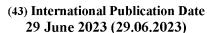
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(57) **Abstract:** The present invention relates to a liquid anhydrous composition comprising: • (a) at least one water soluble dye, • (b) at least one polyol, and • (c) a polyglyceryl-10 pentaoleate • (d) at least one volatile oil, wherein the relative mass amount of (a)/(b)/(c) is ranging between $1/0.6/8 \le (a)/(b)/(c) \le 1/6/18$, and a method for preparing the same. The composition may be used as a liquid lipstick formulation. The method for preparing the composition comprises adding the volatile oil (d) and all other optional ingredients into a premix prepared from components (a), (b) and (c).

LONG WEAR LIQUID ANHYDROUS COMPOSITION

Technical field of the invention

The invention relates to the field of cosmetic products, and more particularly long wear liquid cosmetic compositions allowing for improving the intensity of the color of the makeup and the long-lasting of their intense color. These cosmetic products can for example be used as liquid lipsticks formulations.

Technical background

The use of lipsticks as means for colouring lips has undergone rapid growth, such that lipsticks are now widespread beauty products. Originally, lipstick compositions were solid products melting on the lips upon application but with the growth of the lip makeup market, different textures from liquid to solid have been developed. Independently of its texture, a cosmetically acceptable lipstick should have a homogeneous colour and exhibit long wear properties.

In order to be attractive for the costumer, it is also desirable to offer one lip product under an expanded range of shades. However, colouring materials are either compatible with the water phase (dyes) or with the oily phase (pigments). It is for example complicated to combine dyes and pigments in anhydrous compositions since water-soluble dyes are incompatible in anhydrous compositions. In order to incorporate water soluble dyes in fatty continuous phase compositions, document WO 2017/162599 for example discloses formulating an emulsion, where the dispersed water phase contains a water-soluble dye. The incorporation of dyes in aqueous dispersed phases of emulsions often leads to bleeding of dyes resulting in non-uniform formulas.

Formulating liquid compositions containing pigments and dyes while having a uniform color and a low viscosity (below 300 mPa.s) remains a technical challenge.

Furthermore, when long wear properties are sought, it is conventional to use volatile oils that will evaporate rapidly after application of the makeup and therefore leave a nongreasy and long-lasting coloured film on the lips. However, the presence of volatile oils further decreases the viscosity of liquid formulations which renders the suspension of pigments even more complicated and can affect the homogeneity of the formulation.

It is therefore an objective of the present invention to provide a low viscosity liquid anhydrous composition comprising at least one water soluble dye, where said water soluble dye is homogeneously dispersed, and wherein the liquid anhydrous composition exhibits long wear properties, intensity of colour and long-lasting of the intense color.

It is indeed of the merits of the applicant to have discovered that water soluble dyes could be homogeneously and stably incorporated into low viscosity liquid anhydrous compositions, in the presence of at least one volatile oil, when combined with a specific surfactant, namely polyglyceryl-10 pentaoleate, and a polyol in a particular mass proportion.

Thus, the invention relates according to a first aspect, to a liquid anhydrous composition comprising:

- (a) at least one water soluble dye,
- (b) at least one polyol, and
- (c) a polyglyceryl-10 pentaoleate
- (d) at least one volatile oil,

wherein the relative mass amount of (a)/(b)/(c) is ranging between $1/0.6/8 \le (a)/(b)/(c) \le 1/6/18$.

The liquid anhydrous composition of the invention can be any makeup product for application to the keratinous materials, in particular skin or nails. In a preferred embodiment, the liquid anhydrous composition of the invention is a lip product, such as a liquid lipstick or a lipgloss.

The invention also relates, according to a further aspect, to a method for the preparation of said liquid anhydrous composition comprising the flowing steps:

- i. preparing a premix of
 - (a) at least one water soluble dye,
 - (b) at least one polyol, and
 - (c) polyglyceryl-10 pentaoleate

ii. incorporating of at least one volatile oil (d) and all other optional ingredients into the premix.

Within the meaning of the invention, the term "liquid composition" intends to mean a composition that flows under its own weight, as opposed to a solid composition.

In a preferred embodiment, the liquid anhydrous composition of the invention has a viscosity, as measured on TA Instruments DHR2 rheometer. Mobile: 40mm smooth cone and 4° angle; 96µm air gap; 40mm smooth support. Method: rotation at 100s-1 for 120s at 25°C, after a warm-up time of 60s., between 1 to 300mPa.s, preferably between 20 and 150mPa.s.

The composition of the invention is anhydrous. Within the meaning of the invention, the term "anhydrous composition" intends to mean a composition comprising very low amounts of water, in particular less than 5% by weight, with respect to the total weight of the composition, preferably less than 3% by weight, and more preferably less than 1% by weight of water. In a preferred embodiment, the composition of the invention is free from water.

Water soluble dye

The composition of the invention comprises at least one water soluble organic dye. The water-soluble dye can either be natural or of natural origin, or can be an organic dye

"Natural compound" means a compound that is obtained directly from the earth or the soil, or from plants or animals, via, where appropriate, one or more physical processes, for instance milling, refining, distillation, purification or filtration, or else resulting from a biotechno logical process, especially resulting from cell or microbiological cultures, for example of fungi or of bacteria.

"Compound of natural origin" means a natural compound that has undergone one or more additional chemical or industrial treatments, giving rise to modifications that do not affect the essential qualities of this compound and/or a compound predominantly comprising natural constituents that may or may not have undergone transformations as indicated above. Mention may be made, as nonlimiting example of additional chemical and industrial treatment bringing about modifications which do not affect the essential qualities of a natural compound, of those allowed by the controlling bodies such as Ecocert (Reference system for biological and ecological cosmetic products, January 2003), or defined in recognized handbooks in the field, such as Cosmetics and Toiletries Magazine, 2005, Vol. 120, 9: 10.

According to the invention, a compound is considered to be natural or of natural origin when it is predominantly composed of natural constituents, that is to say when the weight ratio of natural constituents to non-natural constituents which make up the compound is greater than 1.

"Water-soluble dye", and more generally a "water-soluble compound" means a dye or compound which has a solubility in water, measured at 25°C, at least equal to 0.01 g/1 (production of a macroscopically isotropic, transparent, colored or colorless solution).

By way of water-soluble dyes suitable for the invention, the following may especially be mentioned: water-soluble natural dyes such as, for example, betanin (beetroot), carmine, copper chlorophyllin, methylene blue, ortho-diphenol derivatives such as anthocyanins (radish, red cabbage, purple yam, purple corn, black carrot, hibiscus, elderberries), caramel, sandalwood, gardenia, spirulina and riboflavin.

It is more particularly a natural ortho-diphenol derivative.

The ortho-diphenols which may be used according to the invention are preferably chosen from anthocyanidins, such as cyanidin, delphinidin and petunidin; anthocyanins or anthocyans, such as myrtillin; proanthocyanidins and especially the proanthocyanidins Al, A2, B1, B2, B3 and CI; proanthocyanins and mixtures of the previous compounds.

In a preferred embodiment, the water-soluble dye is a water-soluble organic dye.

The water soluble organic dye can be chosen from CI 12085 (RED 36), CI 15850 (RED 6), CI 15850 (RED 7) [E180], CI 15985 (YELLOW 6) [E110], CI 17200 (RED 33), CI 19140 (YELLOW 5) [E102], CI 42090 (BLUE 1) [E133], CI 45370 (ORANGE 5), CI 45370 (ORANGE 5), CI 45380 (RED 22), CI 45410 (RED 27), CI 45410 (RED 28), CI 47000 (YELLOW 11), CI 47005 (YELLOW 10) [E104], CI 61570 (GREEN 5), Acid Red 18, CI 16255, Red 21, CI 45380, and mixtures thereof.

For obvious reasons, the amount of this dye or mixture of dyes present in a composition according to the invention is able to vary significantly with regard to the hue range and the chromatic intensity sought by its presence.

By way of illustration, a composition according to the invention may comprise from 0.01 % to 10% by weight, relative to the total weight of the composition, preferably from 0.1% to 5% by weight, and more preferably 0.5% to 3% by weight of water-soluble dyes.

According to another embodiment, the composition according to the invention contains at least one ortho-diphenol derivative, and preferably at least one anthocyanin derivative, as water-soluble natural dye.

Other colouring agents

In one embodiment of the invention, the liquid anhydrous composition comprises at least one coloring agent other than the water-soluble dye.

The colouring agent may particularly be chosen from among liposoluble colorants, pigments, nacres or one of the mixtures thereof. Preferably,

Examples of mineral pigments are particularly titanium dioxide, iron, zinc or chromium oxides, manganese violets, ultramarines, ferric ferrocyanide known as Prussian Blue, as well as composite pigments and goniochromatic, pearlescent, interferential, photochromic or thermochromic pigments, without this list being exhaustive.

Examples of organic pigments suitable for use in the invention are particularly carbon black, D&C type pigments, FD & C type pigments lacquers based on cochineal carmine, barium, strontium, calcium or aluminium or indeed diketopyrrolopyrrole (DPP) described in the documents EP-A-542669, EP-A-787730, EP-A-787731 and WO-A-96/08537.

The nacres may be chosen from among those conventionally present in makeup products, such as micas / titanium dioxide. Alternatively, they may consist of nacres based on mica / silica / titanium dioxide, based on synthetic fluorphlogopite / titanium dioxide (MAPRECOS SUNSHINE®), calcium sodium borosilicate / titanium dioxide (ENGELHARD REFLECKS®) or calcium aluminium borosilicate / silica / titanium dioxide (MERCK RONASTAR®).

These colouring agents other than the water-soluble dye may be optionally surface-treated with a hydrophobic agent such as silanes, silicones, fatty acid soaps, C₉₋₁₅ fluoroalcohol phosphates, acrylate/dimethicone copolymers, mixed C₉₋₁₅ fluoroalcohol phosphate / silicone copolymers, lecithins, carnauba wax, polyethylene, chitosan and optionally acylated amino acids such as lauroyl lysine, disodium stearoyl glutamate and aluminium acyl glutamate.

The colouring agents other than the water-soluble dye can be present in the composition at a content of between 3% and 10% by weight with respect to the total weight of the composition.

Polyol

The liquid anhydrous composition of the invention also comprises at least one polyol.

Preferably, a polyol in accordance with the present invention is present in liquid form at room temperature.

The polyols that are advantageously suitable for the formulation of a composition according to the present invention are those especially containing from 2 to 32 carbon atoms, preferably 3 to 16 carbon atoms and in particular from 3 to 7 carbon atoms.

Advantageously, the polyol may be chosen, for example, from ethylene glycol, pentaerythritol, trimethylolpropane, propylene glycol, 1,3-propanediol, butylene glycol, isoprene glycol, pentylene glycol, hexylene glycol, glycerol, polyglycerols such as glycerol oligomers, for instance diglycerol, caprylyl glycol, and polyethylene glycols, and mixtures thereof, in particular glycerol.

More preferably, the polyol is chosen from glycerol, butylene glycol, pentylene glycol, hexylene glycol, caprylyl glycol, and mixtures thereof, and preferably glycerol.

The polyol may be used in an amount ranging from 0.01% to 5% by weight, preferably from 0.1% to 3% by weight and even more preferentially from 0.2% to 1% by weight of polyol relative to the total weight of the composition.

Polyglyceryl-10 pentaoleate

The liquid anhydrous composition of the invention also comprises a polyglyceryl-10 pentaoleate.

This polyglyceryl fatty acid ester of oleic acid comprises 10 glycerol units and has a HLB value: 3.5 and is for example available under the commercial reference Nikkol Decaglyn 5-OV from NIKKO CHEMICALS Co. LTD.

The amount of polyglyceryl-10 pentaoleate in the composition according to the present invention may be 1% to 20% by weight, preferably 2% to 15% by weight, and more preferably 3% to 13% by weight, relative to the total weight of the composition.

The oils

The liquid anhydrous composition of the invention also comprises at least one volatile oil.

According to the present invention, the term "oil" denotes a liquid compound at ambient temperature (25°C), and which, when introduced at a rate of at least 1% by weight into water at 25°C, is not at all soluble in water, or soluble at a rate of less than 10% by weight, with respect to the weight of oil introduced into the water.

Volatile oil

"Volatile oil" denotes an oil suitable for evaporating in contact with skin in less than one hour, at ambient temperature and atmospheric pressure.

The volatile oil is a volatile cosmetic oil, liquid at ambient temperature, having particularly a non-zero vapour pressure at ambient temperature and atmospheric pressure, in particular having a vapour pressure of between 0.13 Pa and 40,000 Pa (0.001 to 300 mm of Hg), preferably between 1.3 Pa and 13,000 Pa (0.01 to 100 mm of Hg), and more preferentially between 1.3 Pa and 1,300 Pa (0.01 to 1,000 mm of Hg).

The volatile oils comprise volatiles silicone oils and/or volatile hydrocarbon oil.

The volatile silicone oils optionally used in the compositions according to the invention are linear or cyclic, have particularly from 2 to 7 silicon atoms, optionally alkyl or alkoxy groups having from 1 to 10 carbon atoms, and having a viscosity, at ambient temperature, less than 5 cSt.

By way of examples of volatile silicone oil, mention may more particularly be made of hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, cyclotetradimethylsiloxane, cyclopentadimethylsiloxane, cyclohexadimethylsiloxane, hexamethyldisiloxane, octamethyltrisiloxane, hexamethyltrisiloxane, decamethyltrisiloxane, decamethyltetrasiloxane, dodecamethylpentasiloxane, heptamethyltrisiloxane, heptamethyloctyl trisiloxane or one of the mixtures thereof.

The hydrocarbon volatile oil may be chosen from hydrocarbon oils having from 7 to 16 carbon atoms. As a hydrocarbon volatile oil having from 7 to 16 carbon atoms, mention can be made in particular of C₈-C₁₆ branched alkanes such as C₈-C₁₆ iso-alkanes (also referred to as isoparaffins), isododecane, isodecane, isohexadecane and for example the oils sold under the trade names Isopars or Permetyls, C₈-C₁₆ branched esters such as iso-hexyl neopentanoate, and mixtures thereof. Preferably, the hydrocarbon volatile oil having from 8 to 16 carbon atoms is chosen from isododecane, isodecane, isohexadecane and mixtures thereof, and is in particular isododecane.

Concerning the volatile hydrocarbon oil, mention may more particularly be made of a short-chain hydrocarbon oil, volatile linear alkanes such as for example described in document FR2933865 incorporated by way of reference.

By way of examples of short-chain hydrocarbon oil(s), mention may particularly be made of that/those chosen in the group comprising isododecane, dodecane or mixtures thereof.

By way of example of volatile linear alkanes, mention may be made of those hydrocarbon chains in:

- C9-C17, C10-C14, such as a mixture of undecane (C_{11}) and tridecane (C_{13}) , marketed by BASF Care Creations under the trade name Cetiol® Ultimate,
 - C15-19, such as those marketed by Seppic under the trade name Emogreen L15,
- C12-14, such as those marketed by Biosynthis under the trade name Vegelight 1214LC, n-dodecane (C12) marketed by Sasol under the tradename Parafol12-97 and n-tetradecane (C14) marketed by Sasol under the tradename Parafol 14-97,
- C9-12 alkane, such as those marketed by Daito under the trade name Makigreen D10 or those marketed by Biosynthis under the tradename Vegelight Silk.

In a preferred embodiment, the volatile oil is chosen from isododecane, the mixture of n-undecane (C_{11}) and n-tridecane (C_{13}), dodecane, or mixtures thereof.

The volatile oil is present in an amount between 20% and 99% by weight, relative to the total weight of the composition, preferably between 20 and 49% by weight, and wherein the mass amount of (a) + (b) + (c) represents from 1 to 80% of the total weight of the composition, preferably from 1 to 60% by weight.

Non-volatile oil

The liquid anhydrous composition of the invention can also comprise at least one non-volatile oil.

"Non-volatile oil" denotes an oil which has a boiling point generally greater than 300°C under 760 mm of Hg (101325 Pa) and which has little or no vapour tension.

The non-volatile oils may particularly be chosen from among non-volatile silicon oils, non-volatile hydrocarbon oils, and mixtures thereof.

"Silicone oil" denotes an oil comprising at least one silicon atom, and particularly at least one Si-O group.

As a non-volatile silicone oil, mention may particularly be made of polydimethylsiloxanes containing at least 8 silicon atoms, polyalkylmethylsiloxanes wherein the alkyl chain contains from 8 to 20 carbon atoms and the oils identified by the INCI name phenyl trimethicone.

"Hydrocarbon oil" denotes an oil containing only hydrogen and carbon atoms.

Mention may be made for example of hydrocarbons such as squalane, phytosqualane, polybutene, hydrogenated polyisobutene, hydrogenated polydecene, synthetic (poly) esters also known as "ester oils" and (poly) ethers, in particular C6-C20 acid and C6-C20 alcohol (poly) esters, advantageously branched such as isononyl isononanoate; plant oils; branched and/or unsaturated fatty acids; branched and/or unsaturated fatty alcohols such as octyldodecanol; or one of the mixtures thereof.

"Ester oil" denotes a mono-, di-, tri- or tetra-ester oil. The ester oils are obtained by reacting a mono-, di-, tri- and more generally a polyol with a mono- di- tri- and more generally a carboxylic polyacid, said reagents optionally being linear or branched, saturated or unsaturated, aliphatic or aromatic, and optionally comprising alkoxylated groups. The ester oils may particularly be hydroxylated.

In particular, the non-volatile ester oil may comprise from 18 to 70 carbon atoms.

The non-volatile ester oil may particularly be chosen from among:

- monoesters comprising 18 to 40 carbon atoms, in particular monoesters of formula R1COOR2 wherein R1 represents the residue of a linear or branched fatty acid comprising from 6 to 20 carbon atoms and R2 represents a hydrocarbon chain, particularly branched, containing from 6 to 20 carbon atoms, such as for example Purcellin oil (cetostearyl octanoate), isononyl isononanoate, isodecyl neopentanoate, C12 to C15 alkyl benzoates, 2-ethylhexyl palmitate, octyldodecyl neopentanoate, 2-octyldodecyl stearate, 2-octyldodecyl erucate, isostearyl isostearate, 2-octyldodecyl benzoate, alkyl octanoates, decanoates or ricinoleates, isopropyl myristate, isopropyl palmitate, butyl stearate, hexyl laurate, 2-ethylhexyl palmitate, 2-hexyldecyl laurate, 2-octyldocyl palmitate, 2-octyldodecyl palmitate, 2-diethylhexyl succinate;
- diesters comprising 18 to 60 carbon atoms, in particular from 18 to 50 carbon atoms, such as diesters of carboxylic acid and monoalcohols, such as diisostearyl malate; diesters of glycol and carboxylic monoacids, such as neopentylglycol diheptonoate, polyglyceryl-2 diisostearate,

- triesters comprising 35 to 70 carbon atoms, such as triesters of carboxylic triacid, such as triisostearyl citrate or tridecyl trimellitate; or triesters of glycol and carboxylic monoacids such as polyglyceryl-2 triisostearate;

- tetraesters comprising 35 to 70 carbon atoms, such as such as tetraesters of penthaerythritol or polyglycerol and a carboxylic monoacid, for example pentaerythrityl tetrapelargonate, pentaerythrityl tetraisostearate, pentaerythrityl tetraisononanoate, glyceryl tridecyl-2 tetradecanoate, polyglyceryl-2 tetraisostearate or indeed pentaerythrityl tetradecyl-2 tetradecanoate; pentaerithrityl adipate/caprate/caprylate/heptanoate
- polyesters obtained by condensation of dimer and/or trimer of unsaturated fatty acid and diol such as polyglyceryl-2 isostearate/dimerdilinoleate copolymer marketed by the company Estenity under the tradename Hailucent ISDA or the polyester of dilinoleic acid and 1,4-butanediol;
- esters and polyesters of dimer diol and mono- or dicarboxylic acid, such as the esters of dimer diol and fatty acid and the esters of dimer diols and carboxylic diacid dimer, in particular those obtained from a dimer of a C8 to C34, particularly C12 to C22, in particular C16 to C20, and more particularly C18 unsaturated fatty acid, such as the esters of dilinoleic diacids and dilinoleic dimer diols, for example those marketed by the company NIPPON FINE CHEMICAL under the trade name LUSPLAN DD-DA5® and DD-DA7®;
- triglycerides of fatty acids (liquid at ambient temperature), particularly of fatty acids having from 7 to 40 carbon atoms, such as heptanoic or octanoic acid triglycerides or jojoba oil; saturated triglycerides such as caprylic/capric triglyceride, glyceryl triheptanoate, glycerin trioctanoate; C18-36 acid triglycerides such as those marketed under the reference DUB TGI 24 marketed by Stéarineries Dubois); C10-18 triglyceride, glyceryl dioleate, C8-12 acid triglyceride and unsaturated triglycerides such as castor oil, olive oil, ximenia oil, pracaxi oil;

- or one of the mixtures thereof.

An additional non-volatile oil may also be used to add further properties to the composition according to the invention.

By way of example, diisostearyl malate, polyglyceryl-3 diisostearate, polyglyceryl-2 triisostearate, pentaerythrityl adipate/caprate/caprylate/heptanoate may be added as it makes it possible to obtain good pigment dispersion.

Further additional oils may be added to enhance the sensory properties of the formula.

According to an embodiment, the non-volatile oil is chosen from among hydrogenated polyisobutene, polyglyceryl-2 triisostearate, polyglyceryl-3 diisostearate, diisostearyl malate, octyldodecanol, isostearyl isostearate, pentaerythrityl adipate/caprate/caprylate/heptanoate C10-18 triglyceride, glyceryl dioleate, C8-12 acid triglyceride and polyglyceryl-2 isostearate/dimerdilinoleate copolymer or one of the mixtures thereof.

According to a particular embodiment of the invention, the non-volatile oil introduced into the composition according to the invention is a mixture of hydrogenated polyisobutene, polyglyceryl-2 triisostearate, diisostearyl malate, octyldodecanol and isostearyl isostearate.

The non-volatile oil may be present in the composition according to the invention at a content of between 10% and 40% by weight, preferably between 15% and 35% by weight, more preferentially between 20% and 30% by weight, with respect to the weight of the composition.

Additional ingredients

The liquid anhydrous composition of the invention may also comprise additional ingredients such as structuring agents such as a wax and/or at least a lipophilic gelling agent, film-forming agents, active ingredients, fillers, preservatives or mixtures thereof.

According to an embodiment, the film-forming agent is chosen from among silicon resins such as trimethylsiloxysilicate as those markeketed by the company Wacker under the tradename Belsil TMS803, or dextrin esters such as dextrin isostearate & C8-12 acid triglyceride & isododecane as those marketed by the company Chiba Flour Milling under the tradename Unifilma HVY

The preservatives should be chosen with particular care to avoid any interaction with the water-soluble dye.

Method

The present invention also relates to providing a method for for the preparation of a liquid anhydrous composition as previously disclosed, comprising the flowing steps:

i. preparing a premix of

- (a) at least one water soluble dye,
- (b) at least one polyol, and
- (c) polyglyceryl-10 pentaoleate wherein the relative mass amount of (a)/(b)/(c) is ranging between $1/0.6/8 \le (a)/(b)/(c) \le 1/6/18$,

ii. incorporating of at least one volatile oil (d) and all other optional ingredients into the premix.

The following examples are given to illustrate the invention. These examples merely being given by way of illustration, the invention may in no way be restricted to the subject matter thereof.

Ingredients			Comparative	Comparative	Comparative	Comparative	Example
			example B1	example B2	example B3	example B4	of the
							invention
							B5
Fonction	INCI name	Trademark	%	%	%	%	%
		(company)					
hydrosoluble	CI 15985	UNICERT	0.6	0.6	0.6	0.6	0.6
organic dye	(YELLOW 6)	YELLOW					
		08006-J					
hydrosoluble	CI 17200	UNICERT	0.29	0.29	0.29	0.29	0.29
organic dye	(RED 33)	RED					
		K7057-J					
hydrosoluble	CI 45410	UNICERT	0.42	0.42	0.42	0.42	0.42
organic dye	(RED 28)	RED					
		K7054-J					
Humectant	glycerin		12.48	6.7	1.04	1.04	1.04
Surfactant	Polyglycerin-	Decaglyn	0	5.78	0	0	11.44
	10 pentaoleate	5OV					
		(Nikko					
		Chemicals					
		Co.LTD					
Non volatil	Polyglyceryl-3	Cithrol PG	0	0	11.44	0	0
oil	Diisostearate	32 IS LQ					
		MV					
		(Croda)					
Non volatil	Polyglyceryl-3	Imwittor	0	0	0	11.44	0
oil	Polyricinoleate	600 (IOI					
		OLEO					

		GMBH)					
Volatile oil	isododecane		86.21	86.21	86.21	86.21	86.21
Total (%)		100	100	100	100	100	

Procedure:

All the dyes were weighted and mixed. The humectant, the surfactant and/or non-volatile oil depending on the formula were further added, mixed well and heated to 95°C for 1 hour while mixing. The mixture was then allowed to cool down to room temperature. Isododecane was then added to the cooled solution while mixing to obtain a homogeneous composition.

Viscosity for Example B5: 2.07 mPa.s

Viscosity of only dye solution without isododecane: 13821 mPa.s

Procedure for measuring viscosities: Instrument: TA Instruments DHR2 rheometer. Mobile: 40mm smooth cone and 4° angle; 96 μ m air gap; 40mm smooth support. Method: rotation at 100s-1 for 120s at 25°C, after a warm-up time of 60s.

The composition of Example B5 of the invention provides a mixture which is homogeneous, stable at room temperature for 1 month.

The composition of comparative example B1 comprising a volatile oil (isododecane) but no polyglyceryl-10 pentaoleate results in a solution which **is not homogeneous and shows two immiscible phases**.

The formula of comparative example B2, comprising relative mass amount of (a)/(b)/(c) outside of the claimed scope, provides a solution which is **not homogeneous and shows** liquid on top after mixing.

The formula of comparative example B3, using polyglyceryl-3 diisostearate instead of polyglycerin- 10 pentaoleate results in a composition which is **not homogeneous after** mixing: the dye did not dissolve and formed aggregates.

The formula of comparative example B4, using polyglyceryl-3 polyricinoleate instead of polyglycerin- 10 pentaoleate results in a composition which is **not homogeneous and shows liquid on top after mixing.**

Example II

Fonction	INCI name	Trademark	% of ingredient
		(company)	
hydrosoluble	CI 15985 (YELLOW 6)	UNICERT	0.23
organic dye		YELLOW	
		08006-J	
hydrosoluble	CI 17200 (RED 33)	UNICERT RED	0.11
organic dye		K7057-J	
hydrosoluble	CI 45410 (RED 28)	UNICERT RED	0.16
organic dye		K7054-J	
Humectant	glycerin		0.38
surfactant	Polyglycerin- 10 pentaoleate	Nikkol	4.38
		Decaglyn 5OV	
		(Nikko	
		Chemicals	
		Co.LTD	
Non volatil oil	Hydrogenated polyisobutene &	Parleam 3	0.75
	tocopherol	(Rossow)	
Non volatil oil	Polyglyceryl-2 triiisostearate	Salacos 43 V	3
		(MB) (Saci	
		CPFA)	
Non volatil oil	Diisostearyl malate		16.66
Non volatil oil	octyldodecanol	Eutanol-G	7
Non volatil oil	Isostearyl isostearate	Crodamol Isis	1.7
		LQ (MV)	
		(Croda)	
Active	C10-18 TRIGLYCERIDES &	TECHNOHYAL	0.3
	SODIUM HYALURONATE &	HYAPEARL	
	TRIOLEIN & GLYCERYL	(Roelmi HPC	
	DIOLEATE	SRL)	
Oil gellant	Isododecane (12.18%) &	Bentone gel	14
	disteardimonium hectorite (1.4%) &	ISDV	
	propylene carbonate (0.42%)	(Elementis)	
Film-forming	VP/Hexadecene copolymer	Unimer U-151	4.5
agent			
Film-forming	polybutene	Indopol H-100	8.5
agent			
Film-forming	Trimethylsiloxysilicate	Belsil TMS 803	9
agent			
inorganic &			5.7
organic pigments			
Optical effect	silica	Creaspheres Sil	2
Filler		WL9	
		(cosmo Chem	
		SARL)	
Hydratant	actif		0.3

Anti-oxydant	Tocopheryl acetate	0.5
Volatile oil	Total isododecane	33.06
	(including bentone solvant)	
	Total (%)	100

The liquid anhydrous composition of the following formula (% by weight) was prepared:

Procedure:

- 1) Pre-mix octyldodecanol with VP/Hexadecene copolymer.
- 2) Add isostearyl isostearate, hydrogenated polyisobutene & tocopherol, polybutene, Polyglyceryl-2 triiisostearate and C10-18 TRIGLYCERIDES & SODIUM HYALURONATE & TRIOLEIN & GLYCERYL DIOLEATE and heat to 70°C, and mix well.
- 3) Then turn the heat off and add bentone gel (isododecane (12.18%) & disteardimonium hectorite (1.4%) & propylene carbonate (0.42%)) and mix until Bentone gel is fully dispersed (about 15 min). Then cool down to room temperature to constitute phase 1.
- 4) Combine all dyes and glycerin and polyglyceryl-10 pentaoleate in a beaker, heat to 95°C and mix for 1 hour and then cool down to room temperature to constitute phase 2.
 - 5) Add phase 2 to phase 1 and mix for 5-10 min untill dyes are well dispersed.
- 6) Add the pigments to phase 1 and mix for 10 minutes until the pigments are well dispersed.
 - 7) Slowly add silica to phase 1while mixing and continue mixing for 10 minutes.
- 8) Pre-disperse trimethylsiloxysilicate into isododecane and slowly pour into phase 1while mixing and continue mixing for 10 min.

Steps 5-8 are operated at room temperature.

Viscosity of the composition: 84.22 mPa.s

Procedure for measuring viscosities: Instrument: TA Instruments DHR2 rheometer. Mobile: 40mm smooth cone and 4° angle; 96 μ m air gap; 40mm smooth support. Method: rotation at 100s-1 for 120s at 25°C, after a warm-up time of 60s.

Claims

- 1. A liquid anhydrous composition comprising:
 - (a) at least one water soluble dye,
 - (b) at least one polyol, and
 - (c) a polyglyceryl-10 pentaoleate
 - (d) at least one volatile oil,

wherein the relative mass amount of (a)/(b)/(c) is ranging between $1/0.6/8 \le (a)/(b)/(c) \le 1/6/18$.

- 2. The liquid anhydrous composition according to claim 1, wherein the volatile oil is present in an amount between 20% and 99% by weight, relative to the total weight of the composition, and wherein the mass amount of (a) + (b) + (c) represents from 1 to 80% of the total weight of the composition.
- 3: The liquid anhydrous composition according to claim 1 or 2, comprising (e) at least one non-volatile oil, wherein the amount of non-volatile oil is between 20% and 50% by weight, relative to the total weight of the composition.
- 4: The liquid anhydrous composition according to any of claims 1 to 3, having a viscosity between 1 to 300mPa.s and preferably between 20 to 150mPa.s.
- 5: The liquid anhydrous composition according to claims 1 to 4, wherein the water-soluble dye is either natural or of natural origin, or is an organic dye.
- 6. The liquid anhydrous composition according to claim 5 wherein the water soluble organic dye is chosen from CI 12085 (RED 36), CI 15850 (RED 6), CI 15850 (RED 7) [E180], CI 15985 (YELLOW 6) [E110], CI 17200 (RED 33), CI 19140 (YELLOW 5) [E102], CI 42090 (BLUE 1) [E133], CI 45370 (ORANGE 5), CI 45370 (ORANGE 5), CI 45380 (RED 22), CI 45410 (RED 27), CI 45410 (RED 28), CI 47000 (YELLOW 11), CI 47005 (YELLOW 10) [E104], CI 61570 (GREEN 5), Acid Red 18, CI 16255, Red 21, CI 45380, and mixtures thereof.

7. The liquid anhydrous composition according to claims 1 to 6 wherein the polyol is chosen among glycerol, butylene glycol, pentylene glycol, hexylene glycol, caprylyl glycol, and mixtures thereof.

- 8. The liquid anhydrous composition according to claim 7 wherein the polyol is glycerol.
- 9. The liquid anhydrous composition according to claims 1 to 8 wherein the volatile oil is chosen from hydrocarbon oils, silicone oils and mixtures thereof.
- 10 The liquid anhydrous composition according to claim 9 wherein the volatile oil is chosen from isododecane, the mixture of n-undecane (C_{11}) and n-tridecane (C_{13}), dodecane, or mixtures thereof.
- 11 The liquid anhydrous composition according to claims 1 to 10 wherein the non-volatile oil is chosen from isostearyl isostearate, C8-12 acid triglyceride, polyglyceryl-3 diisostearate, polyglyceryl-2 triisostearate, pentaerythrityl adipate/caprate/caprylate/heptanoate, C10-18 triglyceride, glyceryl dioleate, C8-12 acid triglyceride and polyglyceryl-2 isostearate/dimerdilinoleate copolymer or mixtures thereof.
- 12. The liquid anhydrous composition according to claims 1 to 11 further comprising at least one coloring agent other than the water-soluble dye.
- 13. A method for the preparation of a liquid anhydrous composition according to any of claims 1 to 12 comprising the flowing steps:
 - i. preparing a premix of
 - (a) at least one water soluble dye,
 - (b) at least one polyol, and
 - (c) polyglyceryl-10 pentaoleate
 - wherein the relative mass amount of (a)/(b)/(c) is ranging between $1/0.6/8 \le (a)/(b)/(c) \le 1/6/18$,
 - ii. incorporating of at least one volatile oil (d) and all other optional ingredients into the premix.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2022/087330

A. CLASSIFICATION OF SUBJECT MATTER

A61K8/39

INV. A61K8/31

A61K8/34

A61K8/46

A61K8/49

A61Q1/04

ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A61K A61Q

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	WO 01/62217 A2 (COLOR ACCESS INC [US]) 30 August 2001 (2001-08-30) page 1, paragraph 2 - page 2, paragraph 1 example I	1-13
Y	US 3 677 691 A (KOCH LOUIS) 18 July 1972 (1972-07-18) column 2, lines 40-47 column 4, line 37 - column 5, line 46 column 5, lines 63-65	1-13

* Special categories of cited documents :	"T" later document published after the international filing date or priority
"A" document defining the general state of the art which is not considered to be of particular relevance	date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance;; the claimed invention cannot be considered novel or cannot be considered to involve an inventive
"L" document which may throw doubts on priority claim(s) or which is	step when the document is taken alone
cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance;; the claimed invention cannot be considered to involve an inventive step when the document is
"O" document referring to an oral disclosure, use, exhibition or other means	combined with one or more other such documents, such combination being obvious to a person skilled in the art
"P" document published prior to the international filing date but later than the priority date claimed	"&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
· ·	

26 April 2023

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See patent family annex.

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016

Further documents are listed in the continuation of Box C.

Ovens, Annabel

09/05/2023

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2022/087330

		101, 112022, 00,330		
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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