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(54) **Title**: PROCESS FOR PREPARING A STABLE HIGH OIL-PHASE CONTENT O/W EMULSION, THUS-PREPARED EMULSION AND USE THEREOF

(57) **Abstract**: The present invention relates to a process for preparing a stable high oil-phase content O/W emulsion, an O/W emulsion prepared by this process and use thereof. The emulsion prepared by this process has a high oil-phase content and can be widely used in personal care and cosmetic products. The O/W emulsion has a milk-white cream-like appearance, or becomes a transparent gel appearance as desired. This emulsion, when applied to the skin, releases emollient oil rapidly, serves for make-up removing, massage or humectant function or the like, and can also be used as a substitute for baby oil.

Process for preparing a stable high oil-phase content O/W emulsion, thus-prepared emulsion and use thereof

Technical Field

The present invention relates to a process for preparing a stable high oil-phase content O/W emulsion, an O/W emulsion prepared by this process and use thereof. The emulsion can be widely used in personal care and cosmetic products.

Background Art

Pure oil or pure oil composition products have existed in personal care and cosmetic field for a very long time. But they still have many disadvantages, for example, permeability is so high that requirements on package are high, the liquid nature of products results in inconvenient application. Although the above shortcomings can be overcome if thickening oil directly, but new problems will occur, like oil release with difficulty, sharply-reduced spreadability and remarkably-increased cost and the like. So, is it possible to prepare one system which may release oil rapidly after application, has a cream appearance, is convenient for using and packaging and also a long enough shelf-life?

To this end, oil and fat are generally made into an oil-in-water (O/W) emulsion form and the above problems are thus solved. However, conventional O/W emulsions in the market have an oil-phase content between 10-40 wt. %, and they are usually in the form of cream, lotion or ointment. It is difficult to prepare an O/W emulsion with an oil-phase content of more than 50 wt. %. An O/W emulsion with a high oil-phase content can be obtained only with difficulty by means of conventional emulsification processes, and even if it is emulsified successfully in a short time, it is prone to be demulsified and hard to be storied stably.

Chinese patent CN1175805C discloses an emulsion stabilizer system and an emulsion prepared with the same, wherein said stabilizer system includes an emulsifier for oil and a polysaccharide combination of xanthan polysaccharide and polyglucomannan polysaccharide. In the emulsion, the oil phase is generally silicone oil such as polydimethicone, and the emulsifier is nonionic emulsifier such as fatty acid glycol ester, and also it contains polysaccharide stabilizers such as xanthan polysaccharide and polyglucomannan polysaccharide, and may also contain humectants such as glycol. The amount and combination of the polysaccharide stabilizer in the above mentioned emulsion are narrowly restricted: the amount of the polysaccharide combination of xanthan polysaccharide and polyglucomannan polysaccharide used is 0.02-0.5% by weight and the weight ratio

of xanthan polysaccharide to polyglucomannan polysaccharide is defined to be from 1:10 to 10:1. In most examples of CN1175805C, only such cases in which the oil phase is comprised at an amount of 20 wt. % have been exemplified. The emulsion prepared in Example 18 with an oil-phase content of 80% is just an intermediate product. Although Example 19 discloses a formula with a high oil-phase content, it has a stability of only one week and thus is incapable of meeting needs for practical production and application. That is to say, by means of the formulas in this invention, it is actually difficult to obtain a high oil-content emulsion meeting practical production, especially a stable high oil-phase content O/W emulsion.

Disclosure of the Invention

In view of the problems existing in the prior art, the present inventors made extensive and intensive studies on a high oil-phase content O/W emulsion and found surprisingly that the technical problem can be solved by using a particular process for preparing an O/W emulsion different from conventional ones, obtaining a stable high oil-phase content O/W emulsion. In conventional processes for preparing an O/W emulsion, when adding an oil phase into a water phase, the oil phase is directly poured into the water phase, which is carried out under agitation or followed by agitation, to then obtain an O/W emulsion. The present inventors found that an O/W emulsion with a high oil-phase content, which may be more than 65 wt. %, can be prepared if the oil phase is added to the water phase at a controlled speed so that the whole system maintains a homogeneous emulsification state during the addition. Besides, this emulsion is stable for storage and able to meet needs for practical applications. Moreover, by the selection of an oil phase with a comparatively low starting freezing point in the O/W emulsion, the so-obtained O/W emulsion can meet wide application conditions. The oil-phase content in the emulsion can be very high, even up to 80-95wt.% and even at these high oil-phase contents, the emulsion is stable enough. The emulsion has a milk-white cream-like appearance, or has a transparent gel appearance as desired by adjusting reflective indexes of the two phases. This emulsion, when applied to the skin, releases emollient oil rapidly, serves for make-up removing, massage or humectant function or the like, and can also be used as a substitute for baby oil.

Thus, one object of the present invention is to provide a process for preparing an O/W emulsion, comprising the steps of:

(a) mixing a water phase and an emulsifier to obtain a homogeneous water phase mixture; and

(b) adding an oil phase in a liquid state into the water phase mixture obtained in step (a) under agitation at such an addition speed that the oil phase is added at an amount of 2 to 30% by weight per min, preferably 2 to 20% by weight per min, more preferably 5 to 20% by weight per min, based on the total weight of the emulsion, and that the whole system maintains a homogeneous emulsification state during this addition.

In a preferred embodiment of the present process, in order to prepare the O/W emulsion, the oil phase is used at an amount of 50 to 95% by weight, the water phase is used at an amount of 4.5-45% by weight and the emulsifier is used at an amount of 0.5 to 5 % by weight, based on the total weight of the O/W emulsion.

In the process for preparing the O/W emulsion according to the present invention, preferably, the oil phase is selected so that the temperature at which the whole oil phase begins to freeze when gradually cooled from liquid (that is, starting freezing temperature) shall be lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C.

For the oil phase, it may comprise only one oil ingredient or as well as two or more oil ingredients. When only one oil ingredient is comprised, the melting point of this oil ingredient must meet the above requirements, that is, lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C. When two or more oil ingredients are comprised, the melting point of every oil ingredient shall be lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C, or as well as at least one of the oil ingredients has a melting point of lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C, with the provision that the starting freezing temperature of the whole oil phase is lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C.

For oil ingredients which can be selected in the present invention, they can be those commonly used in the oil phase of personal care and cosmetic O/W emulsions, as long as the starting freezing temperature of the whole oil phase is lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C. For example, they can be vaseline or mineral oil (white oil).

In a further preferred embodiment of the process according to the present invention, the oil phase comprises one or more components selected from the group consisting of ethylhexyl palmitate (TEGO®SOFT OP), diethylhexyl carbonate (TEGO®SOFT DEC), octyldodecanol (TEGO®SOFT G20), PPG-14 butyl ether (TEGO®SOFT PBE) and triisostearin (TEGO®SOFT TIS). Especially, the oil phase

comprises two ingredients from this group, for example, the combination of ethylhexyl palmitate and diethylhexyl carbonate, or the combination of ethylhexyl palmitate and PPG-14 butyl ether, or the combination of octyldodecanol and triisostearin.

Besides, the oil phase may additionally comprise one or more selected from the group consisting of polysiloxane compounds, aliphatic hydrocarbons, aliphatic alcohols and aliphatic esters. The polysiloxane compounds are preferably selected from cyclomethicone, dimethicone and silicone elastomer. Preferred silicone elastomer includes Dow Corning® 9701. These additional oil ingredients shall be selected in such a way that the starting freezing temperature of the resulting whole phase also meets the above requirement, that is, lower or equal to 0°C, preferable lower or equal to -10°C, more preferable lower or equal to -15°C.

In order to prepare the O/W emulsion, generally, the oil phase is used at an amount of 50-95 wt.%, preferably 55-90 wt.%, more preferably 60-85 wt.%, particularly preferably 65-80 wt.%, based on the total weight of the O/W emulsion.

In the process for preparing the O/W emulsion according to the present invention, the water phase is used to form a continuous phase of the O/W emulsion while the oil phase forms a dispersing phase of the O/W emulsion. In the water phase, it can comprise only water or other water soluble substances besides water. These substances can be those usually contained in the water phase of personal care and cosmetic O/W emulsions. They especially include ethanol and/or C_2-C_5-polyols with two or more hydroxyls (for example three hydroxyls), the latter preferably being glycol, propylene glycol, 1,3-butanediol or any combination thereof, especially glycol. In addition, water soluble polymer(s) can also be comprised in the water phase, which is one or more selected from the group consisting of xanthan gum, guar gum, chondroitin sodium sulfate, sodium hyaluronate, arabic gum, sodium alginate, carrageen, hydroxypropyl cellulose, methyl cellulose, substituted methyl cellulose (e.g. hydroxypropyl methyl cellulose), polyacrylics, and polyacrylics with alkyl substitutent(s) in the main chain (e.g. TEGO® Carbomer 341 ER). The water soluble polymer is xanthan gum in particular.

In order to prepare the O/W emulsion, generally, the water phase is used at an amount of 4.5-45 wt.%, preferably 9.5-40 wt.%, more preferably 14.5-35 wt.%, particularly preferably 19.5-30 wt.%, based on the total weight of the O/W emulsion.

In the process for preparing the O/W emulsion according to the present invention,

besides the oil and water phases, emulsifier(s) for emulsifying these two phases to form the O/W emulsion is used. For the purpose of the present invention, the emulsifier(s) is one(s) commonly used for personal care and cosmetic O/W emulsions. It may be nonionic or anionic or cationic emulsifier(s).

As nonionic emulsifier(s), the present invention preferably uses one or more selected from the group consisting of ethoxylated fatty acid esters, polyglyceryl esters of fatty acid, ethoxylated ether of fatty alcohol, fatty acid sugar esters (e.g. fatty acid glucose ester and fatty acid sucrose ester) and ethoxylated dimethicone. More preferred nonionic emulsifier(s) is one or two selected from the group consisting of ethoxylated fatty acid sorbitol esters, polyglyceryl-3-methylglucose di-stearate (TEGO® Care 450), ceteareth-15 and glyceryl stearate (TEGO® Care 215), and cetearyl glucoside (TEGO® Care CG90).

As anionic emulsifier(s), glyceryl stearate citrate (Axol C62) is particularly preferred. As cationic emulsifier(s), distearyldimonium chloride (Varisoft TA100) is particularly preferred.

In the process for preparing the O/W emulsion according to the present invention, emulsifier(s) is in particular polyglyceryl-3-methylglucose di-stearate (TEGO® Care 450)、Ceteareth-15 and glyceryl stearate (TEGO® Care 215)、glyceryl stearate citrate (Axol C62)、distearyldimonium chloride (Varisoft TA100) or cetearyl glucoside (TEGO® Care CG90).

In the process for preparing the O/W emulsion according to the present invention,, generally, the emulsifier is used at an amount of 0.5-5 wt.%, based on the total weight of the O/W emulsion.

Of course, a skilled person in the art should understand that in the water or oil phase used in the process according to the present invention, other conventional ingredients for personal case and cosmetic O/W emulsions can also be contained. Whether these ingredients are appropriately contained in the water phase or in the oil phase is determined according to the common knowledge in the art.

In the process according to the present, use may also be made, as emollients, of all cosmetic oils, in particular mono- or diesters of linear and/or branched mono- and/or dicarboxylic acids having 2 to 44 carbon atoms with saturated or unsaturated and linear and/or branched alcohols having 1 to 22 carbon atoms. Use may likewise be made of the esterification products of bifunctional aliphatic alcohols having 2 to 36 carbon atoms with monofunctional aliphatic carboxylic acids having 1 to 22 carbon atoms. Furthermore, long-chain arylcarboxylic acid

esters, such as, e.g., esters of benzoic acid, e.g. benzoic acid esters of saturated or unsaturated and linear or branched alcohols having 1 to 22 carbon atoms, or also isostearyl benzoate or octyldodecyl benzoate, are suitable. Additional monoesters suitable as emollients and oil components are, e.g., the methyl esters and isopropyl esters of fatty acids having 12 to 22 carbon atoms, such as, e.g., methyl laurate, methyl stearate, methyl oleate, methyl erucate, isopropyl palmitate, isopropyl myristate, isopropyl stearate or isopropyl oleate. Other suitable monoesters are, e.g., n-butyl stearate, n-hexyl laurate, n-decyl oleate, isooctyl stearate, isononyl palmitate, isononyl isononanoate, 2 ethylhexyl palmitate, 2-ethylhexyl laurate, 2 hexyl-decyl stearate, 2-octyldodecyl palmitate, oleyl oleate, oleyl erucate or erucyl oleate, and also esters which can be obtained from industrial aliphatic alcohol cuts and industrial aliphatic carboxylic acid mixtures, e.g. esters of unsaturated fatty alcohols having 12 to 22 carbon atoms and saturated and unsaturated fatty acids having 12 to 22 carbon atoms, such as are accessible from animal and vegetable fats. However, naturally occurring monoester or wax ester mixtures, such as are present, e.g., in jojoba oil or in sperm oil, are also suitable. Suitable dicarboxylic acid esters are, e.g., di(n-butyl) adipate, di(n-butyl) sebacate, di(2-ethyl-hexyl) adipate, di(2-hexyldecyl) succinate or diiso-tridecyl azelate. Suitable diol esters are, e.g., ethylene glycol dioleate, ethylene glycol diisotri-decanoate, propylene glycol di(2-ethylhexanoate), butanediol diisostearate and neopentyl glycol dicaprylate. Additional fatty acid esters which can be used as emollients are, e.g., C12-15-alkyl benzoate, dicaprylyl carbonate or diethylhexyl carbonate. Use may likewise be made, as emollients and oil components, of relatively long-chain triglycerides, i.e. triple esters of glycerol with three acid molecules, at least one of which is a relatively long-chain acid molecule. Mention may be made here, by way of example, of fatty acid triglycerides; use may be made as such, as emollients and oil components, of, for example, natural vegetable oils, e.g. olive oil, sunflower oil, soybean oil, peanut oil, rapeseed oil, almond oil or palm oil, but also the liquid portion of coconut oil or palm kernel oil, and also animal oils, such as, e.g., neatsfoot oil or the liquid portions of beef tallow, or also synthetic triglycerides of caprylic/capric acid mixtures, triglycerides of industrial oleic acid, triglycerides with isostearic acid or triglycerides of palmitic/oleic acid mixtures. Use may furthermore be made of hydrocarbons, in particular also liquid paraffins and isoparaffins. Examples of hydrocarbons which can be used are paraffin oil, isohexadecane, polydecene, petroleum jelly, light liquid paraffin or squalane. Use may further also be made of linear or branched fatty alcohols, such as oleyl alcohol or octyldodecanol, and also fatty alcohol ethers, such as dicaprylyl ether. Suitable silicone oils and waxes are, e.g., polydimethylsiloxanes, cyclomethylsiloxanes and also aryl- or alkyl- or alkoxy-substituted polymethyl-siloxanes or cyclomethylsiloxanes.

All thickening agents known to a person skilled in the art are possible as thickeners for the thickening of oil phases. Mention may in particular be made, in this connection, of waxes, such as hydrogenated castor wax, beeswax or microcrystalline wax. Furthermore, use may also be made of inorganic thickening agents, such as silica, alumina or layered silicates (e.g. hectorite, laponite or saponite). These inorganic oil-phase thickeners can in this connection by hydrophobically modified. Use may in this connection be made, for the thickening/stabilizing of water-in-oil emulsions, of in particular aerosils, layered silicates and/or metal salts of fatty acids, such as, e.g., zinc stearate.

Possible viscosity regulators include, e.g., NaCl, low molecular weight nonionic surfactants, such as cocamide DEA/MEA and laureth-3, or polymeric high molecular weight associative highly-ethoxylated fatty derivatives, such as PEG-200 hydrogenated glyceryl palmate.

Use may be made, as UV/light screening agents, for example, of organic substances which are in a position to absorb ultraviolet radiation and to readmit the absorbed energy in the form of longer wavelength radiation, e.g. heat. UV-B screening agents may be oil-soluble or water-soluble. Mention may be made, as oil-soluble UV-B/light screening agents, e.g., of:

3-Benzylidenecamphor and the derivatives thereof, e.g. 3-(4-methylbenzylidene)camphor,
4-Aminobenzoic acid derivatives, such as, e.g., 2 ethylhexyl 4-(dimethylamino)benzoate and amyl 4 (dimethylamino)benzoate,
Cinnamic acid esters, such as, e.g., 2-ethylhexyl 4 methoxycinnamate, isopentyl 4-methoxycinnamate or 2-ethylhexyl 2-cyano-2-phenylcinnamate (octocrylene),
Salicylic acid esters, such as, e.g., 2-ethylhexyl salicylate, 4-isopropylbenzyl salicylate or homo-menthyl salicylate,
Benzophenone derivatives, such as, e.g., 2-hydroxy-4-methoxybenzophenone, 2-hydroxy-4-methoxy-4'-methylbenzophenone or 2,2'-dihydroxy-4-methoxy-benzophenone
Benzalmalonic acid esters, such as, e.g., di(2-ethyl-hexyl) 4-methoxybenzalmalonate,
Triazine derivatives, such as, e.g., 2,4,6-trianilino-(p-carbo-2'-ethyl-1'-hexyloxy)-1,3,5-triazine and octyl triazone,
Propane-1,3-diones, such as, e.g., 1-(4-(tert-butyl)phenyl) -3-(4'-methoxyphenyl) propane-1,3 -dione.

Possible water-soluble UV-B/light screening agents are:
2-Phenylbenzimidazole-5-sulphonic acid and the alkali metal, alkaline earth metal, ammonium, alkyl-ammonium, alkanolammonium and glucammonium salts thereof,
Sulphonic acid derivatives of benzophenone, such as, e.g., 2-hydroxy-4-methoxybenzophenone -5-sulphonic acid and its salts,
Sulphonic acid derivatives of 3-benzylidenecamphor, such as, e.g., 4-(2-oxo-3-bornylidene- methyl) benzenesulphonic acid and 2-methyl-5-(2-oxo-3-bornylidene) benzenesulphonic acid and the salts thereof.

Derivatives of benzoylmethane are possible in particular as typical UV-A/light screening agents, such as, for example, 1-(4'-(tert-butyl)phenyl)-3-(4'-methoxyphenyl)propane-1,3-dione or 1-phenyl-3-(4'-iso-propylphenyl)propane-1,3-dione. The UV-A and UV-B screening agents can obviously also be used in mixtures.

In addition to the soluble substances mentioned, insoluble pigments, namely finely dispersed metal oxides or salts, are also possible for this purpose, such as, for example, titanium dioxide, zinc oxide, iron oxide, aluminium oxide, cerium oxide, zirconium oxide, silicates (talc), barium sulphate and zinc stearate. The particles shall, in this connection, exhibit a mean diameter of less than 100 nm, e.g. between 5 and 50 nm and in particular between 15 and 30 nm. They may exhibit a spherical form; however, use may also be made of those particles which have an ellipsoidal form or a form deviating in another way from the spherical shape. A relatively new category of light screening agents comprises micronized organic pigments, such as, for example, 2,2'-methylenebis{6-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethyl-butyl)phenol}, with a particle size of less than 200 nm, which is available, e.g., as a 50% aqueous dispersion.
Additional suitable UV/light screening agents can be found in the review by P. Finkel in SÖFW-Journal 122, 543 (1996).

In addition to the two groups of primary UV/light screening agents mentioned above, secondary light screening agents of the antioxidants type can also be used which interrupt the photochemical reaction chain, which is triggered if UV radiation penetrates the skin. Use may be made, as antioxidants, e.g., of superoxide dismutase, tocopherols (vitamin E), dibutylhydroxy-toluene and ascorbic acid (vitamin C).

Use may be made, as solids, for example of iron oxide pigments, titanium dioxide or zinc oxide particles and those additionally mentioned under "UV protecting agents". Furthermore, use may also be made of particles which result in special

sensory effects, such as, for example, nylon-12, boron nitride, polymer particles, such as, for example, polyacrylate or polymethacrylate particles, or silicone elastomers.

Use may be made, as pearlescent additives, e.g., of glycol distearates or PEG-3 distearate.
Use may be made, as insect repellents, for example of N,N-diethyl-m-toluamide, 1,2-pentanediol or insect repellent 3535.

Use may be made, as self-tanning agents, e.g. of dihydroxyacetone and erythrulose.

Use may be made, as preservatives, for example of mixtures of individual or several alkylparaben esters with phenoxyethanol. The alkylparaben esters can be methylparaben, ethylparaben, propylparaben and/or butylparaben. Use may also be made, in place of phenoxyethanol, of other alcohols, such as, for example, benzyl alcohol or ethanol. In addition, use may also be made of other normal preserving agents, such as, for example, sorbic or benzoic acid, salicylic acid, 2-bromo-2-nitropropane-1,3-diol, chloroacetamide, diazolidinyl urea, DMDM hydantoin, iodopropynyl butyl-carbamate, sodium hydroxymethylglycinate, methyliso-thiazoline, chloromethylisothiazoline, ethylhexyl-glycerine or caprylyl glycol.

Use may be made, as fragrances, of natural or synthetic odoriferous substances or mixtures thereof. Natural odoriferous substances are extracts of flowers (lily, lavender, rose, jasmine, neroli or ylang-ylang), stems and leaves (geranium, patchouli, petitgrain), fruits (anis, coriander, caraway, juniper), fruit rinds (bergamot, lemon, orange), roots (mace, angelica, celery, cardamom, costus, iris, thyme), needles and twigs (spruce, fir, pine, mountain pine) and resins and balsams (galbanum, elemi, benzoin, myrrh, frankincense, opoponax). Animal raw materials are also possible, such as, for example, civet and castoreum. Typical synthetic perfume compounds are products of the ester, ether, aldehyde, ketone, alcohol and hydrocarbon types. Perfume compounds of the ester type are, e.g., benzyl acetate, phenoxyethyl isobutyrate, p-(tert-butyl)cyclo-hexyl acetate, linalyl acetate, dimethylbenzylcarbinyl acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, ethylmethyl phenylglycinate, allylcyclohexyl propionate, styrallyl propionate and benzyl salicylate. The ethers include, for example, benzyl ethyl ether, the aldehydes include, e.g., linear alkanals having 8 to 18 carbon atoms, citral, citronellal, citronellyloxy-acetaldehyde, cyclamen aldehyde, hydroxycitronellal, lilial and bourgeonal, the ketones include, e.g., the ionones, isomethyl ionone and methyl cedryl ketone, the alcohols include anethole,

citronellol, eugenol, isoeugenol, geraniol, linalool, phenylethyl alcohol and terpineol, and the hydrocarbons include mainly the terpenes and balsams. Use may be made of mixtures of different odoriferous substances which together generate an attractive scent. Essential oils of low volatility, which are generally used as flavouring components, are also suitable as fragrances, e.g. sage oil, camomile oil, clove oil, balm oil, peppermint oil, cinnamon leaf oil, linden blossom oil, juniper berry oil, vetiver oil, frankincense oil, galbanum oil, labdanum oil and lavandin oil. Use may be made of bergamot oil, dihydromyrcenol, lilial, lyral, citronellol, phenylethyl alcohol, hexyl-cinnamaldehyde, geraniol, benzylacetone, cyclamen aldehyde, linalool, Boisambrene Forte, Ambroxan, indole, Hedione, Sandelice, lemon oil, mandarin oil, orange oil, allyl amyl glycolate, cyclovertal, lavandin oil, clary sage oil, damascone, geranium oil Bourbon, cyclohexyl salicylate, Vertofix Coeur, Iso-E-Super, Fixolide NP, Evernyl, Iraldein gamma, phenylacetic acid, geranyl acetate, benzyl acetate, rose oxide, Romillat, Irotyl and Floramat, alone or in mixtures.

Use may be made, as colorants, of the substances suitable and authorized for cosmetic purposes, such as are compiled, for example, in the publication "Kosmetische Färbemittel" [Cosmetic Colouring Agents] of the Farbstoffkommission der Deutschen Forschungsgemeinschaft [Colorant Commission of the German Research Association], Verlag Chemie, Weinheim, 1984, pp. 81-106. These colorants are only used in concentrations of 0.001 to 0.1% by weight, based on the complete mixture.

Biogenic active substances with conventional volume can be used in the present O/W emulsion. The term "biogenic active substances" is to be understood as meaning, for example, tocopherol and derivatives, ascorbic acid and derivatives, retinol and derivatives, deoxyribonucleic acid, coenzyme Q10, bisabolol, allantoin, phytantriol, panthenol, hydroxy acids, salicylic acid, amino acids, amino acid derivatives, hyaluronic acid, glucans, creatine and creatine derivatives, guanidine and guanidine derivatives, ceramides, phytosphingosine and phytosphingosine derivatives, sphingosine and sphingosine derivatives, pseudoceramides, essential oils, peptides, protein hydrolysates, plant extracts and vitamins and vitamin mixtures.

It is possible to have present, as care additives, e.g., ethoxylated glycerol fatty acid esters, such as, for example, PEG-7 glycerol cocoate, or cationic polymers, such as, for example, polyquaternium-7, or polyglycerol esters.

In step (b) of the process according to the present invention, an oil phase in a liquid state is added into the water phase mixture obtained in step (a) under

agitation at such an addition speed that the oil phase is added at an amount of 2 to 30% by weight per min, preferably 2 to 20% by weight per min, more preferably 5 to 20% by weight per min, based on the total weight of the emulsion, and that the whole system maintains a homogeneous emulsification state during this addition. For the purpose of the present invention, said "homogeneous emulsification state" means that the whole system has a uniform appearance and no phase separation occurs. In fact, the addition speed employed in step (b) may be any one at which the oil phase is added into the water phase mixture without breaking the emulsification state of the system. However for the sake of economic interests, said addition speed is intended to be one at which the oil phase is added into the water phase mixture as soon as possible but without breaking the homogeneous emulsification state of the system. Those skilled in this art can select an appropriate speed according to ratio of oil phase to water phase, total amount of the emulsion, agitation mode and rotation speed of agitator so that the whole system maintains a homogeneous emulsification state during this addition. In a preferred embodiment of the process according to present invention, the addition speed is relatively slow when the weight ratio of the oil phase already added at an earlier stage in step (b) to the water phase is lower than one third; afterwards, the addition speed can be raised to a higher one appropriately. Taking a laboratory dosage of 100-1000 g for example, it generally takes 5-15 minutes to add the oil phase to the water phase mixture while keeping the whole emulsion system homogenously. For mass production in factories, it takes 5-30 minutes to add the oil phase to the water phase mixture while maintaining a homogeneous emulsification state of the whole system during this addition.

In preferred embodiments, the process according to the present invention further comprises the step of

(c) increasing the agitation speed for homogenization after the completion of the addition of the oil phase in step (b), to obtain the O/W emulsion.

In step (c) of the process according to the present invention, after the completion of the addition of the oil phase in step (b), the agitation speed is increased for homogenization, to obtain the O/W emulsion. The purpose of step (c) is to homogenize the mixture system. This homogenization normally requires a higher agitation speed or shearing speed, than the one employed in step (b). The agitation speed in step (b) is usually 600-1000 rpm, preferable 700-1000 rpm; while that in step (c) is usually 1000-16000 rpm, preferable 1000-13000 rpm. To this end, jet-flow agitators provided by IKA® can be used in the present invention, including IKA® RW 28, IKA® RW 20, IKA® RW 14 and IKA® RW 16.

A homogenous emulsion is already obtained in step (b). The purpose of step (c) is to further homogenize the emulsion system and thus improve the appearance and stability of the final product.

In steps (a)-(c), in order to obtain the mixture systems desired in every step faster, or obtain a more homogenous mixture system desired in every step, or for both of them or other purposes, steps (a)-(c) can be carried out under heating. Of course, they can also be carried out without heating.

The process according to the present invention is suitable for the preparation of not only skin care products but hair care products.

According to the second aspect, the present invention provides an O/W emulsion, which is prepared according to the process according to the present invention. The O/W emulsion prepared by this process may have a high oil-phase content and is stable for storage.

The O/W emulsion having a high oil-phase content according to the present invention has a milk white cream appearance, or becomes a transparent gel appearance as desired. This emulsion, when applied to the skin, releases emollient oil rapidly, serves for make-up removing, massage or humectant function or the like, and can also be used as a substitute for baby oil. Therefore, The O/W emulsion having a high oil-phase content according to the present invention can be widely used in various personal care and cosmetic products.

Thus, according to the final aspect, the present invention provides the use of the O/W emulsion having a high oil-phase content prepared according to the process of the present invention in make-up removers, massage preparations, baby care products and sun care preparations.

Except for indication, the wt.% of the components used in the present invention are all presented based on the total weight of the emulsion.

The following examples are intended to illustrate the present invention and should not be construed to restrict the scope of the present invention.

Examples

Examples 1-3

The formulas of Examples 1-3 are as shown in Table 1.

Table 1

		Example1	Example2	Example3
	Ingredients	wt. %	wt. %	wt. %
A	ethylhexyl palmitate(TEGO® SOFT OP)	45		34
	diethylhexyl carbonate(TEGO® SOFT DEC)	40	95	41
B	polyglyceryl-3-methylglucose di-stearate (TEGO® Care 450)	1		
	ceteareth-15 and glyceryl stearate (TEGO® Care 215)		1	5
	glycerol	3	1	3
	xanthan gum	0.1	0.1	0.2
	chloroacetamide(preservative)	0.1	0.1	0.1
	water	10.8	2.8	16.7
		Product 1	Product 2	Product 3

Product 1 is prepared according to the following steps:

(i) Adding 3 wt.% of glycerol, 0.1 wt.% of xanthan gum, 1 wt.% of emulsifier TEGO® Care 450 and 0.1 wt.% of preservative chloroacetamide into 10.8 wt.% of water in sequence, mixing and heating up to 80℃ with agitation, to obtain phase B;

(ii) adding the oil phase A composed of 45 wt.% of ethylhexyl palmitate (TEGO® SOFT OP) and 40 wt.% of diethylhexyl carbonate(TEGO® SOFT DEC) in a liquid state, into the phase B obtained in step (i) at an agitation speed of 800 rpm over 5 minutes (that it, adding 19 wt.% of the oil phase per min), during which the whole system always maintains a homogeneous emulsification state; and

(iii) after the completion of the addition of the oil phase A, increasing the agitation speed up to 10,000 rpm for homogenization, to obtain a high oil-phase content O/W emulsion Product 1 at a total of 200g.

A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

Product 2 is prepared in a similar way to that for preparing Product 1 in Example 1, wherein the oil phase A composed of 95 wt.% of diethylhexyl carbonate(TEGO®

SOFT DEC) in a liquid state, is added into the phase B over 15 minutes (that it, adding 6.3wt.% of the oil phase per min), and finally Product 2 is obtained at a total of 800g. A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

Product 3 is prepared in a similar way to that for preparing Product 1 in Example 1, wherein the oil phase A composed of 34 wt.% of ethylhexyl palmitate (TEGO® SOFT OP) and 41 wt.% of diethylhexyl carbonate(TEGO® SOFT DEC) in a liquid state, is added into the phase B over 10 minutes (that it, adding 7.5 wt.% of the oil phase per min), and finally Product 3 is obtained at a total of 2000g. A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

Each of these three products subjects to ambient-temperature stability test, heat-resistant test and freezing-melting test. The ambient-temperature stability test is storing samples at room temperature (between 20 and 25°C) for 3 months. The heat-resistant test is storing samples at 45°C for 3 months. And the freezing-melting test is samples are cooled at -15 °C and then heated at room temperature for three cycles in total. Test results shows that these products after the above tests still maintain homogenous appearances without discoloration and layer-separation and the like. From these, it is evident that Products 1-3 still have excellent storage stability at a high oil-phase content.

Products 1-3 are in the form of cream, of no apparent fluidity, convenient to use, and have low requirements on package. When applied to the skin, large quantities of emollient oil are released, and serve for make-up removing, massage or humectant function or the like. These products can exhibit the function of oil preparations very well but without common advantages of the same, including high fluidity and permeability.

Example 4

The formula of the O/W emulsion of Example 4 is as shown in Table 2.

Table 2

	Ingredients	wt. %
A	ethylhexyl palmitate(TEGO®SOFT OP)	30
	PPG-14 butyl ether(TEGO®SOFT PBE)	55
B	glyceryl stearate citrate(Axol C62)	1
	xanthan gum	0.1
	carbomer	0.1
	sodium hydroxide	0.02
	chloroacetamide(preservative)	0.1
	water	13.68

In a similar way to that for preparing Product 1 in Example 1, the O/W emulsion having the formula of Table 2 is prepared, wherein the oil phase A composed of 30 wt.% of ethylhexyl palmitate (TEGO® SOFT OP) and 55 wt.% of PPG-14 butyl ether(TEGO®SOFT PBE) in a liquid state, is added into the phase B over 15 minutes (that it, adding 5.7 wt.% of the oil phase per min), and finally Product 4 is obtained at a total of 1000g. A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

The emulsion obtained in this Example has a milk white cream appearance and has a good stability. Product 4 subjects to ambient-temperature stability test, heat-resistant test and freezing-melting test. The ambient-temperature stability test is storing samples at room temperature (between 20 and 25°C) for 3 months. The heat-resistant test is storing samples at 45°C for 3 months. And the freezing-melting test is samples are cooled at -15 °C and then heated at room temperature for three cycles in total. Test results shows that this product after the above tests still maintains homogenous appearance without discoloration and layer-separation and the like. From these, it is evident that Product 4 still has excellent storage stability. Likewise, when applied to the skin, large quantities of emollient oil are rapidly released, and serve for make-up removing, massage and humectant function very well.

Example 5

The formula of the O/W emulsion of Example 5 is as shown in Table 3.

Table 3

	Ingredients	wt. %
A	octyldodecanol(TEGO®SOFT G20)	25
	triisostearin(TEGO®SOFT TIS)	35
B	distearyldimonium chloride(Varisoft TA100)	1
	glycerol	3.0
	chloroacetamide(preservative)	0.1
	water	35.9

In a similar way to that for preparing Product 1 in Example 1, the O/W emulsion having the formula of Table 3 is prepared, wherein the oil phase A composed of 25 wt.% of octyldodecanol(TEGO®SOFT G20) and 35 wt.% of triisostearin(TEGO®SOFT TIS) in a liquid state, is added into the phase B over 5 minutes (that it, adding 12 wt.% of the oil phase per min), and finally Product 5 is obtained at a total of 2000g. A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

The emulsion obtained in this Example has a milk white cream appearance. Product 5 subjects to ambient-temperature stability test, heat-resistant test and freezing-melting test. The ambient-temperature stability test is storing samples at room temperature (between 20 and 25°C) for 3 months. The heat-resistant test is storing samples at 45°C for 3 months. And the freezing-melting test is samples are cooled at -15 °C and then heated at room temperature for three cycles in total. Test results shows that this product after the above tests still maintains homogenous appearance without discoloration and layer-separation and the like. From these, it is evident that Product 5 still has excellent storage stability. When applied to the skin, appropriate quantities of emollient oil are rapidly released, and serve for make-up removing, massage and humectant function very well.

Example 6

The formula of the O/W emulsion of Example 6 is as shown in Table 4.

Table 4

	Ingredients	wt. %
A	white oil	35
	triisostearin(TEGO®SOFT TIS)	35
B	cetearyl glucoside(TEGO® Care CG90)	1
	glycerol	3.0
	chloroacetamide(preservative)	0.1
	water	25.9

In a similar way to that for preparing Product 1 in Example 1, the O/W emulsion having the formula of Table 4 is prepared, wherein the oil phase A composed of 35 wt.% of white oil and 35 wt.% of triisostearin(TEGO®SOFT TIS) in a liquid state, is added into the phase B over 10 minutes (that it, adding 7 wt.% of the oil phase per min), and finally Product 6 is obtained at a total of 1000g. A homogenous emulsion is already obtained in step (ii) of this Example and step (iii) aims at further homogenizing the emulsion system and thus improves the appearance and stability of the final product.

The emulsion obtained in this Example has a milk white cream appearance. Product 6 subjects to ambient-temperature stability test, heat-resistant test and freezing-melting test. The ambient-temperature stability test is storing samples at room temperature (between 20 and 25°C) for 3 months. The heat-resistant test is storing samples at 45°C for 3 months. And the freezing-melting test is samples are cooled at -15 °C and then heated at room temperature for three cycles in total. Test results shows that this product after the above tests still maintains homogenous appearance without discoloration and layer-separation and the like. From these, it is evident that Product 6 still has excellent storage stability. When applied to the skin, appropriate quantities of emollient oil are rapidly released, and serve for make-up removing, massage and humectant function very well.

Claims

1. A process for preparing an O/W emulsion, comprising the steps of:

   (a) mixing a water phase and an emulsifier to obtain a homogeneous water phase mixture; and

   (b) adding an oil phase in a liquid state into the water phase mixture obtained in step (a) under agitation at such an addition speed that the oil phase is added at an amount of 2 to 30% by weight per min, preferably 2 to 20% by weight per min, more preferably 5 to 20% by weight per min, based on the total weight of the emulsion, and that the whole system maintains a homogeneous emulsification state during this addition.

2. The method of claim 1, wherein the oil phase is at an amount of 50 to 95% by weight, the water phase is at an amount of 4.5-45% by weight and the emulsifier is at an amount of 0.5 to 5 % by weight, based on the total weight of the emulsion.

3. The method of claim 1 or 2, wherein the oil phase is at an amount of 55-90 wt.%, preferably 60-85 wt.%, particularly preferably 65-80 wt.%, and the water phase is at an amount of 9.5-40 wt.%, preferably 14.5-35 wt.%, particularly preferably 19.5-30 wt.%, based on the total weight of the O/W emulsion.

4. The method of any of claims 1-3, wherein the oil phase comprises one or more components selected from the group consisting of ethylhexyl palmitate, diethylhexyl carbonate, octyldodecanol, PPG-14 butyl ether and triisostearin.

5. The method of any of claims 1-4, wherein the emulsifier is nonionic, anionic or cationic emulsifier, wherein

   the nonionic emulsifier is one or more selected from the group consisting of ethoxylated fatty acid esters, polyglyceryl esters of fatty acid, ethoxylated ether of fatty alcohol, fatty acid sugar esters (e.g. fatty acid glucose ester and fatty acid sucrose ester) and ethoxylated dimethicone; the nonionic emulsifier is preferably one or two selected from the group consisting of ethoxylated fatty acid sorbitol esters, polyglyceryl-3-methylglucose di-stearate, ceteareth-15 and glyceryl stearate, and cetearyl glucoside;

   the anionic emulsifier is glyceryl stearate citrate; and

   the cationic emulsifier is distearyldimonium chloride.

6. The method of any of claims 1-5, wherein the water phase comprises a water soluble polymer, which is one or more selected from the group consisting of xanthan gum,

guar gum, chondroitin sodium sulfate, sodium hyaluronate, arabic gum, sodium alginate, carrageen, hydroxypropyl cellulose, methyl cellulose, hydroxypropyl methyl cellulose, polyacrylics, and substituted polyacrylics.

7. The method of any of claims 1-6, wherein the oil phase comprises one or more selected from the group consisting of polysiloxane compounds, aliphatic hydrocarbons, aliphatic alcohols and aliphatic esters, and said polysiloxane compounds is preferably selected from the group consisting of cyclomethicone, dimethicone and silicone elastomer.

8. The method of any of claims 1-7 wherein the water phase, besides water, comprises ethanol and/or C_2-C_5-polyols with two or more hydroxyls, the latter preferably being glycol, propylene glycol, 1,3-butanediol or any combination thereof.

9. The method of any of claims 1-8, further comprising the step of

(c) increasing the agitation speed for homogenization after the completion of the addition of the oil phase in step (b), to obtain the O/W emulsion.

10. An O/W emulsion prepared according to the process of any of claims 1-9.

11. The use of the O/W emulsion of claim 10 in make-up removers, massage preparations, baby care products and sun care preparations.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2008/073859

A. CLASSIFICATION OF SUBJECT MATTER

See the extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: A61k7-/; 8-/; A61Q; C08L

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)

CPRS,CNKI, WPI, EPODOC,PAJ: (OIL AND IN AND WATER) OR ("O" W "W") OR (OIL W WATER) EMULSION? (OIL? W PHASE?) AND ((AQUEOUS OR WATER) W PHASE?) PALMITATE? OR CARBONATE? OR +STEARIN? OR+ DODECANOL? XANTHAN+ OR GUAR+ OR HYALURONATE? OR ARABIC OR CELLULOSE? OR SU LFATE? OR SULPHATE? OR ALGINATE? OR CARRAGEEN? OR POLYACYLIC?

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN1832722A(PROCTER & GAMBLE CO)13 Sep.2006(13.09.2006) Page 6, lines 1-19; example 1	1-3, 9-11
Y		4-8
X	CN1832723A(PROCTER & GAMBLE CO) 13 Sep.2006(13.09.2006) Page 7 line 25 – page 8 line 5; example 1	1-3,9-11
Y		4-8

☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&"document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
19 Aug.2009(19.08.2009)	**03 Sep. 2009 (03.09.2009)**
Name and mailing address of the ISA/CN The State Intellectual Property Office, the P.R.China 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088 Facsimile No. 86-10-62019451	Authorized officer HE,Fang Telephone No. (86-10)62084472

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INTERNATIONAL SEARCH REPORT

International application No.
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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	JP4368313A(KAO CORP)21 Dec.1992(21.12.1992) Claims	10-11
A	US2005/0238677A1(CHASE J ET AL.)27 Oct.2005(27.10.2005) The whole document	1-11

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2008/073859

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INTERNATIONAL SEARCH REPORT

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A61K8/81(2006.01)i

A61K8/84(2006.01)i

A61Q17/04(2006.01)i

A61Q19/10(2006.01)i