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(54) Title: PROCESS FOR PREPARING AND COMPOSITIONS OF MACROSPHERE FORMULATIONS

(57) Abstract: There is provided herein an aqueous product and a process for preparing the same where the aqueous product has visible, discrete, and stabilized droplets, and where the aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents.

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PROCESS FOR PREPARING AND COMPOSITIONS OF MACROSPHERE
FORMULATIONS

FIELD OF THE INVENTION

5 [0001] There is provided herein an aqueous product and a process for preparing the same where the aqueous product has visible, discrete, and stabilized droplets, and where the aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium
10 comprising water and one or more alkali swellable aqueous thickening agents.

BACKGROUND

[0002] The varied and unique properties of various oils create a strong need to include them in aqueous-based compositions. For example, oils such as silicone oils, fluids and gums, mineral oils, and water-insoluble organic esters such as isopropyl
15 palmitate and isopropyl myristate, are useful in cosmetic formulations for the skin and hair. In these compositions, the lubricity properties of the oil can impart conditioning action for the user. However, oils are generally immiscible in water making it very difficult to prepare a stable aqueous dispersion that does not quickly separate into phases of oil and water.

20 [0003] To solve the problem of providing effective dispersibility of such materials in water, it has generally been necessary to include a surfactant in aqueous cosmetic compositions that also contain a cosmetically-active oil. The surfactant works to maintain dispersed droplets of the oil in the aqueous solution. However, the use of surfactants increases the cost of the product and may affect the quality of the
25 composition. In addition, even with a surfactant present, the stability of the dispersion is often not satisfactory.

[0004] An alternative approach is to form macroscopic capsules of an oil by in situ aqueous polymerization of oil soluble monomers. For example, Brynko, in U.S. Pat. 2,969,330 and U.S. Pat. 2,969,331, describe the preparation of pressure-
30 rupturable capsules of a chlorinated diphenyl oil in water by dissolving styrene, or an acrylate or vinyl acetate monomer, in the oil, dispersing the monomer-containing oil in water with the aid of an emulsifier to form droplets, and polymerizing the

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monomer to form an encapsulating wall of solid polymer material around each droplet of oil. Berg, in J. Microencapsulation (1989) 6, No. 3, 327-337, also described a process for the microencapsulation of emulsified oil droplets by in situ vinyl polymerization. De Luca, in U.S. Pat. 4,741,872, described the preparation of biodegradable microspheres having a three-dimensional network in which biologically active macromolecular agents were physically entrapped therein. Kopolow, in U.S. Pat. 5,073,296, describes a method for preparing discrete microdroplets of an oil stabilized by in situ polymerization of a water-soluble vinyl monomer. However, each of these approaches has its problems and the stability of their resulting dispersions is still often not satisfactory.

[0005] Accordingly, it is an object of this invention to provide an aqueous product comprising visible, discrete, and stabilized droplets of oil and processes of making the same.

SUMMARY

[0006] The disclosed technology provides an aqueous product and a process for preparing the same. The aqueous product has visible, discrete, and stabilized droplets, and where the aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents.

[0007] The disclosed technology includes compositions and methods of making such compositions that, in some embodiments, may be described as visible macro-spheres formulations containing anhydrous or pre-emulsified droplets suspended in an emulsifier-free, partially neutralized acrylic-based aqueous system. Additional color, pigment, glitters, and/or fragrances, and/or anhydrous compatible active ingredients may be added into the composition to enhance the visual effects and the functional performance from the ingredients above. Other than controlling the size of these suspended oil droplets by mechanical shearing and apparatus, the size and the hardness of these droplets may be altered by including anhydrous rheology modifiers, emulsifiers, or higher melting waxes in the compositions and the rheology modifier in aqueous phase. The macro-spheres dispersion may be thickened by neutralization or in conjunction with additional rheology modifying agent. The macro-spheres

compositions may be advantageously applied to both leave-on and rinse-off skin care, color cosmetic, and sun protection applications such as serum, lotion, cream, facial cleanser, alphabetic cream, lip gloss, make-up remover, body wash, shampoo, conditioner and liquid detergent with appealing aesthetics and the functional performance from the ingredients containing in the anhydrous composition. The mechanical processing, ingredient selection, and order of addition of the components are essential to make the visually appealing anhydrous oil or pre-emulsified droplets in suspension (beads). The disclosed technology includes a process for dispersing the droplets in the pre-neutralized acrylate homopolymer aqueous solution under the specific conditions, which may include a pH 4.0 ± 1.0 and viscosity $< 3,000$ cps, or even $< 1,000$ cps. The rheology and surface tension of the macro-spheres may be adjusted to desired levels by adding anhydrous oil thickener, emulsifiers, waxes, or any combination thereof in order to achieve the desired droplet size (both macro and micro), hardness, and sensory properties.

[0008] The disclosed technology provides a process for preparing an aqueous product with visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and/or flavor delivery, and/or active delivery and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; wherein said process comprises the steps of: (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (II) neutralizing the resulting aqueous product to achieve one or more desired rheological properties; resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

[0009] The disclosed technology further provides the described process where the anhydrous composition further comprises one or more cosmetic pigments, colorants, fragrances, glitters, anhydrous compatible actives, or any combination thereof. The disclosed technology further provides the process where the aqueous medium further comprises one or more cosmetic pigments, colorants, fragrances, glitters, aqueous compatible actives, or any combination thereof. The disclosed technology further

provides the process where the anhydrous composition further comprises one or more cosmetic pigments, colorants, fragrances, glitters, anhydrous compatible actives or any combination thereof and the aqueous medium further comprises one or more cosmetic pigments, colorants, fragrances, glitters, aqueous compatible actives or any combination thereof.

5 **[0010]** The disclosed technology further provides the described process where the process further comprises the step: (III) adding to the aqueous product one or more cosmetic pigments, colorants, fragrances, glitters, aqueous compatible actives or any combination thereof and mixing the resulting aqueous product.

10 **[0011]** The disclosed technology further provides the described process where the oil of said anhydrous composition comprises a crude oil, a synthetic oil, an animal derived oil, a naturally derived vegetable oil, a hydrocarbon oil, an ester oil, a silicone oil, or any combination thereof.

15 **[0012]** The disclosed technology further provides the described process where the anhydrous rheological modifying agent of said anhydrous composition comprises crude wax, synthetic wax, animal derived wax, natural wax, naturally derived wax, ester, silicone derived wax, fatty acid, emulsifier, amino acid derived gelling agent, polymeric wax, poly esters, polyamide, polyethylene, polyurethane, polyurea, organically modified clays, alkyl acrylate, styrene based polymer, polystyrene, polybutylene, polyisobutylene, fumed silica, hydrophobically modified silica, aerogel, or any mixture thereof.

20 **[0013]** The disclosed technology further provides the described process where the aqueous thickener comprises a natural gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any combination thereof. In some embodiments, the aqueous thickening agents comprises pre-neutralized acrylate based homopolymer, neutralized acrylate based homopolymer, polymer of vinylpyrrolidone, crosslinked acrylate based homopolymer, polyurethane, polyacrylamide or any combination thereof. In still other embodiments, the aqueous thickener comprises an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymers), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination thereof.

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[0014] The disclosed technology further provides the described process where the alkali swellable aqueous thickener comprises an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymer, etc.), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination thereof.

[0015] The disclosed technology further provides the described process where the aqueous medium comprises an additional aqueous thickener, where said aqueous thickener comprises a natural gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any combination thereof. In some embodiments, the aqueous thickening agents comprises pre-neutralized acrylate based homopolymer, neutralized acrylate based homopolymer, polymer of vinylpyrrolidone, crosslinked acrylate based homopolymer, polyurethane, polyacrylamide or any combination thereof. In still other embodiments, the aqueous thickener comprises an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymer, etc.), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination thereof.

[0016] The disclosed technology further provides the described process where the aqueous medium is partially neutralized by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0 before it is combined with the anhydrous composition.

[0017] The disclosed technology further provides the described process where the neutralization of step (II) comprises adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or even 5.0 to 9.0.

[0018] The disclosed technology further provides a process for preparing an aqueous product with visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; wherein said process comprises the steps of: (I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0; (II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets

of said anhydrous composition in said aqueous medium; and (III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or even 5.0 to 9.0; resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

5 **[0019]** The disclosed technology further provides an aqueous product made by any of the processes described herein.

10 **[0020]** The disclosed technology further provides a aqueous product comprising visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; where said aqueous product is prepared by a process comprising the steps of: (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (II) neutralizing the resulting aqueous product to achieve one or more desired rheological properties; resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

20 **[0021]** The disclosed technology further provides a aqueous product aqueous product comprising visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; where said aqueous product is prepared by a process comprising the steps of: (I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0; (II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or even 5.0 to 9.0; resulting in an aqueous product where

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said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

DETAILED DESCRIPTION

[0022] Various preferred features and embodiments will be described below by way of non-limiting illustration.

[0023] The disclosed technology provides an aqueous product and a process for preparing the same.

The Aqueous Product

[0024] The disclosed technology provides an aqueous product. The aqueous product includes visible, discrete, and stabilized droplets of an anhydrous composition suspended in an aqueous medium.

[0025] As used herein, the term “visible” with regards to the droplets means the droplets may be seen by the naked human eye. As used herein, the term “discrete” with regards to the droplets means the droplets are individually separate and distinct from one another. As used herein, the term “stabilized” with regards to the droplets means the droplets will remain suspended in the aqueous product for a sufficient period of time. In some embodiments, a sufficient period of time is the product life of the composition in which the droplets are suspended. In some embodiments, a sufficient period of time means the composition will pass a given stability test, for example: At least 1 month at 50 °C, 3 months at room temperature, 40 or 45 °C, or 3-5 cycles of freeze and thaw stability. In some embodiments, a sufficient period of time means a specific period of time, for example: at least 1 month, 3 months, 6 months, 1 year, 2 years, 5 years, or even indefinitely (subject to moderate storage conditions and the like). As used herein, the term “anhydrous” means a non-aqueous material, composition, and/or phase, or in other words a water insoluble and/or water incompatible material, composition, and/or phase. An anhydrous component, as used herein, will not have any water intentionally added to it, and in some embodiments, is substantially free of water and/or is substantially free of dissolved water. In some embodiments, the anhydrous components described herein are free of water.

[0026] The described aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying

agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents.

The Anhydrous Composition

5 [0027] The anhydrous compositions comprises one or more oils and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment.

[0028] The anhydrous compositions may further comprise one or more cosmetic pigments, colorants, fragrances, glitters, anhydrous compatible actives, any of the additional additives described herein, or any combination thereof. These additional
10 additives may be added and suspended in any step in the processes described herein. Examples include Soliens™ Glitter Gold SL-6003E commercially available from CQV Co. Ltd. and LIPOCHROMAN® synthetic molecule commercially available from The Lubrizol Corporation.

[0029] The anhydrous compositions may comprise one or more anhydrous
15 rheological modifying agents to suspend the anhydrous additives as droplets in the overall composition, adding a visual aesthetic and expanding the personal & home care applications in which the compositions herein are useful.

[0030] Suitable oils includes crude oil, synthetic oil, animal derived oil, naturally
20 derived vegetable oil, hydrocarbon oil, ester oil, silicone oil, or any combination thereof. Examples included but not limited to mineral oil, petrolatum oil, vegetable oil, triglycerides, coconut oil, corn oil, sunflower oil, peanut oil, jojoba oil, lanolin derived wax, lanolin oil, acetylated lanolin alcohol, alkyl myristate, alkyl palmitate, alkyl adipate, C12 to C15 alkyl benzoate, dimethicone, cyclomethicone, and the like. More specific examples include Schercemol™ ester oils such as Schercemol CO ester,
25 Schercemol LL ester, commercially available from The Lubrizol Corporation, mineral oil, sunflower oil, dimethicone, caprylic/capric triglyceride, and coconut oil.

[0031] The anhydrous rheological modifying agent of said anhydrous composition
30 may include crude wax, synthetic wax, animal derived wax, natural wax, naturally derived wax, ester, silicone derived wax, fatty acid, emulsifier, amino acid derived gelling agent, polymeric wax, poly esters, polyamide, polyethylene, polyurethane, polyurea, organically modified clays, alkyl acrylate, styrene based polymer, polystyrene, polybutylene, polyisobutylene, fumed silica, hydrophobically modified

silica, aerogel, or any mixture thereof. Additional examples included but are not limited to cetyl palmitate, polyurethane-79, diisostearyl fumarate, diisostearyl malate, Poly C10-30 alkyl acrylate, coconut oil, polyethylene, polyamide 3, polyamide 8, dibutyl lauroyl glutamide, dibutyl ethylhexanoyl glutamide, quaternium-90 sepiolite, quaternium-90 montmorillonite, silica silyate, dextrin palmitate/ethylhexanoate, 5 stearic acid, 12-hydroxy stearic acid, cetyl alcohol, polyglyceryl-6 dipalmitate, stearyl alcohol, steareth-2, steareth-20, silica, styrene/acrylates copolymer and the like. More specific examples include C10 to C30 alkyl acrylate crosspolymer, Intelimer[®] 13-1 polymer from Evonik Inc., Gelatinization Agent GP-1 and EB-21 from Ajinomoto Co., Inc., OleoCraft[™] LP-20 from Croda Inc., and polyurethane-79 from The Lubrizol Corporation. 10

[0032] Still further rheology modifiers and thickeners may be used in the described process and compositions described herein. Suitable rheology modifiers and thickeners include the oil soluble, organic and/or anhydrous compatible synthetic and semi-synthetic rheology modifiers described below (some of which may also be used 15 in the aqueous medium described below).

[0033] In some embodiments, the anhydrous composition comprises an ester oil, an acrylate crosspolymer, and optionally one or more cosmetic pigments, colorants, fragrances, glitters, anhydrous compatible actives or any combination thereof. Examples include Soliens[™] Glitter Gold SL-6003E commercially available from CQV Co. Ltd. and LIPOCHROMAN[®] synthetic molecule commercially available from The Lubrizol Corporation. 20

[0034] The ester oil may make up from 0.1 to 10, 1 to 10, 2 to 5, 2 to 4, 2.5 to 3.5, or even about 3 pbw of the anhydrous composition.

[0035] The anhydrous rheological modifying agents may make up from 0.1 to 5, 0.2 to 1, 0.3 to 0.9, 0.4 to 0.6, or even about 0.5 pbw of the anhydrous composition. 25

[0036] The cosmetic pigments, colorants, fragrances, glitters, anhydrous compatible active or any of the additional additives described herein may make up, either each individually or overall in combination with one another, from 0.001 to 5, 0.001 to 1, 0.005 to 0.5, 0.005 to 0.05, or even about 0.01 pbw of the anhydrous composition. 30

The Aqueous Medium Composition

[0037] The described aqueous product also includes an aqueous medium. This aqueous medium comprises water and one or more alkali swellable aqueous thickening agents.

5 **[0038]** Suitable aqueous thickeners include a natural gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any combination thereof. In some embodiments, the aqueous thickening agents comprises pre-neutralized acrylate based homopolymer, neutralized acrylate based homopolymer,
10 polymer of vinylpyrrolidone, crosslinked acrylate based homopolymer, polyurethane, polyacrylamide or any combination thereof. In still other embodiments, the aqueous thickener comprises an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymers), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination thereof.

15 **[0039]** In some embodiments, the alkali swellable aqueous thickener includes an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymer, etc.), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination thereof.

[0040] In some embodiments, the aqueous medium includes an additional aqueous
20 thickener, where this additional aqueous thickener is different from the first and may include a natural gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any combination thereof. In some embodiments, the aqueous thickening agents comprises pre-neutralized acrylate based homopolymer, neutralized
25 acrylate based homopolymer, polymer of vinylpyrrolidone, crosslinked acrylate based homopolymer, polyurethane, polyacrylamide or any combination thereof. In still other embodiments, the aqueous thickener comprises an acrylate based polymer (acrylate homopolymer, acrylate copolymer, and acrylate crosspolymer, etc.), for example, a 2-acrylamido-2-mehtylpropane sulfonic acid (AMPS) based polymer or any combination
30 thereof.

[0041] The aqueous medium may further comprise one or more rheological modifying agents (including the anhydrous rheological modifying agents described

herein). These agents may also act to help suspend the droplets of anhydrous composition in the aqueous medium. Suitable aqueous thickening agents include comprises a natural gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any combination thereof. More specific examples include Novemer™ EC-1 polymer, Novemer™ EC-2 polymer, and Avalure™ Flex-6 polymer from The Lubrizol Corporation, SEPIPLUS™ 400 from Seppic Inc., Aristoflex® AVC from Clariant Inc., BENTONE® LT from Elementis Specialties Inc., xanthan gum, and guar gum.

10 **[0042]** Still further rheology modifiers and thickeners may be used in the described process and compositions described herein. Suitable rheology modifiers and thickeners include synthetic and semi-synthetic rheology modifiers (some of which may also be used in the anhydrous composition described above).

15 **[0043]** Exemplary synthetic rheology modifiers include acrylic based polymers and copolymers. One class of acrylic based rheology modifiers are the carboxyl functional alkali-swellable and alkali-soluble thickeners (ASTs) produced by the free-radical polymerization of acrylic acid alone or in combination with other ethylenically unsaturated monomers. The polymers can be synthesized by solvent/precipitation as well as emulsion polymerization techniques. Exemplary synthetic rheology modifiers of this class include homopolymers of acrylic acid or methacrylic acid and copolymers polymerized from one or more monomers of acrylic acid, substituted acrylic acid, and salts and C1-C30 alkyl esters of acrylic acid and substituted acrylic acid. As defined herein, the substituted acrylic acid contains a substituent positioned on the alpha and/or beta carbon atom of the molecule, wherein in one aspect the substituent is independently selected from C1-4 alkyl, -CN, and -COOH. Optionally, other ethylenically unsaturated monomers such as, for example, styrene, vinyl acetate, ethylene, butadiene, acrylonitrile, as well as mixtures thereof can be copolymerized into the backbone. The foregoing polymers are optionally crosslinked by a monomer that contains two or more moieties that contain ethylenic unsaturation. In one aspect, 25 the crosslinker is selected from a polyalkenyl polyether of a polyhydric alcohol containing at least two alkenyl ether groups per molecule. Other exemplary crosslinkers are selected from allyl ethers of sucrose and allyl ethers of pentaerythritol,

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and mixtures thereof. These polymers are more fully described in U.S. Patent No. 5,087,445; U.S. Patent No. 4,509,949; and U.S. Patent No. 2,798,053 herein incorporated by reference.

[0044] In one aspect, the AST rheology modifier or thickener is a crosslinked homopolymer polymerized from acrylic acid or methacrylic acid and is generally referred to under the INCI name of Carbomer. Commercially available Carbomers include Carbopol® polymers 934, 940, 941, 956, 980 and 996 available from Lubrizol Advanced Materials, Inc. In a further aspect, the rheology modifier is selected from a crosslinked copolymer polymerized from a first monomer selected from one or more monomers of acrylic acid, substituted acrylic acid, salts of acrylic acid and salts of substituted acrylic acid and a second monomer selected from one or more C10-C30 alkyl acrylate esters of acrylic acid or methacrylic acid. In one aspect, the monomers can be polymerized in the presence of a steric stabilizer such as disclosed in U.S. Patent No. 5,288,814, which is herein incorporated by reference. Some of the forgoing polymers are designated under INCI nomenclature as Acrylates/C10-30 Alkyl Acrylate Crosspolymer and are commercially available under the trade names Carbopol® 1342 and 1382, Carbopol® Ultrez 20 and 21, Carbopol® ETD 2020 and Pemulen® TR-1 and TR-2 from Lubrizol Advanced Materials, Inc.

[0045] In another aspect, the auxiliary rheology modifier can be a crosslinked, linear poly(vinyl amide/acrylic acid) copolymer as disclosed in U.S. Patent No. 7,205,271, the disclosure of which is herein incorporated by reference.

[0046] Another class of optional synthetic rheology modifiers and thickeners suitable for use in the present invention includes the hydrophobically modified ASTs, commonly referred to as hydrophobically modified alkali-swellaable and alkali-soluble emulsion (HASE) polymers. Typical HASE polymers are free radical addition polymers polymerized from pH sensitive or hydrophilic monomers (e.g., acrylic acid and/or methacrylic acid), hydrophobic monomers (e.g., C1-C30 alkyl esters of acrylic acid and/or methacrylic acid, acrylonitrile, styrene), an "associative monomer", and an optional crosslinking monomer. The associative monomer comprises an ethylenically unsaturated polymerizable end group, a non-ionic hydrophilic midsection that is terminated by a hydrophobic end group. The non-ionic hydrophilic midsection comprises a polyoxyalkylene group, e.g., polyethylene oxide, polypropylene oxide, or

mixtures of polyethylene oxide/polypropylene oxide segments. The terminal hydrophobic end group is typically a C8-C40 aliphatic moiety. Exemplary aliphatic moieties are selected from linear and branched alkyl substituents, linear and branched alkenyl substituents, carbocyclic substituents, aryl substituents, aralkyl substituents, arylalkyl substituents, and alkylaryl substituents. In one aspect, associative monomers can be prepared by the condensation (e.g., esterification or etherification) of a polyethoxylated and/or polypropoxylated aliphatic alcohol (typically containing a branched or unbranched C8-C40 aliphatic moiety) with an ethylenically unsaturated monomer containing a carboxylic acid group (e.g., acrylic acid, methacrylic acid), an unsaturated cyclic anhydride monomer (e.g., maleic anhydride, itaconic anhydride, citraconic anhydride), a monoethylenically unsaturated monoisocyanate (e.g., α,α -dimethyl-m-isopropenyl benzyl isocyanate) or an ethylenically unsaturated monomer containing a hydroxyl group (e.g., vinyl alcohol, allyl alcohol). Polyethoxylated and/or polypropoxylated aliphatic alcohols are ethylene oxide and/or propylene oxide adducts of a monoalcohol containing the C8-C40 aliphatic moiety. Non-limiting examples of alcohols containing a C8-C40 aliphatic moiety are capryl alcohol, iso-octyl alcohol (2-ethyl hexanol), pelargonic alcohol (1-nonanol), decyl alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol, cetyl alcohol, cetearyl alcohol (mixture of C16 C18 monoalcohols), stearyl alcohol, isostearyl alcohol, elaidyl alcohol, oleyl alcohol, arachidyl alcohol, behenyl alcohol, lignoceryl alcohol, ceryl alcohol, montanyl alcohol, melissyl, lacceryl alcohol, geddyl alcohol, and C2-C20 alkyl substituted phenols (e.g., nonyl phenol), and the like.

[0047] Exemplary HASE polymers are disclosed in U.S. Patent Nos. 3,657,175; 4,384,096; 4,464,524; 4,801,671; and 5,292,843, which are herein incorporated by reference. In addition, an extensive review of HASE polymers is found in Gregory D. Shay, Chapter 25, "Alkali-Swellable and Alkali-Soluble Thickener Technology A Review", *Polymers in Aqueous Media - Performance Through Association*, Advances in Chemistry Series 223, J. Edward Glass (ed.), ACS, pp. 457-494, Division Polymeric Materials, Washington, DC (1989), the relevant disclosures of which are incorporated herein by reference. Commercially available HASE polymers are sold under the trade names, Aculyn® 22 (INCI Name: Acrylates/Steareth-20 Methacrylate Copolymer), Aculyn® 44 (INCI Name: PEG-150/Decyl Alcohol/SMDI Copolymer), Aculyn 46®

(INCI Name: PEG-150/Stearyl Alcohol/SMDI Copolymer), and Aculyn® 88 (INCI Name: Acrylates/Steareth-20 Methacrylate Crosspolymer) from Rohm & Haas, and Novethix™ L-10 (INCI Name: Acrylates/Beheneth-25 Methacrylate Copolymer) from Lubrizol Advanced Materials, Inc.

5 **[0048]** The aqueous medium may further comprise one or more cosmetic pigments, colorants, fragrances, glitters, aqueous compatible active, any of the additional additives described herein, or any combination thereof. These additional additives may be added and suspended in any step in the processes described herein. Examples include Soliens™ Glitter Gold SL-6003E commercially available from CQV Co. Ltd.
10 Additional additives include glycols and glucose ethers. Examples include Glucam™ material such as Glucam E-20, commercially available from the Lubrizol Corporation, and butylene glycol, propylene glycol, glycerin, a preservative such as euxyl, or any combination thereof.

[0049] The water may make up from 10 to 100, 20 to 80, 30 to 70, 50 to 70, 55 to
15 65 or even about 60 pbw of the aqueous medium.

[0050] The alkali swellable aqueous thickening agents may make up from 0.01 to 40, 0.1 to 40, 1 to 40, 10 to 30, 15 to 25, or even about 20 pbw of the aqueous medium.

[0051] The cosmetic pigments, colorants, fragrances, glitters, or any of the additional additives described herein may make up, either each individually or overall
20 in combination with one another, from 0.001 to 5, 0.001 to 1, 0.005 to 0.5, 0.005 to 0.05, or even about 0.01 pbw of the aqueous medium; or from 0.01 to 20, 0.01 to 10, 0.5 to 10, 1 to 10, 5 to 10, 6 to 8, or even about 7 pbw of the aqueous medium.

The Process

[0052] The disclosed technology provides a process for preparing the aqueous
25 product. The process produces the described aqueous product, which has visible, discrete, and stabilized droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment dispersed in an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents.

30 **[0053]** In one embodiment, the process comprises the steps of: (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium;

and (II) neutralizing the resulting aqueous product to achieve one or more desired rheological, visual, and/or sensorial properties; resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

5 **[0054]** In another embodiment, the process comprises the steps of: (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (II) neutralizing the resulting aqueous product; resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously
10 throughout the aqueous medium.

[0055] In another embodiment, the process comprises the steps of: (I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0; (II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous
15 composition in said aqueous medium; and (III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or 5.0 to 9.0. Where the process results in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

20 **[0056]** It is noted that in some embodiments, the process includes the step of partially neutralizing the aqueous medium before adding the anhydrous composition and then completing the neutralization of the resulting aqueous composition. While not wishing to be bound by theory, it is believed this partial neutralization followed by the combination of the aqueous medium and the anhydrous composition, which is then
25 followed by the complete neutralization of the overall composition leads to improved stability of the resulting composition.

[0057] In some embodiments, the anhydrous composition used in the described process further comprises one or more cosmetic pigments, colorants, glitters, anhydrous compatible active, or any combination thereof. In some embodiments, the
30 aqueous medium used in the described process further comprises one or more cosmetic pigments, colorants, glitters, aqueous compatible active, or any combination thereof. In still other embodiments, the anhydrous composition used in the described process

further comprises one or more cosmetic pigments, colorants, glitters, or any combination thereof; and the aqueous medium used in the described process further comprises one or more cosmetic pigments, colorants, glitters, anhydrous compatible actives, or any combination thereof.

5 **[0058]** The process described herein may further comprises the step: (III) adding to the aqueous product one or more cosmetic pigments, colorants, glitters, aqueous compatible actives, or any combination thereof and mixing the resulting aqueous product.

10 **[0059]** In some embodiments, the neutralization of step (II) described above, comprises adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or from 5.0 to 9.0. This adjustment may be done with any neutralizing agent, for example, sodium hydroxide, more specifically, an 18 percent by weight solution of sodium hydroxide in water.

15 **[0060]** In some embodiments, the process described herein includes an initial step where the aqueous medium is partially neutralized. In some embodiments, partially neutralized means the pH of said aqueous medium is adjusted to within the range of 3.0 to 6.0 before it is combined with the anhydrous composition. This adjustment may be done with any neutralizing agent, including those described above.

20 **[0061]** The disclosed technology further provides a process for preparing an aqueous product with visible, discrete, and stabilized droplets, wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; and wherein said process comprises the
25 steps of: (I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0; (II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (III)
30 neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or from 5.0 to 9.0; resulting in an aqueous product where said droplets of

said anhydrous composition are suspended homogeneously throughout the aqueous medium.

[0062] The disclosed technology further provides for the resulting aqueous product itself.

5 **[0063]** In some embodiments, the aqueous product comprises visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous
10 thickening agents; where said aqueous product is prepared by a process comprising the steps of: (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (II) neutralizing the resulting aqueous product to achieve one or more desired rheological properties; resulting in an aqueous product where said
15 droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

[0064] In some embodiments, the aqueous product comprises visible, discrete, and stabilized droplets; wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological
20 modifying agents for rheological, visual, and/or sensorial adjustment; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents; where said aqueous product is prepared by a process comprising the steps of: (I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0; (II) adding said anhydrous
25 composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and (III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 3.5 to 10.0 or from 5.0 to 9.0; resulting in an aqueous product where said droplets of
30 said anhydrous composition are suspended homogeneously throughout the aqueous medium.

[0065] In still other embodiments, the anhydrous composition described above is prepared by mixing all the component except the anhydrous rheological modifying agent, and then adding the anhydrous rheological modifying agent last and mixing the composition until it is homogenous, resulting in the anhydrous composition.

5 **[0066]** In still other embodiments, the process may include the steps of (1) preparing an aqueous medium by combining an alkali swellable aqueous thickening agents and water; (2) optionally adding one or more additional additives and mixing the resulting composition to provide the aqueous medium; (3) optionally adding a neutralizing agent the aqueous medium, adjusting the aqueous medium to a pH of
10 about 3.5-3.6; (4) separately mixing an oil, an anhydrous rheological modifying agent, and optionally one or more additional additives, where the anhydrous rheological modifying agent is added last, resulting in the anhydrous composition; (5) adding the anhydrous composition to the aqueous medium and mixing the combined mixture to form droplets of the anhydrous composition in the aqueous
15 medium; and (6) add a neutralizing agent to fully neutralize the composition while mixing, adjusting the composition to a pH of about 5.0 to 9.0 or from 5.5 to 7.5.

[0067] Technical and scientific terms used herein have the meanings commonly understood by one of ordinary skill in the art, unless defined otherwise. Reference is made herein to various methodologies known to those of ordinary skill in the art, as
20 well as publications and other materials setting forth such known methodologies, all of which are incorporated herein by reference in their entireties. Any suitable materials and/or methods known to those of ordinary skill in the art can be utilized in carrying out the present invention. However, specific materials and methods are described. Materials, reagents and the like to which reference is made in the
25 following description and examples are obtainable from commercial sources, unless otherwise noted.

[0068] As used herein, the singular forms "a," "an," and "the" designate both the singular and the plural, unless expressly stated to designate the singular only.

30 **[0069]** The term "about" and the use of ranges in general, whether or not qualified by the term about, means that the number comprehended is not limited to the exact number set forth herein, and is intended to refer to ranges substantially within the quoted range while not departing from the scope of the invention. As used herein,

"about" will be understood by persons of ordinary skill in the art and will vary to some extent on the context in which it is used. If there are uses of the term which are not clear to persons of ordinary skill in the art given the context in which it is used, "about" will mean up to plus or minus 10% of the particular term.

5 **[0070]** The phrase "substantially free of" as used herein means that the described composition (e.g., polymer matrix, etc.) comprises less than about 5%, less than about 3%, or less than about 1% by weight, based on the total weight of the composition at issue, of the excluded component(s). The phrase "free of" as used herein means that
10 the described composition (e.g., polymer matrix, etc.) is formulated without adding the excluded component(s) as an intended component, although trace amounts may be present in other components or as a by-product or contaminant, such that the composition comprises at most only trace amounts of the excluded component(s).

[0071] By the term "for rheological, visual, and/or sensorial adjustment" it is meant the additive and/or component in question is being used to adjust the rheological,
15 visual, and/or sensorial properties of the overall composition. In some embodiments the term "for rheological, visual, and/or sensorial adjustment" may be replaced herein with the term "for rheological adjustment." By rheological adjustment, we mean one or more rheological properties of the overall composition is modified by the addition of the additive and/or component in question, for example, viscosity and/or shear
20 stress. In some embodiments, the term "for rheological, visual, and/or sensorial adjustment" may be replaced herein with the term "for visual adjustment." By visual adjustment, we mean one or more visual properties of the overall composition is modified by the addition of the additive and/or component in question, for example, the droplets suspended in the composition are suspended more homogeneously across
25 the entire composition, there are clear color distinctions between the overall compositions and the droplets, etc. In some embodiments, the term "for rheological, visual, and/or sensorial adjustment" may be replaced herein with the term "for sensorial adjustment." By sensorial adjustment, we mean one or more sensorial properties of the overall composition is modified by the addition of the additive and/or component in
30 question, for example, the droplets suspended in the composition are firm and when used in personal care applications such as hand soaps, they "burst" or yield when used, such as when they are under the pressure of the user, having applied the composition

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to their hands, rubbing their hands together or applying similar pressure to the compositions.

[0072] The amount of each chemical component described is presented exclusive of any solvent which may be customarily present in the commercial material, that is, on an active chemical basis, unless otherwise indicated. However, unless otherwise indicated, each chemical or composition referred to herein should be interpreted as being a commercial grade material which may contain the isomers, by-products, derivatives, and other such materials which are normally understood to be present in the commercial grade.

[0073] It is known that some of the materials described above may interact in the final formulation, so that the components of the final formulation may be different from those that are initially added. For instance, metal ions can migrate to other acidic or anionic sites of other molecules. The products formed thereby, including the products formed upon employing the composition of the technology described herein in its intended use, may not be susceptible of easy description. Nevertheless, all such modifications and reaction products are included within the scope of the technology described herein; the technology described herein encompasses the composition prepared by admixing the components described above.

EXAMPLES

[0074] The technology described herein may be better understood with reference to the following non-limiting examples.

[0075] A series of examples are prepared using the following procedure: (1) an aqueous medium is prepared by combining, in a suitable vessel equipped with a mixed, water and the alkali swellable aqueous thickening agents. The thickening agent is allowed to completely self-wet and then the composition is mixed using a paddle blade at about 500 rpm. (2) The rest of the components for the aqueous medium (identified as Part A in the table below) are added to the mixing vessel and the composition is mixed gently until the resulting aqueous medium is homogeneous. (3) When present, a neutralizing agent (identified as PART B in the table below) is added to the aqueous medium to partially neutralize batch, here by adjusting the aqueous medium to a pH of about 3.5-3.6, within continued gentle mixing. (4) In a separate vessel, mix all the components of the anhydrous composition (identified as

PART C in the table below) adding the anhydrous rheological modifying agent last, and mix until homogeneous. Begin heating the oil phase to about 105°C and not above 110°C. Continue mixing until the anhydrous rheological modifying agent is solubilized, typically about 45 to 60 minutes, resulting in the anhydrous composition.

- 5 (5) Add the anhydrous composition to the aqueous medium, mixing the combined mixture at less than 300 rpm. (6) Add the neutralizing agent to the batch with less than 300 rpm mixing to fully neutralize and suspend oil droplets. Once desired visual effect is achieved immediately turn off the mixer.

10 [0076] The table below summarizes the formulations of the examples prepared using this procedure as well as their resulting appearance.

Table 1

	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6
Ingredient	PBW	PBW	PBW	PBW	PBW	PBW
<u>Part A</u>						
Deionized Water	90.795	90.795	90.795	90.795	90.795	90.795
Carbopol® U10	0.2	0.2	0.2			
Carbopol® U21				0.2	0.2	0.2
Glycerin	3	3	3	3	3	3
Propylene Glycol	2	2	2	2	2	2
Euxyl	0.5	0.5	0.5	0.5	0.5	0.5
<u>Part B</u>						
Sodium Hydroxide Added to adjust pH to	3.5-3.6	3.5-3.6	n/a	3.5-3.6	3.5-3.6	n/a
<u>Part C</u>						
Schercemol™ NGDO Ester	3	3	3	3	3	3
Polyurethane oil thickener	0.5	0.5	0.5	0.5	0.5	0.5
Oil Soluble dye	0.005	0.005	0.005	0.005	0.005	0.005
<u>Part D</u>						
Sodium Hydroxide Added to adjust pH to	5.0-5.5	n/a	5.0-5.5	5.0-5.5	n/a	5.0-5.5
pH	5.03	3.62	5.2	5.01	3.61	5.28
Viscosity (cps)	14,020	1,250	10,100	16,260	1,090	6,620
Yield Value	1,070	70	522	730	48	352
Appearance	more uniform small droplets	a lot of small uniform droplets	very large and non-uniform droplets	more uniform small droplets	a lot of small uniform droplets	a lot of small non-uniform droplets
Average Anhydrous Macrosphere Size (mm)	1.0	1.0	5.0	1.0	1.0	1.5

[0077] The results show the compositions of the invention provide useful composition with visible, discrete, and stabilized droplets.

[0078] A visual panel testing was conducted by 10 panelists to rate the aesthetic preference by viscosity, macrosphere size, and macrosphere shape. A satisfaction point was given to each attribute when meeting the panelist's visual satisfaction on a scale of 0 to 3 with a higher score being a better score. The table below summarizes the panel result, which indicated that the pre-neutralized step of the alkali swellable polymer in the aqueous phase is critical for optimal aesthetics.

Table 2

Panelist	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6
1	3	2	1	3	2	1
2	3	2	0	3	2	1
3	3	1	1	2	1	1
4	3	2	0	3	2	2
5	2	2	1	3	2	2
6	3	3	1	2	3	1
7	3	3	0	2	3	2
8	2	2	1	2	3	2
9	3	1	1	1	1	1
10	2	1	0	3	1	2
Visual Satisfaction Point (Average)	2.7	1.9	0.6	2.4	2.3	1.5

10

[0079] Panel data further indicated the visually acceptable macrosphere size ranges in between 0.01 mm to 2.0 mm with the optimal preference ranges in between 1.0 mm to 1.5 mm.

15

[0080] Each of the documents referred to above is incorporated herein by reference, including any prior applications, whether or not specifically listed above, from which priority is claimed. The mention of any document is not an admission that such document qualifies as prior art or constitutes the general knowledge of the skilled person in any jurisdiction. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." It is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. Similarly, the ranges and amounts for each element of the

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technology described herein can be used together with ranges or amounts for any of the other elements.

[0081] As used herein, the transitional term “comprising,” which is synonymous with “including,” “containing,” or “characterized by,” is inclusive or open-ended and does not exclude additional, un-recited elements or method steps. However, in each 5 recitation of “comprising” herein, it is intended that the term also encompass, as alternative embodiments, the phrases “consisting essentially of” and “consisting of,” where “consisting of” excludes any element or step not specified and “consisting essentially of” permits the inclusion of additional un-recited elements or steps that do not materially affect the basic and novel characteristics of the composition or 10 method under consideration. That is “consisting essentially of” permits the inclusion of substances that do not materially affect the basic and novel characteristics of the composition under consideration.

[0082] While certain representative embodiments and details have been shown for 15 the purpose of illustrating the subject technology described herein, it will be apparent to those skilled in this art that various changes and modifications can be made therein without departing from the scope of the subject invention. In this regard, the scope of the technology described herein is to be limited only by the following claims.

What is claimed is:

1. A process for preparing an aqueous product with visible, discrete, and stabilized droplets;

5 wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents;

wherein said process comprises the steps of:

10 (I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and

(II) neutralizing the resulting aqueous product to achieve one or more desired rheological properties;

15 resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

2. The process of claim 1 wherein:

20 (a) the anhydrous composition further comprises one or more cosmetic pigments, colorants, fragrances, glitters, or any combination thereof;

(b) the aqueous medium further comprises one or more cosmetic pigments, colorants, fragrances, glitters, or any combination thereof; or

(c) any combination thereof.

25 3. The process of any of the claims 1 to 2 wherein the process further comprises the step:

(III) adding to the aqueous product one or more cosmetic pigments, colorants, fragrances, glitters, or any combination thereof and mixing the resulting aqueous product.

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4. The process of any of the claims 1 to 3 wherein the oil of said anhydrous composition comprises a crude oil, a synthetic oil, an animal derived oil, a naturally

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derived vegetable oil, a hydrocarbon oil, an ester oil, a silicone oil, or any combination thereof.

5 5. The process of any of the claims 1 to 4 wherein the anhydrous rheological
modifying agent of said anhydrous composition comprises crude wax, synthetic wax,
animal derived wax, natural wax, naturally derived wax, ester, silicone derived wax,
fatty acid, emulsifier, amino acid derived gelling agent, polymeric wax, poly esters,
polyamide, polyethylene, polyurethane, polyurea, organically modified clays, alkyl
10 acrylate, styrene based polymer, polystyrene, polybutylene, polyisobutylene, fumed
silica, hydrophobically modified silica, aerogel, or any mixture thereof.

6. The process of any of the claim 1 to 5 wherein the alkali swellable aqueous
thickening agents comprises one or more natural gum and acrylate based thickeners.

15 7. The process of any of the claim 1 to 6 wherein the alkali swellable aqueous
thickening agent comprises a natural gum, a clay, an alkali swellable synthetic
polymer, a pre-neutralized alkali swellable synthetic polymer, polymer of
vinylpyrrolidone, polyurethane, polyacrylamide, or any combination thereof.

20 8. The process of any of the claim 1 to 7 wherein the aqueous medium further
comprises an aqueous thickener, where said aqueous thickener comprises a natural
gum, a clay, an alkali swellable synthetic polymer, a pre-neutralized alkali swellable
synthetic polymer, polymer of vinylpyrrolidone, polyurethane, polyacrylamide or any
combination thereof. In some embodiments, the aqueous thickening agents comprises
25 pre-neutralized acrylate based homopolymer, neutralized acrylate based homopolymer,
polymer of vinylpyrrolidone, crosslinked acrylate based homopolymer, polyurethane,
polyacrylamide or any combination thereof.

30 9. The process of any of the claims 1 to 8 wherein the aqueous medium is partially
neutralized by adjusting the pH of said aqueous medium to within the range of 3.0 to
6.0 before it is combined with the anhydrous composition.

10. The process of any of the claims 1 to 8 wherein the neutralization of step (II) comprises adjusting the pH of said resulting aqueous product to within the range of 5.0 – 9.0.

5 11. A process for preparing an aqueous product with visible, discrete, and stabilized droplets;

wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents;

10

wherein said process comprises the steps of:

(I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0;

15

(II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and

(III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 5.0 – 9.0;

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resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

12. An aqueous product comprising visible, discrete, and stabilized droplets;

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wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents;

where said aqueous product is prepared by a process comprising the steps of:

30

(I) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and

(II) neutralizing the resulting aqueous product to achieve one or more desired rheological properties;

resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

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13. An aqueous product comprising visible, discrete, and stabilized droplets;

wherein said aqueous product comprises: (i) droplets of an anhydrous composition comprising an oil and one or more anhydrous rheological modifying agents; and (ii) an aqueous medium comprising water and one or more alkali swellable aqueous thickening agents;

10

where said aqueous product is prepared by a process comprising the steps of:

(I) partially neutralizing the aqueous medium by adjusting the pH of said aqueous medium to within the range of 3.0 to 6.0;

(II) adding said anhydrous composition to the aqueous medium and mixing the resulting composition to form suspended droplets of said anhydrous composition in said aqueous medium; and

15

(III) neutralizing the resulting aqueous product to achieve one or more desired rheological properties by adjusting the pH of said resulting aqueous product to within the range of 5.0 – 9.0;

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resulting in an aqueous product where said droplets of said anhydrous composition are suspended homogeneously throughout the aqueous medium.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2018/059939

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. Claims Nos.: **1-13(partially)**
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No PCT/US2018/059939

A. CLASSIFICATION OF SUBJECT MATTER				
INV. A61K8/11	A61Q5/00	A61Q17/04	A61Q19/00	C11D11/00
C11D17/00	A61K8/37	A61K8/87	A61K8/81	A61K8/88
A61K8/891	A61K8/92	A61K8/85	A61K8/65	A61K8/73
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) A61Q C11D A61K				
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, CHEM ABS Data, WPI Data				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the relevant passages			Relevant to claim No.
X	US 2016/015622 A1 (RAFFERTY DENISE W [US] ET AL) 21 January 2016 (2016-01-21)			1,4-8,
Y	claims 1,43,51,52,54-58,62,65 page 20, paragraph 225-227 examples 2,3,7,9,10; table 1 page 25, paragraph 265-270; examples 11-24			10,12,13 1-13
X	----- DATABASE GNPD [Online] MINTEL; 23 August 2017 (2017-08-23), anonymous: "Hydro-Pearl Serum", XP055545170, retrieved from www.gnpd.com Database accession no. 4996627			12,13
Y	abstract			1-13
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<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C.		<input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents :				
"A" document defining the general state of the art which is not considered to be of particular relevance		"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		
"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 step when the document is taken alone		
"L" document which may throw doubts on priority claim(s) or which is 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 combined with one or more other such documents, such combination being obvious to a person skilled in the art		
"O" document referring to an oral disclosure, use, exhibition or other means		"&" document member of the same patent family		
"P" document published prior to the international filing date but later than the priority date claimed				
Date of the actual completion of the international search		Date of mailing of the international search report		
23 January 2019		05/02/2019		
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		Authorized officer Grillenberger, Sonja		

INTERNATIONAL SEARCH REPORT

International application No PCT/US2018/059939

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	<p>Nn: "Carbopol (TM) Ultrez 10 Polymer for Personal Care Applications TDS-225", ¹ January 2002 (2002-01-01), pages 1-4, XP055545296, Retrieved from the Internet: URL:https://www.ulprospector.com/documents/1172658.pdf?bs=77&b=3758&st=20&r=eu&ind=personalcare [retrieved on 2019-01-21] incorporating...; formulating emulsions ...; universal properties ... -----</p>	1-13
X	<p>Nn: "Glass Marble After-Sun Chamomile Gel SU-0074", ²⁸ August 2017 (2017-08-28), pages 1-2, XP055545139, Retrieved from the Internet: URL:https://www.ulprospector.com/documents/1548721.pdf?bs=77&b=741525&st=20&r=eu&ind=personalcare [retrieved on 2019-01-21]</p>	1-13
Y	<p>introduction; formulation; procedure -----</p>	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2018/059939

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2016015622	A1	21-01-2016	
		AR 095065 A1	16-09-2015
		BR 112015021530 A2	18-07-2017
		CN 105007884 A	28-10-2015
		EP 2964181 A1	13-01-2016
		JP 2016516674 A	09-06-2016
		KR 20150128792 A	18-11-2015
		TW 201446283 A	16-12-2014
		US 2016015622 A1	21-01-2016
		WO 2014137859 A1	12-09-2014

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 1-13(partially)

Present independent claims 1, 11, 12 and 13 relate to an extremely large number of possible compounds to be used in the claimed methods ("oil", "anhydrous rheological modifying agent", "alkali swellable aqueous thickening agent"). Support and disclosure in the sense of Article 6 and 5 PCT is to be found however for only a very small proportion of the compounds used in the methods claimed, see table 1 (p.21), where a specific ester oil is used ("Schercemol NGDO"), a single class of "polyurethane oil thickener" is mentioned, and two specific "alkali swellable aqueous thickening agents" are used ("Carbopol Ultrez 10" = U10, and "Carbopol Ultrez 21" = U21). It is noted that there is no support for performance of the processes of claims 1 and 11 for any of the "aqueous thickening agents" listed in dependent claims 6-8 besides "acrylate based thickeners" and/or "alkali swellable synthetic polymer".

The non-compliance with the substantive provisions is to such an extent, that the search was performed taking into consideration the non-compliance in determining the extent of the search of independent claims 1, 11, 12 and 13 (PCT Guidelines 9.19 and 9.23).

The search of independent claims 1, 11, 12 and 13 was restricted to those claimed compounds to be used in the claimed methods which appear to be supported and a generalisation of their structural formulae, i.e. ester oils (selection in claim 4), the anhydrous rheological modifying agents of claim 5, and acrylate based thickeners as alkali swellable synthetic polymer (selection in claim 6).

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guidelines C-IV, 7.2), should the problems which led to the Article 17(2) declaration be overcome.