche 1 bis 5 und 7 bis 9.

14. Nahrungsmittel- oder Getränkezusammensetzung, hergestellt unter Verwendung des Konzentrats nach Anspruch 11 oder 12.

## 20 Revendications

1. Microémulsion utilisée pour incorporer des substances lipophiles insolubles dans l'eau dans des compositions de type aliment et boisson, comprenant

25 (a) une phase huileuse comprenant ladite substance insoluble dans l'eau et un émulsifiant à basse valeur HLB ayant une HLB de 1 à 5, choisi parmi l'ester de glycérol d'acides gras, les monoglycérides, les diglycérides, les monoglycérides éthoxylés, l'ester de polyglycérol d'acides gras, la lécithine, les esters de sorbitan d'acides gras, les esters de saccharose d'acides gras, et leurs mélanges ;

(b) une phase aqueuse ; et

30 (c) un système d'émulsifiants de qualité alimentaire comprenant :

(i) un émulsifiant à valeur HLB élevée ayant une HLB de 10 à 16, choisi parmi les esters d'acide acétique des mono- et des diglycérides, les esters d'acide lactique des mono- et des diglycérides, les esters d'acide succinique des mono- et des diglycérides, les esters diacétyltartriques des mono- et des diglycérides, les esters de polysorbitan, les esters de polyglycérol d'acides gras, la lécithine, les esters de saccharose d'acides gras, et leurs mélanges ; et

(ii) un émulsifiant à valeur HLB moyenne ayant une HLB de 6 à 8, choisi parmi les monoglycérides, les diglycérides, les monoglycérides éthoxylés, les esters de sorbitan d'acides gras, les esters d'acide phosphorique des mono- et des diglycérides, les esters de polyglycérol d'acides gras, la lécithine, les esters de saccharose d'acides gras, et leurs mélanges ;

dans laquelle ladite phase huileuse est dispersée sous forme de particules ayant un diamètre moyen de 1 à 300 nm, de préférence, de 1 à 200 nm, dans ladite phase aqueuse,

45 qui comprend la phase aqueuse à raison de 50 à 90 %, de préférence, à raison de 70 à 85 % du total, le reste étant la phase huileuse et le système d'émulsifiants de qualité alimentaire, la phase huileuse représentant de 1 à 15 %, de préférence, de 2 à 6 %, et qui comprend de 0,1 à 5 % d'émulsifiant à basse valeur HLB, de 5 à 25 % d'émulsifiant à valeur HLB élevée, et de 0,1 à 5 % d'émulsifiant à valeur HLB moyenne, et

50 dans laquelle la phase aqueuse peut contenir des substances dissoutes choisies parmi les colorants, les vitamines, les jus, les antioxydants, les extraits de composants naturels (tels que racines végétales, feuilles, graines, fleurs, etc.), les médicaments, les phénols simples, les polyphénols, les bioflavonoïdes, les produits laitiers, les protéines, les peptides, les sels, les sucres, les édulcorants, les arômes, les précurseurs d'arômes, les nutriments, les oligo-éléments, les acides et les assaisonnements, et leurs mélanges.

2. Microémulsion selon la revendication 1, dans laquelle l'émulsifiant à basse valeur HLB est choisi parmi le monooléate de glycérol, le ricinoléate de polyglycérol, le décaoléate de décaglycérol, l'éruçate de saccharose, l'oléate de saccharose, et l'émulsifiant à valeur HLB moyenne est choisi parmi les esters de polyglycérol, tels que l'hexaoléate de décaglycérol, et les monoacides gras de triglycérol, tels que le monooléate de triglycérol, et le stéarate de saccharose, et l'émulsifiant à valeur HLB élevée est choisi parmi le polysorbate 80, le monolaurate de polyoxysorbitan, le poly-

glycérol-6-laurate, les esters d'acide laurique de décaglycérol, les esters d'acide oléique de décaglycérol et les esters de saccharose.

- 5 3. Microémulsion selon la revendication 1, dans laquelle l'émulsifiant à basse valeur HLB est choisi parmi le monooléate de glycérol, le ricinoléate de polyglycérol, le décaoléate de décaglycérol, l'éruçate de saccharose, l'oléate de saccharose, et leurs mélanges, ou l'émulsifiant à valeur HLB moyenne est choisi parmi l'hexaoléate de décaglycérol, le monooléate de triglycérol, le stéarate de saccharose, et leurs mélanges, ou l'émulsifiant à valeur HLB élevée est choisi parmi le polysorbate 80, le monolaurate de polyoxysorbitan, le polyglycérol-6-laurate, les esters d'acide laurique de décaglycérol, les esters l'acide oléique de décaglycérol et leurs mélanges.
- 10 4. Microémulsion selon la revendication 1, dans laquelle la substance insoluble dans l'eau est choisie parmi les caroténoïdes et leurs dérivés, les huiles essentielles, les huiles comestibles, les acides gras, les protéines et les peptides, les acides gras polyinsaturés et leurs esters, la vitamine A et ses dérivés, la vitamine E et ses dérivés, la vitamine D et ses dérivés, la vitamine K et ses dérivés, les colorants, les aromatisants, les nutriments, les nutraceutiques, les agents thérapeutiques, les antioxydants, les extraits de composants naturels, les médicaments, les conservateurs, l'acide lipoïque, les phytostérines, la quercétine, les phytostérols et leurs esters, la co-enzyme Q10 (ubidécarone), les isoflavones végétales, et leurs mélanges, de préférence, le bêta-carotène, l'épocaroténal, la vitamine E, la vitamine A, le palmitate de vitamine A, l'acétate de vitamine E, et leurs mélanges.
- 15 5. Microémulsion selon la revendication 1, dans laquelle les émulsifiants utilisés dans la micro-émulsion comprennent le monooléate de glycérol, le polysorbate 80, et les esters de polyglycérol.
- 20 6. Procédé de préparation de la microémulsion selon la revendication 1, comprenant les étapes suivantes :
- 25 (a) mélange des composants lipophiles insolubles dans l'eau avec l'émulsifiant à basse valeur HLB pour former la phase huileuse ;  
 (b) incorporation du système d'émulsifiants dans la phase huileuse ; et  
 (c) ajout de la phase aqueuse dans le produit de l'étape (b) et mélange pour former la microémulsion.
- 30 7. Microémulsion utilisée pour incorporer des substances solubles dans l'eau dans des compositions de type aliment et boisson, comprenant :
- 35 (a) une phase aqueuse comprenant ladite substance soluble dans l'eau et un émulsifiant à valeur HLB élevée ayant une HLB de 9 à 17, choisi parmi le polysorbate 80, le monolaurate de polyoxysorbitan, le polyglycérol-6-laurate, les esters d'acide laurique de décaglycérol, les esters d'acide oléique de décaglycérol et les esters de saccharose ;  
 (b) une phase huileuse ; et  
 (c) un système d'émulsifiants de qualité alimentaire comprenant :
- 40 (i) un émulsifiant à basse valeur HLB ayant une HLB de 1 à 5, choisi parmi le monooléate de glycérol, le ricinoléate de polyglycérol, le décaoléate de décaglycérol, l'éruçate de saccharose, l'oléate de saccharose ;  
 (ii) un émulsifiant à valeur HLB moyenne ayant une HLB de 6 à 8, choisi parmi les esters de polyglycérol, tels que l'hexaoléate de décaglycérol, et les monoacides gras de triglycérol, tels que le monooléate de triglycérol, et le stéarate de saccharose ; dans laquelle ladite phase aqueuse est dispersée sous forme de
- 45 particules ayant un diamètre moyen de 1 à 300 nm, dans ladite phase huileuse,
- qui comprend la phase huileuse à raison de 50 à 90 % du total, le reste étant la phase aqueuse et le système d'émulsifiants de qualité alimentaire, la phase aqueuse représentant environ 1 à environ 15 % du total, le reste étant la phase huileuse et le système d'émulsifiants de qualité alimentaire, et qui comprend de 0,1 à 5 % d'émulsifiant à valeur HLB élevée, de 0,1 à 5 % d'émulsifiant à valeur HLB moyenne, et de 5 à 30 % d'émulsifiant à basse valeur HLB.
- 50 8. Microémulsion selon la revendication 7, dans laquelle la phase huileuse comprend des substances dissoutes choisies parmi les caroténoïdes et leurs dérivés, les huiles essentielles, les huiles comestibles, les acides gras, les protéines et les peptides, les acides gras polyinsaturés et leurs esters, la vitamine A et ses dérivés, la vitamine E et ses dérivés, la vitamine D et ses dérivés, la vitamine K et ses dérivés, les colorants, les aromatisants, les nutriments, les nutraceutiques, les agents thérapeutiques, les antioxydants, les extraits de composants naturels, les médicaments, les conservateurs, l'acide lipoïque, les phytostérines, la quercétine, les phytostérols et leurs esters, la co-
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enzyme Q10 (ubidécarrone), les isoflavones végétales, et leurs mélanges.

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9. Microémulsion selon la revendication 7, dans laquelle la substance soluble dans l'eau est choisie parmi les additifs alimentaires, les conservateurs alimentaires, les antioxydants, les nutraceutiques, les extraits végétaux, les médicaments, les acides gras, les protéines, les peptides, les glucides, le sel, les arômes naturels, les arômes synthétiques, les colorants, les vitamines, et leurs mélanges.
10. Procédé de préparation de la microémulsion selon la revendication 7, comprenant les étapes suivantes :
- 10
- (a) mélange des composants constituant la substance soluble dans l'eau avec l'émulsifiant à valeur HLB élevée pour former la phase aqueuse ;
  - (b) incorporation du système d'émulsifiants dans la phase aqueuse ; et
  - (c) ajout de la phase huileuse dans le produit de l'étape (b) et mélange pour former la microémulsion.
- 15
11. Concentré utilisé pour former une microémulsion huile dans l'eau, destiné à être incorporé dans des produits de type aliment ou boisson, comprenant :
- (a) de 1 à 40 % d'une phase huileuse dispersée contenant un composant lipophile à incorporer dans ladite composition de type aliment ou boisson ;
  - 20 (b) de 1 à 10 % d'un émulsifiant de qualité alimentaire à basse valeur HLB ayant une HLB de 1 à 5 selon la revendication 1 ;
  - (c) de 1 à 10 % d'un émulsifiant de qualité alimentaire à valeur HLB moyenne ayant une HLB de 6 à 8 selon la revendication 1 ; et
  - 25 (d) de 65 à 95 % d'un émulsifiant de qualité alimentaire à valeur HLB élevée ayant une HLB de 10 à 16 selon la revendication 1.
12. Concentré utilisé pour former une microémulsion eau dans l'huile, destiné à être incorporé dans des produits à base d'huile ou de matière grasse, comprenant :
- 30
- (a) de 1 à 40 % d'une phase aqueuse dispersée contenant un composant à incorporer dans ladite composition de type aliment ou boisson ;
  - (b) de 65 à 95 % d'un émulsifiant de qualité alimentaire à basse valeur HLB ayant une HLB de 1 à 5 selon la revendication 7 ;
  - 35 (c) de 1 à 10 % d'un émulsifiant de qualité alimentaire à valeur HLB moyenne ayant une HLB de 6 à 8 selon la revendication 7 ; et
  - (d) de 1 à 10 % d'un émulsifiant de qualité alimentaire à valeur HLB élevée ayant une HLB de 9 à 17 selon la revendication 7.
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13. Composition de type aliment ou boisson comprenant la microémulsion selon l'une quelconque des revendications 1 à 5 et 7 à 9.
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14. Composition de type aliment ou boisson préparé à l'aide du concentré selon la revendication 11 ou 12.
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**REFERENCES CITED IN THE DESCRIPTION**

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