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Flavors & Fragrance Delivery Systems

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Abstract: This article focusses on the art of encapsulating flavors and fragrances into carrier materials, emphasizing the scientific challenges imposed by the particular nature of these essentially volatile encapsulants. The expected benefits of F&F delivery systems are discussed in terms of protection against ageing, enhanced impact during use and sustained release from substrates. Special attention is given to the preparation and use of hydrophilic delivery systems based on carbohydrates. The discussion is illustrated by selected applications of current delivery systems in the fields of flavor formulations and perfumed laundry care detergent powders.

Keywords: Controlled release · Delivery system · Encapsulation · Flavors · Fragrances · Protection

1. Introduction

Fragrances and flavors are subtle and complex mixtures of molecules having various physicochemical properties such as volatility, water solubility, and chemical reactivity. A principal feature of these mixtures is their evanescence in open systems. F&F formulations have indeed a limited lifetime because their constituents evaporate, are degraded during storage or are lost by dissolution in water during use. On the other hand, because their constituents neither evaporate at the same rate nor dissolve at the same extent, the hedonic attributes of scents and tastes change as a function of time and environmental conditions. One of the striking consequence of this situation is the rapid loss of very volatile constituents – also called top notes – during storage and use, which often results in a loss of freshness or impact. As a consequence, a large part of the volatile composition is lost without being perceived by the consumer. Such a loss is, of course, undesirable.

The flavor industry has first addressed the issue of performance loss of aged flavors by developing specific delivery systems like microcapsules and carriers. The function of these delivery systems is to protect the volatile components against oxidation and evaporation during storage and to enhance flavor release during food processing and eating. Today, flavor encapsulation is a well-established area of food technology, as apparent from the large body of literature available on this topic [1]. Generally, flavors are incorporated into hydrophilic or, less often, into hydrophobic matrix materials, emulsion droplets and coacervates. Food regulations restrict the palette of materials allowed for flavor encapsulation to native biopolymers and their thermally, acid, alkaline or enzymatically modified analogues, as well as to a few surfactants, waxes and fats. The encapsulated flavors are usually commercialized in the form of powders and granulated materials obtained by extrusion and grinding, spray-drying, spray-chilling, spray-coating and coacervation. Some of these classical techniques are discussed in the present article.

Fragrances are very similar to flavors in their nature and properties and, in practice, similar stability and performance issues are often encountered with perfumes. Additionally, fragrances need to be transferred to various substrates and need to be perceived over a prolonged period of time once deposited on these substrates. Therefore, tremendous effort is made in order to increase the efficiency of F&F formulations and to make them perceivable where and when they are needed. In perfumery, a classical way of solving this issue is to design molecules with a low perception threshold, a low vapor pressure, and high capability to deposit on substrate [2][3]. These molecules are referred to as being *substantive*. Although this approach has considerably improved the performance of dry-down notes, *i.e.* of notes that remain on the substrate at the end of the perfume life, it fails in extending the longevity of top notes. The use of chemical precursors, or pro-fragrances, can extend the perception of some individual volatile molecules [4], but not of accords. Consequently perfumery ends up with a restricted choice of long-lasting scents available on substrates.

The development of specific delivery systems that protect the volatile components during storage, assist their delivery in the form of *whole* accords to substrates, and control their release during and after use constitutes an alternative strategy to this chemical approach. This strategy and its scientific challenges will be discussed and exemplified in the present paper.

2. Scientific Challenges

The general scheme of our F&F delivery strategy is shown in Fig. 1a. The scheme is divided into five specific technical tasks: (i) process retention, (ii) protection, (iii) deposition (fragrances only),

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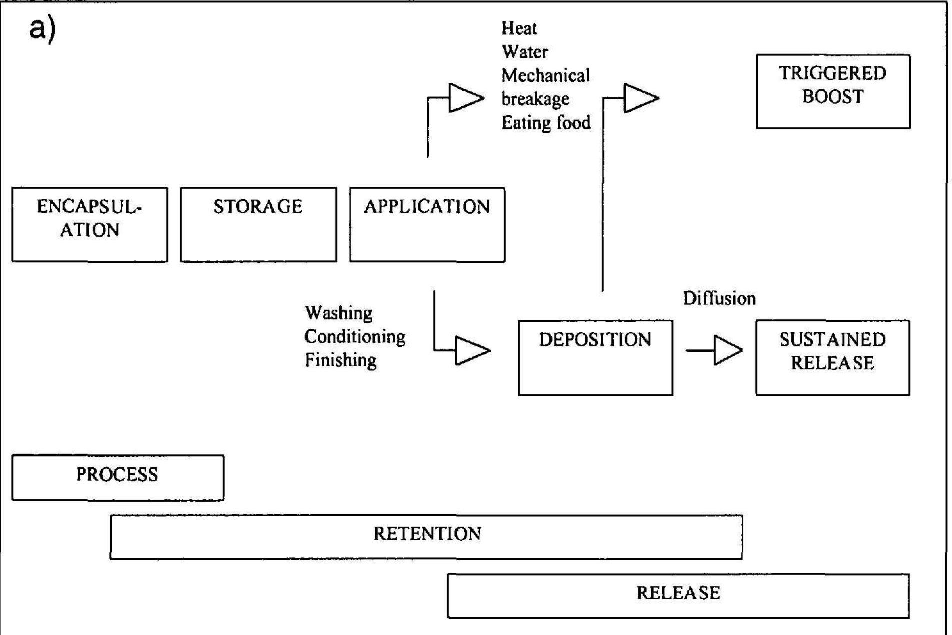
(iv) triggered release, and (v) sustained release of volatiles. Solving each of these tasks requires a thorough understanding of the interactions between the volatile ingredients and the carrier or encapsulating material on the one hand, and of the behavior of this material under various application conditions on the other.

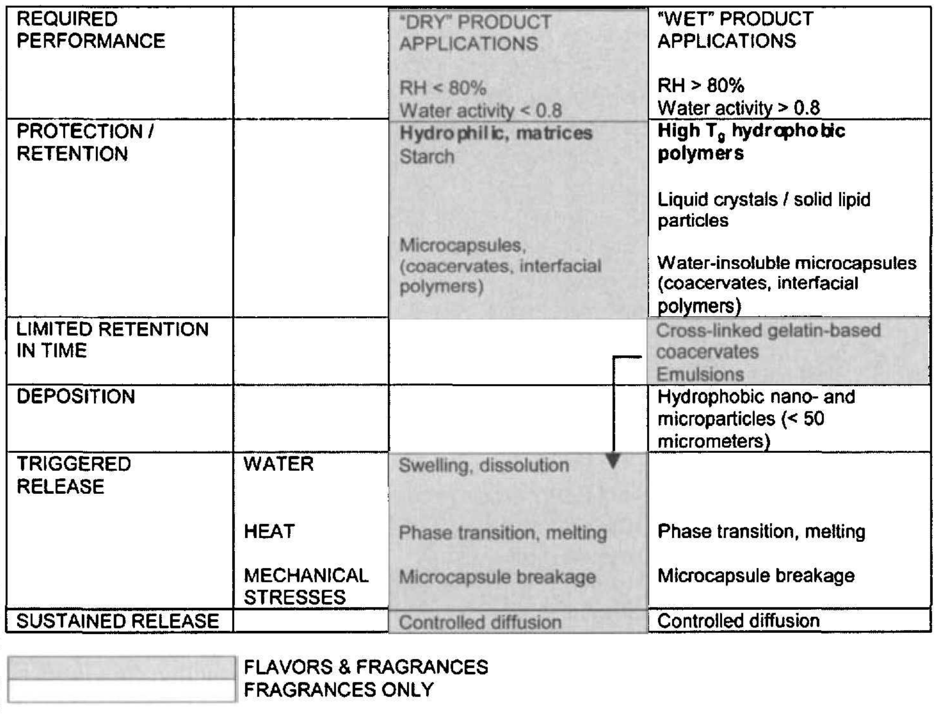
The selection of a particular delivery system depends on the nature of the product in which this delivery system is to be used, as well as on the kind of release mechanism required. The way this selection is made is summarized in a matrix representation (Fig. 1b).

The first selection criterion is whether the delivery system will be used in food applications or not. Fragrance applications offer a broader choice of potential carriers, as long as the olfactory properties and cost level of the latter meet consumer acceptance.

The second selection criterion is the presence or absence of water in the product and the level of humidity during storage. At low water activities and relative humidities lower than 80%, glassy or crystalline *hydrophilic matrices* can be used. This case is referred as 'dry product applications' in Fig. 1b. Water has the adverse effect of plasticizing, swelling and ultimately dissolving these matrices, which results in poor retention and early release of encapsulated flavors and fragrances. In liquid aqueous products and high relative humidity ('wet product applications'), *hydrophobic carriers* must be used. However, these materials are intrinsically permeable to both hydrophobic substances and oxygen and their ability to retain and protect the volatile actives during storage is limited. The reduction of volatile losses through such hydrophobic matrices and the development of hydrophilic delivery systems that survive high humidity storage conditions, such as those encountered in tropical countries, are two major tasks of F&F delivery research.



b)



REQUIRED PERFORMANCE		"DRY" PRODUCT APPLICATIONS RH < 80% Water activity < 0.8	"WET" PRODUCT APPLICATIONS RH > 80% Water activity > 0.8
PROTECTION / RETENTION		**Hydrophilic, matrices** Starch Microcapsules, (coacervates, interfacial polymers)	**High T_g hydrophobic polymers** Liquid crystals / solid lipid particles Water-insoluble microcapsules (coacervates, interfacial polymers)
LIMITED RETENTION IN TIME			Cross-linked gelatin-based coacervates Emulsions
DEPOSITION			Hydrophobic nano- and microparticles (< 50 micrometers)
TRIGGERED RELEASE	WATER	Swelling, dissolution	
	HEAT	Phase transition, melting	Phase transition, melting
	MECHANICAL STRESSES	Microcapsule breakage	Microcapsule breakage
SUSTAINED RELEASE		Controlled diffusion	Controlled diffusion

▨ FLAVORS & FRAGRANCES
☐ FRAGRANCES ONLY

Fig. 1. a) F&F delivery strategy. b) Selection of delivery systems.

The third selection criterion is based on the kind of *release mechanism* and release kinetics required during application. A burst-like release is generally generated by the action of 'triggers' such as heat, mechanical stresses or water. In contrast, controlling the transport kinetics in the encapsulating material produces sustained release.

The constraints imposed by the *hedonic character* of flavors and fragrances create an additional challenge, which has no equivalent in other areas of microencapsulation such as pharmaceutical or industrial applications. In order to meet consumer acceptance, the selected delivery system must be olfactory neutral and tasteless. On the other hand, the selectivity of both retention and permeability must be as low as possible in order to prevent composition distortion during product storage and use.

Finally, fragrance delivery systems are often considered as commodity products. This imposes the use of cheap barrier material and low-cost encapsulation technologies.

3. F&F Microencapsulation Processes

The term 'microencapsulation' refers nowadays to any technologies where an active substance is entrapped into a carrier material. We follow this extended definition and will not comment on the various types of delivery systems and on their classification, which can be found elsewhere [5]. The classification proposed by many authors is based on the morphology of the 'microcapsules'. Objects consisting of a reservoir of active substance(s) surrounded with a barrier material are often referred to as 'true' microcapsules, while particles containing the active substance(s) molecularly distributed throughout a matrix material are called 'matrix particles'. We consider that these two structural patterns are actually limiting cases and that, in real life, both structures can coexist in the same entity.

3.1. *Encapsulation in Hydrophilic Matrices*

Encapsulation in glassy or crystalline matrices is the most classical way to prepare powders having a high volatile retention and extended shelf life. Starches, modified starches and sugars are known for their exceptional encapsulating capability for hydrophobic actives [6]. This behavior is related to the low solubility of the volatiles in the matrix and to the low free volume available for molecular transport. Furthermore, hydrophilic materials have a low permeability with respect to oxygen [7]. Typical enrobment procedures involve spray-drying, spray-coating, extrusion and matrix encapsulation [6][8][9].

3.2. *Spray Drying*

A widely used procedure to encapsulate flavors and fragrances involves emulsifying the actives with an aqueous suspension of matrix material and drying this emulsion by spray-drying [10]. The emulsion is dispersed by atomization into a chamber with circulating hot air, where rapid evaporation occurs. The dry material is recovered as a fine powder at the bottom of the chamber. Due to the very short residence time of the powder particles in the air stream and to the endothermic nature of evaporation, the temperature of the powder remains below 100 °C at any time of the drying process. Hence, spray-drying can be used even for heat-sensitive ingredients. The ultimate properties of the powder encapsulate depend on many process parameters as shown on Fig. 2. The key factors allowing high encapsulation quality are (i) the quality of the emulsion, (ii) the nature of the barrier materials and (iii) the drying conditions. The overall spray-drying scheme is illustrated in Fig. 3.

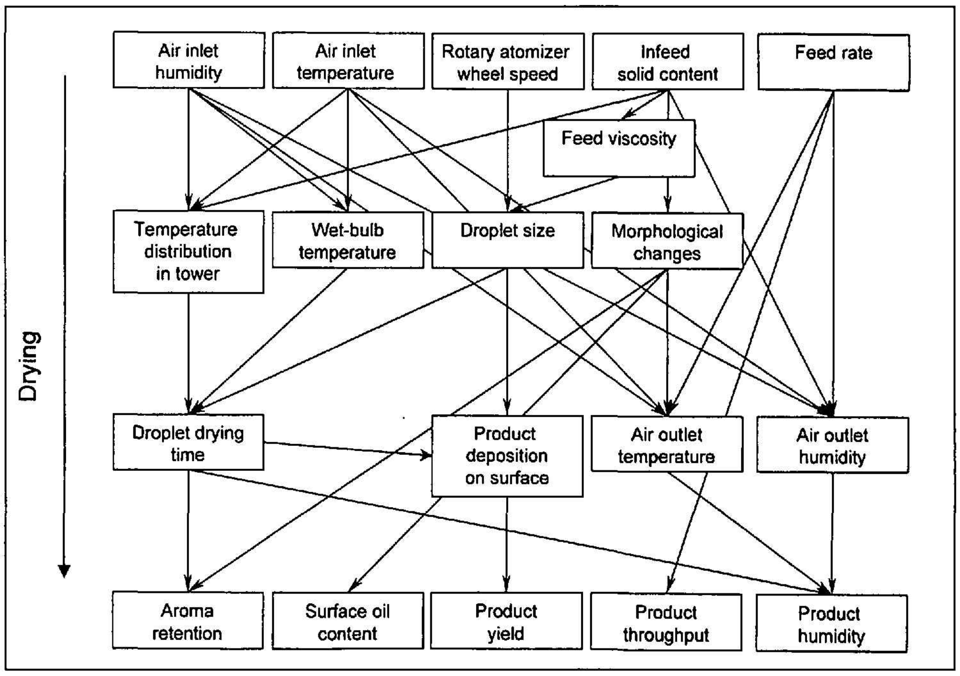


Fig. 2. Overview of the process parameters involved in spray drying.

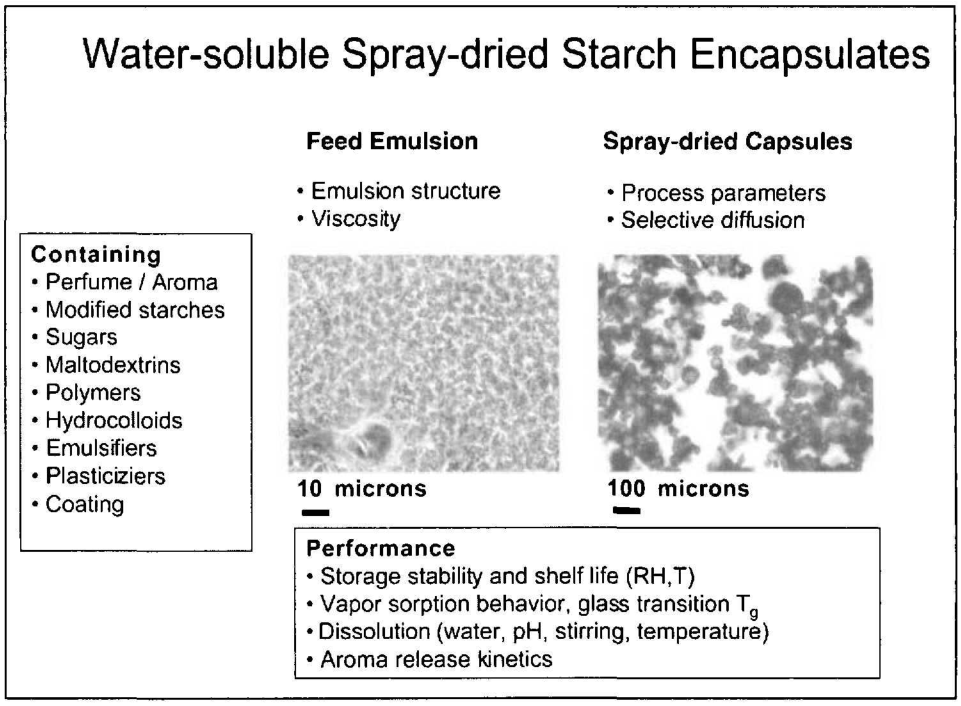


Fig. 3. Spray-drying formulation and process

High quality emulsions are characterized by stable monodisperse oil droplets, ideally having a size below one micrometer. Electron microscopy studies show that, under suitable drying conditions, stable emulsions retain their morphology on drying, yielding oil inclusions dispersed in the carbohydrate matrix and having the same size as the original oil droplets in the emulsion. Such dry emulsions can contain more than 50% (w/w) of encapsulated oil and yield powders with a level of surface oil not exceeding 1%. The term surface oil refers to the non-encapsulated fragrance or flavor oils present on the powder surface. The amount of surface oil can be taken as a measure of the quality of the encapsulation process.

The selection of matrix materials is often a matter of know how and expertise. The ability of carbohydrates to bind flavors and fragrances, complemented by their diversity, low cost, and widespread use in foods, make them the preferred choice for encapsulation [11]. Optimal retention and chemical stability during storage are achieved by determining the right balance between oil retention, and oxygen and water permeabilities. In hydrophilic materials, the free volume controls the diffusion of apolar species and oxygen, while the diffusion of water involves complex mechanisms [12]. The principal difficulty resides in the fact that hydration and plasticization occur in the presence of water, which in turn decreases the barrier properties of the matrix with respect to the other species. A short glance through the patent literature [13] and our own expertise show that mixtures of hydrolyzed starches, like maltodextrins (*e.g.* low molecular weight polysaccharides having from five to ten glucose units and high maltose content), starch octenyl succinate and small molecular weight sugars impart a good volatile retention and a high resistance to humidity even at high oil levels (*e.g.* 50% (w/w)). In such modified starch encapsulates, starch octenyl succinate, sucrose esters, glycerol esters and proteins act normally as emulsifiers [14]. The emulsifier helps to finely disperse the hydrophobic composition in the carrier solution and is crucial for the retention and stability during storage [10]. Maltodextrin and sugars act as matrix fillers that impart additional barrier properties and mechanical stability.

The advantages of spray-drying include diversity and availability of machinery, good keeping quality of microcapsules, and excellent redispersability of particles in aqueous media. However, the maximal particle size of spray-dried powders is limited to about 200 micrometers when a nozzle is used and 100 micrometers when a rotary disc atomizer is used. This can lead to very dusty and less freely flowing materials. Using multistage dryers, where recycling and secondary agglomeration of the fine particle fraction occur, particles up to 400 micrometers can be produced with a narrower particle size distribution but a lower mechanical stability. Some other disadvantages of spray drying are loss of very low boiling actives [15] and oxidation of some flavor compounds during processing [16].

The role of the various formulation ingredients and process conditions with respect to the quality of spray-dried fragrances and flavors has been extensively reviewed by Reineccius [10]. These discussions are based, however, on practical observations and remain qualitative.

Our understanding of the mechanisms involved in spray drying is based on the works of Thijssen [17], Furuta *et al.* [18] and, more recently, Coumans *et al.* [19]. According to these authors, the drying process is ideally divided into three subsequent regimes. The initial regime is characterized by a high water content and a low drying rate. Water elimination at this stage is accompanied by an intensive loss of volatile and water-soluble substances by distillation. Constant drying rate and linearly decreasing water content characterize the intermediate regime. The extent of volatile loss at this stage depends on the water concentration at the end of the first stage and on the drying rate. The third regime starts at very low water concentrations and is characterized by an exponentially decreasing drying rate and a slowly decreasing moisture level. Volatile loss at this stage is negligible, unless structural imperfections like micropores are present. Minimal encapsulant loss is obtained by minimizing very rapidly the water content of the drying particle below a critical value and by working at constant and homogeneous drying rate to avoid water entrapment and subsequent micropore formation. This regime is reached by tailoring the temperature and airflow profiles in the drying chamber and the way the droplets are formed. Under these conditions, the loss of the more polar or water-soluble volatile actives by distillation is minimized. Concomitantly the diffusion coefficient of all actives is considerably reduced and typically a process retention higher than 90% is observed, even for critical substances.

3.3. *Granulation and Spray-Coating*

For many applications, larger particles than those produced by spray-drying may be preferred. This is achieved by secondary agglomeration of pre-existing spray-dried particles or by spraying the feed emulsion onto a particular core material. In both cases, a layer is produced at the surface of the particles, which remains tacky during the agglomeration regime and becomes glassy after water elimination and cooling. The agglomeration is carried out in common fluidized bed dryers [6].

A typical feature of agglomeration processes is the persistence of high moisture levels during the agglomeration regime. This can lead to important volatile losses by water distillation. As apparent from Fig. 4, which shows the mean retention of a collection of key fragrance molecules calculated from data obtained from various perfumes, there is a rough correlation between retention, vapor pressure and water solubility. In order to define an optimal operation window of the agglomeration process, process variables such as inlet temperature, air velocity, feed spray and atomization pressure, can be controlled. These process parameters influence response variables such as bed temperature and humidity and, therefore, are crucial with respect to volatile retention during agglomeration [20][21]. Recent experimental investigations of liquid binder-particle interactions [22] and computer simulation [23] have considerably improved our understanding of wet granulation.

Applying an uniform layer of any material onto fluidized particles is commonly referred to as spray-coating [20]. The protective layer can be a hydrophobic material, which offers an additional barrier against water. Hydrophobically modified silica [24] and fats [25] are commonly used to improve the shelf life of encapsulates.

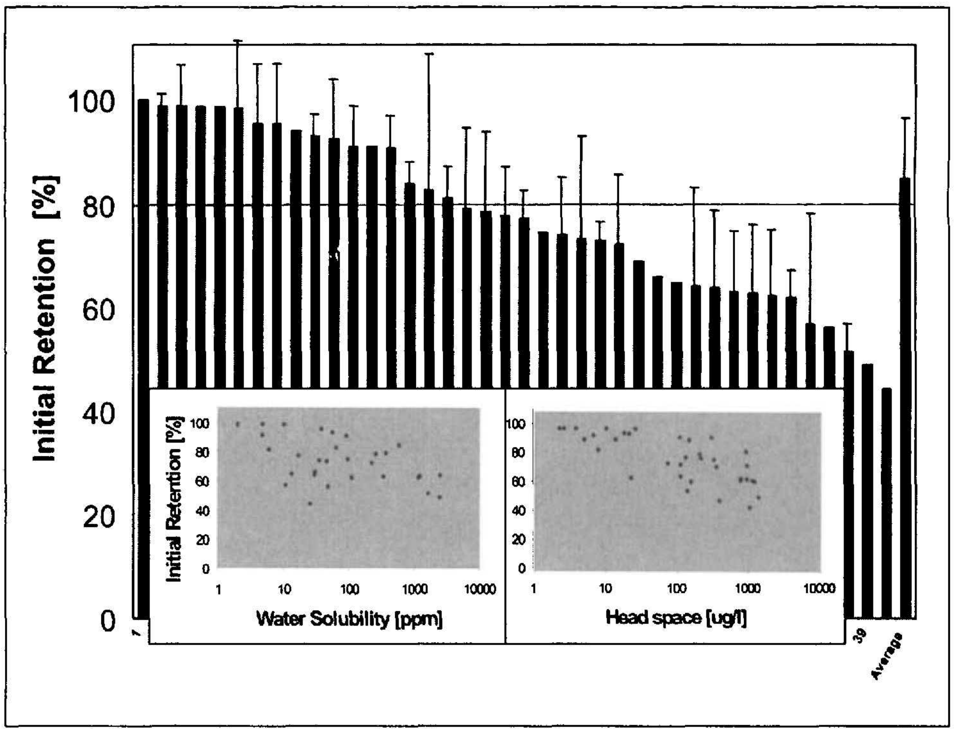


Fig. 4. Retention of fragrance components during granulation (see text).

Givaudan's granulation technology [25] is a typical example of a mixed process combining both granulation and spray-coating. By carefully selecting the core particles, the feed emulsion and the coating material, a granulated composite material can be obtained which possesses a high functionality, like controlled release of fragrances in wash liquors.

3.4. *Encapsulation by Coacervation and Interfacial Polymerization*

Surrounding oil droplets with a polymer layer is another classical microencapsulation process, which leads to 'true' capsules, *i.e.* capsules having a reservoir core and a more or less permeable wall. Coacervation involves typically the following steps: (i) preparing an oil-in-water emulsion by dispersing the oil into an aqueous solution of surface active hydrocolloid (*e.g.* gelatin), (ii) precipitating the hydrocolloid onto the oil-water interface by decreasing the solubility of the hydrocolloid (*e.g.* by adding a non-solvent or varying both pH and temperature) and/or (iii) inducing the formation of a polymer-polymer complex at the interface (*e.g.* by adding a second, complex-forming hydrocolloid). Optionally, a cross-linking agent may be added in order to improve both mechanical stability and barrier properties of the wall. The coacervation can also be followed by a drying stage to yield solid beads having sizes ranging typically from 10 to 250 micrometers. Extensive discussions of the factors affecting both encapsulation process and bead properties can be found elsewhere [26].

In interfacial polymerization, oil-soluble and water-soluble monomers are added to both oil and aqueous phases, respectively. The system is emulsified to form an oil-in-water emulsion and polymerization starts upon addition of an initiator. This leads to the formation of an interfacial film between the oil droplets and the aqueous phase. Preferred polymers encompass polyesters and polyamides obtained by interfacial polycondensation [27].

The partition of fragrance components between the aqueous and oil phases constitutes the principal drawback of both coacervation and interfacial polymerization. The loss of water-soluble and highly volatile species induces severe distortion of the hedonic profile of the encapsulated oils. On the other hand, chemically reactive fragrances like aldehydes can participate to the interfacial polymerization and can be partially lost.

Recently, the partition issue has been successfully solved by using a new encapsulation two-step process developed by Givaudan [28]. In a first step, the microcapsules are produced by coacervation of gelatin and gum arabicum on neutral oil droplets and then separated from the coacervation medium, and dried by conventional techniques. In a second step, the perfume or flavor is allowed to diffuse into the dry microcapsules. Complete incorporation of the volatile actives occurs within a few hours yielding free flowing beads containing up to 40% of encapsulants. The beads can be incorporated into various products with a water activity lower than 0.8. These microcapsules provide a dual release mechanism. The volatile actives can either diffuse slowly through the shell of the bead at a permeation rate depending on the water activity or relative humidity or be released according to a burst-like release mechanism when the microcapsules are mechanically broken. The extent of slow release and the mechanical stability of the bead can furthermore be modified by cross-linking with, *e.g.* glutaraldehyde. The process proved very efficient in encapsulating quantitatively critical water-soluble flavors.

3.5. *Colloidal Carriers for Application in Aqueous Products*

Flavors and fragrances are traditionally incorporated into aqueous formulations by simply mixing the oil with the other ingredients at some stage of the manufacturing process. The volatile components readily partition between the different phases present in the system, *i.e.* water, micellar entities, emulsion droplets, porous particles, *etc.* With the use of synthetic packaging materials, many products must be considered as non-equilibrium open systems and partitioning includes also the diffusion of the volatile components through the packaging into the atmosphere. The time-dependent behavior of the overall oil composition profile is therefore a complex function of the activity and diffusion coefficient of the individual components and of their volatility.

In most cases, the application of perfumed aqueous products involves a dilution step in a large quantity of water, which induces a dramatic change of the chemical potential of each fragrance ingredient. This is the case for detergents, cleansing compositions and rinse-off conditioners. At this stage, a considerable amount of perfume is lost by dissolution in the large excess of water and ends up down the drain. Concomitantly, displacement of the most volatile species into the air occurs. While the latter effect may be beneficial to the consumer in terms of sensory experience, the loss by dilution is not desirable. Of course, both effects decrease the quantity of perfume available to the substrate on which the product is applied, *e.g.* fabrics, skin, hair, *etc.*

The remaining fragrances deposit on the substrate either molecularly or associated with other ingredients. Some correlation has been found between the extent of molecular deposition and the hydrophobicity of the fragrance, as measured by its octanol-water partition coefficient [2]. After rinsing, the fragrances remaining on the substrate evaporate more or less rapidly, depending on their individual volatility and on their affinity with the substrate or with co-adsorbed species. Generally, the most volatile fragrances (*e.g.* top notes) disappear first, while the heaviest (*e.g.* bottom notes) remains perceivable over a prolonged period of time.

A basic strategy to minimize the loss and improve the deposition of perfume involves the incorporation of the perfume in surfactant aggregates such as multi-lamellar vesicles. The incorporation occurs spontaneously in concentrated fabric care conditioners containing a large amount of cationic surfactant and accounts for the excellent perfume transfer observed in this kind of product. However, this strategy does generally not provide any sustained release of the very volatile fragrances, as shown from various release kinetics measurements performed [29]. This is attributed to the spreading of the cationic surfactant molecules on the fibers and the concomitant breakdown of the multilayer structures.

In order to circumvent the drawbacks of the above classical approaches, new colloidal entities with improved perfume retention must be considered. The patent literature offers a broad selection of potential delivery systems developed for this sake, including wax particles [30], composite microspheres [31], emulsion polymers [32], associative polymers like block and graft copolymers [33], and liposomes [34]. Most of the systems disclosed so far are, however, not efficient in retaining the fragrance over a long period of time, especially in the presence of surfactants in the surrounding aqueous medium. In fact, progress in this area requires solid, glassy, highly cross-linked, or (partially) crystalline polymers and elaborate encapsulation or enrobment procedures, which are still at a very early research stage.

4. Selected Applications

4.1. *Stable Solid Flavor Formulations*

According to Baisier and Reineccius [35], the shelf life of a spray-dried citrus flavor is limited by the formation of 1,2-limonene epoxide. As soon as the concentration reaches levels above 2 mg/g of oil, the product develops an easily noticeable off taste.

Our investigations on different carrier systems showed a significantly improved stability against oxidation by using maltodextrins with higher dextrose equivalents (DE). By combining sugars with maltodextrins or adding ascorbic acid it was possible to improve the resistance against oxidation. Fig. 5 shows the formation of 1,2-limonene epoxide in an accelerated stability test at 37 °C. Improved carrier systems are able to at least double or triple the shelf life of spray-dried products with respect to oxidation.

Further improvement can be obtained by using specific blends of carbohydrates and emulsifiers. Givaudan's latest development, based on an improved carrier blend, is an example of this. The new formulation is spray-dried under particular conditions, resulting in a dense and glassy matrix showing an excellent resistance against oxidation.

4.2. *Laundry Detergents*

Laundry products are generally perfumed at a perfume level ranging from 0.2 to 1%, depending on the product category. Perfume addition fulfills three missions: (i) to mask the unpleasant odor of cleansing actives, (ii) to signal cleanness performance during storage and use, and (iii) to impart a nice smell to fabrics. Most consumers consider the smell as one of the most important attributes of laundry categories.

When added to wash powders and tablets, perfumes tend to fade by evaporation, oxidation, chemical degradation or interactions with other ingredients. The extent of these effects can be sufficient to dramatically alter the quality of the product, especially when packages are repeatedly opened over an extended period of time or stored under humid conditions. Encapsulation provides an elegant way to stabilize the perfume during storage and to counteract the above losses. In particular, the top notes can be advantageously preserved. An additional benefit of encapsulation is the possibility to increase the level of perfume in the product without affecting the solubility and dispersibility of the detergent in the wash liquor.

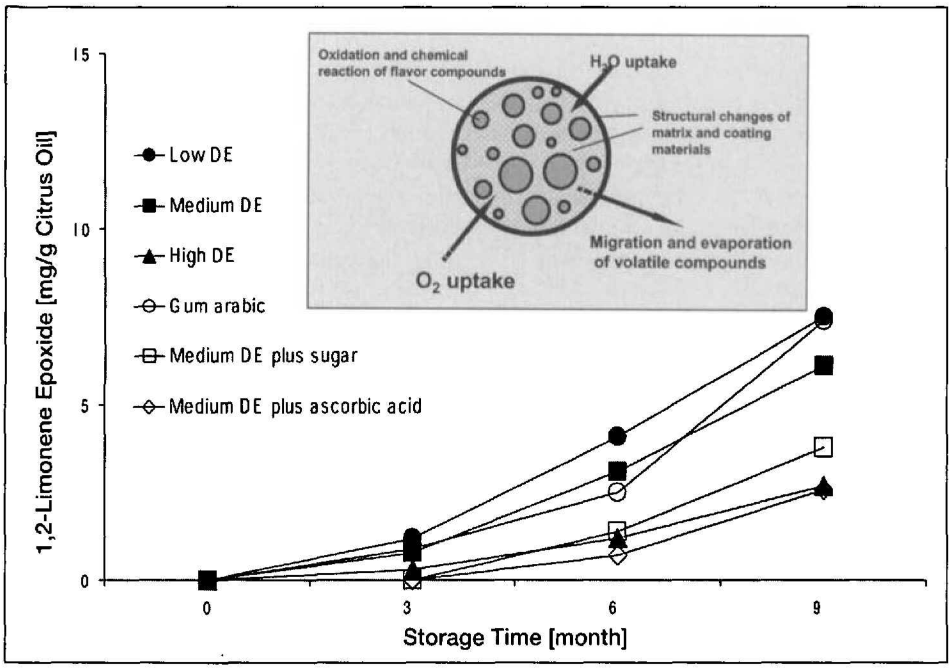


Fig. 5. Limonene stability during storage as a function of matrix composition.

4.3. *Fragrance Boosters through High Volatile Retention*

Using carefully selected hydrophilic matrices, spray-dried encapsulates with a high resistance to moist environment (70% relative humidity, rH) can be produced to protect the most volatile and fragile fragrances in wash powders during storage, even under tropical conditions. Once encapsulates come in contact with liquid water, a boost of top notes is generated, which enhances the olfactory impact of the wash powder. Varying the balance between barrier properties and hygroscopicity of the matrix materials can increase or decrease the duration of the boost. A typical example is shown on Fig. 6. In this example, a perfume consisting of fragrances varying in volatility and water solubility was emulsified in an aqueous solution containing maltodextrins, whereas the emulsifier was a starch octenyl succinate. The powdered fragrance was blended at a level of 0.3% into a standard non-perfumed powder detergent, filled into carton boxes and stored at 37 °C and 70% rH.

The average initial retention of the example described exceeds 95%, whereas after one month at 37 °C and 70% rH an average level of 80% fragrances was still present in the detergent powder. For comparison, less than 40% fragrances survived such drastic storage conditions.

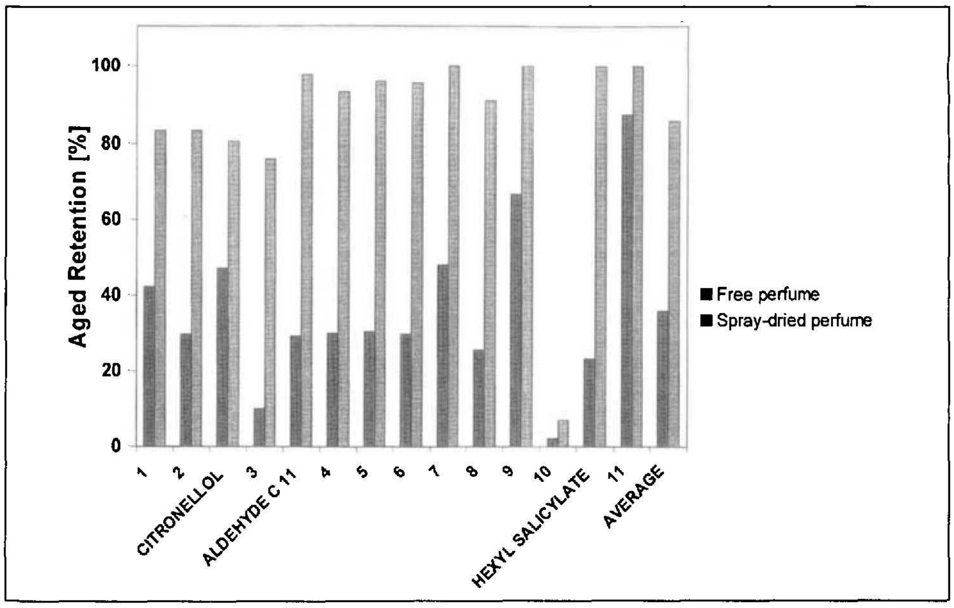


Fig. 6. Retention of key fragrance components in wash powder after one month storage at 70% relative humidity and 37 °C. Perfume load = 30% (w/w).

4.4. *Controlled Release of Perfume During Laundry Machine Wash*

The classical pathway of delivering the non-encapsulated (free) perfume onto a fabric washed in a typical washing machine is characterized by the following general steps: (i) dissolution of powder detergent, (ii) partitioning of perfume between emulsion/water/air/micellar phase, (iii) deposition of perfume onto fabric and (iv) partial loss of perfume during rinsing.

Once the fragranced powder detergent is immersed in the wash liquor, the perfume oil droplets will be entrapped in surfactant micelles and kept in 'solution'. This solvation of free perfume is also related to the aqueous solution solubility of each perfume component. Additionally, there will be partitioning of the perfume between the air-liquid interface depending on Henry's partition coefficient of each perfume component which is related to the partial vapor pressure in the head space. Furthermore, the dissolved perfume droplet will partition between the liquid-fabric interface and deposit onto the fabric.

Using a tailored delivery system may be advantageous compared to the classical pathway. The release profile of the perfume from a delivery system is characterized *e.g.* by the dissolution behavior of delivery systems such as spray-coated and granulated particular materials. The dissolution may be controlled *e.g.* by applying hydrophobic coatings with a distinct melting temperature and coating thickness. The retarded dissolution leads to a different partition kinetics of the perfume in the wash liquor compared to the classical pathway. This in turn enhances perfume deposition onto the fabric as the release profile of volatile notes can be adapted to the specific wash cycle characteristics.

In this example, we discuss the case of a delivery system capable of protecting key fragrances in wash powders during storage and delivering these fragrances on a controlled manner during the wash cycle. The target product category encompasses laundry detergent powders for machine wash under European conditions.

Granulated starch encapsulates were produced by spray-coating of coarse sugar core particles (0.3 mm) with a feed emulsion, as described above. The granulated particles were further coated with a vegetable fat with a melting point of 40 °C. The size of the granulated particles ranged between 0.6 and 1 mm, as measured by Fraunhofer laser diffraction (Malvern Mastersizer). The perfume load expressed in weight perfume per weight of granulated material was 22%.

After one month of ageing at 70% rH and 37 °C, the wash powder as well as one liter of tap water were poured into an experimental model wash machine (Fig. 7), consisting of a thermostated vessel fitted with an oscillatory U-shaped mixer and a cooling unit. The amount of perfume released into the headspace of the model wash machine was measured using a photon ionization detector (ppbRAE) with a detection limit lower than a few ppb fragrance per liter of air. Optionally, a piece of terry cotton towel was placed into the vessel for subsequent deposition studies. Model wash cycles were performed at 40 °C for 120 min. Typical perfume release curves are shown in the inset of Fig 7. Various analytical results and panel evaluations confirm that the use of the granulated material in conventional laundry detergent powders considerably improves the deposition of key fragrances onto fabrics.

5. Future Trends

In this review, we have emphasized the scientific challenges associated with the protection and controlled release of flavor and fragrances in various products

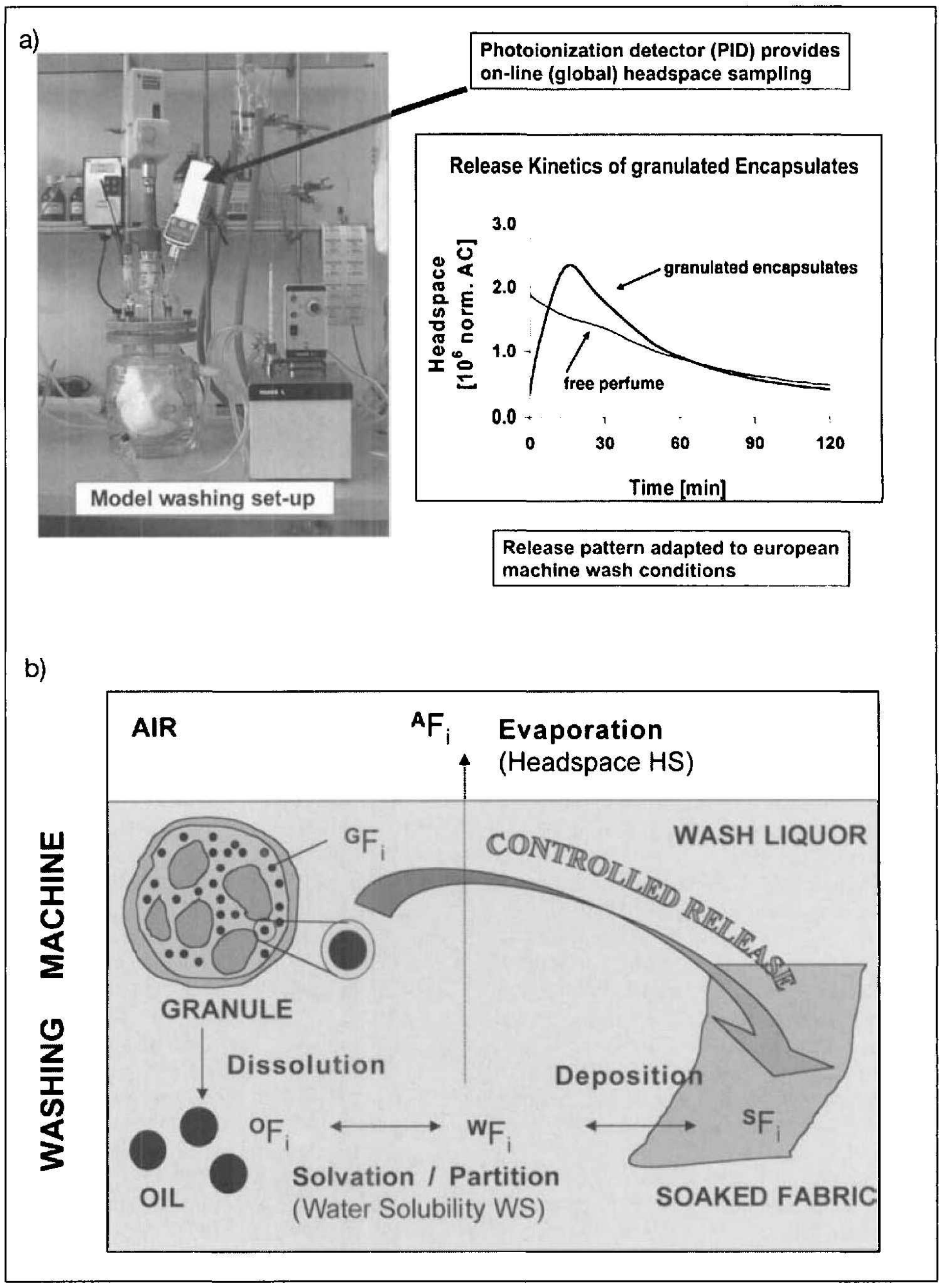


Fig. 7. a) Experimental set-up for laundry study. The curves in the inset show the controlled release effect of the granulated encapsulates. b) Release and deposition mechanisms of encapsulated fragrances (F_i) during laundry. G: granule, O: oil, W: aqueous phase, A: headspace, S: substrate.

and applications. It appears that much work is still to be done in three major areas. Firstly, the retention of volatile actives in carrier materials should be improved, especially in the case of products that are subjected to drastic storage or handling conditions. Secondly, the potential of triggered and sustained release systems in terms of hedonic benefits should be investigated more in depth. Finally, the cost level of current delivery systems is still an issue in many fragrance applications. We believe that progress in all of these areas will require a better understanding of the materials science aspects underlying encapsulation as well as a better integration of the delivery systems into the perfume/flavor creation process.

In parallel with the continuing work on classical encapsulation technologies, new developments are expected to arise from current scientific advances, including molecular recognition, new polymers, composite materials and nanotechnologies.

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