P-009	**Perfume release from PSf capsules**	

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INTRODUCTION AND OBJECTIVES

Nowadays, perfume encapsulation technology is employed in the industrial process of textile detergents and softeners. It is used with the aim to preserve the fragrance until the final use and to provide a long lasting fragrance release. However, certain industrial microcapsules present some problems, such as: low material stability, low perfume encapsulation capacity and, in addition, it is not possible to encapsulate hydrophilic perfumes (Peña 2011). With the aim to solve these problems polysulfone (PSf) capsules were proposed.

PSf macrocapsules containing vanillin, a hydrophilic component commonly used in perfume and cosmetic formulations, have been previously investigated (Peña 2009). They showed promising results related to material stability, high encapsulation capacity and long lasting perfume release. However, in order to fulfill with commercial applications, capsules need to be obtained at micro-scale rather than at macro-scale. Therefore, the aim of this investigation is to determine if PSf macrocapsules may be scaled down and maintain the same properties than that presented at macro-scale and, hence confirm, their suitability for textile detergents and softeners products.

MATERIALS AND METHODS

Materials

Polysulfone (PSf) and vanillin reagentPlus® 99% were purchased from Sigma-Aldrich (Spain). N,N-Dimethylformamide (DMF) reagent grade ACS ISO and Acetonitrile multisolvent® HPLC grade ACS UV-VIS were obtained from Scharlau (Spain). MilliQ water was used for all experiments.

Microcapsules preparation

PSf microcapsules were prepared from a polymeric solution by dissolving 15% w/w of PSf and 10% w/w of vanillin in DMF. The mixture was stirred at 500 rpm during 24 h at room temperature. PSf microcapsules were obtained by phase inversion precipitation by atomization. The polymeric solution was atomized into a coagulation bath containing 200 mL of milliQ water. Micro-droplets precipitation took place, because of an exchange of water and DMF, which leads to vanillin encapsulation. Finally, PSf microcapsules were collected by filtration.

Morphological characterization of microcapsules

Morphological characterization of PSf microcapsules was analysed by Scanning Electron Microscopy SEM (JEOL JSM-6400 Scanning Microscopy Series), with an acceleration voltage of 15 – 20 KV.

Release experiments

Release experiments were performed adding 1g of PSf microcapsules into 80 ml of MilliQ water. Capsules were stirred during at least 96 h at 700 rpm in a SBS multipoint magnetic stirrer, Spain. The working temperature for all the experiments was 20ºC. Table 1 shows the experiment working conditions. Release medium samples of 1 ml were periodically withdrawn and hermetically stored until the quantitative analysis.

Table 1 : Experimental parameters

Experiment	Release medium	Capsules Weight	Time
Release experiment	MiliQ water	1 ± 0.01 g	96 h
Vanillin solution treatment	Vanillin saturated solution	1 ± 0.01 g	96 h
Release experiment after vanillin treatment	MiliQ water	1 ± 0.05 g	96 h

Encapsulation capacity

Perfume encapsulation capacity of PSf microcapsules was determined measuring the vanillin remaining in a coagulation bath (200 ml of millQ water) after capsule precipitation from 20 ml of polymeric solution. Encapsulation capacity was obtained as the difference between the vanillin added to the polymeric solution and the vanillin detected in the precipitation water bath.

Analytical techniques

The concentration of vanillin in the release medium samples was determined by High-performance liquid chromatography (HPLC) using an Agilent 1100 chromatograph with photodiode array detector. The column used was a supelcosil LC-8 (SUPELCO). The mobile phase was 80:20 water:acetonitrile. Vanillin concentration was determined at 229 nm, showing a typical retention time of 4.2-4.5 min.

RESULTS AND DISCUSSION

Macro and microcapsules comparison

In order to prove whether PSf macrocapsules performance may be extrapolated to microcapsules they both have been compared in terms of morphology, release be-

havior and encapsulation capacity. Figure 1 shows SEM micrographs of the PSf micro and macrocapsules.

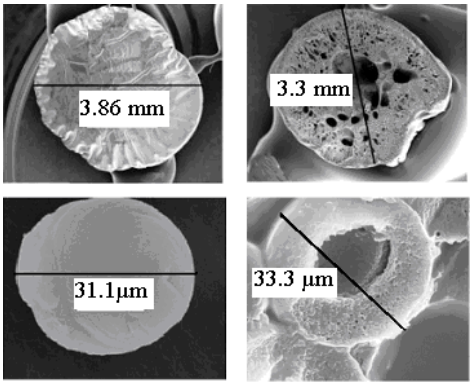

Figure 1 : SEM micrographs. Top images: PSf macrocapsules (Peña 2009). Bottom image: PSf microcapsules.

SEM micrographs show that PSf microcapsules present diameters around 30 µm and a well-defined spherical shape while PSf macrocapsules show diameters around 3 mm and a lentil shape. Cross-section micrographs show, in both capsules, an empty space that ensures a significant volume for perfume encapsulation. Previous investigations showed that PSf capsules present a good physical stability (Peña 2011).

Figure 2 shows the release of vanillin from PSf microcapsules. Vanillin is rapidly released during the first 10 hours of experiment. Afterwards, from 10 to 30 h, a plateau is reached. Similar release tendencies were also observed in previous works with PSf macrocapsules (Peña 2009).

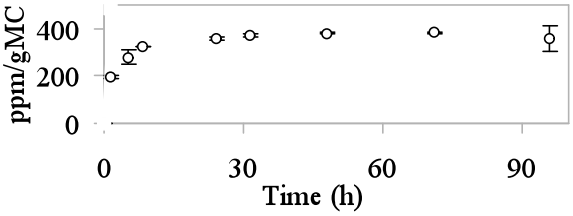

Figure 2: Vanillin release form PSf microcapsules

In another hand, it was detected that not only vanillin was encapsulated but also DMF as well. In order to remove the DMF from PSf capsules, capsules were stored in a vanillin-saturated solution, as Table 1 shows. After vanillin treatment, capsules were recovered an analysed according Table 1. Table 2 shows the results.

As can be seen in Table 2, DMF was successfully removed from PSf microcapsules. The encapsulation capacity of PSf microcapsules was found to be around 45% of the total perfume added to the polymeric solution. Similar results were encountering in PSf macrocapsules (Peña 2009).

Table 2 : PSf macro and microcapsules comparison

	PSf macrocapsules	PSf microcapsules
Encapsulation capacity of vanillin	> 50 %	45%
Perfume release	96 h	96 h
DMF removed after vanillin treatment	99 %	100 %
Vanillin added after vanillin treatment	-30 % (lost)	Around 50 % (added)

PSf microcapsules presented similar results to PSf macrocapsules according to encapsulation capacity, release time and percent of DMF removed after vanillin treatment. However, during vanillin treatment nearly 30% of vanillin was lost in PSf macrocapsules. This effect was completely contrary in PSf microcapsules, in which the amount of vanillin is greatly increased after vanillin treatment. This difference could be attributed to the capsule size. Lower capsule diameter leads to lower amount of DMF encapsulated and this fact decreases the solubility of vanillin in DMF. Vanillin is highly soluble in DMF. Hence, in the case of macrocapsule formation, certain part of the encapsulated vanillin was solubilized into DMF also encapsulated. Afterwards, the vanillin solubilized in DMF was released together with the DMF to the bulk solution media.

In PSf microcapsules the amount of DMF encapsulated was not enough to solubilize part of the vanillin encapsulated. Therefore it was not released togheter with the DMF. In addition, as PSf microcapsules present a large amount of micro-pores, the vanillin from the vanillin solution could be trapped into the pores when it enters in contact with the microcapsule

CONCLUSIONS

PSf microcapsules containing vanillin were prepared successfully by phase inversion precipitation technique. PSf microcapsules not only show the same adventages of PSf macrocapsules, but also they can incorporate new vanillin after capsule formation if conveniently treated. Therfore PSf microcapsules present promising results to be used in textile detergents and softheners.

REFERENCES

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