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Emerging application of vanillin microcapsules

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1 Introduction

1.1 Vanillin

Vanillin (4-hydroxy-3-methoxybenzaldehyde) is one of the most important aromatic aldehydes and also one of the most-used flavoring agents worldwide. Its functional groups include aldehyde, hydroxyl, and ether. It is the primary component of the extract of the vanilla bean. However, synthetic vanillin is now used more often than natural vanilla extract. Figure 1 shows the chemical structure of vanillin [1] and Table 1 summarizes its main physicochemical properties [2].

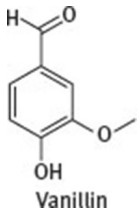

Figure 1: Chemical structure of vanillin.

Table 1 Physicochemical properties of vanillin.

Vanillin physicochemical properties	
Boiling point	170 °C
Molar mass	152.15 g/mol
Water solubility	1 g/100 mL
Vapor pressure	0.01 mmHg at 25 °C
Melting point	81 °C
Flash point	147 °C
Odor threshold [3]	pp l09[1]

Natural vanillin is extracted from vanilla pods, but this production cannot meet market demand alone [4]. Thus, vanillin is also produced industrially, through chemical synthesis, on a scale of more than 10,000 tonnes per year. Chemical synthesis is a well-established approach because it is economical. However, it has many drawbacks, such as the consequent environmental pollution and the lack of substrate selectivity. These factors can reduce process efficiency and increase downstream processing cost [5]. Vanillin production is also not ideal through tissue culture techniques, because plants are slow growing and vanillin biosynthetic pathway is not very actively expressed [6]. Thus, alternative biotechnology-based approaches have been developed for the production of vanillin from lignin, phenolic stilbenes, isoeugenol, eugenol, ferulic acid, or aromatic amino acids and through biosynthesis by applying fungi, bacteria, plant cells, or genetically engineered microorganisms.

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2 Properties and applications

Vanillin is considered to be one of the most appreciated flavor compounds, thus it is widely used for enhancing flavor in the food and beverage industry.

In addition, vanillin also has several bioactive properties [7] such as antimicrobial activities against yeasts, moulds [8] and bacteria [9, 10] and antioxidant properties [11, 12]. Because of this it is also used as a biopreservative.

Furthermore, it also possesses antimutagenic, anticlastogenic and antitumor activities, thus it is an important raw material in the pharmaceutical industries for production of drugs such as aldomet, dopamine, papaverine, and L-DOPA [13].

Finally, among other uses, vanillin is used to manufacture antifoaming agents for lubrication oils, and a great number of household products, deodorants, air fresheners, floor polishes, and herbicides [5, 14].

3 Microencapsulation of vanillin

3.1 Purpose of vanillin microencapsulation

In general, aromatic compounds are instable; they tend to degrade over time and their stability is mainly determined by chemical parameters. However, if they are protected from the environment then degradation could be stopped, or at least slowed down. Thus, the aim of microencapsulating a fragrance is to build a physical barrier between the aromatic molecule and its environment, in order to protect it or control its release. There are five principal reasons for encapsulating perfumes [1]:

- to protect perfume from oxygen in the air to avoid oxidation;
- to avoid hydrolysis;
- to avoid reactions of flavored ingredients with each other;
- to avoid evaporation;
- to convert liquids into powders, making them easier to handle.

In the particular case of vanillin, reported show that it is vulnerable to be degradation by sunlight in air, with a half-life of 4.7 h. Conversely, it is rather stable to hydrolysis in water. Finally, vanillin is readily biodegradable under aerobic conditions and it also degrades rapidly under anaerobic conditions [15].

Additionally, vanillin may be easily volatilized under some food processing conditions (baking, spray-drying, etc.) or – in the case of being added to detergents – it could be easily lost during the laundering operation.

Thus, vanillin microencapsulation could be mainly intended to increase its half-life in aggressive environments and/or to slow down its volatilization by controlling its release.

It is important to point out that vanillin, as with most flavors, is an expensive compound. Avoiding its degradation and controlling its release means that the same aromatic long-term effect could be obtained with the addition of less of the compound, which would lead to money savings for industry.

3.2 Materials and methods

Several materials and different methods for vanillin encapsulation have been reported.

Spray drying has been the most employed technique, possibly because it is the most commonly used microencapsulation method for food ingredients [16]; chitosan has been used as a wall material in order to encapsulate vanillin by this technique [17]. Mixtures of maltodextrin with gum arabic have been also employed in spray-drying processes [18].

However, carnauba wax has been employed as wall material for vanillin encapsulation by means of a melt dispersion technique [19].

Ethylcellulose microcapsules have been obtained by an oil-in-water solvent evaporation method. In addition, these were dip-coated by chitosan and the coating was crosslinked with nontoxic 1,2,3,4-butanetratecarboxylic acid. This modification was shown to improve the vanillin release [20].

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Moreover, photosensitive microcapsules have been prepared by using a phase inversion technique. In this case the wall material was based on poly (α-methylstilbenesebacoate-co-α-methylstilbeneisophthalate), containing the photosensitive α-methylstilbene moiety [21].

Finally, this chapter is mainly focused on summarizing the work done within our research group on polysulfone/vanillin microcapsules prepared by a phase inversion precipitation technique [22].

4 Applications

Applications of vanillin microcapsules are found in different fields. Their main application is in the food industry, followed by cosmetics and household products.

Because it is a well known and widely used aromatic molecule, vanillin is also used in research as a reference compound for determining the encapsulation capacity of several preparations [21–23].

Finally, the encapsulation of vanillin and its controlled release offer potential applications for textile products. The antibacterial and aromatic properties of vanillin could be used in several fabric products [17, 24].

5 Polysulfone/vanillin microcapsules

5.1 Introduction

Polysulfone is one of the most employed polymers for the preparation of flat membranes and microcapsules, owing to its excellent chemical properties together with its thermal and mechanical resistance [25, 26]. In addition, it is a biocompatible polymer and thus can be used for medical applications [27–31].

Among applications of polysulfone capsules, special mention has to be given to their use for the elimination of toxic compounds from residual water [25], and the production of medical materials [32]. Nevertheless, the interest of this chapter is focused on the entrapment and controlled release of vanillin.

5.2 Preparation methods

The most-used methods for polysulfone microcapsule production are solvent evaporation [33] and phase inversion precipitation, with the latter method more common because capsule preparation is easy and fast [25, 34–38]. Phase inversion precipitation methods – immersion in liquid non-solvent or contact with vapor non-solvent – are described below.

5.3 Materials

For preparing microcapsules by phase inversion precipitation, at least three compounds are required: the polymer, the solvent and a non-solvent. As has been mentioned, polysulfone (Sigma Aldrich, St. Louis, MO, USA) was used as wall material because it is a polymer with excellent properties. Water has been the non-solvent used. As core material, vanillin was acquired from Sigma-Aldrich S.A., with purity > 99 %.

Solvent needs to be miscible with water and able to dissolve polysulfone. In that sense, dimethylformamide (DMF; scharlab > 99.8 %) was selected from a list of compatible solvents [39]. The reasons why more effective solvents were discarded lay in the fact that they were not completely miscible with water and/or they were flammable and thus, could not be used for safety reasons.

5.4 Methods

Phase inversion precipitation was used for the preparation of the capsules. This method is based on the interaction of at least three compounds – the polymer, a solvent in which the polymer is soluble and another substance, miscible with the solvent and in which the polymer is not soluble.

Mainly, it consists in contacting a polymeric solution (composed by the polymer, the solvent and the core material if required) either with a liquid or a vapor containing a non-solvent. This contact causes a change in the composition of the polymeric solution. By diffusion of the substances, the non-solvent concentration

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increases in the solution, together with the decrease of solvent concentration, and this leads the solution to a thermodynamically unstable state. When the solution is thermodynamically unstable, it spontaneously splits up into two different phases: in one of the phases polymer concentration is high (rich phase), whereas in the other one it is very low (poor phase). This process is known as liquid-liquid demixing. The rich in polymer phase precipitates, leading to the production of a solid membrane. The poor in polymer phase nuclei are responsible for pore formation in the membrane. After polymer precipitation, membrane morphology remains defined and invariable. Thus, membrane morphology is determined by the liquid-liquid demixing process. In the case of the ternary system used (PSf, DMF, water), when polymeric solution gets in contact with water, liquid-liquid demixing is fast and a solid structure is obtained in less than a second.

The first step in the production of microcapsules by the phase inversion precipitation is the preparation of a polymeric solution. It is obtained by solving the polymer in a suitable solvent, usually in a concentration range between 10 wt % and 30 wt %. This solution needs to be stirred for long enough to get the polymer completely dissolved in this case a 15 wt % polysulfone solution in DMF was used, which required stirring for 24 h. In addition, the bottles in which the solution is kept need to be hermetically closed, in order to avoid contact with atmospheric air, which contains moisture that would lead to premature precipitation of the polymer. In the case of the polysulfone/vanillin capsules herein described, the solution also contained a 10 wt % of vanillin, thus, bottles needed to be protected from light exposure and so amber bottles were preferred.

The second step is to disperse the solution into microdroplets, in order to consequently obtain microcapsules constituted by a spherical polysulfone membrane; atomization is used as the dispersion technique.

Two different atomization setup configurations have been reported for the preparation of polysulfone/-vanillin microcapsules (see Figure 2). In Figure 2(a) precipitation is caused by the immersion in a precipitation bath containing a liquid non-solvent. The airbrush is positioned so that the outlet flow is perpendicular to the surface of the precipitation bath. When the polymeric solution microdroplets fall in the precipitation bath, microcapsules immediately floated on the surface and could be collected by filtration. In this chapter these capsules are referred to as IPS (immersion phase separation).

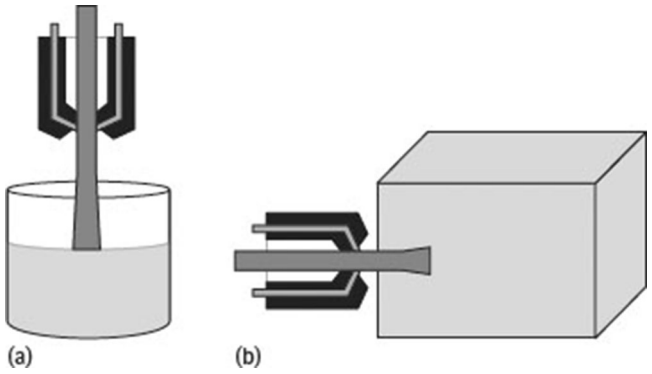

Figure 2: Scheme of atomization setup for capsules production by phase inversion: (a) by immersion in liquid non solvent (IPS), (b) by contact with non solvent vapor (VIPS).

In Figure 2(b) precipitation is caused by the contact with vapor non-solvent inside a chamber. When water is the non-solvent the recommended relative humidity inside the chamber is 95% at 20 °C. When the walls of the chamber are covered with microcapsules production should be stopped. The product is collected using a spatula. These capsules are identified in this chapter as VIPS (vapor induced phase separation).

6 Characterization

Polysulfone/vanillin microcapsules have been characterized using several techniques.

6.1 Scanning electron microscopy

Scanning electron microscopy (SEM), produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition.

In Figure 3 we can see surface images of microcapsules prepared by immersion precipitation or vapor non-solvent. In order to be observed by SEM, samples need to be electron conductive, thus the first step is to cover the samples with gold. The images on Figure 3 were obtained using a Jeol JSM-6400 working at 15–20 kV [40].

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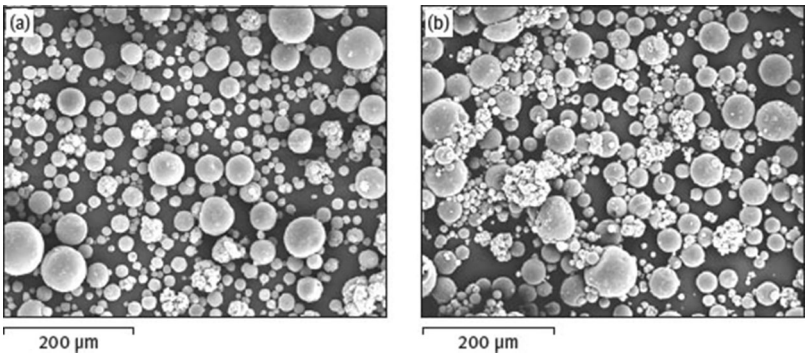

Figure 3: Scanning electron microscope (SEM) micrographies of capsules obtained: (a) by IPS, (b) by VIPS.

Images like Figure 3 were analyzed at low magnifications using image-J® software in order to determine the mean size and size distribution of the samples. Table 2 shows the results obtained from measuring 500 capsules of each type. As can be observed, significant differences in size and size distribution between both preparations were not reported. This is explained by the fact that the size of the capsules is mainly determined not by the precipitation method but by the dispersion step [40].

Table 2 Mean size and size distribution of capsules prepared by IPS and by VIPS.

	Immersion precipitation	vapor-induced precipitation
Mean diameter (µm)	21.00	24.04
Standard deviation (µm)	12.62	16.10
Maximum diameter (µm)	83.32	95.23
Minimum diameter (µm)	6.17	4.71

In addition, if characterization of the capsules cross-section is required, capsules need to be cut. It has been found that cryogenic breaking is the best technique for cutting the capsules [41]. Figure 4 shows images of polysulfone/vanillin microcapsules obtained by both described methods. In order to obtain these images, capsules were mixed with a freezing medium (Jung Tisseu Freezing Medium, Leica Instrumental) and then immersed in liquid nitrogen. This media freezes fast and generates a matrix that keeps the capsules entrapped. Then frozen samples were introduced inside a cryochamber (Leica CM1850) at – 22 °C and they were cut with a blade in slides of 15 µm thickness. Slides were place on a microscope glass and covered with gold for SEM observation [40, 41].

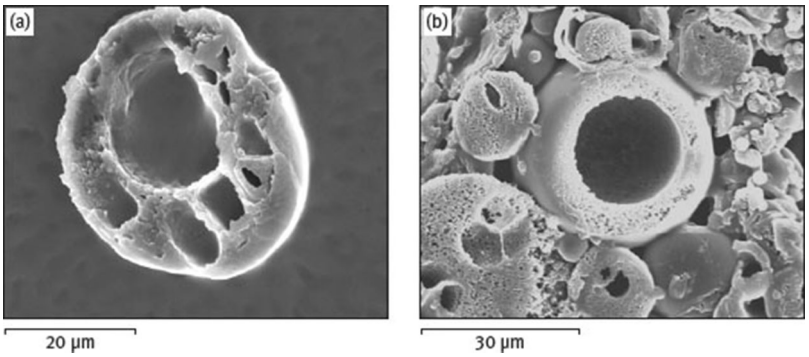

Figure 4: Cross section images of capsules prepared by: (a) IPS, (b) VIPS.

From Figure 4 it can be observed that microcapsules prepared by VIPS showed a uniform sponge-like structure, whereas on capsules prepared by VIPS very big pores (macrovoids) appeared. Differences on membrane cross-sections lay on the basis of the different precipitation techniques. More detailed information about liquid-liquid demixing processes in phase inversion can be found in the literature [40].

6.2 High performance liquid chromatography

Due to its versatility high performance liquid chromatography (HPLC) is one of the most employed analysis techniques based on separation, which allows its use in several fields.

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The technique is based on the selective retention of the compounds in a sample when they flow through a column. Thus, components are solved in a suitable solvent (mobile phase) and they are forced (by applying high pressures) through the chromatographic column which contains a filling (stationary phase) that is able to selectively retain the different compounds in the sample. Because of this retention, different compounds flow out of the column separately and at different times, according to its retention time. In this way, a chromatogram is obtained, which allows the determination and quantification of the compounds in the sample.

In the work herein described, this technique was used to determine vanillin and DMF concentration in an aqueous media. The equipment used was an Agilent 1100 with photodiode array detector. The column used was a Supelcosil LC-8 (SUPELCO). The mobile phase was 80:20 water:acetonitrile. For all analyzes the flow rate was set at 1 mL/min, the column temperature was set at 40 °C, the analysis time was 8 min and the injection volume was 4 μL. Vanillin and DMF concentrations were determined at 229 nm, showing a typical retention time of 4.2–4.5 min and 2.0–2.2 min, respectively [42–44].

For the vanillin release experiments the medium was composed of 100 mL of distilled water in which 1 g of microcapsules was added. The preparation was stirred at 700 rpm for 72 h. Samples of 0.5 mL were taken from the release medium periodically and hermetically stored until analyzed by HPLC. Figure 5 shows the release results obtained for both types of microcapsules and the total mg of vanillin released to the medium in time, together with the percentage of vanillin released (related to the maximum amount released at the end of the experiment).

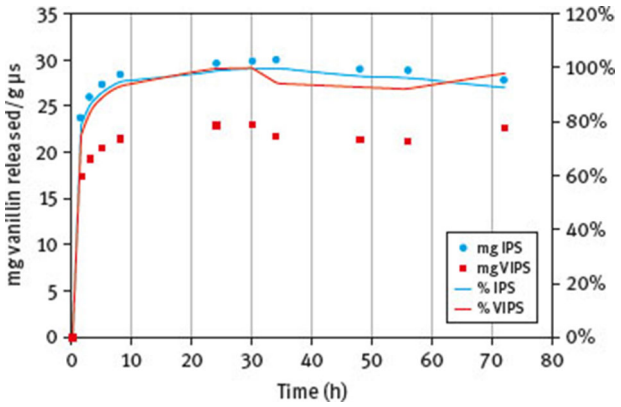

Figure 5: Vanillin release from PSf/vanillin microcapsules.

As can be observed, in both preparations the release was fast during the first 10 h of experiment but slowed down until it reached a plateau. This release tendency for IPS capsules has been observed in several studies [22, 42]. These results showed that both preparations have similar permeability to vanillin (because the shape of the curve is the same). Conversely, although it may seem that microcapsules prepared by the VIPS methodology had encapsulated less vanillin, this is not necessarily true. It has been demonstrated in previous studies [40] that this difference was the result of higher amounts of DMF encapsulated in capsules prepared by VIPS, which affected their density.

6.3 Nitrogen adsorption/desorption analysis

Pore size distribution in the range of mesopores (from 2 to 50 nm) was determined by N_2 gas adsorption–desorption analysis. Prior to adsorption measurements, the samples were degassed under vacuum for 12 h at 50 °C. Afterwards they were analyzed at 77 K with a Quadrasorb SI device [45]. Samples of microcapsules, taken before and after release experiments underwent N_2 adsorption/desorption analysis. Results are shown in Figure 6.

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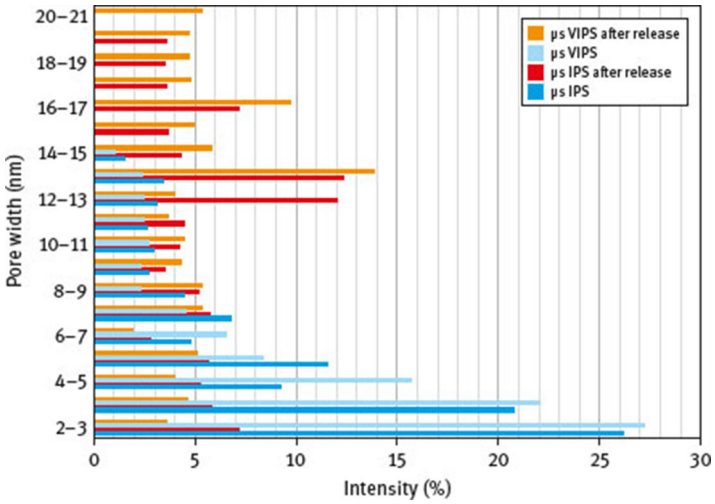

Figure 6: Mesopore size distribution in microcapsules samples.

Significant differences between the two types of capsules were not detected. In both, before the release, the higher density was identified for pores in the range 2–6 nm. There were no pores over 15 nm. However, after the release, intensity of pores under 6 nm decreased, while larger pores were detected in the range between 12–20 nm. Thus, it is possible that many of the pores under 6 nm were, in fact, not small pores but larger ones partially blocked by vanillin, as determined in previous studies [45]. Thus, the precipitation technique is not affecting the mesopore size distribution.

6.4 Differential scanning calorimetry

In order to determine the effect of temperature on polysulfone/vanillin microcapsules, calorimetric curves were obtained by using a Mettler-Toledo 822 Differential Scanning Calorimetry (DSC) (Mettler-Toledo Inc., Schwerzenbach, Switzerland). DSC curves were obtained in a nitrogen atmosphere at 10 °C/min heating rate. The pan used was a 40 μL aluminium sealed crucible. Samples weight was approximately 8 mg. Capsules behavior in the range 20–100 °C was assessed. In addition, pure polysulfone and vanillin were analyzed.

As can be observed in Figure 7, the polymer did not suffer any changes in the range of temperatures assessed, because its glass transition temperature is found at 185 °C.

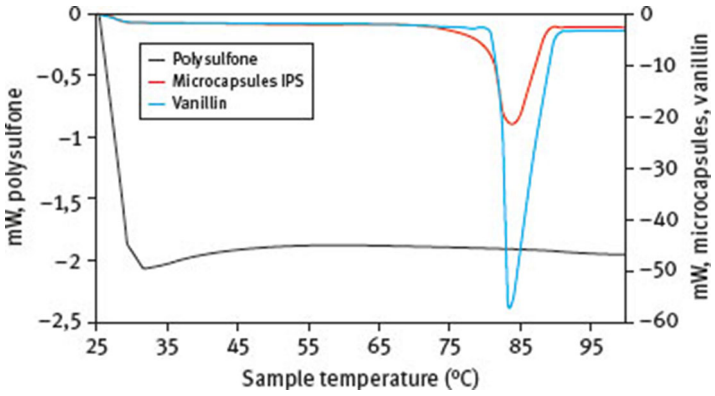

Figure 7: DSC curves for vanillin, polysulfone and microcapsules.

This means that the wall of the capsules can resist temperatures considerably higher. Conversely, an endothermic peak was encountered for vanillin at 85 °C. This peak may corresponds to the melting of the compound, which occurs at 81.5 °C [2]. The same peak was appreciated in PSf/vanillin capsules, which again demonstrated the presence of vanillin in the capsules.

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7 Antibacterial and aromatic finishing of fabrics

7.1 Introduction

As mentioned above, vanillin possesses several bioactive properties, such as antimicrobial activity. In particular, in this work the attention was focused on the inhibitory effect of vanillin against the growth of *Staphylococcus aureus* [13], which is one of the most common bacteria in postsurgical wound infections, causing several diseases ranging from mild to severe [46].

7.2 Antibacterial activity

Vanillin had previously shown significant inhibitory activities against *S. aureus* [13]. Thus, research was focused to determine if PSF/vanillin microcapsules could maintain this activity. A modified agar-well diffusion technique was employed [47] and petri dishes containing 15 mL of nutrient agar were filled with a mixture of 100 μL of standard 108 colony forming unit (CFU)/mL *S. aureus.* Different holes of 5 mm diameter were made, and they were filled with:

- Different solutions of vanillin in ethanol at 1.25 wt %, 2.5 wt %, 5 wt %, 10 wt %.
- Solid vanillin, 0.1 g.
- PSf/vanillin microcapsules prepared by IPS, 0.1 g.
- Ultra pure water and ethanol as control solutions.

The inhibitory activity of vanillin against *S. aureus* was tested by incubating the plates for 1 week at 37 °C. A clear zone around the holes indicated the inhibitory effect. Figure 8 shows images obtained one day after the beginning of the experiment.

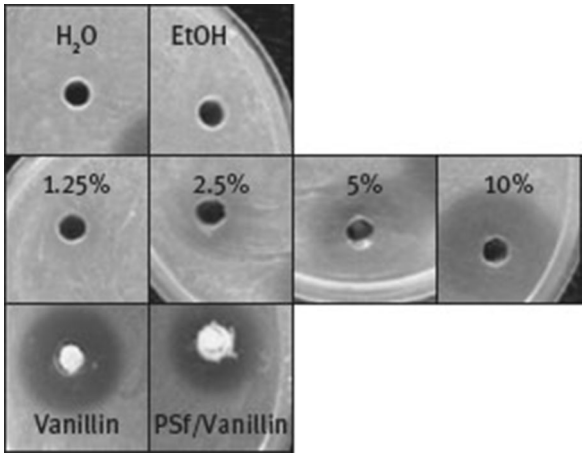

Figure 8: Inhibitory activity of vanillin against *S. aureus*.

Significant inhibitory effects could be observed in the most concentrated vanillin/ethanol solutions. Similar results had been previously reported [13]. This is logical because dissolved vanillin is able to diffuse through the cultivation medium. However, the most interesting results were that significant inhibitions were also observed when vanillin was added in a solid state, or even in the encapsulated product. Although inhibition diameters in the solid products were shorter than in the ethanol solutions, this could be explained by the fact that when vanillin was in solid state its diffusion to the medium was hindered. According to the published results [24], the inhibition diameter remain constant for at least 1 week, which means that PSf/vanillin microcapsules inhibited *S. aureus* for at least 1 week, showing promising results for the inhibition of the bacteria growth.

7.3 Microcapsules adhesion to fabrics

Microcapsules prepared by IPS, which had shown to have an antibacterial effect, were incorporated in cotton fabric samples. For this purpose, the first step was to coat the fabric samples with Seitex 100 using a casting knife,

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which provides coatings of uniform thickness (50 μm) pushed by an applicator working at constant velocity (K-Pain applicator, UK).

The second step was to place a layer of capsules on a tray.

Finally, fabric samples were deposited over the tray, allowing their coated side to be in contact with the capsules while exerting a slight pressure in order to facilitate the adhesion of the capsules to the coating. This procedure ensured fixing the maximum number of capsules per area.

The durability of the adhesion was tested by exposing the fabrics to several washing cycles. The experiments were conducted in a commercial washing machine (Bosch Maxx WFO-2063). According to ASTM D2960-05 (standard test method of controlled laundering test using naturally soiled fabrics and household appliances), the load weight was fixed to 3 kg. The laundry program was intended to be as close as possible to the standard normal home laundry test conditions fixed by the American Association of Textile Chemists and Colorists. Thus, washing time was 15 min, followed by rinsing for 10 min and finally spinning for 10 min. The pieces of fabrics were weighted and observed by SEM before and after each washing in order to determine the weight loss and the number of capsules per area.

Figure 9 shows images of the fabric with capsules added, before the laundry and after the first, second, third, fourth and fifth washing cycles. It can be observed that the density of capsules suffered an important decrease after the second washing cycle.

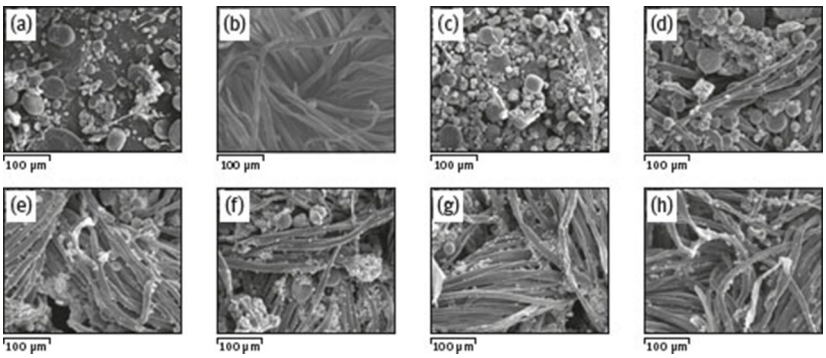

Figure 9: Scanning electron microscope (SEM) images from (a) microcapsules, (b) fabrics, (c) fabrics with microcapsules before washing, (d) fabrics with microcapsules after first washing, (e) after second washing, (f) after third washing, (g) after fourth washing, (h) after fifth washing.

Although several rounded particles were observed (Figure 9 f, h, g) they were not PSf/vanillin microcapsules but were zeolites from the detergent, as was demonstrated by elemental analysis and justified in an already published study [24].

As can be observed, the number of capsules decreased every washing cycle (see Table 3, extracted from a previous study [24]). However, an interesting fact was that smaller capsules seemed to resist more washing cycles.

Table 3 Amount of capsules and diameter range after each washing cycle.

Microcapsules/cm^2	Diameter range (μm)	Washing cycles
110000 ± 18000	2.5–40	0
79000 ± 18000	2.5–28	1
14000 ± 1100	2.5–24	2
12000 ± 4900	2.5–28	3
2900 ± 460	2.5–17	4
1900 ± 460	2.5–13	5

This observation makes us think that maybe durability of the adhesions could be improved by adjusting the diameter of the microcapsules to 10 μm. However, more precise nozzles were required and this was out of the scope of the work.

The relation between the amount of capsules encountered and weight loss is shown in Figure 10.

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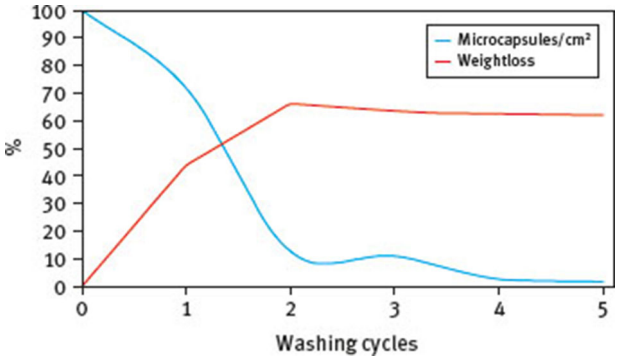

Figure 10: Relation between microcapsules/cm^2 encountered and the weight loss measured after every washing cycle.

The most loss of weigh happened during the second washing cycle. After that the weight lost reached a plateau. However, in the case of capsules, even more of them were lost during the first and second washing cycles, and they were still being lost along all the experimentation. It is comprehensible that the loss of capsules did not affect significantly the weight of the samples, due to its low density (144 kg/m^3). In addition, zeolites were being attached to the fabrics. However, it was interpreted that the main weight changes were due to the binder, Seitex 100, which was used to attach the capsules. According to that, most of the binder would be lost in the first and second washing cycles, but that a thin layer (in direct contact with the textile) probably remained, maintaining some capsules stuck to the fabrics [24].

7.4 Aroma durability

A small survey was conducted in order to assess the perception of vanillin aroma and its durability in the fabrics coated with vanillin microcapsules. A population sample of three volunteers were asked to rate the aroma intensity for each piece of fabric. Thus, reproducibility and inclusion of different smelling sensitivities was assured. The volunteers smelled and rated their perception of vanillin aroma before and after each washing cycle.

The experimental design included performing five washing cycles. From every cycle, five different pieces of fabrics were assessed. Thus, variables in the study were: the observer (persons 1–3), the aroma intensity (not detecting aroma = 0, detecting = 1, strong detection = 2), the washing cycle (1–5) and the aroma detection (yes = 1, no = 0).

The two main hypotheses of the survey were:

- All the observers perceive the same aroma intensity.
- Washing cycles influence aroma intensity.

These hypotheses validated their statistical significance for a confidence interval of 95% against their null hypotheses. The statistical analysis and hypothesis contrast were performed by using the statistical software JMP® Pro 9.0.3 from SAS Institute Inc. (Cary, NC, USA).

Finally, a model was constructed that was able to predict the probability of smelling vanillin in fabrics after being subjected to different number of washings.

Before the first washing cycle, all the observers agreed that all the samples released a strong vanillin aroma, thus scoring a value of 2 in terms of aroma intensity. More details about the statistical analysis can be found in the published paper [24]. However, its main conclusions were:

- There existed differences in the aroma perception among the three observers. Thus, survey data was recorded from people with different olfactory sensibilities, which gives robustness to the modeling done.
- There was a statistically significant correlation between the perfume release of a fabric and the number of washing cycles. Validation of this hypothesis allowed building a model in order to quantify this relationship.
- A model was built using the number of washing cycles as the factor, and the aroma detection as the response variable.
- The model was validated against the experimental data and – as can be observed in Figure 11 – it fitted well with the sample data. A black line plots the model fit equation that represents the whole population, while a gray line plots the data obtained from the survey sample.

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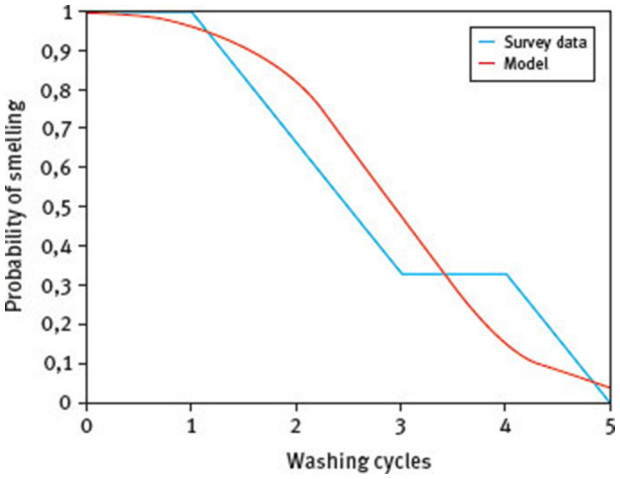

Figure 11: Validation of the model against survey data.

This model was useful because it allowed the determination of the probability to maintain fabrics perfumed for any washing time. Finally, according to the validated model, we could predict that the probability of maintaining the aroma after two washing cycles was 82%, while after the third and the fourth washing cycles the probability was only 48% and 15%, respectively.

8 Conclusions

In conclusion, polysulfone/vanillin microcapsules can be effectively produced by phase inversion precipitation. Two different approaches – IPS and VIPS – have shown to be successful for this purpose when using DMF as solvent and water as non-solvent. Vanillin encapsulation was a challenge because it is a polar compound, which could easily leak to the water phase. However, the fast precipitation of the polymer succeeded in entrapping the flavor. In addition, the performance of both products, in terms of vanillin release, was similar. Presence of vanillin in the capsules has been demonstrated by DSC and HPLC analysis.

Conversely, SEM analysis showed that the products had a different cross-section structure. Macrovoids appeared in microcapsules produced by IPS, whereas these large pores were not found in capsules prepared by VIPS.

After having successfully prepared and characterized polysulfone/vanillin microcapsules, the objective of the research was to use them for providing fabrics of antimicrobial and aromatic properties through a microcapsules coating. Thus it was neceaasry to design and assess a suitable method.

First of all, antimicrobial activity of the capsules against *S. aureus* was confirmed. Capsules were shown to inhibit the growth of the bacteria for at least 1 week.

Afterwards, microcapsules were incorporated to 100% cotton fabric samples, which underwent several washing cycles in a conventional washing machine. Resistance of the adhesion and durability of the aroma were investigated.

Over 50% of the capsules were lost during the first and second washing cycles; however, smaller capsules (around 10 μm) resisted more washing cycles. In fact, they were still being encountered after the fifth washing.

Aroma durability was determined by a perception survey, which concluded that there was a correspondence between durability of the aroma and the washing cycle. A model was built and validated in order to predict the probability of maintaining the aroma after different washing cycles.

The studies in the literature that have been described in this chapter set the basis for further development of fabrics with antimicrobial activity and pleasant aroma finishing based on polysulfone/vanillin capsules. Further work in this area should be focused on narrowing the size distribution of the capsules and improving their adhesion to the fabrics.

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Notes

1 Parts of compound in 10^9 parts of water (volume/volume).

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