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(73) Proprietor: **THE PROCTER & GAMBLE COMPANY**
301 East Sixth Street
Cincinnati Ohio 45202 (US)

(72) Inventor: **Whyte, David Denzil**
319 Circlewood Lane
Cincinnati Ohio 45215 (US)

(74) Representative: **Gibson, Tony Nicholas et al**
Procter & Gamble (NTC) Limited Whitley Road Longbenton Newcastle upon Tyne NE12 9TS (GB)

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Description

Technical field

This invention relates to the manufacture of heavily perfumed particles for use in detergent compositions. More particularly it relates to the manufacture of perfume particles having both immediate and long lasting perfume emitting properties.

The use of perfume in various consumer products for aesthetic reasons is well known. Detergent compositions in particular generally contain a perfume. The perfume is normally simply admixed with the remainder of the detergent composition, whether it is a liquid or solid detergent composition. While the perfume does not add to cleaning performance, it does make the product more aesthetically pleasing to the consumer, and, in some cases, imparts a pleasant fragrance to treated articles or surfaces. The consumer has come to expect such detergent products to have a pleasing odor.

Perfumes are, by their nature, made of a combination of volatile substances. Because of this, the perfume is continuously emitted from simple solutions and dry mixes to which it has been added. Various techniques have been developed to hinder or delay the release of the perfume from the composition so that the composition remains aesthetically pleasing for a prolonged length of time. For example, see West German Patent 825,293, December 17, 1951; East German Patent 15,693, October 12, 1975; U.S. Patent 3,772,215, issued November 13, 1973; and U.S. Patent 3,567,119, issued March 2, 1971. While such methods of prolonging the release of perfume from the composition are effective to a limited extent, there is still a need to economically formulate a perfumed particle which continually emits perfume for a substantial length of time.

It will be recognized that a product should desirably have an initial pleasant smell and be capable of delivering that pleasant smell over a long length of time. Encapsulation techniques have a tendency to enclose the perfume so that it is not noticeable until actual use of the product, when the encapsulating material dissolves and the perfume is released. Such techniques are generally also expensive. Other techniques, such as absorption techniques, of "fixing" perfumes have the disadvantage of generally low perfume loads and high perfume losses during manufacture. It is desirable in detergent composition usage that the product have a pleasant smell while in storage and provide strong fragrance impact upon addition to water. U.S. Patent 4,209,417 provided a solution to these requirements in the form of heavily perfumed particles having both immediate and long lasting perfume emitting properties consisting essentially of from 30% to 70% water-insoluble perfume, from 25% to 65% of a water soluble polymer, and emulsifier. The particles were comprised of a continuous polymer matrix having dispersed substantially uniformly therethrough perfume/emulsifier droplets. Those perfume particles were taught to be prepared by forming an aqueous dispersion consisting essentially of from 2% to 40% perfume, from 5 to 20% water-soluble polymer, from 40 to 90% water, and sufficient emulsifier to form a stable emulsion of the perfume in the water-soluble polymer solution. The aqueous dispersion was cast upon a surface for drying and dried to form a film. The film was comminuted to form particles of the desired size for use in detergent compositions.

The drying step of this process is disadvantageous in that it is energy-expensive, time consuming, and adds nothing, other than dry, granular form, to the finished product.

It is an object of this invention to formulate a perfumed particle having a high level of perfume and having the capability of emitting perfume over a prolonged time period.

It is another object of this invention to provide a perfumed particle which gives an immediate and long lasting perfume effect and additionally releases perfume upon contact with water.

It is yet another object of this invention to provide such heavily perfumed particles which additionally contain other hydratable materials useful in detergent compositions.

A still further object of this invention is to provide a strongly perfumed particle containing a hydratable material useful in detergent compositions which can be made by a process which does not require a separate drying step.

In the present invention, an emulsion of perfume in an aqueous solution of water-soluble polymer is mixed with a powdered, anhydrous, hydratable material, useful in detergent compositions, in such a ratio that all of the water in the emulsion can be held as the hydrate of the material.

Disclosure of the invention

According to the present invention there is provided a process for producing heavily perfumed particles having both immediate and long lasting perfume emitting properties which comprises the steps of

a) forming an aqueous dispersion comprising by weight of the dispersion from 2% to 40% of a water-insoluble perfume, from 5% to 20% of a water-soluble polymer which will dissolve in water at a temperature of less than 100°C, from 40% to 90% water, and sufficient emulsifier to emulsify the perfume in the dispersion;

b) cooling the dispersion of step (a) to 0°C or less;

c) mixing the cooled dispersion of step (b) with an anhydrous, hydratable material which in its hydrated form is stable solid at ambient temperatures, in an amount sufficient to hold at least 85% of the water in the dispersion when fully hydrated; and

d) warming the mixture of step (c), while mixing, to ambient temperature.

In the process of this invention, two materials are prepared and mixed. One is a liquid, the

perfume/water/emulsifier emulsion. In this emulsion, the ingredients can have the same relative proportions as disclosed in U.S. Patent 4,209,417. In particular, the emulsion consists essentially of by weight of the emulsion, from 2% to 40%, preferably 4% to 25% perfume, from 5% to 20%, preferably 10% to 15% water soluble polymer, from 40% to 90%, preferably 60% to 85% water, and sufficient emulsifier, preferably 0.1% to 3%, to form a stable emulsion of the perfume in the water-soluble polymer solution.

In processing, the first step involves forming an emulsion of the perfume emulsifier, and polymer in water. This emulsion is then chilled to a temperature of 0°C or below. Although it is not essential, it is preferred that this temperature is below the freezing point of the dispersion. In such a preferred process, the frozen dispersion may then be comminuted prior to mixing with the hydratable material. Finally, the chilled emulsion is mixed with the hydratable material, in such a manner that substantially all of the free water is taken up to form the hydrate of the added material. In the preferred process the hydratable material is also cooled to 0°C or below prior to mixing with the aqueous dispersion. The resulting particles will consist essentially of from 0.5% to 40% water-insoluble perfume, from 1.5% to 20% of a water-soluble polymer which will dissolve in water at a temperature of less than 100°C, from 0.01% to 5% of an emulsifier, from 10% to 90% water, and from 1.5% to 75% by weight of a hydratable material, the amount of the hydratable material being sufficient to hold at least 85% of the water in said particles when fully hydrated. The granular material can be used as is, or can be further comminuted or agglomerated to achieve a desired particle size. The perfumed particles of this invention desirably have an ultimate particle size of from 40 to 1400 µm preferably 175 to 1000 µm. The perfume/emulsifier droplets contained within the particles have diameters of from 0.01 to 0.5 µm preferably 0.02 to 0.2 µm.

Hydratable materials

In general, any hydratable material which is either compatible with, or not deleterious to, detergent compositions can be used in the practice of this invention. Such materials include, without limitation, alkali metal halides such as sodium chloride, potassium chloride, sodium fluoride and potassium fluoride. Other hydratable materials include the variety of commonly known detergency builder materials, including polyvalent inorganic or organic salts or mixtures thereof. Examples include alkali metal carbonates, borates, phosphates, polyphosphates, bicarbonates, silicates, and sulfates. Specific examples of such salts include the sodium and potassium tetraborates, perborates, bicarbonates, tripolyphosphates, pyrophosphates, orthophosphates and hexametaphosphates.

Still other materials which may be used as the hydratable materials herein include the commonly used zeolite molecular sieves, also known as zeolites or aluminosilicates, having, in the anhydrous state, a hydration capacity of from 15 to 30 grams of water per gram of material. Other, related materials include expandable clays, including sodium and calcium montmorillonites, sodium saponites, and sodium hectorites. The term expandable as used to describe these smectite clays relates to the ability of the layered clay structure to be swollen, or expanded, on contact with water.

In general, the water-soluble polymer and hydratable material employed should be selected so that the hydratable material is more hydrophilic than the water-soluble polymer employed. However since one object of this invention is to produce a dry, free flowing, granular perfume product, excessively hygroscopic or frankly deliquescent materials, which cannot be hydrated to form a stable solid, are unacceptable.

It can also be appreciated that, within the realm of acceptable hydratable materials, those with higher hydration capacities can be used in smaller amounts per volume of emulsion to provide products with higher (wt%) perfume loads, and are therefore preferred.

Regardless of which hydratable material is selected, the amount of hydratable material to be used will be determined by the hydration capacity of the material in relation to the amount of water to be absorbed from the emulsion. The hydratable material must absorb at least 85% by weight of the water in the emulsion. It can be appreciated that absorption of amounts in excess of 85% of the water will improve the flowability of the resulting product, while adding an amount of hydratable material sufficient to absorb more than 100% of the water will not only insure a free-flowing, granular material, but confer caking resistance to bulk quantities of the perfume particles which result from the practice of this invention.

Thus, for example, sodium tripolyphosphate (STP) is hydratable to form the hexahydrate, i.e., 6 molecules of water are associated with each molecule of STP in the fully hydrated form. Therefore, each mole of STP added to the emulsion in the final processing step will absorb a maximum of 6 moles of water from the emulsion. The actual amount absorbed will be determined by the relative hydrophilicities of the hydratable material and the water-soluble polymer selected. In most cases, complete transfer of water from the polymer solution to the hydratable material will not be achieved, but a dry, granular, free flowing material is produced nonetheless.

The hydration capacity of the hydratable materials used can be determined from standard reference texts, by standard test methods for measuring water of hydration of hydrophilicity, or by simple preparation of test mixtures at various hydration levels and observation of their flowability and propensity for caking. The temperature of the hydratable material and the polymer/per-

fume emulsion must be reduced so that the rate of hydration is initially low enough that an intimate mixture of emulsion and hydratable material can be made. If this is not done, large gummy lumps form and it is very difficult to uniformly mix the two components. In general, temperatures of 0°C or less allow easy and complete mixing to be effected. The heat of hydration will then help to increase the temperature of the resulting mix to ambient levels.

Industrial applicability

Perfume

As used herein the term "perfume" is used to indicate any water-insoluble odoriferous material characterized by a vapor pressure below atmospheric pressure at ambient temperatures. The perfume material will most often be liquid at ambient temperatures. A wide variety of chemicals are known for perfume uses, including materials such as aldehydes, ketones and esters. More commonly, naturally occurring plant and animal oils and exudates comprising complex mixtures of various chemical components are known for use as perfumes. The perfumes herein can be relatively simple in their composition or can comprise highly sophisticated complex mixtures of natural and synthetic chemical components, all chosen to provide any desired odor.

Typical perfumes can comprise, for example, woody/earthy bases containing exotic materials such as sandalwood oil, civet and patchouli oil. The perfumes can be of a light floral fragrance, e.g. rose extract, violet extract, and lilac. The perfumes can also be formulated to provide desirable fruity odors, e.g. lime, lemon and orange. Any chemically compatible material which exudes a pleasant or otherwise desirable odor can be used in the perfumed particles herein.

Water-soluble Polymer

The matrix of the perfumed particles comprises a water-soluble polymer. As used herein, by "water-soluble polymer" is meant a polymer that will dissolve completely in water at a temperature less than 100°C. Any polymer can be used, provided it is water-soluble. Examples include water-soluble polyvinyl alcohols, polyethylene glycols, polyvinyl pyrrolidone, poly(ethylene oxide), cellulose derivatives, e.g. cellulose ethers such as methyl-, ethyl-, propyl- and butylcellulose ether, gelatin, pectin, starches, gum arabic, poly(acrylic acid) and its derivatives, polyacrylamides, styrene maleic anhydrides, poly(vinyl methyl ether maleic anhydrides), amorphous poly(vinyl methyl ether), poly(vinyl 2-methoxyethyl ethers), poly(vinyl sulfonic acid) or its sodium salt, poly(4-vinyl-phthalic acid), and low m.w. melamine formaldehyde resins. Any of the aforementioned polymers which are water-soluble are used herein. Preferred polymers include polyvinyl alcohols, polyethylene glycol, polyvinyl pyrrolidones, cellulose derivaties, poly(acrylic acid) and its derivatives, the poly(acrylamides) and poly(ethylene oxides), and poly(methyl vinyl ether/maleic anhydride) co-polymers. Most preferred for use herein are the poly(methyl vinyl ether maleic anhydride) co-polymers.

Emulsifier

The emulsifier is used to emulsify the perfume into an aqueous solution of the water-soluble polymer. The perfume, as used as the high levels of this invention, and the water-soluble polymer are not miscible. Processing to form homogeneous particles would not be possible since phase separation of the two components would occur. However, use of an emulsifier causes the perfume to form droplets which are uniformly distributed throughout the polymer solution. The distribution of perfume/emulsifier droplets allows for a heavy loading of perfume in the particles. The emulsifier is used in an amount sufficient to emulsify the perfume in the aqueous solution of the water-soluble polymer. This amount can vary widely depending on the particular perfume, water-soluble polymer and particular emulsifiers.

Emulsifiers are of a nonionic, anionic or cationic nature. Examples of satisfactory nonionic emulsifiers include fatty alcohols having 10 to 20 carbon atoms condensed with 2 to 20 moles of ethylene oxide and or propylene oxide; alkyl phenols with 6 to 12 carbon atoms in the alkyl chain, condensed with 2 to 20 moles of ethylene oxide, mono- and di-fatty acid esters of ethylene glycol, wherein the fatty acid moiety contains from 10 to 20 carbon atoms; fatty acid monoglycerides, wherein the fatty acid moiety contains from 10 to 20 carbon atoms; sorbitan esters; polyoxyethylene sorbitol; polyoxyethylene sorbitan; and hydropholic wax esters. Suitable anionic emulsifiers include the fatty acid soaps, e.g. sodium, potassium and triethanolamine soaps, wherein the fatty acid moiety contains from 10 to 20 carbon atoms. Other suitable anionic emulsifiers include the alkali metal, ammonium or substituted ammonium alkyl sulfates, alkyl arylsulfonates, and alkyl ethoxy ether sulfonates having 10 to 30 carbon atoms in the alkyl moiety. The alkyl ethoxy ether sulfonates contain from 1 to 50 ethylene oxide units. Satisfactory cationic emulsifiers are the common quaternary ammonium, morpholinium and pyridinium compounds.

Optional Components

Optional components such as dyes, antioxidants, etc. can be included as a part of the perfumed particles in minor amounts.

Detergent Compositions

The perfumed particles described above are especially useful when included as part of a detergent composition. The detergent composition contains a water-soluble organic surfactant and other detergency adjacent materials in addition to the perfumed particles. The level of

surfactant depends upon the type of detergency product, but generally ranges from 0.05% to 35%. The organic surfactants are selected from the group consisting of anionic surfactants, nonionic surfactants, ampholytic surfactants, zwitterionic surfactants, and mixtures thereof. U.S. Patent 3,664,961, issued May 23, 1972, describes suitable surfactants. The detergent composition can be a pre-soak detergent composition, main wash detergent composition, or household cleaner detergent composition and can be prepared in any suitable solid granular or powder form. Pre-soak and household cleaner detergent compositions contain a low level of surfactant, primarily for dispersion and composition throughout the aqueous bath. A level of surfactant from 0.05% to 2%, preferably 0.25% to 1% is used. A main wash detergent composition contains from 5% to 35%, preferably 8% to 20% surfactant.

The balance of the detergent composition consists essentially of detergency adjust materials. The detergency adjunct materials include builders, soil suspending agents, processing aids, brighteners, enzymes, and bleaches. The particular nature of the adjunct materials is dependent on the use of the product. A preferred detergent composition is a built detergent composition containing from 10% to 80%, preferably 25% to 75% detergency builder. Any of the known compounds possessing builder properties are useful herein. U.S. Patent 3,664,961 also describes satisfactory detergency builders. Many of these detergency builders, in particular, sodium tripolyphosphate, are particularly useful as the hydratable materials in the perfumed particles of this invention. Thus, the perfumed particles of this invention can supply a small proportion of the detergency builder in the total detergent composition.

To provide appropriate levels of fragrance, the detergent compositions herein preferably comprise from 0.1% to 1%, more preferably 0.2% to 0.5% of the perfumed particles. The balance of the composition comprises surfactant and detergency adjunct materials as described above.

Detergent compositions containing the above described perfume particles possess a pleasant smell immediately after manufacture, and also after storage for a substantial period. In addition, when the detergent composition is ultimately used in an aqueous solution an additional burst of perfume is released. That is, as the particles dissolve in water, additional perfume entrapped within the polymeric matrix is released. Thus a slight but noticeable perfume effect is obtained during storage while a stronger perfume effect is noticed upon use of the detergent composition. The following examples are illustrative of the invention, while not intending to be limitative thereof.

Example I

An aqueous solution is made containing 25.0 g polyvinyl alcohol (PVA) (M.W.=90,000; 98.8% hydrolyzed), 0.5 g ditallow dimethyl ammonium chloride DTDMAC) (as the emulsifier) and 225.0 gm water. The solution is put into a blender and agitated until the PVA is dissolved. The solution is then dearated and cooled. Mixing is resumed and 25.0 gm of Cedar Pine perfume is added to the vortex of the agitated solution. The mixture is agitated vigorously for 1 minute and again deaerated. The resulting emulsion is cooled to 0°C. At the same time, 776 g of anhydrous sodium tripolyphosphate (STP) are also cooled to 0°C. The emulsion and STP are mixed together at moderate shear and gradually allowed to warm up ambient temperature during mixing. The resulting product is a dry, free-flowing, granular material having a mild fragrance. The average analysis is

PVA	2.4%
DTDMAC	.05%
H_2O	21.4%
Perfume	2.4%
STP	74%

Example II

13 grams of Gantrez (RTM) AN 139 poly(methyl vinyl ether/maleic anydride) are dissolved in 67 grams H_2O. The solution is heated to hydrolyze the anhydride groups and 1.42 g of Atlas (RTM) G-1702 (polyoxyethylene sorbitol beeswax derivative) and 1.42 g Myrj (RTM) 45 (polyoxyethylene (8) stearate) are added as emulsifiers. To this mixture is added 13 grams "Bloom" perfume, and a stable emulsion is formed by high shear mixing. After emulsification, 1 gram of $Ca(OH)_2$ is added to crosslink the polymer chains for better perfume retention. The emulsion is deaerated and cooled to below 0°C using pulverized dry ice. The frozen particles of emulsion are mixed with 117 grams of similarly cooled anhydrous sodium sulfate at moderate shear and the entire batch is allowed to warm to ambient temperature during mixing. The resulting product is a dry, free-flowing, granular material.

Another "control" batch is made as above, but without the Gantrez AN 139. Samples of each batch are placed in open Petri dishes and exposed to ambient conditions. After 1 week, little fragrance is detectable over either sample. Upon addition of H_2O, "Bloom" fragrance is strongly emitted from the sample containing the Gantrez polymer, non is detectable over the control sample.

Example III

A household cleaning composition is formulated as follows:

Sodium sequicarbonate: 63.8%

Sodium C_{12} alkyl benzene sulfonate: 0.9%

Tall oil ethoxylated with an average of 8 moles ethylene oxide: 0.1%
Trisodium phosphate: 10.0%
Sodium tripolyphosphate: 22.1%
Perfumed particles of Example II: 2.1%
Misc. (coloring matter and water): 1.0%

The composition has a pleasant odor during storage and when made into an aqueous solution prior to use (15 gm product per liter water) emits a strong but pleasing perfume smell.

All percentages herein are by weight, unless otherwise indicated.

Claims

1. A process for producing heavily perfumed particles having both immediate and long lasting perfume emitting properties by

a) forming an aqueous dispersion comprising by weight of the dispersion from 2% to 40% of a water-insoluble perfume, from 5% to 20% of a water-soluble polymer which will dissolve in water at a temperature of less than 100°C, from 40% to 90% water, and sufficient emulsifier to emulsify the perfume in the dispersion; characterised in that it comprises the additional steps of

b) cooling the dispersion of step (a) to 0°C or less;

c) mixing the cooled dispersion of step (b) with an anhydrous, hydratable material which in its hydrated form is a stable solid at ambient temperatures, an an amount sufficient to hold at least 85% of the water in the dispersion when fully hydrated; and

d) warming the mixture of step (c), while mixing, to ambient temperature.

2. A process according to Claim 2, characterised in that the dispersion is cooled without freezing the dispersion.

3. A process according to Claim 1 characterised in that in step (b) the dispersion is cooled to below its freezing point, and in that the frozen dispersion is comminuted prior to mixing with the anhydrous hydratable material.

4. A process according to any one of Claims 1—3 characterised in that the anhydrous hydratable material is cooled to 0°C or less prior to mixing with the aqueous dispersion.

5. A process according to any one of Claims 1—4 wherein the water soluble polymer is selected from

poly(methyl vinyl ether/maleic anhydride),
poly(ethylene oxides),
poly(acrylamides),
poly(acrylic acid),
poly vinyl pyrrolidones,
polyethylene glycols,
cellulose derivatives,
polyvinyl alcohols, and
poly(acrylic acid) derivatives.

6. A process according to any one of Claims 1—5 wherein the hydratable material is selected from

alkali metal halides,
alkali metal carbonates,
alkali metal silicates,
alkali metal sulfates,
alkali metal borates,
alkali metal phosphates,
alkali metal polyphosphates,
alkali metal bicarbonates,
zeolite molecular sieves, and expandable smectite clays.

7. A process according to claim 6, wherein the hydratable material is sodium tripolyphosphate.

8. A process according to Claim 6 wherein the hydratable material is a zeolite molecular sieve material having, in the anhydrous state, a hydration capacity of from 15 to 30 grams of water per gram of material.

Patentansprüche

1. Ein Verfahren zur Herstellung von stark parfümierten Teilchen, die die Eigenschaft besitzen, sowohl sofort als auch lang anhaltend Parfum auszuströmen durch

a) Bilden einer wäßrigen Dispersion, enthaltend, bezogen auf das Gewicht der Dispersion,
2 bis 40% eines wasserunlöslichen Parfums,
5 bis 20% eines wasserlöslichen Polymers, das in Wasser bei einer Temperatur von weniger als 100°C sich löst, 40 bis 90% Wasser und ausreichend Emulgator, um das Parfum in der Dispersion zu emulgieren, dadurch gekennzeichnet, daß es die folgenden zusätzlichen Stufen enthält;

b) Kühlen der Dispersion von Stufe (a) auf 0°C oder weniger;

c) Mischen der gekühlten Dispersion von Stufe (b) mit einem wasserfreien hydratisierbaren Material, das in seiner hydratisierten Form ein bei Umgebungstemperatur stabiler Feststoff ist, in einer Menge, die ausreicht, um mindestens 85% des Wassers in der Dispersion, well sie voll hydratisiert ist, zu halten und

d) Erwärmen des Gemischs von Stufe (c) unter Mischen auf Umgebungstemperatur.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Dispersion ohne Gefrieren der Dispersion gekühlt wird.

3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß in Stufe (b) die Dispersion unterhalb ihres Gefrierpunkts gekühlt wird und daß die gefrorene Dispersion vorm Mischen mit dem wasserfreien hydratisierbaren Material zerkleinert wird.

4. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß das wasserfreie hyratisierbare Material auf 0°C oder weniger vor dem Mischen mit der wäßrigen Dispersion gekühlt wird.

5. Verfahren nach einem der Ansprüche 1 bis 4, worin das wasserlösliche Polymer ausgewählt ist aus

Polymethylvinylether/Maleinsäureanhydriden,
Polyethylenoxiden,
Polyacrylamiden,
Polyacrylsäure,
Polyvinylpyrrolidonen,

Polyethylenglykolen,
Cellulosederivaten,
Polyvinylalkoholen und
Polyacrylsäurederivaten.

6. Verfahren nach einem der Ansprüche 1 bis 5, worin das hydratisierbare Material ausgewählt ist aus
Alkalimetallhalogeniden,
Alkalimetallcarbonaten,
Alkalimetallsilikaten,
Alkalimetallsulfaten,
Alkalimetallboraten,
Alkalimetallphosphaten,
Alkalimetallpolyphosphaten,
Alkalimetallbicarbonaten,
Zeolithmolekularsieben und expandierbaren Smectittonen.

7. Verfahren nach Anspruch 6, worin das hydratisierbare Material Natriumtripolyphosphat ist.

8. Verfahren nach Anspruch 6, worin das hydratisierbare Material ein Zeolithmolekularsiebmaterial ist, das im wasserfreien Zustand eine Hydratationskapazität von 15 bis 30 g Wasser je g des Materials aufweist.

Revendications

1. Procédé de production de particules fortement parfumées ayant des propriétés d'émission de parfum à la fois immédiates et de longue durée en

a) formant une dispersion aqueuse comprenant, en poids de la dispersion, de 2% à 40% d'un parfum insoluble dans l'eau, de 5% à 20% d'un polymère hydrosoluble qui se dissoudra dans l'eau à une température inférieure à 100°C, de 40% à 90% d'eau et une quantité suffisante d'émulsifiant pour émulsionner le parfum dans la dispersion; caractérisé en ce qu'il comprend les étapes supplémentaires de

b) refroidissement de la dispersion de l'étape (a) jusqu'à 0°C ou moins;

c) mélange de la dispersion refroidie de l'étape (b) avec un matériau hydratable anhydre qui est, sous sa forme hydratée, un solide stable aux températures ambiantes, en quantité suffisante pour contenir au moins 85% de l'eau de la dispersion lorsqu'il est entièrement hydraté; et

d) réchauffement du mélange de l'étape (c), sous agitation, jusqu'à la température ambiante.

2. Procédé selon la revendication 1, caractérisé en ce que la dispersion est refroidie sans congeler la dispersion.

3. Procédé selon la revendication 1, caractérisé en ce que, dans l'étape (b), la dispersion est refroidie jusqu'à une température inférieure à son point de congélation et en ce que la dispersion gelée est broyée avant d'être mélangée avec le matériau hydratable anhydre.

4. Procédé selon l'une quelconque des revendications 1 à 3, caractérisé en ce que le matériau hydratable anhydre est refroidi jusqu'à 0°C ou moins avant d'être mélangé avec la dispersion aqueuse.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que le polymère hydrosoluble est choisi parmi le poly(méthylvinyléther/anhydride maléique), les poly(oxydes d'éthylène), les poly(acrylamides), le poly(acide acrylique), les polyvinylpyrrolidones, les polyéthylène-glycols, les dérivés de la cellulose, les poly(alcools vinyliques) et les dérivés du poly(acide acrylique).

6. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel le matériau hydratable est choisi parmi les halogénures de métaux alcalins, les carbonates de métaux alcalins, les silicates de métaux alcalins, les sulfates de métaux alcalins, les borates de métaux alcalins, les phosphates de métaux alcalins, les polyphosphates de métaux alcalins, les bicarbonates de métaux alcalins, les tamis moléculaires de type zéolite et les argiles de type smectite expansibles.

7. Procédé selon la revendication 6, dans lequel le matériau hydratable est le tripolyphosphate de sodium.

8. Procédé selon la revendication 6, dans lequel le matériau hydratable est un tamis moléculaire de type zéolite ayant, à l'état anhydre, une capacité d'hydratation de 15 à 30 grammes d'eau par gramme de matériau.