



(86) Date de dépôt PCT/PCT Filing Date: 2003/11/03
(87) Date publication PCT/PCT Publication Date: 2004/07/01
(45) Date de délivrance/Issue Date: 2011/05/31
(85) Entrée phase nationale/National Entry: 2005/05/20
(86) N° demande PCT/PCT Application No.: EP 2003/012419
(87) N° publication PCT/PCT Publication No.: 2004/054695
(30) Priorité/Priority: 2002/12/16 (US10/320,029)

(51) Cl.Int./Int.Cl. *B01F 3/08* (2006.01),
B01F 15/04 (2006.01), *B01F 5/10* (2006.01)

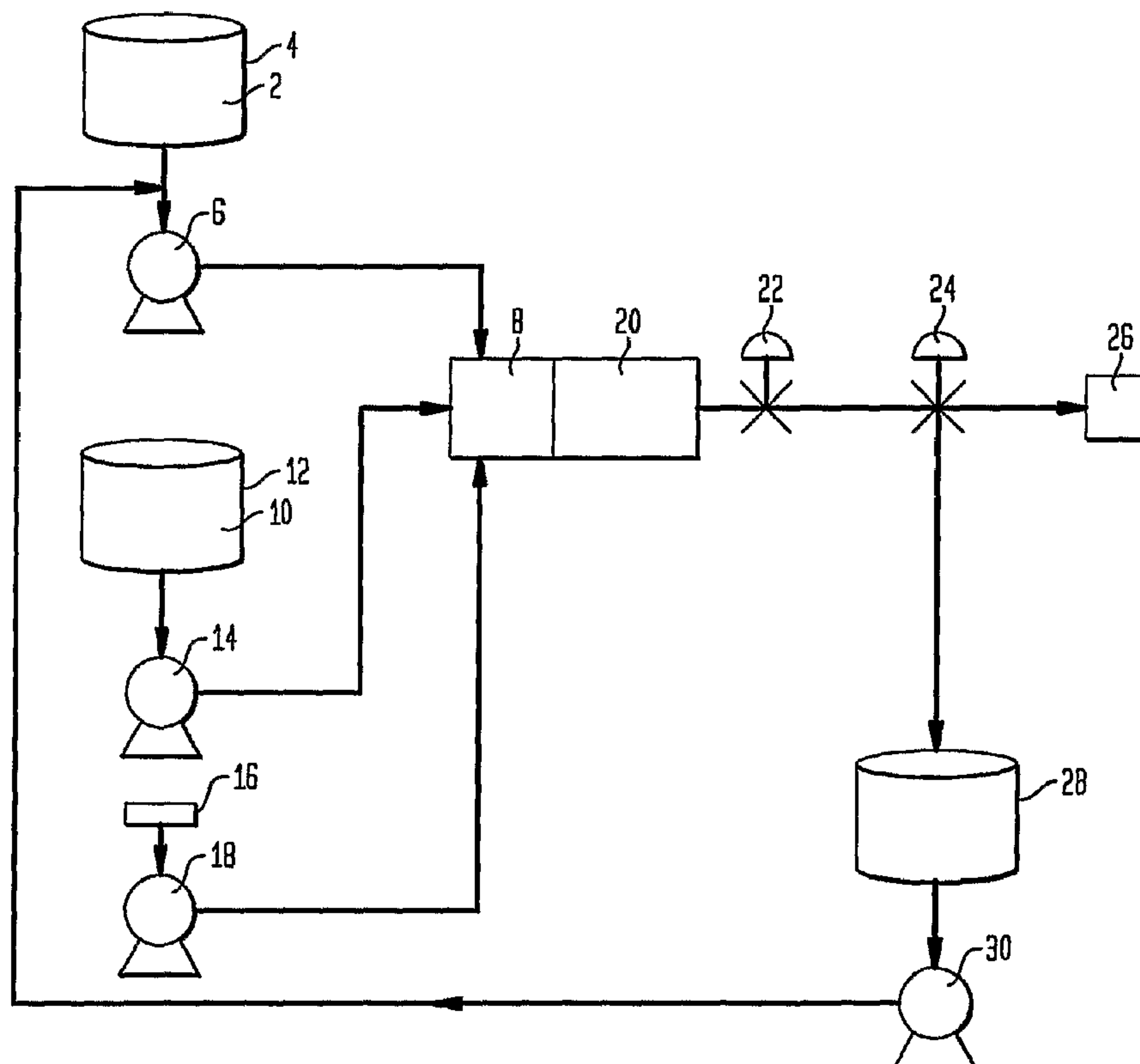
(72) Inventeurs/Inventors:
DIVONE, PETER ANTHONY, US;
BIERCEVICZ, WALTER ANTHONY, US;
REGAN, JOSEPH JAMES, US;
BRIDGES, CHRISTY ANN, US;
PRIEST, KIMBERLY ANN, US;
JUNGBLUT, MATTHEW, US

(73) Propriétaire/Owner:
UNILEVER PLC, GB

(74) Agent: BERESKIN & PARR LLP/S.E.N.C.R.L.,S.R.L.

(54) Titre : PROCÉDE DE FABRICATION DE PRODUITS DE SOIN PERSONNEL AU MOYEN D'UNE PHASE D'EAU
CONCENTREE

(54) Title: PROCESS FOR MANUFACTURE OF PERSONAL CARE PRODUCTS UTILIZING A CONCENTRATE WATER
PHASE



(57) Abrégé/Abstract:

A process which may be continuous is provided for manufacture of personal care product compositions. The process involves feeding a first water phase which is a concentrate containing most if not all water soluble ingredients of the composition into a



(57) **Abrégé(suite)/Abstract(continued):**

blending tube. A second phase which can be oily or aqueous and a third water phase, the latter being essentially pure water, are also fed into the blending tube. All of the phases are transported through the tube at a flow rate of 5 to 5,000 lbs./minute and at a pressure of 10 to 5,000 psi. Preferably the tube leads into a homogenizer such as a sonolator.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
1 July 2004 (01.07.2004)

PCT

(10) International Publication Number
WO 2004/054695 A1

(51) International Patent Classification⁷: **B01F 3/08**

(21) International Application Number:
PCT/EP2003/012419

(22) International Filing Date:
3 November 2003 (03.11.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
10/320,029 16 December 2002 (16.12.2002) US

(71) Applicant (for AE, AG, AU, BB, BZ, CA, EG, GB, GD, GH, GM, IL, KE, LC, LK, LS, MN, MW, NZ, OM, PG, SC, SD, SG, SL, TT, TZ, UG, VC, ZA, ZM, ZW, SZ, CY, IE only): **UNILEVER PLC** [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).

(71) Applicant (for AL, AM, AT, AZ, BA, BE, BF, BG, BJ, BR, BY, CF, CG, CH, CI, CM, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, FR, GA, GE, GN, GQ, GR, GW, HR, HU, ID, IS, IT, JP, KG, KP, KR, KZ, LR, LT, LU, LV, MA,

MC, MD, MG, MK, ML, MR, MX, MZ, NE, NI, NL, NO, PH, PL, PT, RO, RU, SE, SI, SK, SN, SY, TD, TG, TJ, TM, TN, TR, UA, UZ, VN, YU only): **UNILEVER NV** [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).

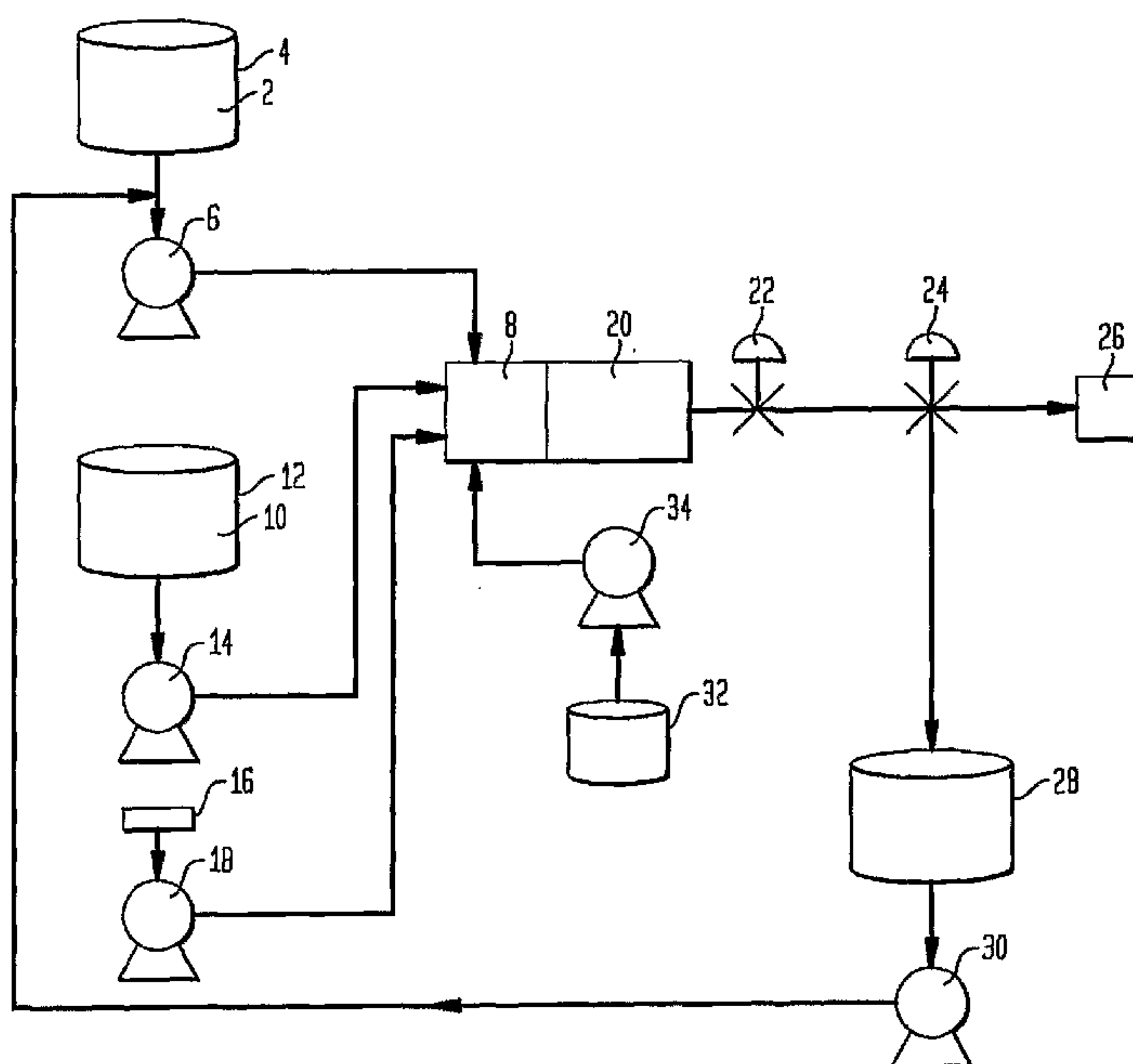
(71) Applicant (for IN only): **HINDUSTAN LEVER LIMITED** [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, 400 020 Mumbai (IN).

(72) Inventors: **DIVONE, Peter, Anthony**; Unilever Home & Personal Care USA, 40 Merritt Boulevard, Trumbull, CT 06611 (US). **BIERCEVICZ, Walter, Anthony**; 9 Wagon Wheel Drive, Prospect, CT 06712 (US). **REGAN, Joseph, James**; Unilever Home & Personal Care USA, 40 Merritt Boulevard, Trumbull, CT 06611 (US). **BRIDGES, Christy, Ann**; Unilever Home & Personal Care USA, 40 Merritt Boulevard, Trumbull, CT 06611 (US). **PRIEST, Kimberly, Ann**; Unilever Home & Personal Care USA, 40 Merritt Boulevard, Trumbull, CT 06611 (US). **JUNG-BLUT, Matthew**; Unilever Home & Personal Care USA, 325 North Wells, Chicago, IL 60610 (US).

(74) Agent: **MULDER, Cornelis, Willem, Re**; Unilever PLC, Patent Department, Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB).

[Continued on next page]

(54) Title: PROCESS FOR MANUFACTURE OF PERSONAL CARE PRODUCTS UTILIZING A CONCENTRATE WATER PHASE



(57) Abstract: A process which may be continuous is provided for manufacture of personal care product compositions. The process involves feeding a first water phase which is a concentrate containing most if not all water soluble ingredients of the composition into a blending tube. A second phase which can be oily or aqueous and a third water phase, the latter being essentially pure water, are also fed into the blending tube. All of the phases are transported through the tube at a flow rate of 5 to 5,000 lbs./minute and at a pressure of 10 to 5,000 psi. Preferably the tube leads into a homogenizer such as a sonolator.

WO 2004/054695 A1

WO 2004/054695 A1



(81) **Designated States (national):** AE, AG, AL, AM, AT (utility model), AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ (utility model), CZ, DE (utility model), DE, DK (utility model), DK, DM, DZ, EC, EE (utility model), EE, EG, ES, FI (utility model), FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT (utility model), PT, RO, RU, SC, SD, SE, SG, SK (utility model), SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) **Designated States (regional):** ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE,

ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

PROCESS FOR MANUFACTURE OF PERSONAL CARE
PRODUCTS UTILIZING A CONCENTRATE WATER PHASE

The invention concerns a process for manufacture of liquid
5 personal care products which minimizes equipment requirements
increases capacity and reduces manufacturing times.

Among the most widely used personal care products of the
liquid type are shampoos, shower gels and skin lotions.
Ordinarily each of these products are marketed in a variety
10 of colors with different fragrances and promotional
ingredients. More efficient ways are continuously being
sought for their manufacture.

Particularly sought are systems which can minimize
equipment, increase capacity and reduce formulation time.

15 In a first aspect, the invention provides a process for
manufacture of personal care compositions which includes:

- (i) feeding a first water phase having a viscosity from
about 50 to about 30,000 cps into a blending tube;
- (ii) feeding a second phase into the blending tube;
- 20 (iii) feeding a third phase having a viscosity of less
than 30 cps into the blending tube, the third
phase being at least about 15 % of the
composition;
- (iv) mixing together all three of the phases, each of
25 the phases being pumped into the blending tube as

- 2 -

a liquid stream at a pressure from about 10 to about 5,000 psi and at a flow rate of about 2.27 to 2,270 kg (5 to about 5,000 pounds) per minute; and

- 5 (v) recovering a resultant mixture as a personal care composition.

Advantages and features of the present invention will become more readily apparent from consideration of the drawings, in which:

- 10 Fig. 1 is a schematic of a first embodiment according to the present process; and

Fig. 2 is a schematic of a second embodiment according to the present process.

Now it has been found that significant manufacturing
15 efficiencies can be achieved for the production of personal care compositions. The advance is based upon a first water phase concentrate incorporating most of any water soluble ingredients found in the final personal care composition. The concentrate is then blended with a second phase. Where
20 the eventual personal care composition is an emulsion such as a skin cream, the second phase will be an oil phase. Other products such as shampoos which do not contain an oil phase will be manufactured with a second phase that is aqueous and formulated with one or more surfactants. A
25 third phase provides additional water. Normally the third

- 3 -

phase will be purely water, but sometimes it may carry a minor amount of further ingredients.

All three phases are blended in a tube. For the purposes of this invention, the term "blending tube" may include any
5 chamber, vessel and piping wherein all three phases come together for mixing. Ordinarily the volume of the blending tube will be relatively small. The volume may range from about 0.0016387 to 1638.7 cm³ (0.0001 to about 100 cubic inches), preferably from about 0.016387 to 327.74 cm³ (0.001
10 to about 20 cubic inches), more preferably from about 0.16387 to 81.94 cm³ (0.01 to about 5 cubic inches), and optimally from about 1.6387 to 16.387 cm³ (0.1 to about 1 cubic inch).

Even though much of the water is eliminated from the first
15 water phase concentrate, most water-soluble ingredients can still be placed into solution or suspended. The benefit is found with the holding/blending tank which can be significantly downsized from the traditional non-concentrate process. Less mixing equipment is necessary and less tank
20 space is required. Top-off water in the form of an additional water (third) phase reconstitutes the concentrate at the point of mixing. No holding tanks or mixers are ordinarily necessary for preparing the third (water) phase. This phase may be directly pumped into the blending tube
25 from any water source such as a well or municipal water carrier.

- 4 -

Viscosities of the first water phase concentrate will be much higher than that of the third (water) phase. More specifically, the first water phase will have a viscosity ranging from about 50 to about 30,000 cps, preferably from
5 about 150 to about 20,000 cps, optimally from about 300 to about 5,000 cps as measured on a Brookfield RVT Viscometer, Spindle No. RV6 at 20 rpm for 1 minute at 25°C.

The third (water) phase ordinarily will have virtually no other additives. Viscosity of this phase is usually less
10 than 5 cps, preferably from about 1 to 4 cps, as measured on a Brookfield RVT Viscometer, Spindle No. RV6 at 20 rpm for 1 minute at 25°C.

Figure 1 is a schematic flow diagram of the process according to the present invention. A concentrated first
15 water phase 2 is formulated within a tank 4. Reconstitution of the concentrate may require anywhere from two to fifty times the amount of pure water by weight to achieve the desired composition. The first phase is delivered via a pump 6 through piping to a blending tube 8.

20 A second phase 10 is held within tank 12. Often when the intended composition is a skin care product, formulation will be an emulsion requiring a water and oil phase. In this instance, the second phase will be an oil phase. Products such as shampoos and shower gels often do not
25 require an oil phase. For these types of products, the second phase will be aqueous and usually deliver one or more surfactants. The levels of surfactants in shampoos and shower gels generally are well above 10 %. Consequently,

- 5 -

these types of products may utilize an aqueous second phase which can include total surfactant levels ranging from about 15 % to about 90 %, preferably from about 25 % to about 75 % by weight of the second phase.

- 5 Delivery of the second phase into the blending tube is mediated by a pump 14. Typical pumps can be Triplex Cat or Progressive Cavity Pumps (ex Moyno/Seepex).

A third phase 16 normally constituted only by water, but occasionally may have a few additives, is also fed into the
10 blending tube. Transfer is facilitated by a pump 18. The latter may be a Moyno/Seepex Progressive Cavity pump, a Waukesha or Positive Displacement pump.

Subsequent to leaving blending tube 8, the combination of phases 2, 10 and 16 enter a Sonolator[®] 20 which operates as
15 a homogenizer thoroughly mixing all phases together under high pressure and shear. The resultant fluid is then transmitted through a back pressure valve 22. Downstream a 3-way valve 24 leads most of the product to a storage tank or directly to a package filler 26. Normally a small amount
20 of product is diverted via the 3-way valve into re-work reservoir 28 for recycling via pump 30 into the stream of the first phase. Amounts of rework may range from 0 to 50 %, preferably from 1 % to 20 %, optimally about 5 % of total product that has exited the sonolator from the blending
25 tube.

Typically the process involves formation of a first water phase in a first reactor, wherein components are mixed and

- 6 -

maintained at a temperature from about 10 to about 70°C, preferably from about 18 to about 58°C, optimally from about 24 to about 52°C. In most but not all instances it will be preferable to have the first water phase at a temperature at least about 5°C, preferably at least about 11°C, cooler than the second phase (especially where this is oil) at point of mutual blending.

The second phase will be formed in a reactor by mixing components at a temperature ranging from about 10 to about 150°C, preferably from about 40 to about 165°C, optimally from about 66 to about 95°C.

The third phase can be maintained at a temperature from about 0 to about 57°C, preferably from about 5 to about 45°C, optimally from about 15 to about 38°C.

- 15 Viscosities of the second phase as measured with a Brookfield RVT Viscometer, Spindle No. RV6 at 20 rpm for 1 minute at 25°C, may range from about 50 to about 200,000 cps, preferably from about 1,000 to about 100,000, optimally from about 5,000 to about 50,000 cps.
- 20 Viscosities of the final personal care products normally may range between about 1,000 and about 30,000 cps, preferably between about 5,000 and about 30,000 cps, and optimally between 10,000 and 25,000 cps using the Brookfield RVT Viscometer, Spindle No. RV6 at 20 rpm for 1 minute at 25°C.
- 25 Deliveries of fluids from tanks containing the first and second phases into the blending tube 8 can be achieved

- 7 -

through a pair of electronically controlled servo-driven rotary pumps. Careful control of flow is achieved by use of blending valves available from the Oden Corporation monitored by E&H mass flow meters. The equipment may
5 include static mixing elements and back-pressure valves.

Flow rate for the first water phase and second phase into blending tube 8 may range from about 2.27 to 2,270 kg (5 to about 5,000 lbs.) per minute. Preferably the flow rate may range from about 22.7 to 454 kg (50 to about 1,000 lbs.) per
10 minute, and optimally from about 68.10 to 363.20 kg (150 to about 800 lbs.) per minute.

The geometry of the blending tube 8 should be such that residence time of the blended first, second and third phases may range from about 0.0001 seconds to about 5 minutes,
15 preferably from about 0.001 to about 120 seconds, optimally from about 0.01 to about 10 seconds.

In systems where the second phase is an oil, the temperature of the emulsion in blending tube 8 normally should be less than the temperature of the second (oil) phase as the latter
20 leaves its reactor tank. Typical emulsion temperatures within the blending tube 8 may range from about 10 to about 82°C, preferably from about 27 to about 65°C, optimally from about 35 to about 55°C.

In a preferred embodiment, oil and both water (first and
25 third) phases are pumped at relatively high pressures which may range from about 10 to about 5,000 psi, preferably from

- 8 -

about 100 to about 1000 psi, and optimally from about 150 to about 250 psi.

In one embodiment of the present invention, oil and first water phases are transferred from their respective reactors
5 through a positive displacement feed pump such as a Waukesha PD Gear Pump. Thereafter each of the separate phases are delivered through a high pressure pump such as a triplex plunger type available from the Giant Corporation, Toledo, Ohio or from the Cat Corporation. From there, each of the
10 separate water and oil phases are fed into the blending tube 8 which in a preferred embodiment is a Sonolator[®] available from the Sonic Corporation, unit of General Signal.

The Sonolator[®] is an in-line device capable of converting the kinetic energy of a high velocity stream of liquid into
15 a high intensity mixing action. Conversion is accomplished by pumping the liquid through an orifice against a blade-like obstacle immediately in the jet stream of the liquid. The liquid itself oscillates in a stable vortexing pattern, which in turn causes the blade-like obstacle to resonate,
20 resulting in a high level of cavitation, turbulence and shear. The blade or knife is brought into an ultrasonic vibration by the fluid motion which causes cavitation in the liquid.

Alternative high-pressure fed homogenizers other than the
25 sonolator are the Manton Gaulin type homogenizer available from the APV Manton Corporation and the Microfluidizer available from Microfluidics Corporation. These type high pressure homogenizers contain a valve which is pressed

- 9 -

(hydraulically or by a spring) against a fixed valve seat. Under high pressure, fluid flows through the opening in the seat and then through a gap between the valve and seat. Although geometries of different high pressure homogenizers
5 may differ in details, and may even be roughened with sharp edges, they all are generally similar. Often the high pressure homogenizer may consist of two or more valve-seat combinations.

The weight ratios of the first water phase (concentrate) to
10 the third (water) phase will be dependent upon the type of personal care composition and particular formulation ingredients. The weight ratio may range from about 1:1 to about 1:40, preferably from about 1:1 to about 1:10, optimally from about 1:1 to about 1:6.

15 The first water phase and second phase are present in a weight ratio ranging from about 50:1 to about 1:50, preferably from about 10:1 to about 1:10.

Ordinarily the third phase will be constituted solely of water. However, in certain instances this phase may contain
20 a small amount of additives. Normally the amount of those additives will constitute no more than 10 %, preferably no more than 5 %, optimally no more than 2 % by weight of the third phase. By contrast, the first water phase (concentrate) can generally include more than 5 %,
25 preferably more than 10 %, most preferably more than 15 %, and optimally more than 40 % by weight of the concentrate of ingredients other than water.

- 10 -

Fig. 2 illustrates a slightly modified process from the one described by Fig. 1. The modification involves addition of a thickener in water as a fourth phase 32 via a pump 34 into the blending tube. Ordinarily, thickeners can be formulated
5 via the first phase. However, some types of final product are required to be exceptionally viscous. For those instances, a separate phase is found to be more efficient for incorporating relatively large amounts of a thickener.

Personal care compositions according to this invention may
10 include shampoos, shower gels, liquid hand cleansers, liquid dental compositions, skin lotions and creams, hair colorants, facial cleansers, and impregnated fluids absorbed on wiping articles.

Generally the first phase and/or the second phase will
15 contain a surfactant. Useful surfactants include nonionic, anionic, cationic, amphoteric, zwitterionic and surfactant combinations thereof. Overall amount of surfactant may range from about 0.1 % to about 50 %, preferably from about 2 % to about 40 %, optimally from about 15 % to about 25 %
20 by weight of the total personal care composition.

Illustrative but not limiting examples of suitable nonionic surfactants include C₁₀-C₂₀ fatty alcohol or acid hydrophobes condensed with from 2 to 100 moles of ethylene oxide or propylene oxide per mole of hydrophobe; C₂-C₁₀ alkyl phenols
25 condensed with from 2 to 20 moles of alkylene oxides; mono- and di- fatty acid esters of ethylene glycol such as ethylene glycol distearate; fatty acid monoglycerides;

- 11 -

sorbitan mono- and di- C₈-C₂₀ fatty acids; and polyoxyethylene sorbitan available as Polysorbate 80 and Tween 80[®] as well as combinations of any of the above surfactants.

- 5 Other useful nonionic surfactants include alkyl polyglycosides (APGs), saccharide fatty amides (e.g. methyl gluconamides) as well as long chain tertiary amine oxides. Examples of the latter category are: dimethyldodecylamine oxide, oleyldi(2-hydroxyethyl)amine oxide,
- 10 dimethyloctylamine oxide, dimethyldecylamine oxide, dimethyltetradecylamine oxide, di(2-hydroxyethyl)tetradecylamine oxide, 3-didodecyloxy-2-hydroxypropyldi(3-hydroxypropyl) amine oxide, and dimethylhexadecylamine oxide.
- 15 Illustrative but not limiting examples of anionic surfactants include the following:

(1) Alkyl benzene sulfonates in which the alkyl group contains from 9 to 15 carbon atoms, preferably 11 to 14 carbon atoms in straight chain or branched chain configuration. Especially

20 preferred is a linear alkyl benzene sulfonate containing about 12 carbon atoms in the alkyl chain.

(2) Alkyl sulfates obtained by sulfating an

25 alcohol having 8 to 22 carbon atoms, preferably 12 to 16 carbon atoms. The alkyl sulfates have the

- 12 -

formula $\text{ROSO}_3\text{-M}^+$ where R is the C₈-22 alkyl group and M is a mono- and/or divalent cation.

(3) Paraffin sulfonates having 8 to 22 carbon atoms, preferably 12 to 16 carbon atoms, in the alkyl moiety.

(4) Olefin sulfonates having 8 to 22 carbon atoms, preferably 12 to 16 carbon atoms. Most preferred is sodium C₁₄-C₁₆ olefin sulfonate, available as Bioterge AS 40[®].

(5) Alkyl ether sulfates derived from an alcohol having 8 to 22 carbon atoms, preferably 12 to 16 carbon atoms, ethoxylated with less than 30, preferably less than 12, moles of ethylene oxide. Most preferred is sodium lauryl ether sulfate formed from 2 moles average ethoxylation, commercially available as Standopol ES-2[®].

(6) Alkyl glyceryl ether sulfonates having 8 to 22 carbon atoms, preferably 12 to 16 carbon atoms, in the alkyl moiety.

(7) Fatty acid ester sulfonates of the formula: $\text{R}^1\text{CH}(\text{SO}_3\text{-M}^+)\text{CO}_2\text{R}^2$ where R¹ is straight or branched alkyl from about C₈ to C₁₈, preferably C₁₂ to C₁₆, and R² is straight or branched alkyl from about C₁

- 13 -

to C₆, preferably primarily C₁, and M⁺ represents a mono- or divalent cation.

(8) Secondary alcohol sulfates having 6 to 18, preferably 8 to 16 carbon atoms.

5 (9) Fatty acyl isethionates having from 10 to 22 carbon atoms, with sodium cocoyl isethionate being preferred.

10 (10) Mono- and dialkyl sulfosuccinates wherein the alkyl groups range from 3 to 20 carbon atoms each.

(11) Alkanoyl sarcosinates corresponding to the formula RCON(CH₃)CH₂CH₂CO₂M wherein R is alkyl or alkenyl of about 10 to about 20 carbon atoms and M is a water-soluble cation such as ammonium,
15 sodium, potassium and trialkanolammonium. Most preferred is sodium lauroyl sarcosinate.

Illustrative of cationic surfactants are C₈-C₂₂ alkyl C₁-C₄ di-alkyl ammonium salts such as cetyl dimethyl ammonium chloride, stearyl dimethyl ammonium methosulfate, oleyl
20 diethylammonium phosphate, and lauryl dimethyl ammonium borate. Particularly preferred is cetrimonium chloride which is a generic term for cetyl dimethyl ammonium chloride.

Amphoteric surfactants useful for the present invention
25 include betaines which may have the general formula

- 14 -

$RN^+(R^1)_2R^2-COO^-$ wherein R is a hydrophobic moiety selected from the group consisting of alkyl groups containing from 10 to 22 carbon atoms, preferably from 12 to 18 carbon atoms; alkyl aryl and aryl alkyl groups containing 10 to 22 carbon
5 atoms with a benzene ring being treated as equivalent to about 2 carbon atoms, and similar structures interrupted by amido or ether linkages; each R^1 is an alkyl group containing from 1 to 3 carbon atoms; and R^2 is an alkylene group containing from 1 to about 6 carbon atoms.

10 Sulfobetaines such as cocoamidopropyl hydroxysultaine are also suitable.

Examples of preferred betaines are dodecyl dimethyl betaine, cetyl dimethyl betaine, dodecyl amidopropyldimethyl betaine, tetradecyldimethyl betaine, tetradecylamidopropyldimethyl
15 betaine, and dodecyldimethylammonium hexanoate. Most preferred is cocoamidopropyl betaine available as Tegobetaine F[®] sold by Th. Goldschmidt AG of Germany.

Polyols are frequently present in the water phase of the present invention. Typical polyhydric alcohols include
20 glycerol (also known as glycerin), polyalkylene glycols and more preferably alkylene polyols and their derivatives, including propylene glycol, dipropylene glycol, polypropylene glycol, polyethylene glycol and derivatives thereof, sorbitol, hydroxypropyl sorbitol, hexylene glycol,
25 butylene glycol, 1,2,5-hexanetriol, ethoxylated glycerol, propoxylated glycerol and mixtures thereof. Most preferred is glycerin. Amounts of the polyols may range from about

- 15 -

0.5 % to about 50 %, preferably between about 1 % and about 15 % by weight of the total personal care composition.

Thickeners/viscosifiers in amounts from about 0.01 % to about 10 % by weight of the total personal care composition
5 may also be included in the first water phase and/or in a separate fourth phase. As known to those skilled in the art, the precise amount of thickeners can vary depending upon the consistency and thickness of the composition which is desired. Exemplary thickeners are xanthan gum, sodium
10 carboxymethyl cellulose, hydroxyalkyl and alkyl celluloses (particularly hydroxypropyl cellulose), and cross-linked acrylic acid polymers such as those sold by B.F. Goodrich under the Carbopol trademark.

Thickeners such as modified starches and clays may also be
15 used to thicken the water phase. For instance, aluminum starch octenyl succinate (available as DryFlo[®] from the National Starch and Chemical Company) is particularly useful. Among the clays are included magnesium aluminum silicate (available as Veegum[®]), hectorite clays,
20 montmorillonite clays, bentonites (e.g. Bentone[®] 38) and combinations thereof.

Water soluble conditioning agents may also be incorporated into the water phase. Cationic agents in monomeric and polymeric form are particularly useful for this purpose.
25 Cationic cellulose derivatives, cationic starches, copolymers of a diallyl quaternary ammonium salt and an acrylamide, quaternized vinylpyrrolidone vinylimidazole polymers,

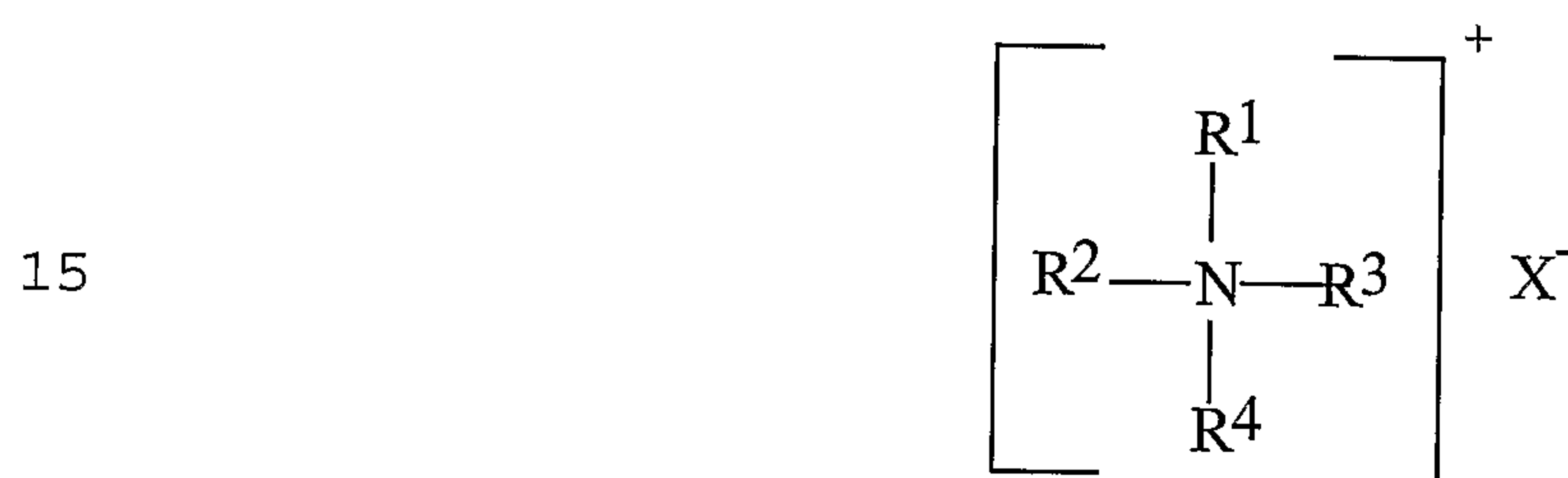
- 16 -

polyglycol amine condensates, quaternized collagen polypeptide, polyethylene imine, cationized silicone polymer (e.g. Amodimethicone), cationic silicone polymers provided in a mixture with other components under the trademark Dow

5 Corning 929 (cationized emulsion), copolymers of adipic acid and dimethylaminohydroxypropyl diethylenetriamine, cationic chitin derivatives, cationized guar gum (e.g. Jaguar[®] C-B-S, Jaguar[®] C-17, and Jaguar[®] C-16) and quaternary ammonium salt polymers (e.g. Mirapol[®] A-15, Mirapol[®] AD-1, Mirapol[®], ZA-1,

10 etc., manufactured by the Miranol Division of the Rhone Poulenc Company).

Examples of the monomeric cationic conditioning agents are salts of the general structure:



wherein R¹ is selected from an alkyl group having from 12 to 22 carbon atoms, or aromatic, aryl or alkaryl groups having from 12 to 22 carbon atoms; R², R³, and R⁴ are independently

20 selected from hydrogen, an alkyl group having from 1 to 22 carbon atoms, or aromatic, aryl or alkaryl groups having from 12 to 22 carbon atoms; and X⁻ is an anion selected from chloride, bromide, iodide, acetate, nitrate, sulfate, methyl sulfate, ethyl sulfate, tosylate, lactylate, citrate,

- 17 -

glycolate, and mixtures thereof. Additionally, the alkyl groups can also contain either linkages or amino group substituents (e.g., the alkyl groups can contain polyethylene glycol and polypropylene glycol moieties).

5 Compositions of the present invention that are emulsions may be oil-in-water or water-in-oil, although the former is preferred. Relative weight ratios of water to oil phases of the emulsion may range from about 1,000:1 to about 1:10, preferably from about 100:1 to about 1:5, optimally from
10 about 10:1 to about 1:2 by weight of the total personal care composition.

Another component often present in the first water phase and sometimes in the other water phase(s) are preservatives. These are incorporated to protect against the growth of
15 potentially harmful microorganism. Suitable traditional preservatives are EDTA salts and alkyl ester of parahydroxybenzoic acid. Other preservatives which have more recently come into use include hydantoin derivatives, propionate salts, and a variety of quaternary ammonium
20 compounds. Cosmetic chemists are familiar with appropriate preservatives and routinely choose them to satisfy the preservative challenge test and to provide product stability.

Particularly preferred preservatives are iodopropynyl butyl
25 carbamate, phenoxyethanol, methyl paraben, propyl paraben, imidazolidinyl urea, sodium dehydroacetate and benzyl alcohol. The preservatives should be selected having regard for the use of the composition and possible

- 18 -

incompatabilities between the preservatives and other ingredients in the composition. Preservatives are typically employed in amounts ranging from 0.01 % to 2 % by weight of the total personal care composition.

- 5 The second phase when an oil phase will contain hydrophobic components. Most often the oil phase will incorporate an emollient which may be selected from hydrocarbons, silicones and synthetic or vegetable esters. Amounts of the emollients may range anywhere from about 0.1 % to about 30 %, preferably
10 between about 0.5 % and about 10 % by weight of the total personal care composition.

Hydrocarbons suitable for the present invention include isoparaffins, mineral oil, petrolatum and hydrocarbon waxes such as polyethylenes.

- 15 Silicones may be divided into the volatile and non-volatile variety. The term "volatile" as used herein refers to those materials which have a measurable vapor pressure at ambient temperature. Volatile silicone oils are preferably chosen from cyclic or linear polydimethylsiloxanes containing from
20 about 3 to about 9, preferably from about 4 to about 5, silicone atoms.

- Non-volatile silicones useful as an emollient material include polyalkyl siloxanes, polyalkylaryl siloxanes and polyether siloxane copolymers. The essentially non-volatile polyalkyl
25 siloxanes useful herein include, for example, polydimethyl siloxanes with viscosities of from about 5 to about 100,000 centistokes at 25°C.

- 19 -

Among suitable ester emollients are:

(1) Alkenyl or alkyl esters of fatty acids having 10 to 20 carbon atoms. Examples thereof include isopropyl; palmitate, isononyl isononoate, oleyl myristate, oleyl
5 stearate, cetearyl stearate and oleyl oleate.

(2) Ether-esters such as fatty acid esters of ethoxylated fatty alcohols.

(3) Polyhydric alcohol esters. Ethylene glycol mono- and di-fatty acid esters, diethylene glycol mono- and di-fatty
10 acid esters, polyethylene glycol (200-6000) mono- and di-fatty acid esters, propylene glycol mono- and di-fatty acid esters, polypropylene glycol 2000 monooleate, polypropylene glycol 2000 monostearate, ethoxylated propylene glycol monostearate, glyceryl mono- and di-
15 fatty acid esters, polyglycerol fatty esters, ethoxylated glyceryl monostearate, 1,3-butylene glycol monostearate, 1,3-butylene glycol distearate, polyoxyethylene polyol fatty acid ester, sorbitan fatty acid esters, and polyoxyethylene sorbitan fatty acid esters are
20 satisfactory polyhydric alcohol esters.

(4) Wax esters such as beeswax, spermaceti, myristyl myristate, stearyl stearate.

(5) Steroid esters, of which soya sterol and cholesterol fatty acid esters are examples thereof.

25 Most preferred vegetable ester emollients are sunflower seed oil, soy sterol esters, borage seed oil, maleated soybean oil,

- 20 -

sucrose polycottonseedate, tribehenin, sucrose polybehenate and mixtures thereof.

Fatty acids may also be included in the oil phase. These fatty acids may have from 10 to 30 carbon atoms. Illustrative
5 of this category are pelargonic, lauric, myristic, palmitic, stearic, isostearic, hydroxystearic, oleic, linoleic, ricinoleic, arachidic, behenic and erucic acids. Amounts may range from 0.1 % to 25 % by weight of the total personal care composition.

10 Fragrances, colorants and promotional ingredients may also be included in the oil and either of the water phases. Colorants illustrative of the present invention include Red No. 4, Red No. 40 and the FD&C colorants Red No. 3, Red No. 6, Red No. 28, Red No. 33, Blue No. 1, Green No. 5, Yellow No. 5, all
15 the foregoing being water soluble. Oil soluble dyes may also be utilized such as Green No. 6 and D&C Violet No. 2. Active levels of these colorants may range from about 0.0001 % to about 1 %, preferably from about 0.001 % to about 0.1 % by weight of the total personal care composition.

20 The term "fragrance" is defined as a mixture of odoriferous components, optionally mixed with a suitable solvent diluent or carrier, which is employed to impart a desired odor.

Fragrance components and mixtures thereof may be obtained from
25 natural products such as essential oils, absolutes, resinoids, resins and concretes, as well as synthetic products such as hydrocarbons, alcohols, aldehydes, ketones, ethers, carboxylic acids, esters, acetals, ketals, nitriles and the like,

- 21 -

including saturated and unsaturated compounds, aliphatic, carbocyclic and heterocyclic compounds.

Suitable solvents, diluents or carriers for fragrance ingredients as mentioned above are for example: ethanol,
5 isopropanol, diethylene glycol monoethyl ether, dipropyl glycol and triethyl citrate.

Particularly preferred fragrance ingredients are cyclic and acyclic terpenes and terpenoids. These materials are based
10 upon isoprene repeating units. Examples include alpha and beta pinene, myrcene, geranyl alcohol and acetate, camphene, dl-limonene, alpha and beta phellandrene, tricyclene, terpinolene, allocimmane, geraniol, nerol, linanool, dihydrolinanool, citral, ionone, methyl ionone, citronellol,
15 citronellal, alpha terpineol, beta terpineol, alpha fenchol, borneol, isoborneol, camphor, terpinen-1-ol, terpin-4-ol, dihydroterpineol, methyl chavicol, anethole, 1,4 and 1,8 cineole, geranyl nitrile, isobornyl acetate, linalyl acetate, caryophyllene, alpha cedrene, guaiol, patchouli alcohol,
20 alpha and beta santalol and mixtures thereof.

Amounts of the fragrance may range from about 0.00001 % to about 2 %, preferably from about 0.0001 % to about 1 %, optimally from about 0.01 % to about 0.5 %, most preferably
25 from about 0.05 % to about 0.25 % by weight of the personal care composition.

Vitamins are illustrative of promotional ingredients. These include Vitamin A (retinol), Vitamin A derivatives (retinyl palmitate, retinyl linoleate, retinyl acetate and retinoic

- 22 -

acid), Vitamin C, Vitamin C derivatives (such as ascorbyl tetraisoalmitate and magnesium ascorbyl phosphate), Vitamin E, Vitamin E derivatives (such as tocopherol acetate), biotin, niacin and DL-panthenol and combinations thereof.

- 5 Amounts of the promotional ingredients may range from about 0.00001 % to about 2 %, preferably from about 0.0001 % to about 1 %, optimally from about 0.001 % to about 0.5 % by weight of the total personal care composition.

10 The combined first and third water phases may constitute from about 5 % to about 99.5 %, preferably from about 20 % to about 90 %, more preferably from about 35 % to about 80 %, optimally from about 45 % to about 70 % by weight of the final personal care composition.

15 The combined first and third water phases will contain water as a major component. Usually the amount of water may range from about 30 % to about 99.9 %, preferably from about 50 % to about 95 %, more preferably from about 65 % to about 80 %, optimally from about 55 % to about 70 % by weight of the combined first and third water phases.

20 The term "comprising" is meant not to be limiting to any subsequently stated elements but rather to encompass non-specified elements of major or minor functional importance. In other words the listed steps, elements or options need not be exhaustive. Whenever the words "including" or "having"

25 are used, these terms are meant to be equivalent to "comprising" as defined above.

- 23 -

EXAMPLES

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material ought to be
5 understood as modified by the word "about".

The following examples will more fully illustrate the embodiments of this invention. All parts, percentages and proportions referred to herein and in the appended claims are by weight unless otherwise illustrated.

10 Example 1

A pair of skin lotions are prepared to reflect a 2X and a 10X level of concentrate. Both concentrates with the appropriate amount of added water will attain the resultant composition
15 shown in Table I.

- 24 -

TABLE I

Phase	Ingredient Name	Resultant Composition (2 Stream)	2X (3 Stream)	10X (4 Stream)
Oil	Stearic Acid	2.0217	2.0217	2.0217
	Glycol Stearate/ Stearamide AMP	1.1939	1.1939	1.1939
	Glycerol Monostearate	0.5572	0.5572	0.5572
	Cetyl Alcohol	0.3184	0.3184	0.3184
	Petrolatum	0.5	0.5	0.5
	Mineral Oil	1.4	1.4	1.4
	Dimethicone	0.3	0.3	0.3
Water	Water	79.4078	32.5534	3.1699
	Tetrasodium EDTA	0.1017	0.1017	0.1017
	Magnesium Aluminum Silicate	0.2	0.2	0.2
	Glycerin	3.5	3.5	3.5
	Methylparaben	0.1425	0.1425	0.1425
	Titanium Dioxide (Water Dispersible)	0.1	0.1	0.1
	Carbopol 934® (2% Active in Water)	9	9	0
	Triethanolamine	0.7568	0.7568	0.7568
	Aloe Vera Gel	0.09	0.09	0.09
	DMDM Hydantoin	0.25	0.25	0.25
	Fragrance	0.16	0.16	0.16
Water Only		-	46.8544	76.2379
Carbopol® 934 Only		-	-	9

The oil and water phases are each separately contained in
 respective first and second tanks. With the aid of a
 5 progressive cavity pump (ex Moyno/Seepex), these phases are
 separately fed through pipes to a blending tube antechamber
 section of a Sonolator®. The combined flow rate is
 precisely maintained at 9.08 kg (20 lbs.)/minute at 200 psi.
 The water phase concentrate is held at a temperature between
 10 24 and 52°C with capacity of the tank being 3,000 gallons.

- 25 -

The oil phase is held at a temperature from 65 to 93°C in a 250 gallon tank.

From a third tank, the 2X concentrate experiment delivers a third stream which is pure water regulated at the same
5 pressure as that of the first water and oil phases. The pure water phase is maintained at 15 to 38°C and pumped into the blending tube antechamber of the Sonolator[®]. All phases are then homogenized together in the Sonolator[®] at 500 psi.

Thereafter the combined streams are sent to a storage vessel
10 where resultant product is held at 24 to 44°C. Some of the resultant skin lotion composition is pumped by a positive displacement pump as a return stream into the blending tube of the Sonolator[®]. The amount of this return flow is approximately 5 % for purposes of a new start-up or as re-work
15 product. The remainder of the composition held within the storage vessel is sent to a packaging line for fill into empty bottles.

The 10X concentrate employs a fourth stream of water/thickener (Carbopol 934[®]). This fourth stream is pumped at similar
20 pressure and flow rate (proportioned to its proportion in the final composition) into the blending tube antechamber of the Sonolator[®]. All other processing is identical to that described above for the 2X concentrate.

Example 2

25

A set of dry skin remediating lotions are prepared to reflect a 2X, 4X and 6X level of concentrates. All three concentrates

- 26 -

with the appropriate amount of added water will attain the resultant composition shown in Table II.

- 27 -

TABLE II

Phase	Ingredient Name	Resultant Composition (2 Stream)	2X (3 Stream)	4X (3 Stream)	6X (3 Stream)
Oil	Stearic Acid	2.54	2.54	2.54	2.54
	Glycol Stearate/Stearamide AMP	1.5	1.5	1.5	1.5
	Glycerol Monostearate	0.7	0.7	0.7	0.7
	Cetyl Alcohol	0.4	0.4	0.4	0.4
	Sunflower Seed Oil	1.25	1.25	1.25	1.25
	Silicone Fluid 200	0.26	0.26	0.26	0.26
	Soy Sterol	0.08	0.08	0.08	0.08
	Vitamin E Acetate	0.01	0.01	0.01	0.01
	Lecithin	0.04	0.04	0.04	0.04
	Sodium Stearoyl Lactylate	0.1	0.1	0.1	0.1
	Petrolatum	0.9375	0.9375	0.9375	0.9375
	Methyl Palmitate	0.3125	0.3125	0.3125	0.3125
Water	Water	81.6605	35.7205	12.7605	5.1005
	Glycerin	5.5	5.5	5.5	5.5
	Disodium EDTA	0.05	0.05	0.05	0.05
	Mineral Blend	0.001	0.001	0.001	0.001
	Magnesium Aluminum Silicate	0.2	0.2	0.2	0.2
	Oat Protein	0.001	0.001	0.001	0.001
	Methylparaben	0.1425	0.1425	0.1425	0.1425
	Titanium Dioxide (Water Dispersible)	0.1	0.1	0.1	0.1
	Carbopol 934® (2% Active)	3	3	3	3
	Triethanolamine	0.813	0.813	0.813	0.813
	DMDM Hydantoin	0.25	0.25	0.25	0.25
	Fragrance	0.15	0.15	0.15	0.15
	Vitamin A Palmitate	0.001	0.001	0.001	0.001
	Keratin	0.001	0.001	0.001	0.001
	Total	91.87			15.31
Water Only		-	45.94	68.9	76.56

- 28 -

The oil and water phases are each separately contained in respective first and second tanks. With the aid of a progressive cavity pump (ex Moyno/Seepex), these phases are separately fed through pipes to a blending tube antechamber
5 section of a Sonolator[®]. The combined flow rate is precisely maintained at 22.7 kg (50 lbs.)/minute at 800 psi. The water phase concentrate is held at a temperature between 24 and 52°C with capacity of the tank being 5,000 gallons. The oil phase is held at a temperature from 65 to 93°C in a
10 250 gallon tank.

From a third tank, the 2X concentrate experiment delivers a third stream which is pure water regulated at the same pressure as that of the first water and oil phases. The pure water phase is maintained at 15 to 38°C and pumped into
15 the blending tube antechamber of the Sonolator[®]. All phases are then homogenized together in the Sonolator[®] at 1000 psi. Thereafter the combined streams are sent to a storage vessel where resultant product is held at 24 to 44°C. Some of the resultant dry skin lotion composition is pumped by a
20 positive displacement pump as a return stream into the blending tube of the Sonolator[®]. The amount of this return flow is approximately 5 % for purposes of a new start-up or as re-work product. The remainder of the composition held within the storage vessel is sent to a packaging line for
25 fill into empty bottles.

Processing for the 4X and 6X concentrates is identical to that described above for the 2X concentrate.

- 29 -

Example 3

A set of shampoo formulations are prepared to reflect a 2X, 4X and 8X level of concentrates. All three concentrates
 5 with the appropriate amount of added water will attain the resultant composition shown in Table III.

TABLE III

Phase	Ingredient Name	Resultant Composition (2 Stream)	2X (3 Stream)	4X (3 Stream)	8X (3 Stream)
Water	Water	79.5278	38.5288	18.0278	7.7768
	Fragrance	0.5000	0.5000	0.5000	0.5000
	D&C RED #33	0.0002	0.0002	0.0002	0.0002
	Vitamin E Acetate	0.0010	0.0010	0.0010	0.0010
	Raspberry Extract	0.0010	0.0010	0.0010	0.0010
	Citric Acid (50% Active)	0.0300	0.0300	0.0300	0.0300
	Hydroxypropyl- methylcellulose	0.1500	0.1500	0.1500	0.1500
	Tetrasodium EDTA	0.2000	0.2000	0.2000	0.2000
	Glydant®	0.1000	0.1000	0.1000	0.1000
	Kathon CG®	0.0400	0.0400	0.0400	0.0400
	Benzophenone-4	0.0500	0.0500	0.0500	0.0500
	Ammonium Chloride	1.4000	1.4000	1.4000	1.4000
Oil		18	18	18	18
Water Only		-	40.999	61.5000	71.7510

10 Processing of the shampoo is performed in a manner similar to that described under Example 2.

- 30 -

EXAMPLE 4

A set of hair conditioners is prepared to reflect a 2X, 4X and 8X level of concentrates. All the concentrates with the appropriate amount of added water will attain the resultant composition shown in Table IV.

TABLE IV

Phase	Ingredient Name	Resultant Composition (2 Stream)	2X (3 Stream)	4X (3 Stream)	8X (3 Stream)
Water	Water	73.486	32.171	11.514	1.185
	Cocamidopropyl Betaine	6.028	6.028	6.028	6.028
	Aloe Vera Powder	0.005	0.005	0.005	0.005
	Benzophenone-4	0.100	0.100	0.100	0.100
	Glycerin	1.000	1.000	1.000	1.000
	PEG-10 Sunflower Seed Oil	0.100	0.100	0.100	0.100
	Tetrasodium EDTA	0.051	0.051	0.051	0.051
	Sodium Hydroxide (50% Aqueous)	0.048	0.048	0.048	0.048
	Ammonium Chloride	0.325	0.325	0.325	0.325
	Kathon CG®	0.020	0.020	0.020	0.020
	Fragrance	1.000	1.000	1.000	1.000
	Color Sol. Ext Violet #2 (0.2%)	0.333	0.333	0.333	0.333
	Vitamin E Acetate	0.001	0.001	0.001	0.001
	Dequest 2010®	0.033	0.033	0.033	0.033
	Polyquaternium-10	0.100	0.100	0.100	0.100
		82.630			
Oil		17.37	17.37	17.37	17.37
Water Only		-	41.315	61.973	72.301

All of the processing is identical to that described under Example 2 for the respective concentrates.

- 31 -

CLAIMS

1. A process for manufacture of personal care compositions comprising:

(i) feeding into a blending tube a first water phase
5 concentrate having a viscosity from 50 to 30,000
cps and incorporating most of any water soluble
ingredients found in the personal care
composition;

(ii) feeding a second phase into the blending tube;

10 (iii) feeding a third phase having a viscosity of less
than 30 cps into the blending tube, the third
phase being at least 15 % of the composition;

(iv) mixing together all three of the phases, each of
the phases being pumped into the blending tube as
15 a liquid stream at a pressure from 10 to 5,000 psi
and at a flow rate of 2.274 to 2,270 kg (5 to
5,000 pounds) per minute; and

(v) recovering a resultant mixture as a personal care
composition.

20

2. The process according to claim 1 wherein the first phase
comprises more than 5 % by weight of the first phase of
ingredients other than water.

- 32 -

3. The process according to claim 1 or claim 2 wherein the first phase comprises more than 40 % by weight of the first phase of ingredients other than water.

5 4. The process according to claim 1, 2 or 3 wherein the first phase and the third phase are fed to the blending tube at a relative weight ratio of 1:1 to 1:40.

5. The process according to any one of claims 1 to 4,
10 wherein the first phase and the third phase are fed to the blending tube at a relative weight ratio of 1:1 to 1:10.

6. The process according to any one of claims 1 to 5,
wherein the third phase comprises no more than 10 % by weight
15 of the third phase of ingredients other than water.

7. The process according to any one of claims 1 to 6,
wherein the third phase comprises no more than 2 % by weight
of the third phase of ingredients other than water.

20

8. The process according to any one of claims 1 to 7 wherein the blending tube has a volume ranging from 0.0016387 to 1638.7 cm³ (0.0001 to 100 cubic inches).

- 33 -

9. The process according to any one of claims 1 to 8 wherein the blending tube has a volume ranging from 0.16387 to 81.94 cm³ (0.01 to 5 cubic inches).

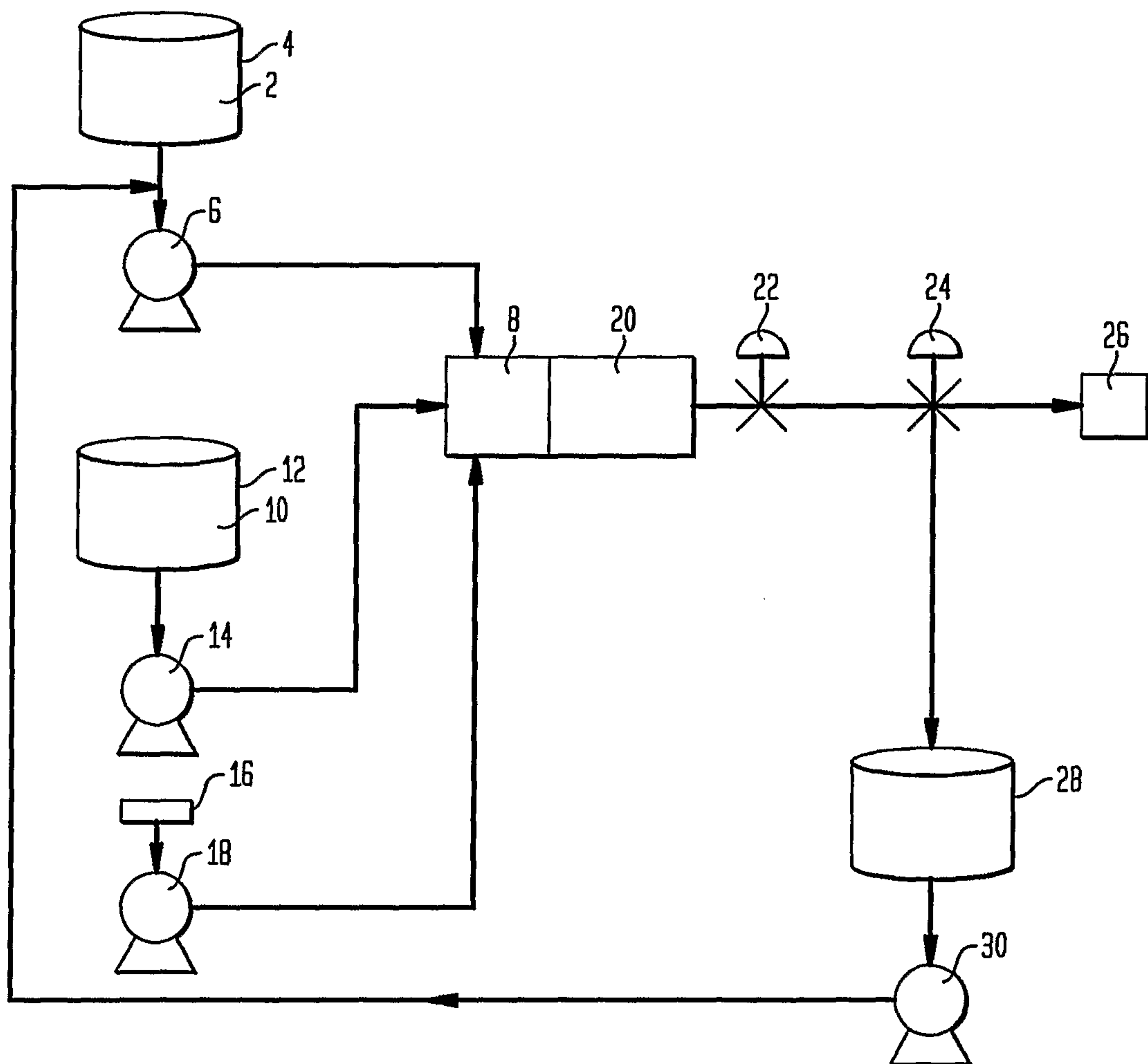
5 10. The process according to any one of claims 1 to 9 wherein mixing of the phases is by sonic agitation.

11. The process according to any one of claims 1 to 10 wherein the first phase entering the tube is at least 5°C
10 lower in temperature than that of the second phase entering the tube.

12. The process according to any one of claims 1 to 11 wherein first, second and third phases are pumped into the
15 tube at a pressure ranging form 100 to 1,000 psi.

1/2

FIG. 1



2/2

FIG. 2

