A Case Study of Product Engineering: Performance of Microencapsulated Perfumes on Textile Applications

Miguel A. Teixeira, Oscar Rodríguez, Sofia Rodrigues, Isabel Martins and Alírio E. Rodrigues
Dept. of Chemical Engineering, Laboratory of Separation and Reaction Engineering, Associate Laboratory LSRE/LCM, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, Porto 4200-465, Portugal

DOI 10.1002/aic.12715
Published online July 20, 2011 in Wiley Online Library (wileyonlinelibrary.com).

The evaluation of the performance of new products to be released on the market is essential to measure its acceptability by consumers. In this work, the performance of an added-value product, microencapsulated perfumes applied on textiles for man suits, was predicted and evaluated. The odor intensity and character of the perfume ingredients was measured by headspace gas chromatography after dry cleaning and abrasion tests on textiles impregnated with microcapsules. The evaporation and diffusion of the fragrances in the encapsulated perfume was assessed. For this type of application, limonene has shown a better performance in the perfume mixture, being the dominant note over time and distance.
 AIChE J, 58: 1939–1950, 2012
Keywords: product engineering, microcapsules, fragrances, performance, textile

Introduction

The last 25 years mark a time of accelerated technological expansion and the emergence of a multitude of innovative products. This has been shown, among other things, by growing demands of new products, devices and services: since the advent of mobile phones up to recording DVD devices, from credit cards to digital cameras, contact lenses or high-tech footwear. Within the last 25 years, the industry has been shifting to product development. This switch has been seen for long time, but only recently a more systematic approach for product development has started to be implemented. For a part of these discoveries, Chemical Engineering has contributed with its knowledge. In fact, a new discipline named Product Engineering[1–5] is the answer to a global change in the chemical industry economy: in the past driven by technological expansion for the large production of commodities, towards a market-driven production of high added-value, specialty and performance products.[6,7] This new paradigm on chemical industry can be summarized in Charpentier's triplet ''molecular processes-product-process''[8] or more recently, by the equation $ChE = M^2{\cdot}P^2\ Eng$—where M stands for molecular and materials and P for process and product.[9,10] This can be understood in the perspective that Chemical Engineering is the combination of both, process and product design, and for that purpose it is necessary to use and understand the behavior of materials at the molecular level.

In what concerns chemical product development, there have been several contributions from the literature addressing this issue. These approaches consider the definition of several stages from the identification of consumer needs for the development of ideas and their selection up to the manufacture of the product (needs-ideas-selection-manufacture).[1,2,11] A similar stepwise framework that is widely used in industry is the stage-gate™ product development process (STPDP) which uses product design strategies based on decision analysis.[12,13] A different approach is that of Bagajewicz,[14] who developed a methodology, mainly oriented for the product development stage, based on the application of microeconomics evaluations from the beginning of product design. The approach of Bagajewicz considers a pre-evaluation of consumer demand by introducing pricing and microeconomics models to assess the right properties for the desired product (see, for example, its application to some products[15,16]). If on one hand, this approach is fundamental to eliminate potential products at an early stage of product design, on the other hand, it may not be applied to all products, and definitely introduces an investment (both in time and money) to perform such analysis. For the case studied here, the stepwise description based on the ''needs-ideas-selection-manufacture'' concept[1,2,11] was followed due to its simplicity and aiming for the development of an innovative product. It is important to note that the time mediating all these steps, from the initial idea to the final product, may vary but is around a couple of years, which is reasonably good compared to the time involved in a good research work.[2] This way, an integrated process can be translated into a series of steps involving studies, decision making and research, in different fields of our society.

In this work it was our intention to combine some parts of this methodology for the development of a product that fulfills a specific market niche that has been growing. A combination of three well known products (fragrances, microcapsules and textiles) is presented here aiming to develop a novel product: added-value perfumed suits for men. Starting

Correspondence concerning this article should be addressed to A. E. Rodrigues at e-mail: arodrig@fe.up.pt.

with a basic description of the product design, we will focus on the evaluation of the product performance, namely on what concerns to the perfume performance. This includes the evaluation of the selected type of microcapsules and binder, textile appearance after impregnation by experts (in particular tactile and visual perception), type of textile used, evaluation panel for perfume olfactive quality, and modeling of fragrance release and perceived odor intensity as a measure of performance. These issues are directly associated to the mechanical and physicochemical properties of the polymeric material (and thus the release mechanism of the perfume), and the olfactive properties of the fragrance mixture. The product design evaluation carried out here incorporates perception as a tool that has been gaining importance in product development.[6] A structured study on its characterization, applicability, and performance will be presented.

Fragrances, Microcapsules, and Textiles

The Flavor & Fragrance business comprises from flavors for foods and beverages to fragrances for cosmetics and perfumes. It is a multibillion dollar market that has been increasing until the recent economic and financial crisis. Within this industry, there are multiple applications for fragrances in the formulation of new products (food, cosmetics, personal care, cleaners, and household products) that are subjected to consumer approval or rejection everyday. Being said that, innovation is the key for success. It should not only be devoted to the discovery of new fragrant chemicals (natural, natural-identical or synthetic), but also to their application, and of course, to the performance of the final product.

An example of new applications is the manufacture of microencapsulated perfumes incorporated on textiles for the production of added-value perfumed cloths which is the purpose of this work.[17–20] This way, value is incorporated in textiles that we can use everyday, by introducing pleasant fragranced microcapsules in it. Microencapsulation is a very good technique to control the release of fragrances and to produce more durable fragrant finishing on textiles. The application of microcapsules into textiles presents also several advantages in terms of fragrance performance and health benefits: reduces perfume dermatitis in humans and protects the fragrance material from aggressive external agents or media.[20,21]

In what concerns the textile industry, it has recently shown great interest on microencapsulation and, more specifically, in the application of durable fragrances and skin softeners.[20,22] Microcapsules are also developed for different purposes, including insect repellants, phase-change materials and medical applications, just to mention a few.[19,23–25] Microcapsules are small capsules (1–100 μm diameter) of a polymeric membrane containing an active compound. They are used either to protect this active compound from external agents as humidity, temperature, light, oxidation and exposure to other substances over their lifetime, or to control its release rate.[26] There are several patented processes for microencapsulation of fragrances for textile applications, such as coacervation, interfacial polymerization and others.[27–33] The industry is also putting big efforts on these techniques pursuing consumer needs for several applications.

In this work, polyurethane-urea (PUU) microcapsules were incorporated in textiles to develop an added-value product: perfumed men suits. This type of clothing is designed, from the beginning, as a luxury product. Thus, although the incorporation of a fragrance scent will make it more expensive, it will also make the product more valuable and exclusive. Using this technique, the encapsulated perfume will be released upon breakage of the microcapsules. This way, during the daily use of the suit, fragrances will diffuse into the air and release a pleasant smell.

To evaluate this product, it is of prime interest to assess the effect of dry cleaning and abrasion cycles on the released odor, as they mimic the washing and day-to-day use of the textiles, respectively. This will allow evaluating the lifetime of the impregnated textiles which is a key point for customer appreciation. This is related to another key parameter of Product Engineering: product performance. High added-value products provide a service or a function to the customer, and so what the customer will evaluate is the performance of the product when used. The optimization and the predictability of its performance are imperative for the success of the product in high competitive markets.

Perfume performance has been evaluated (either qualitatively or quantitatively) with a series of parameters that account for the evolution of the odor intensity and dominant fragrances with time and distance from the perfume source.[34,35] These parameters can be used to characterize the perceived scents as they evaporate and diffuse in air over time (t) and at different distances from the source or point of application of the perfume (z). Among these parameters, have to be mentioned the impact, tenacity, diffusion and volume. Impact deals with the initial perception of the scent, like when a perfume bottle is opened or the perfume is sprayed on the body for example ($z\ \varepsilon$ [0, 0.3]m and $t\ \varepsilon$ [0, 5]min); tenacity represents the persistence of a fragrance after a long period of time but near the source of the perfume. It provides a measurement of how long the perfume lasts in the skin after its application ($z\ \varepsilon$ [0, 0.3]m and $t\ \varepsilon$ [10, 20]h). The last two parameters correspond to odor perception at some distance from the source: diffusion is a measure of the efficacy of a perfume away from the source ($z\ \varepsilon$ [1, 2]m and $t\ \varepsilon$ [0, 5] min) showing the diffusion rate of a fragrance in air; volume is the effectiveness of a perfume over distance with time ($z\ \varepsilon$ [1, 2]m and $t\ \varepsilon$ [10, 20]h).[34–36]

Our research group has previously developed a methodology to quantitatively account for these performance parameters.[35] From a Chemical Engineering point of view, this can be traduced by the diffusion profile of the fragrance mixture with time and distance.

In this work, such methodology has been applied to a microencapsulated perfume attached on textiles. A diffusion model based on Fick's second law together with a model to quantify odor intensity and odor character perception were combined to study the performance of the encapsulated perfume once applied in the fabrics.

Methodology

Throughout this work, we have used a four-step procedure for product design—needs, ideas, selection, and manufacture. These steps were applied to the development of perfumed men suits and the result is presented in Table 1. In the previous section we have focused on the needs and ideas to fulfill a market niche. Now the characterization of the last two steps is overviewed. Some consumer preferences that were taken into account for obtaining the desired properties of the final product are highlighted.

Table 1. Description of the Four-Step Procedure Considered in this Work

Needs	Fragrant suits for men
Ideas	Perfume + microcapsule + textile: define several perfume families and compositions, formaldehyde-free microcapsules and polymerization techniques, types of textile and binders
Selection	Composition of a masculine perfume + polyurethane-urea microcapsules by interfacial polymerization + impregnation in the foulard step + polyurethane binder + wool/polyester textile
Manufacture	Production of microcapsules with perfume + impregnation onto textiles + assessment of finished textile quality + evaluation of olfactive performance after dry washing and abrasion cycles

Perfume design

A renowned perfumer of the past century classified fragrances in top, middle, and base notes (attending to their volatility) and stated that a well structured perfume should contain a blend of the three types of notes.[37] Top notes are considered the most volatile ones, being perceived immediately after the perfume is applied and within the first minutes. Middle notes are less volatile and get noticeable later after application, and within the first hours. Finally, the base notes are the least volatile fragrances of all. They are perceived by the human nose much later and can last for several hours or even days (e.g., when applied in textiles base notes last much longer than in the skin). However, it is easily perceptible that this traditional view is an oversimplification of the evaporation and diffusion processes lying behind fragrance mixtures. In fact, from the moment a perfume bottle is opened or a perfume is sprayed, all fragrant notes start to evaporate and diffuse into the air. The different evaporation rates depend on their physicochemical properties, namely their volatility and diffusion coefficient. Nevertheless, fragranced products and fine perfumes are still made with odorant chemicals belonging to different notes so that the perceived scent will be smooth and pleasant over time. Following this classic perfume structure of top, middle and base notes, a perfume concentrate was designed combining limonene (a citrus, fruity top note), methyl dihydrojasmonate (a floral-jasmine middle note), methyl cedryl ketone (a woody, oriental base note) and galaxolide (a musk base note that dissolved in a 50% diethyl phthalate solution has a particular sweet floral and woody "nuance").[18] A nuance traduces a less perceptible odor among a mixture of odorants, adding an extra sensorial perception to the overall scent. Therefore, it represents a male fragrance belonging to the Citrus-Woody-Musk families according to the classification of pure fragrances from Brechbill.[38] This perfume was also developed having in mind its final application into fabrics and the fact that it was oriented for men. The evaluation of a perfume from the point of view of a consumer is difficult to predict and so its formulation is handled by experts (perfumers). This perfume formulation was developed in partnership with I-Sensis.[39] Their experts selected this perfume mixture among various formulations taking into account its olfactory nature and the desired application. A perfume concentrate (without any solvent matrix) was used to intensify the scent at the release stage.

Microcapsules and impregnation on textiles

Most commercially available microencapsulated fragrances are based on formaldehyde polymeric materials, but these have been subjected to increased restrictions. The use of polyurethane-urea (PUU) systems for the polymeric matrix have revealed to be an economically attractive, environmentally-friendly, and versatile solution.[18] For that reason, PUU microcapsules were used to encapsulate the perfume mixture in this work. For the impregnation it is important to take into account the type of binder and textile selected. The binder plays an important role on the performance of the product, influencing the adhesion to the textile substract. Two different binders were tested, Primal ECO 934 TK (an acrylate emulsion from Horquim) and Peripret Plus (a polyurethane emulsion from Bayer). The latest was selected once it resulted in a higher adhesion of microcapsules to the textile. Finally, different types of fabrics with varied grammages were also tested for impregnation. A wool/polyester textile provided the best results according to tactile and visual evaluations performed by industrial partners (CITEVE and A Penteadora SA) involved in that process.

Perfume performance

The odor intensity of fragrant mixtures can be evaluated in quantitative and qualitative terms using either mathematical models or experimental analysis. The odor value (OV) provides a simple and convenient measurement of the odor intensity for a single component. The OV of a fragrant component i is defined as the ratio between its composition in the gas phase, C_i^g, and its odor detection threshold concentration in air, Thr_i, both in g/m^3:

$$\mathrm{OV}_i = \frac{C_i^g}{\mathrm{Thr}_i} \tag{1}$$

As the odor detection threshold provides the minimum concentration that can be detected by the human nose, only when the OV is higher than unity ($\mathrm{OV}_i > 1$) the component can be perceived.[34,40] However, it is reasonable to admit that odor perception may not be linearly proportional to the concentration throughout the whole scale of magnitudes above the threshold. In fact, as the concentration increases it is expected that the perceived odor will also increase, although at some point in the intensity scale there will be a saturation of the odor receptor cells. Following this line of though, the Stevens Psychophysical Law considers that the perceived sensation (Ψ) is proportional to the stimulus (C_i^g) raised to an exponent (n). This law can be applied to all perceptual continua, so that it follows a Power Law as expressed by Eq. 2 for the case of olfaction:

$$\psi_i = \left(\frac{C_i^g}{\mathrm{Thr}_i}\right)^n \tag{2}$$

In olfaction, the majority of the odorants seem to generate power functions with exponents smaller than unity with a reported median value of 0.35, which can be used whenever experimental data for the exponent are not available.[41] The odor detection thresholds and the exponents for the power law are available in the literature from compilations of data for hundreds of odorant species.[34,40–42]

The concentration in the gas phase can be calculated from the concentration of the liquid perfume using some basic Thermodynamics.

At atmospheric pressure, ideal gas behavior for the gas phase can be assumed and so the fugacity coefficient becomes unity, $\phi_i = 1$. Thus, C_i^g can be calculated as:

$$C_i^g = \frac{y_i M_i P}{RT} = x_i \gamma_i \frac{M_i P_i^{sat}}{RT} \quad (3)$$

where y_i and x_i stand for the vapor and liquid compositions in mole fraction, γ_i is the liquid activity coefficient of component i, P is the total pressure, P_i^{sat} is the saturation pressure of pure i (which can be obtained from the literature[43]), M_i is the molecular mass of component i, R the universal gas constant and T the absolute temperature. In this equation, the activity coefficients in the liquid phase (γ_i) cannot be neglected. Fragrances, which in general present several different functional groups in their chemical structure, are likely to present non-ideal behavior. From Eqs. 2 and 3 we obtain the expression for the odor intensity (Ψ) of each component, which can be predicted from its composition in the liquid phase using the UNIFAC method and some physical properties:

$$\psi_i = \left[\gamma_i x_i \left(\frac{P_i^{sat} M_i}{\text{Thr}_i}\right)\left(\frac{1}{RT}\right)\right]^n \quad (4)$$

As stated above, ψ provides a measurement of the odor intensity for the different odorants present in the surrounding air. Still, it is necessary to account for the perception of odor mixtures. Several attempts have been done to describe the perception of mixtures, but none has shown convincing.[44–47] Along with the models proposed, the Stronger Component Model is among those recommended for truly multicomponent mixtures (>3 components).[44,45] The basic statement of this model is that the odorant with the highest odor intensity will be more strongly perceived. Combining this model with the power law for odor intensity (Ψ) and applying to the N components present in the gas phase results:

$$\psi_{max} = \max\{\psi_i\}, \quad i = 1, ..., N \quad (5)$$

The selection of this model is based on the experimental evidences that the odor intensity of mixtures tend to show a certain compromise..[45,47,48] The model does not provide any information about the remaining components, just stating which one will dominate the perceived scent above all of them. But as far as Eq. 4 can be used to predict odor intensity, Eq. 5 allows the prediction of the dominant note in a mixture or a perfume.

The odor intensity (Ψ) concept can be applied together with some previously developed methodologies like the perfumery ternary diagram (PTD®) and the perfumery quaternary-quinary diagram (PQ2D®) for the odor intensity and character of fragrance mixtures,[10,49] and the perfumery radar (PR) methodology for fragrance mixture classification.[50] The former two methodologies allow representing the odor character of ternary, quaternary, and quinary mixtures with graphical tools. The PTD® uses ternary diagrams and the PQ2D® uses tetrahedric diagrams to map the perceived odor character of fragrance mixtures (e.g., the component with the highest odor intensity, Ψ_{max}) as a function of the composition in the liquid phase together with Eqs. 4 and 5. For that, the UNIFAC method was used to predict the vapor-liquid equilibrium (VLE) considering non-idealities in the liquid phase of the perfume.[10] The last methodology allows classifying perfumes or fragrance mixtures into olfactive families using radar plots. The PR methodology introduces scientific knowledge to predict the character of perfume mixtures using the odor intensity and character models aforementioned.[50] It is based on the classification of pure fragrances into olfactory families, and then predicts the odor intensity for each fragrance using an odor intensity model (OV—Eq. 1, power law—Eq. 2, or other). Finally, it estimates the cumulative odor intensity of each olfactory family and represents that in the Perfumery Radar. Further details can be found elsewhere.[50]

In what concerns the performance parameters previously presented for the effect of time and distance in the perception of fragrances or perfumes, they use the odor intensity and stronger component models together with a diffusion model. In the perspective of a chemical engineer these performance parameters are simply the diffusion effects of the fragrances in air. Thus, combining the power law model from Eq. 2 with a diffusion model to account for the effects of time and distance on the fragrance concentration in air will allow predicting the performance of the different fragrances used in the perfume formulation. For this purpose, a simple diffusion model, previously developed based on the Fick's second law for diffusion, was used.[35] The diffusion model simulates the evaporation rate of small amounts of a perfume mixture, using the UNIFAC thermodynamic method for the VLE. This way, the volume and composition of the liquid are changing with time as the perfume evaporates and it was considered one-dimensional diffusion in air.

In sum, in the present work it is intended to show the different steps involved in product development, which in this case was oriented to the production of microencapsulated perfumed textiles for commercial use (manufacture of man suits). In a first stage, various perfumes were produced by a fragrance company and a quaternary perfume concentrate was selected by their experts taking into account its scent and final application.[18] The odor intensity and character of this perfume was predicted using simulation techniques like the PTD®, PQ2D® and PR methodologies.[10,35,49,50] The performance of the perfume was also analyzed using a previously developed diffusion model for time and distance.[35] Subsequently the perfume was microencapsulated and impregnated in fabrics. These textiles were evaluated by experts in terms of touch and visual quality after impregnation and then subjected to five dry washing cycles and 9000 abrasion tests. The headspace of these textiles was experimentally analyzed and the performance of the impregnated fragrance was simulated with the diffusion model.

Materials and Methods

Perfume formulation

The perfume formulation was obtained using four commercially available fragrances, namely (4R)-1-methyl-4-(1-methylethenyl)-cyclohexene (limonene), methyl-(2-amyl-3-oxocyclopentyl)acetate (methyl dihydrojasmonate, MDJ), methyl cedryl ketone (vetiver) and 1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g]-2-enzopyran (galaxolide) in a 50%

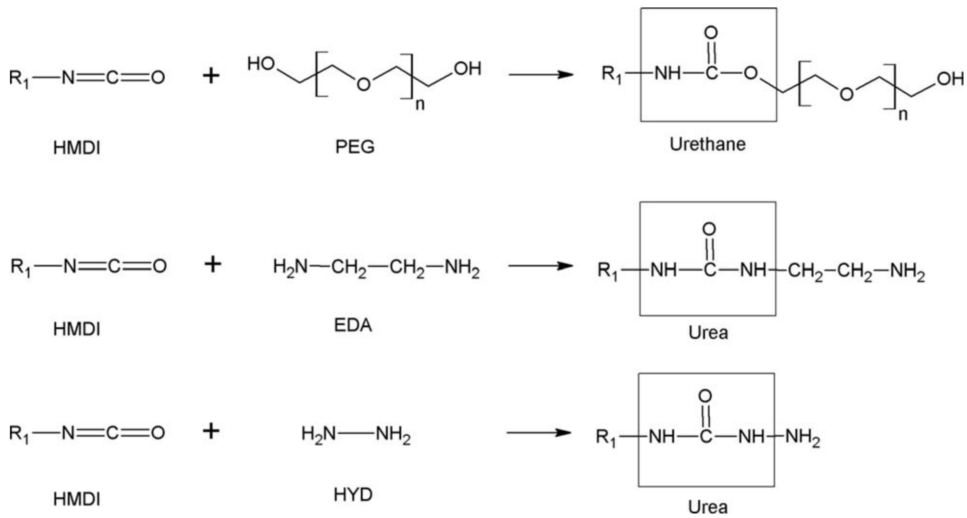

Figure 1. Polymerization reaction mechanism for the synthesis of the polyurethane-urea shell of the microcapsules.

diethyl phthalate (DEP) mixture. All fragrances were supplied by Sigma Aldrich and used without further purification.

PUU microcapsules and impregnation on textiles

The production of PUU microcapsules was performed according to the reaction mechanism presented in Figure 1. The reactants used in this process and its further application on the textile substrate were: hexamethylene-1,6-diisocyanate (HMDI) purchased from Bayer; polyethylene glycol 400 (PEG 400) from Sigma Aldrich; ethylenediamine (EDA) obtained from Panreac; hydrazine monohydrate (HYD purum) was purchased from Sigma-Aldrich; polyvinyl alcohol (PVA) was obtained from Celanese Chemicals acting as a protective colloid, and Triton Ca acquired from Dow company was the emulsifier; dibutyltindilaurate (DBDTL) from Sigma-Aldrich was used as catalyst. PUU microcapsules were prepared by interfacial polymerization and further details on the encapsulation process can be found elsewhere.[18] The microencapsulation process was carried out in a polymerization reactor with controlled temperature and equipped with a mechanical stirrer. The microencapsulation process involves three steps:[18] first, the formation of oil-water emulsion (the emulsion is placed at 80°C and stirred at 100 rpm in an IKA Reactor); then, the formation of the capsule wall (urethane and urea phase membrane around the oil droplets); finally, the separation and washing procedures. The resulting microcapsules were collected by centrifugation and washed with an ethanol solution (30% v/v) and distilled water to remove the remaining reactants. Finally the microcapsules were suspended in a solution of water containing the wetting agent (Triton CA).

PUU microcapsules with the selected masculine fragrance were applied on fabrics of wool/polyester in the finishing process using a foulard.[18] For the impregnation process it was prepared a bath containing: (i) microcapsules at a concentration of 50 g/L, (ii) a softener at 10 g/L, and (iii) a self-cross linking agent at 50 g/L. The conditions required for the impregnation process on the foulard were: pressure at 3 bar, 70–80% of pick-up rate, 100°C during 3 min for drying followed by 140°C during 3 min for thermo-fixation.

Dry cleaning and abrasion tests

After the impregnation of the microcapsules on the textiles, these were subjected to dry cleaning washing cycles or abrasion tests to evaluate their resistance and performance. The dry cleaning process used perchloroethylene as solvent, and according to the European Norm EN ISO 3175-2 the procedure followed a washing time of 15 min and a final rinse of 5 min.[51] For its part, the abrasion tests are intended to evaluate the resistance of the textile substrate when submitted to flexing, rubbing, shock, compression, stretching, and other types of mechanical forces. For that, the sample was placed between two discs driven by a rotor along a "zigzag" course in a circular orbit (cycle), according to the European Norm EN ISO 12947-2.[52]

Headspace/GC/FID analysis

The headspace of fabrics impregnated with fragranced microcapsules was evaluated by GC-FID before and after each test to account for the performance of the final product. Square samples (4 × 4 cm) of the impregnated fabrics were placed in plastic bags (12 × 23 cm) and the microcapsules were broken by mechanical stress. Samples were left for equilibration overnight, and then a volume of 0.5 mL of the gas phase was injected and analyzed by Headspace/GC/FID. Gas chromatographic analyses were carried out on a Varian CP-3800 instrument equipped with split/splitless injector, CP-Wax 52 CB bonded fused silica polar column (50 m × 0.25 mm, 0.2 μm film thickness), and a FID detector. The GC oven temperature was programmed as follows: 50°C for 5 min, ramp of 2°C/min up to 200°C and held at 200°C for 25 min. The injector was set at 240°C with a split ratio of 1/50 for FID, while the FID detector was kept at 250°C. In the case of headspace analysis the injection was performed using gastight syringes (SGE) in splitless mode. The carrier gas used was helium He N60 at a constant flow rate of 1 mL/min.

Results

Perfume characterization

The fragrant components used in the formulation of the perfume together with their compositions and some physicochemical properties are presented in Table 2. In the design of this perfume concentrate, a characterization of its olfactive intensity and character was performed using some perfumery tools previously developed in our research group. First, the odor character of the mixture was studied using the PTD® and PQ2D® methodologies. These are graphical tools showing the

Table 2. Perfume Concentrate Components, Composition, and Some Physical Properties

Component	Molecular Formula	Molar Composition (x_i)	Molecular Weight, M_i (g/mol)	Odor Threshold, C^i_{Thr} (g/m^3)	Vapor Pressure, P_i (Pa)*	$n^{\dagger}$
Limonene	$C_{10}H_{16}$	0.296	136.20	$6.84 \times 10^{-4\ddagger}$	20.5×10^{1}	0.37
Methyl dihydrojasmonate	$C_{13}H_{22}O_3$	0.326	226.31	$9.12 \times 10^{-4\S}$	9.47×10^{-2}	0.35¶
Methyl cedryl ketone	$C_{17}H_{26}O$	0.299	246.39	3.80×10^{-5}**	1.13×10^{-2}	0.35¶
Galaxolide (in 50% DEP)	$C_{18}H_{26}O$	0.037	258.40	$1.38 \times 10^{-5\S}$	5.52×10^{-2}	0.36
Diethyl Phthalate	$C_{12}H_{14}O_4$	0.043	222.23	Odorless	2.22×10^{-1}	–

*Chemspider. Database of Chemical Structures and Property Predictions - Royal Society of Chemistry.[Database] November, 2010; Available from: http://www.chemspider.com/Default.aspx.
†Devos M, Rouault J, Laffort P, Standardized olfactory power law exponents. Dijon - France: Editions Universitaires-Sciences 2002.
‡van Gemert LJ, Compilations of odour threshold values in air, water and other media. The Netherlands: Oliemans Punter & Partners BV 2003.
§Leffingwell JC, Leffingwell D. May 2010; Available from: http://www.leffingwell.com/odorthre.htm.
¶Recommended average value.
**Measurement in our laboratory using an olfactometer.

dominant odor as a function of composition in triangular or tetrahedric diagrams, respectively. The dominant odor is calculated using an odor intensity model (e.g., the power law as in Eq. 4) together with an odor perception model (e.g., the stronger component model as in Eq. 5) as presented before. As the perfume concentrate is composed by four fragrances and an odorless solvent, the PQ2D® is perfectly suited to show in a single 3-D-graph the effect of the composition in the initial odor character near the source of fragrant release. The odor character of the ternary subsystems that compose the perfume mixture used in this work are presented in Figure 2. The presented PTD® diagrams match the faces of the PQ2D® which is presented in Figure 3. Each fragrant odor will dominate above all others within a certain composition range, which in the PTD® represents an odor zone and in the PQ2D® is shown by an odor volume.

In Figure 3 it is possible to visualize the shapes of the different odor volumes as a function of the liquid composition in the PQ2D® for the quaternary mixture (limonene + MDJ + vetiver + galaxolide). It is clear that limonene, the top note (the most volatile) has the dominant odor of the mixture for most of the composition range, and so its odor volume occupies nearly the whole tetrahedron. The galaxolide odor volume dominates the odor space for low limonene concentrations, appearing as a slice opposite to the limonene vertex.

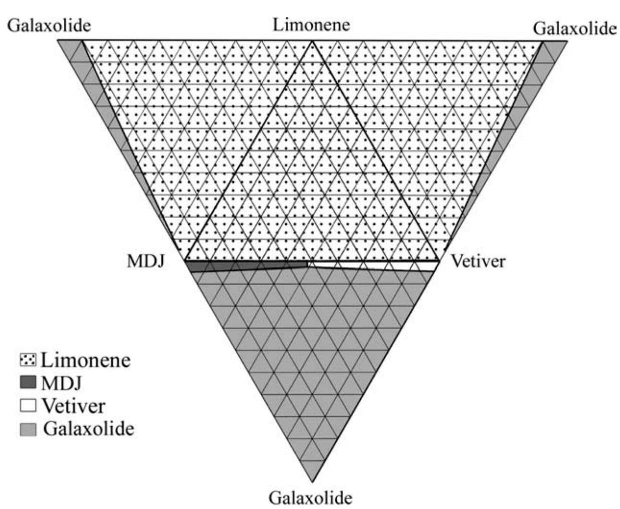

Figure 2. PTD® for the ternary subsystems with the odor zones for each individual fragrance in the mixture.

Both MDJ and vetiver (the floral and woody notes) make themselves more strongly perceptible only for very low concentrations of limonene and galaxolide. Thus, their odor volumes are like fine sticks near the axis linking their vertices. Considering the initial perfume composition and the PQ2D® on Figure 3, it is possible to see that the perfume mixture selected for this work would initially present a dominant limonene scent. To assess the odor character or quality of this perfume formulation, the PR methodology was also applied, considering its molar composition (Table 2) and the classification of the pure fragrances into olfactive families presented in Table 3.

The obtained perfumery radars for the predicted and experimental (headspace) compositions of the perfume mixture are shown in Figure 4. Both perfumery radars present a citrus primary olfactive family, resulting from the dominance of limonene. In this way, in terms of olfactive families both the predicted and the experimental perfumery radars evidence an initial Citrus-Floral odor perception, although the fruity, woody and musk nuances have also their impact. These last two families will be more strongly perceived after the evaporation of the top note (limonene). Thus, considering the proposed olfactive families of the PR methodology, it can be possible to classify this perfume as Citrus-Floral with woody and musk nuances. The experimental classification of this simple perfume mixture obtained from the nose evaluation of experts agreed with the Perfumery Radar, considering the powerful limonene odor (mainly citrus and fruity) to give lift and freshness, together with a background of sweet (musk) and woody nuances.[18]

Nevertheless, the perceived smell of a perfume does not remain the same over time. As mentioned before, perfumes are formulated as a combination of different types of fragrances. Once it is applied in our body or clothes the diverse components will start to evaporate and diffuse at different rates. The same applies for a perfume released from microcapsules. Thus, the odor of the perfume will evolve with time, and this effect must be accounted for the evaluation of its performance. To evaluate the performance of the proposed perfume formulation, a diffusion model previously developed was used to predict the evolution of the odor intensity and character of the selected formulation.[35,49] This diffusion model accounts for the change of the fragrances concentration, both in the liquid and gas phases. The combination of the diffusion model with the odor intensity model (Eq. 2) and the perception model (Eq. 5) provides the tool to predict the odor intensity profiles for a given perfume

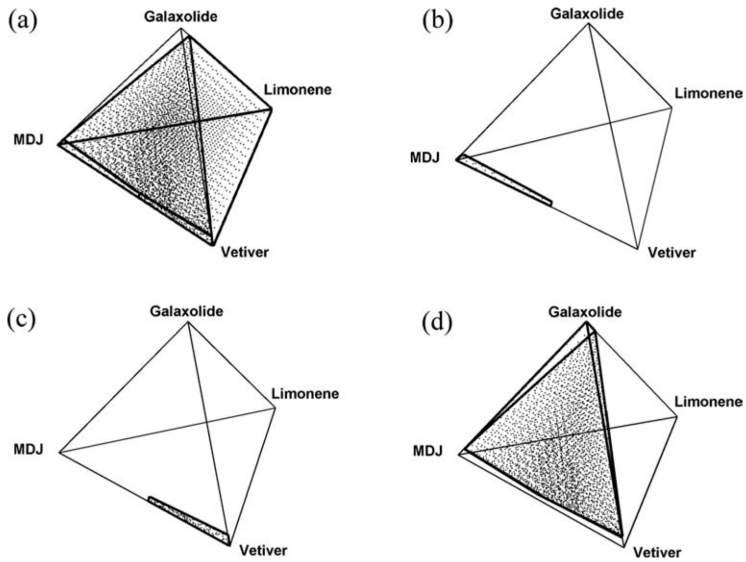

Figure 3. PQ2D® with the odor volumes for each individual fragrance in the mixture: (a) limonene, (b) MDJ, (c) vetiver, and (d) galaxolide.

composition. These intensity profiles are presented in Figure 5 for the selected perfume composition (Table 2) at two different distances from the source: 0 and 2 m. Despite the simplicity of the odor models, they provide basic information about the dominant note (character) of the perfume. This way, the diffusion profiles show that limonene is the odor note more intensely perceived for all the time considered. Even at longer distances from the release point, limonene is more strongly perceived although the middle and the base notes present some background odor effects: vetiver gives an oriental-sweet nuance while the musk galaxolide acts as a fixative by retaining the other fragrances in the liquid and, thus, slowing down their evaporation.

Performance of fabrics with microencapsulated perfume

Figure 6 presents the micrographs obtained from scanning electron microscopy (SEM) for the impregnated microcapsules produced in the laboratory. Through these images it is possible to see that the surface morphology of the PUU microcapsules is spherical and smooth. It can also be observed that the microcapsules are well distributed in the textiles without excessive agglomeration and confirmed the adhesion between textile fibers and microcapsules.[18]

The encapsulated perfume is a homogeneous solution with constant composition in each microcapsule, which will be released to the air from breakage of their polymeric walls. To evaluate the performance of the microencapsulated perfume when applied to the fabrics, the released odor intensity was compared to that of the pure perfume. The comparison was extended to the odor intensity predicted by means of Eq. 4. Odor intensities for the fragrant components were calculated from the concentrations measured experimentally in the headspace of the perfume at equilibrium conditions and in the headspace of the microencapsulated textiles (before any washing cycles or abrasion tests). Besides, the odor intensity was also predicted from the perfume composition, considering the encapsulation efficiency, using Eq. 4. A comparison between the odor intensity of each fragrant component in the three situations is presented in Figure 7.

From Figure 7a, it can be observed that for the perfume mixture, limonene is the dominant fragrant note in all cases, followed by galaxolide, vetiver, and finally MDJ. Comparing the odor intensities obtained in the headspace of the liquid perfume mixture (left) and in the headspace of the impregnated textiles (middle), it is possible to see that the top note (limonene) is the only species that has higher odor intensity when applied in the textiles. This may be attributed to the fact that limonene was the fragrance ingredient with the least encapsulation efficiency, and so much of it remained outside the microcapsules, being directly adhered to the fibers. To simulate the odor intensity of the fragrant compounds, they were also predicted using the composition of the liquid perfume, the encapsulation efficiency obtained, and the UNIFAC method as described before. The predicted odor intensities for each fragrance compound are presented on the right column of Figure 7a. The results show an over prediction for the odor intensity of limonene, while for the other fragrances it presents the same tendency as observed for the perfume and textiles headspace. The general trend of the perceived odor intensities for all the species in all the three cases is Limonene > Galaxolide > Vetiver > MDJ.

In a previous study we reported the microencapsulation of pure limonene for textile applications.[17] The type of microcapsule (PUU) and fabric, the microencapsulation process and the impregnation protocol was the same as in this work. Thus, the same analysis can be performed for these microcapsules with pure limonene. The results obtained are presented in Figure 7b. It is possible to observe that the odor intensity in the headspace of the impregnated fabric was lower than for liquid limonene, as well as the odor intensity predicted with our model (Eq. 4). It is important to recall that the

Table 3. Fragrance Classification in Terms of Volatility and Olfactive Character

Component	Fragrance Note	Odor Character[38]
Limonene	Top	Citrus, fruity, floral
Methyl dihydrojasmonate	Middle	Floral
Methyl cedryl ketone	Base	Woody, oriental
Galaxolide (in 50% DEP)	Base	Musk, floral, woody
Diethyl phthalate	Solvent	odorless

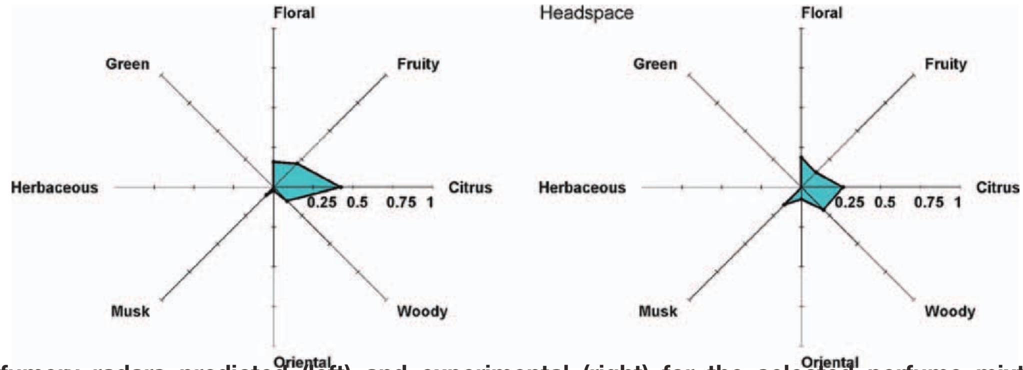

Figure 4. Perfumery radars predicted (left) and experimental (right) for the selected perfume mixture that was encapsulated and impregnated in the fabrics.

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

model for a single component reduces to the saturation pressure. Thus, the model gives a value close to the pure liquid, while the textile with microencapsulated limonene provides a lower odor intensity which can be understood as a matrix effect of the microcapsule application on the odor release, acting as a fixative of the fragrance in the substrate. It should be highlighted that a similar trend was observed for limonene in both textile applications: the predicted value based on our model is higher than what was experimentally measured in the headspace. This is probably due to matrix effects, that is, interactions between the fragrance ingredients and the fibers, the microcapsule wall material, the softener and/or the surfactant, which can act as fixative agents of the fragrances. These effects on the released odor intensity were previously studied by other authors showing that several factors may influence the interaction with fragrance materials.[53–55]

As mentioned before, the impregnated textiles were subjected to dry cleaning and abrasion (mechanical) tests for the evaluation of their performance in a daily use. The effect of the number of dry washing cycles and abrasion tests on the perceived odor intensity of the fragrance encapsulated textiles is presented in Figures 8a, b, respectively.

The influence of the dry washing cycles on the performance of the fragranced textiles shows that there is a decrease in the perceived odor intensity of all the fragrant components. Limonene is still the most strongly perceived component of all fragrances, as expected for a top note. After five dry washing cycles there is a decrease of 58% and 47% of the odor intensity of limonene and vetiver, respectively. For the case of MDJ it is not perceived, since its odor intensity always falls below unity. The base note, galaxolide (musk), was no longer perceived from the fourth dry washing cycle onwards. This variation on the presence and composition of the different fragrant components shows that not only the perceived odor intensity is changing due to the washing cycles but also that the odor character is evolving since the effect of MDJ and galaxolide on the overall scent is being reduced.

The abrasion tests are also intended to evaluate the performance of the fabrics by mimicking the mechanical resistance of the microcapsules during the daily use of a man suit. The variation of the odor intensity of the fragrances as presented in Figure 8b evidences a slight decrease over the abrasion cycles. Once more, it is seen that the odor intensity of MDJ is always below the threshold level, and so it is not perceived by the human nose. However, its presence both in the liquid and vapor phases influences the overall scent of the perfume mixture. It was observed that only limonene and vetiver remained at perceivable concentrations after 9000 abrasion cycles. Nevertheless, it should be highlighted that even after such an intensive test there are fragrances still remaining adhered to the fabrics, and contributing to the perceived scent.

On both tests (washing and abrasion cycles) it was possible to conclude that limonene and vetiver were the most retained fragrances in the fabrics and with suprathreshold odor intensities. It is seen through this analysis that after a number of dry washing cycles or abrasion tests some of the notes (MDJ, galaxolide) have odor intensities below unity

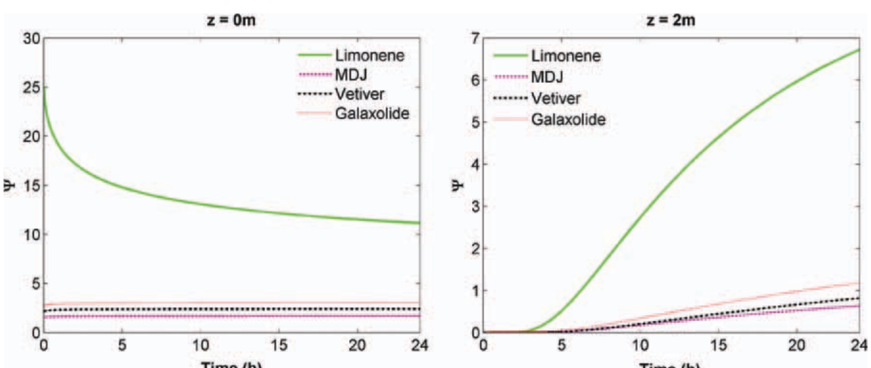

Figure 5. Odor profiles for the perfume concentrate near the point of release (left) and at a distance of 2m from the source point (right).

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

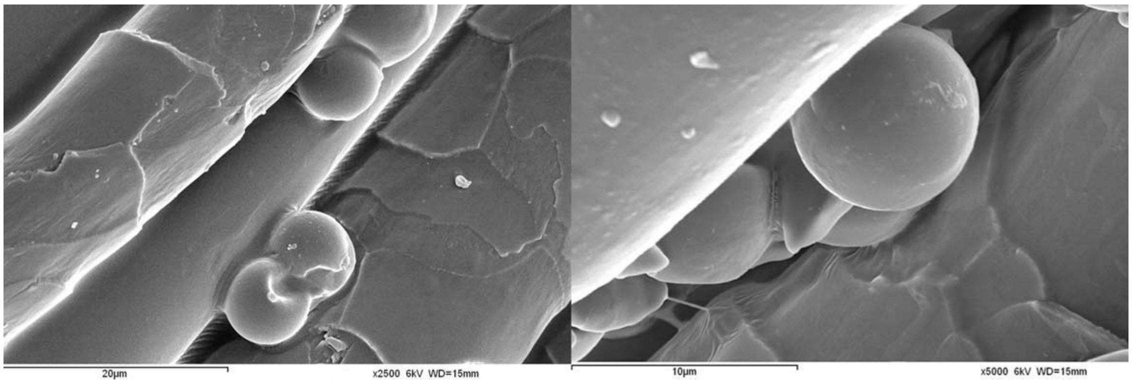

Figure 6. SEM images of the microcapsules impregnated in the tested fabrics at the laboratory scale: ©LSRE 2010, with permission.

(sub-threshold). However, it should be emphasized that this does not mean that the fragrant species is not still impregnated in the fabrics, but that its concentration is below the detection limit. Thus, if the fragrant component remains in the perfume mixture, it continues to play a role in the evaporation rate of the other fragrances, influencing the overall perceived odor.

To assess the performance of these fragranced textiles it is important to evaluate the perceived odor intensity and character of the microencapsulated perfume either over time of usage of the suit, either at different distances from it. This is important to evaluate the perceived sensation of the fragranced suit on the user and surrounding people. The analysis of such parameters is quite difficult to predict (due to the randomness of microcapsule breakage and evolution of the perfume composition over time) or to evaluate experimentally (due to the complexity in sample collection and analysis at low or trace levels). However, for a simple evaluation, a diffusion model was used to estimate the performance of the perfumed suits in terms of the odor intensity and character over time and distance of application: The composition of the liquid perfume remaining on the textiles was estimated from the experimentally measured compositions in the headspace of the impregnated textiles. A flash vapor-liquid equilibrium (VLE) calculation was performed using the UNIFAC method for estimating the activity coefficients in the liquid phase. From that point the diffusion model was applied using an estimated total number of moles of 1.0 mmol and an area of liquid-gas interface of 0.071 m^2. This analysis was performed for each dry washing cycle to evaluate the odor intensity and character of the perceived scents at different distances from the man suit and over time. The odor profiles obtained after the first and third dry washing cycle are shown in Figure 9.

The predicted odor intensity profiles show that, from a very simplistic approach, the top note limonene would be more strongly perceived than the other fragrances after each dry cleaning cycle. Thus, limonene would have higher impact, tenacity, diffusion and volume parameters, though the character of the perceived scent might change as the concentration of the middle and base notes evolve through time and distance. Another important result is that although the perceived scent decreases in intensity after dry cleaning cycles (as expected), its character remains similar. This can be seen in Figure 9: comparison of top and bottom graphics shows that the relative proportion of the ingredients is kept. This is desirable from the consumer point of view, because it is expected that the perfume will maintain the same scent and olfactive family after washing. It should be noted that although convection effects were neglected in the model and the total mass of perfume impregnated in the textile was assumed as small, the predicted odor intensity after 24 h is

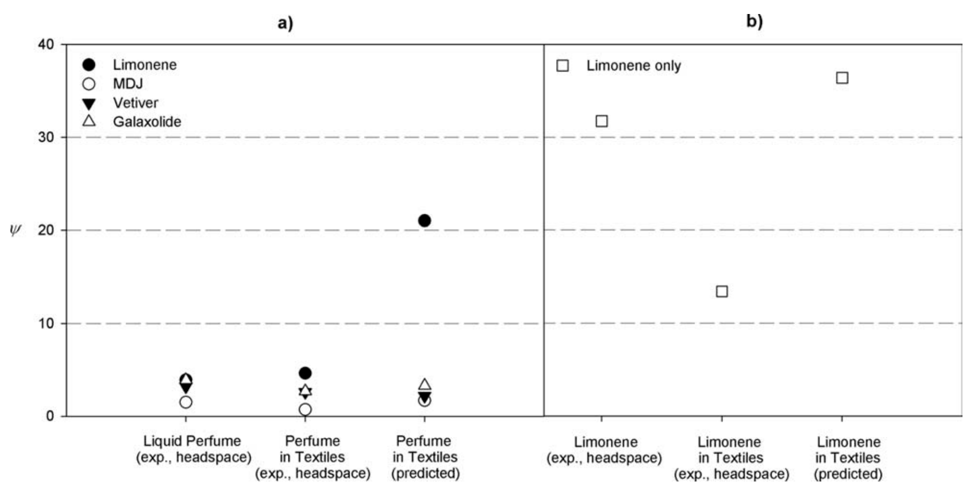

Figure 7. Comparison between the odor intensities of the fragrant species in the headspace: liquid perfume, textiles impregnated with microcapsules, and prediction by Eq. 4.

(a) This work. (b) Previous results with microencapsulated limonene.

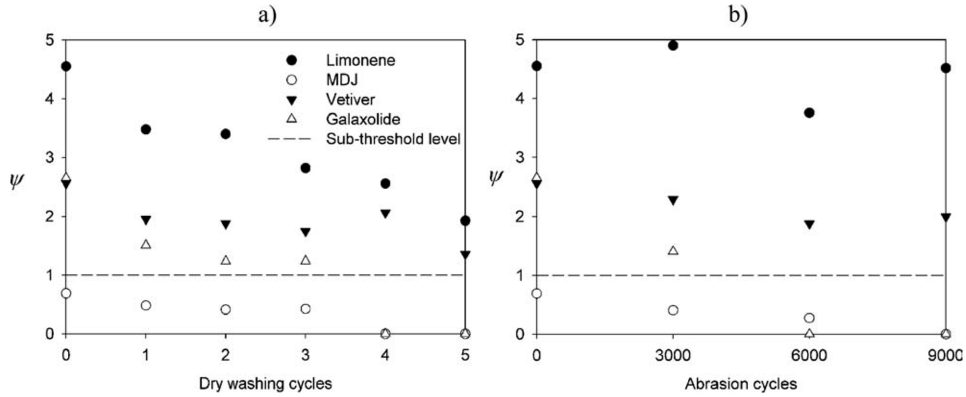

Figure 8. Headspace odor intensity for the perfumed textiles as a function of the number of dry washing cycles (a) and the number of abrasion cycles (b).

still higher than unity for some fragrances. This way, the perfume is expected to be perceived around the people wearing the suit for a considerable time. Nevertheless, it is important to highlight that this evaluation of performance considers only the evaporation and diffusion of the fragrant components from the microcapsules into air, neglecting possible interactions of the fragrances with the textile and between themselves. In this case the releasing process starts by breakage of the microcapsule and then evaporation of the liquid perfume mixture into air. However, the binding of fragrant molecules to textile fibers would reduce the evaporation rate, thus enhancing the performance of the perfumed suit.

Conclusion

In this work different predictive models were combined with experimental methodologies for the development of an added-value product. The performance analyses of perfumed textiles have shown a reduction of the odor intensity of the perfume with the number of dry cleaning and abrasion cycles. However, even after being subjected to these tests, it is possible to detect fragrances released from the suits at suprathreshold concentrations. The prediction of the odor intensity for the headspace of both the perfume and the textiles has shown that limonene was the most strongly perceived

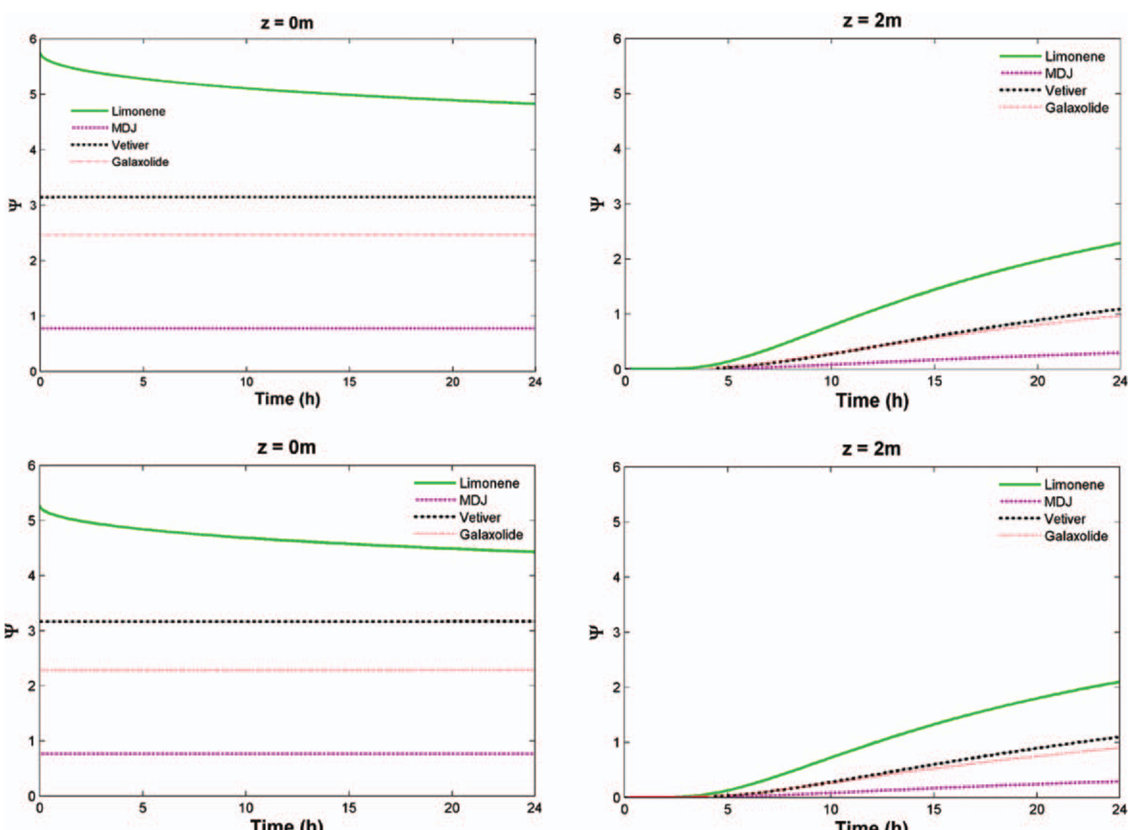

Figure 9. Odor profiles for the perfume concentrate after the first (top) and third (bottom) dry washing cycles: near the point of release (left) and at a distance of 2 m from the source (right).

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

fragrance within the perfume mixture, which was confirmed experimentally. The relative odor intensity of the perfume ingredients predicted with the model was in agreement with the experimental headspace evaluations showing a general trend: Limonene > Galaxolide > Vetiver > MDJ. Moreover, it was observed that although galaxolide was strongly perceived after impregnation, it was less perceived than vetiver after the first dry cleaning or abrasion tests. Moreover, one important parameter to take into account for such evaluations is matrix effects that may retain or push-out fragrance ingredients from the textiles. In sum, this work has presented a performance analysis of fabrics with microencapsulated perfume. The results are relevant for the product design and development of perfumed suits, a valuable alternative for a niche market of fragranced products.

Acknowledgments

This work was carried out in the project SCENTFASHION, contract ADI/2004/M2.3/0015POCI funded by Agência de Inovação (AdI) in the framework of POCI 2010-Medida 2.3-IDEIA. They also acknowledge the support of their partners CITEVE and A Penteadora SA. Oscar Rodríguez acknowledges financial support of Programme Ciência 2007 (FCT). Miguel A. Teixeira acknowledges his Ph.D. grant of FCT (SFRH/BD/37781/2007). Isabel Martins acknowledges her Ph.D. grant of FCT (SFRH/BD/43215/2008) and financial support from LSRE (FEUP/no 424329).

Literature Cited

1. Cussler EC, Moggridge GD.*Chemical Product Design*.Cambridge:-Cambridge University Press, 2001.
2. Wesselingh JA, Kiil S, Vigild ME. *Design & Development of Biological, Chemical, Food and Pharmaceutical Products*. Chichester, UK: Wiley, 2007.
3. Wei J. *Product Engineering. Molecular Structure and Properties*. New York: Oxford University Press, 2007.
4. Ng KM, Gani R, Dam-Johansen K.*Chemical Product Design: Toward a Perspective Through Case Studies*.Amsterdam, The Netherlands:Elsevier, 2007.
5. Costa R, Moggridge GD, Saraiva PM. Chemical product engineering: an emerging paradigm within chemical engineering. *AIChE J*. 2006;52:1976–1986.
6. Cussler EL, Wagner A, Marchal-Heussler L. Designing chemical products requires more knowledge of perception. *AIChE J*. 2010;56:283–288.
7. Hill M. Chemical product engineering-the third paradigm. *Comput Chem Eng*. 2009;33:947–953.
8. Charpentier JC. The triplet "molecular processes-product-process" engineering: the future of chemical engineering? *Chem Eng Sci*. 2002; 57:4667–4690.
9. Rodrigues AE, da Silva FA, Granato M, Lamia N, Grande C, Gomes PS. Propane/propylene separations: from molecular simulation to process development. In: Proceedings of the XXXIII Reunión Ibérica de Adsorción, Madrid, Spain, 2008.
10. Teixeira MA, Rodríguez O, Mata VG, Rodrigues AE. Perfumery quaternary diagrams for engineering perfumes. *AIChE J*. 2009;55:2171–2185.
11. Ulrich KT, Eppinger SD. *Product Design and Development*, 2nd ed. New York: McGraw-Hill, 2000.
12. Seider WD, Seader JD, Lewin DR, Widagdo S. *Product and Process Design Principles: Synthesis, Analysis and Evaluation*, 3rd ed. Asia: Wiley, 2010.
13. Cooper RG. *Winning at New Products: Accelerating the Process from Idea to Launch*, 3 ed. New York: Perseus Publishing, 2001.
14. Bagajewicz M. On the role of microeconomics, multi-scale planning and finances in product design. *AIChE J*. 2007;53:3155–3170.
15. Street C, Woody J, Ardila J, Bagajewicz M. Product design: A case study of slow-release carpet deodorizers/disinfectants. *Ind Eng Chem Res*. 2008;47:1192–1200.
16. Bagajewicz M, Hill S, Robben A, Lopez H, Sanders M, Sposato E, Baade C, Manora S, Coradin JH. Product design in price-competitive markets: a case study of a skin moisturizing lotion. *AICHE J*. 2011;57:160–177.
17. Rodrigues SN, Fernandes I, Martins IM, Mata VG, Barreiro F, Rodrigues AI. Microencapsulation of limonene for textile application. *Ind Eng Chem Res*. 2008;47:4142–4147.
18. Rodrigues SN, Martins IM, Fernandes IP, Gomes PB, Mata VG, Barreiro MF, Rodrigues AE. Scentfashion®: microencapsulated perfumes for textile application. *Chem Eng J*. 2009;149:463–472.
19. Nelson G. Microencapsulation in textile finishing. *Rev Prog Color*. 2001;31:57–64.
20. Soest JJGV. *Encapsulation of fragrances and flavours: a way to control odour and aroma in consumer products*. In: Berger RG, editor. *Flavours and Fragrances: Chemistry, Bioprocessing and Sustainability*. Berlin Heidelberg: Springer-Verlag, 2007.
21. Herrmann A. Controlled release of volatiles under mild reaction conditions: from nature to everyday products. *Angew Chem Int Ed Engl*. 2007;46:5836–5863.
22. Haefliger OP, Jeckelmann N, Ouali L, Leon G. Real-time monitoring of fragrance release from cotton towels by low thermal mass gas chromatography using a longitudinally modulating cryogenic system for headspace sampling and injection. *Anal Chem*. 2010;82:729–737.
23. Wang CX, Chen SL. Anchoring beta-cyclodextrin to retain fragrances on cotton by means of heterobifunctional reactive dyes. *Color Technol*. 2004;120:14–18.
24. Shin Y, Son K, Yoo DI. Development of natural dyed textiles with thermo regulating properties. *Thermochim Acta*. 2010:511:1–7.
25. Specos MMM, García JJ, Tornesello J, Marino P, Vecchia MD, Tesoriero MVD, Hermida LG. Microencapsulated *Citronella* oil for mosquito repellent finishing of cotton textiles. *Trans R Soc Trop Med Hyg*. 2010;104:653–658.
26. Thies C. *A survey of microencapsulation processes*. In: Benita S, Editor. *Microencapsulation: Methods and Industrial Applications*. New York: Marcel Dekker, 1996:1–20.
27. Whitaker DM. Stabilized perfume containing microcapsules and method of preparing same. US Patent 5,051,305. 1991.
28. Kukovic M, Knez E. Process for preparing carries saturated or coated with microencapsulated scents. WO 96/09114. 1996.
29. Soper JC, Kim YK, Thomas MT. Method of encapsulation flavours and fragrances by controlled water transport into microcapsules. US Patent 6,045,835. 2000.
30. Harris L, Blevins L. Process for applying microcapsules to textile materials and products formed by the process. WO 02/090643 A1. 2000.
31. Kleban M, Weisser J, Koch F, Schwaiger W. Leather finished with scent containing microcapsules. US Patent 2002/0198392 A1. 2002.
32. Bochot A, Alphandary H, Duchene D, Fatal E. Microencapsulation systems and applications of same. WO 04/066906 A2. 2004.
33. Ouali L, Benczedi D. Process for producing nano-capsules containing a fragrance. WO 2006/027664 A2. 2006.
34. Calkin R, Jellinek S.*Perfumery: Practice and Principles*.New York:-Wiley, 1994.
35. Teixeira MA, Rodríguez O, Mata VG, Rodrigues AE. The diffusion of perfume mixtures and odor performance. *Chem Eng Sci*. 2009;64:2570–2589.
36. Cortez-Pereira CS, Baby AR, Kaneko TM, Velasco MVR. Sensory approach to measure fragrance intensity on the skin. *J Sensory Stud*. 2009;24:871–901.
37. Carles J. A method of creation in perfumery. *Soap Perfum Cosmet*. 1962;35:328–335.
38. Brechbill GO.*A Reference Book on Fragrance Ingredients*.New Jersey:Creative Endeavor Books, 2006.
39. I-sensis, Lda. CTC-Centro Tecnológico do Calçado, Rua de Fundões, Devesa Velha, 3700-121 S. João da Madeira. Portugal. www.i-sensis.com.
40. van Gemert LJ. *Compilations of Odour Threshold Values in Air, Water and Other Media*. The Netherlands: Oliemans Punter & Partners, 2003.
41. Devos M, Rouault J, Laffort P.*Standardized Olfactory Power Law Exponents*.Dijon, France:Editions Universitaires-Sciences, 2002.
42. Leffingwell JC, Leffingwell D. Available at http://www.leffingwell.-com/odorthre.htm. 2011.
43. Chemspider. Database of Chemical Structures and Property Predictions—Royal Society of Chemistry. Available at http://www.chemspider.com/Default.aspx. 2011.
44. Cain WS, Schiet FT, Olsson MJ, de Wijk RA. Comparison of models of odor interaction. *Chem Senses*. 1995;20:625–637.
45. Laffort P, Dravnieks A. Several models of suprathreshold quantitative olfactory interaction in humans applied to binary, ternary and quaternary mixtures. *Chem Senses*. 1982;7:153–174.

46. Laffort P, Walsh RM, Spillane WJ. Application of the U and Gamma' models in binary sweet taste mixtures. *Chem Senses*. 2002;27:511–520.
47. Olsson MJ. An integrated model of intensity and quality of odor mixtures. *Olf Taste 12*. 1998;855:837–840.
48. Berglund B, Olsson MJ. Odor-intensity interaction in binary and ternary mixtures. *Percept Psychophys.* 1993;53:475–482.
49. Mata VG, Gomes PB, Rodrigues AE. Engineering perfumes. *AICHE J*. 2005;51:2834–2852.
50. Teixeira MA, Rodríguez O, Rodrigues AE. Perfumery radar: a predictive tool for perfume family classification. *Ind Eng Chem Res.* 2010;49:11764–11777.
51. European Norm. Textiles—determination of the slippage resistance of yarns at a seam in woven fabrics—part 2: fixed load method. EN ISO 13936–2. 2004.
52. European Norm. Textiles—determination of the abrasion resistance of fabrics by the Martindale method—part 2: determination of specimen breakdown. EN ISO 12947–2. 1998.
53. Escher SD, Oliveros E. A quantitative study of factors that influence the substantivity of fragrance chemicals on laundered and dried fabrics. *J Am Oil Chem Soc*. 1994;71:31–40.
54. Specos MMM, Escobar G, Marino P, Puggia C, Victoria M, Tesoriero D, Hermida L. Aroma finishing of cotton fabrics by means of microencapsulation techniques. *J Ind Textiles*. 2010;40:13–32.
55. Martel B, Morcellet M, Ruffin D, Vinet F, Weltrowski M. Capture and controlled release of fragrances by CD finished textiles. *J Inclusion Phenom Macrocyclic Chem.* 2002;44:439–442.

Manuscript received Nov. 24, 2010, and revision received May 19, 2011.