


Review

Nanoemulsions as a Form of Perfumery Products

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Abstract: Alcohol-based perfumes, e.g., eau de parfum, eau de toilette, eau de cologne or au fraiche, are the most common type of fragrance products available on the market. There are also alcohol-free fragrance products, mainly in the form of solid or oil perfume. From the consumers' point of view, such perfumery products are of interest; therefore, looking for new solutions is still interesting. Nanoemulsions are liquid, kinetically stable colloidal dispersions, consisting of an aqueous phase, an oil phase and a surfactant, with or without a co-surfactant. They are transparent, not greasy, easy to spray and spread. Additionally, they show capacity to protect fragrances from oxidation. The development of a water-based perfumes in the form of stable nanoemulsions containing fragrance compositions (in the range of 5–15%), stabilized by nonionic surfactants, allows to create safe products for a wider group of consumers, including children, adolescents and people with sensitive skin. In this article, an application of nanoemulsions as a potential form of perfumery products were described.

Keywords: fragrance; alcohol-free perfume; nanoemulsions

1. Introduction

Traditional perfume is a solution of fragrance compounds (e.g., fragrant essential oils, absolutes, aroma compounds, fixatives) dissolved in selected solvents. The most typical solvent used in perfumery products is ethanol or a mix of water and ethanol (aqueous alcoholic solution). Depending on the concentration of aromatic compounds in the solution the following varieties of fragrance can be distinguished: extrait de parfum, known as parfum (up to 40% fragrance composition, in 90–96% alcoholic solution), eau de parfum, EDP (10–15% fragrance composition in 80–90% aqueous alcoholic solution), eau de toilette, EDT (5–10% fragrance composition in 60–85% aqueous alcoholic solution), aftershave (5% fragrance composition in diluted alcoholic solution), eau de Cologne, commonly called cologne (3–8% aromatic compounds in 70–80% aqueous alcoholic solution) and au fraiche also known as body splash, body spray, body mist or body spritz (up to 3% fragrance composition diluted mainly with water) [1]. In these formulations, ethanol is used as a solvent of fragrances but additionally, it is an antimicrobial agent. A further advantage of alcohol is its fast-drying rate. On the other hand, it can dry out the skin and cause cutaneous intolerance or allergic contact dermatitis and as consequence, skin irritation and inflammation [2–4]. One of the alternatives to alcoholic solutions are oil-based perfumes. For their production, virtually any neutral oil can be combined with even the least intense fragrance. Oil as a carrier of aromatic substances cannot mask or distort the scent of the designed fragrance composition. The most commonly used oils are jojoba oil or fractionated coconut oil. The essential advantage of oil-based perfumes is that they do not dry out the skin like alcohol solutions. Quite the opposite, oil-based products moisturize the skin and improve the fragrance longevity. This type of product is commonly known as “skin scents”, and the body temperature allows

the scent to be released gradually. It is estimated that the scent of oil-based perfumes lasts for 6–15 h, while those based on alcohol last for about 3 h. The fragrance concentration in oil products is quite high, usually around 20% [5]. One of the disadvantages of these products is their lack of fresh, lighter notes called “head notes”, making the overall smell heavy. Additionally, dispersing these products on the clothes and in the air causes greasy stains and can make surfaces slippery and sticky.

Another type of perfume in which fatty substances play an essential role as aroma carriers are so called “solid perfumes”. Solid perfumes occur in the form of emulsions, gels and pomades. Various types of waxes, which are melted and mixed with oils to obtain a homogeneous fat base, are responsible for its consistency. Most often, solid perfumes are sold in the form of a stick or mass pressed into a thimble and, in opposite to classical sprayable perfumes, they are applied to the skin by rubbing. They tend to deliver richer, deeper notes which are released slowly. Solid perfumes are most often used as a complementary scent to classic fragrance products. The main disadvantage of this form of perfumes is the method of their application. Solid perfumes are applied directly to the skin, causing a risk of greasy clothes, and cannot be sprayed on skin.

It is also worth mentioning the very popular ‘home scents’ with endless diffusing products to choose from, ranging from oil burners, which have been used for health purposes (aromatherapy) for centuries, to a vast spectrum of atomizers, diffusers and aerosols distributing aroma in the form of a mist. Among others, scents can also be captured and released in forms such as potpourri, wax melts, gel melts, candles, cotton papers and permeable plate diffusers. Additionally, the growing use of nanocapsules (encapsulated fragrances) in ‘home scents’ has been noticed recently. Encapsulation of fragrances in a mixture of lipid, phospholipid and polymer matrix allows controlled release of the scents. The shell of the capsule can perform as a diffusion barrier and enhance the retention of the fragrance [6,7]. A practical use of the encapsulation is to protect fragrance chemicals from a negative influence of environmental factors [8]. Nanoencapsulation also improves the stability of the fragrance composition, protecting the aroma components from oxidation caused by light and/or oxygen exposure [9–12]. Nanoencapsulated fragrances are used for the production of food, fragrance samples for perfuming textiles, footwear, jewelry and other materials intended for marketing purposes e.g., beauty brochures, calendars, and leaflets with scented pages [9]. Recently, devices enabling dosing of household chemical products have appeared on the market. These are mixtures of nanoparticles containing fragrances and other chemicals in water and can be used to sterilize the air, mask unpleasant odors and release pleasant aromas. These devices are mainly used in enclosed spaces like houses, hospitals, or public buildings.

2. Alcohol-Free Fragrance Products

Alcohol-based perfume is the most common type of fragrance products available on the market. However, due to the potential irritating properties of ethanol [4], its flammability and ethical restrictions associated with its use in the Middle East, there is a growing market of alcohol-free perfume products, mainly in the form of solid or liquid oils. Products in these forms have a number of drawbacks discussed above. Therefore, new solutions are still being sought with regard to the physical and chemical form of perfumery goods. To expand the market to new consumer groups like children, adolescents, and allergy sufferers, the elimination of the most commonly used solvent—ethanol—becomes a necessity but it also remains a great challenge.

There are some examples of alcohol-free, water-based products described in the literature. However, replacing ethyl alcohol completely with water is a challenging task for scientists. The essences used in perfumery products are inherently mixtures of hydrophobic (water-insoluble) compounds. Therefore, obtaining stable solutions based on water is very difficult. One of the solutions facilitating the introduction of hydrophobic fragrances into water is the use of solubilizers (substances that increase solubility), e.g., glycols, glycerin or surfactants (compounds that lower the surface tension between two liquids), in order to obtain stable dispersions of hydrophobic fragrances in water, in the form of emulsions [12–16], microemulsions [17–19], micelles [20] and/or liposomes [21].

3. Fragrance Products in the Form of Microemulsions

US patent US5468725A “Alcohol-free perfume” [18] describes a transparent microemulsion (oil in water-O/W), containing a non-alcoholic, hydrophobic fragrance composition. The formulation of alcohol-free perfume is stabilized with a mixture of non-ionic (from the group of ethoxylated fatty acid sorbitan esters, ethoxylated hydrogenated castor oil, fatty alcohol polyglycol ethers, ethylene oxide-propylene oxide block copolymers or fatty acid sorbitan esters) and ionic surfactants (Sodium Lauryl Sulphate, Quaternary salts).

US Patent US8343521B “Ethanol-free aqueous perfume composition” [19] describes a transparent microemulsion, consisting of at least one fragrance, water and optionally one or more surfactants and suggests using 1,2-hexanediol as a solvent. 1,2-hexanediol as an amphiphilic substance with good solubilization properties allows reduction of the surfactant content. For example, a weight composition of ethanol-free perfume described in the patent application is as follow: 12.36% of 1,2-hexanediol, 1% of Brij 30, 1% of stearic acid, 6% of fragrance composition, 79.51% of water and 0.14% of NaOH.

Patent application CN103637942(A) “Alcohol-free transparent perfume composition” [22] describes a formulation with a high concentration of surfactants (more than 10%) and the presence of polyols such as 1,3-butanediol and glycerol as the solvents. The formulation comprises the following materials in parts by weight: 5–20 parts of essential oil, 8–12 parts of polyoxyethylene surfactants, 5–8 parts of phosphate surfactants, 4–7 parts of polyglycerol ester surfactants, 0.5–1.2 parts of glycerol and 15–20 parts of water.

Patent application US5736505(A) “Non-alcoholic perfume or cologne” [17] discloses the composition of a perfume containing as solubilizer-glycereth-7-triacetate. The formulation contains as main ingredients: fragrance compound (preferably 2–20 wt.%), glycereth-7-triacetate (preferably 3–12 wt.%), non-ionic surfactant such the PEG-60 hydrogenated castor oil or isoceteth-20 (preferably less than 6 wt.%), water (preferably 50–80 wt.%) and less than 1% *w/w* of preservatives (alkyl esters of *p*-hydroxybenzoic acid).

The disadvantages of microemulsions are the high surfactants concentration in the composition (up to 40%). It could be a reason of high products viscosity or lack of its transparency. Moreover, fragrances formulated in such compositions can react with surfactant and may undergo degradation processes, which adversely affects the stability and organoleptic properties of the product. In addition, it should be noted that the cationic surfactants described in some of the above patents are known for their irritating properties and therefore should be avoided in ‘leave-on products’ such as perfume goods. Additionally, these products may require the use of special packaging that is resistant to this type of formulation.

4. Perfumes in the Form of Nanoemulsions

An interesting solution for water-based perfume products are nanoemulsions. Recently, nanoemulsions have become more widely used for pharmaceutical or cosmetic applications. They are liquid, kinetically stable colloidal dispersions, consisting of an aqueous phase, an oil phase and a surfactant, with or without a co-surfactant, characterized by a high degree of dispersion. The droplet size of the dispersed phase is in the range from 20 to 500 nm. From an application point of view, nanoemulsions present several advantages. Among others, they are easy to spray and spread; moreover, they show a capacity to solubilize hydrophobic substances and protect them, for example from oxidative degradation [23–29].

Nanoemulsions can be obtained by high- and low-energy emulsification methods. The high-energy methods consist in using mechanical energy, usually from mechanical devices such as high-revolution mixers, high-pressure homogenizers and ultrasound generators [30,31]. Low-energy methods are based on the chemical energy stored in the emulsion ingredients (mainly in surfactants) [32]. The presence of emulsifiers results in reduced interphase tension, and thus in reduction of the energy required for fragmentation of the particles [33].

4.1. Characterization of Nanoemulsions

Nanoemulsions are characterized by a high kinetic stability. The small size of the nanoemulsion particles reduces the gravity force, which is overcome by Brownian motion, preventing the process of sedimentation and creaming. However, from a thermodynamic point of view, nanoemulsions are non-equilibrium systems, which may lead to their destabilization processes (such as flocculation, coalescence and/or Ostwald ripening). In contrast to microemulsions, obtaining nanoemulsions requires the use of smaller amounts of surfactant (about 5–10%), which makes it possible to obtain systems that are safer for the human body [34]. Their transparent appearance, liquidity and low viscosity make nanoemulsions an attractive form for many industrial applications, including cosmetics. Due to their unique properties, they can act as carriers for lipophilic active substances in medicines [26–28,35], in beauty products [26–29,36] and for fragrance compounds in cosmetics [37–40]. In addition, nanoemulsions containing various essential oils, e.g., lemon [41], cinnamon [42], basil [43], orange peel [44], eucalyptus [45], lemongrass [46] and peppermint [47], have been examined as potential carriers of pesticides in agricultural production [48,49], as antimicrobial agents [50–52] and as a modern form of preservative in the food industry [53]. Direct use of essential oils is limited due to their hydrophobic nature and high sensitivity to external factors of volatile substances included in the composition (e.g., phenols, ketones, aldehydes, alcohols, esters, lactones, and terpenes). Application of essential oils in the form of nanoemulsions increases their bioavailability, biocompatibility and protects them against the negative effects of oxidizing factors.

4.2. Nanoemulsions as Fragrance Carriers

As mentioned above, nanoemulsions may also act as a matrix for aroma compounds [54]. The scientific research described in the literature is mainly limited to isolated, individual terpenes e.g., D-limonene. Li and Chiang [45] obtained stable nanoemulsions ($d < 100$ nm), containing D-limonene, with the use of ultrasonic homogenisation. The concentration of D-limonene in the formulation was 10% *w/w*. They were stabilized by the mixture of emulsifiers contained sorbitan trioleate and oleyl alcohol ethoxylated. Application of nanoemulsions as a carrier of D-limonene allowed an increase of its bioavailability and chemical stability, including protection against oxidation.

Most research teams that produced nanoemulsions containing D-limonene in their composition used high-energy methods, such as ultrasonification [37,39] and high-pressure homogenization [30]. However, Li and co-workers [40] obtained nanoemulsions as D-limonene carriers by phase inversion emulsification (PIC), which belongs to the low-energy methods. Polysorbate 80 was used as a stabilizer for these nanoemulsions. It was found that the introduction of olive oil into the system resulted in improved kinetic stability of the obtained formulations (increased resistance to Ostwald ripening, the main destabilizing process of nanoemulsions).

Friberg and co-workers [14] studied the stability of perfume emulsions containing water, a nonionic surfactant from the group of ethoxylated fatty alcohols (Laureth-4) and phenylethyl alcohol (a component of fragrance compositions with a floral aroma). Studies have shown that the stability of the obtained nanoemulsion is affected by the ratio of phenylethanol to the surfactant. The same team [46] also studied the effects of various aroma compounds (e.g., limonene, a mixture of limonene and phenylethyl alcohol or benzaldehyde) on the stability of the described emulsions. During the emulsification process, liquid crystalline systems of high viscosity were created as a transient stage in the formation of nanoemulsions, which was a technological difficulty. An attempt to eliminate the problem related to the presence of liquid crystals was made by Dumanois and Gueyne [55] using isoprene glycol as a cosolvent in a non-alcoholic fragrance composition. The composition consists of 1–6% *w/w* of essential oils, 5–20% *w/w* of isoprene glycol, 0.5–3.5% *w/w* of PEG-40 Hydrogenated Castor Oil, 0.5–3.5% *w/w* of ethoxylated fatty alcohol, 0.1–0.2% *w/w* of parabens and up to 100% water.

In addition, the patent literature describes solutions for non-alcoholic perfumes based on nanoemulsions. In these perfume formulations, additional solubilizers such as polyhydric alcohols, paraffinic hydrocarbons, and cationic or anionic surfactants are used and all these can have the potential

to irritate the skin. Patent application EP2127632A1 “Perfume composition with reduced alcohol content” [56] presents a perfume composition containing glycerol, additional hydrophobic solvents such as isopropyl myristate, isohexadecane or isoeicosan, and in addition to nonionic emulsifiers (Polyglyceryl-4-Laurate), it includes anionic surfactants e.g., disodium lauryl sulfosuccinate.

The patent applications P.426105 [57] and P.426104 [58] describe a method of obtaining alcohol-free perfumes in the form of nanoemulsions (oil in water-O/W), containing 5 to 15% *w/w* of fragrance composition, without additional solubilizers and co-surfactants. Fragrance-loaded nanoemulsions are stabilized using nonionic surfactants (ethoxylated castor oil or esters of polyglycerol-4 and sebacic and lauryl acids), in the range of 5 to 10% *w/w*. Additionally, up to 1% wt. of natural preservatives (sodium anisate and sodium levulinate) are used to obtain microbiologically stable products. The water-based perfume was prepared by used two different methods—a low energy (PIC, Phase Inversion Composition) and a high energy method (ultrasonication). The stable nanoemulsion systems, with a transparent appearance, and the droplets size in the range of 20 to 100 nm were obtained. The systems stability was assessed by dynamic light scattering (DLS) measurements. The size of oil droplets was monitoring during the 16-month of storage, at the ambient temperature. No changes were observed.

5. Conclusions

Nowadays alcohol-based perfumes are a standard solution available on the fragrance market. However, ethyl alcohol is a solvent that has some irritating potential. Perfumery products in the form of perfumed solids or oil perfumes have a number of drawbacks from a consumer’s point of view.

Alcohol-free perfumes, in the form of stable oil-in-water (O/W) nanoemulsions, with regard to the physical and chemical form of perfumery goods, is an interesting solution. Nono-perfumes containing fragrance compositions without the addition of ethanol and co-surfactants, allows the creation of safe products, with good user properties, dedicated for a wider group of consumers, including children, adolescents and people with sensitive skin.

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