

## SYNTHESIS OF NANOCAPSULES CONTAINING ALMOND OIL BY MINIEMULSION POLYMERIZATION

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### Introduction

The situation of the shoe industry today makes it necessary for new materials and concepts to be found, to be used as differentiating elements against competitors and that make products stronger in terms of quality, personal hygiene and safety or respect for the environment. In this sense, microencapsulation presents a new option for the shoe industry as its application can transform traditionally used materials or products into smart materials or products capable of interacting with feet. They can improve quality of life by incorporating different therapeutic products for foot care such as essential oils properly dosed. The microencapsulation of active substances to be incorporated in different footwear components in order to obtain an “active shoe” presents an opening up of a new way of innovation.

Actually, in this study polystyrene nanocapsules containing almond oil were synthesized by a direct miniemulsion polymerization process (O/W) to be applied to footwear materials (lining, insoles, etc.). Sweet almond oil is a natural moisturizing agent very often used in the cosmetic industry due to its high content of essential oils content such as oleic and linoleic oils. Because it is not greasy, it is absorbed quickly. In this case, the nanocapsule can act as nanocontainer in order to retain the liquid and therefore to prevent it from leaking into the continuous phase. In addition, nanocapsules formation improves the oil stability, reduces its evaporation rate and it allow a release control when are applied on a substrate.

Nanocapsules are generally considered as spherical, hollow structures with an average diameter of less than 1  $\mu\text{m}$ . Typically, the capsule consists of a polymeric wall with a thickness in the nanometer region, filled with an oil. To enable a stable dispersion, the capsule is stabilized by surface charges or by absorption of an amphiphile. The approach to synthesize nanocapsules is based on the principle of miniemulsion using the differences of interfacial tension and the phase separation process during polymerization to obtain a nanocapsule morphology. A miniemulsion polymerization is described that yields an encapsulation of a nonsolvent hydrocarbon by the polymer being formed. Using this process, it is possible to prepare latex particles having voids with facile control of the particle diameter, void fraction and structure. The process initially involves polymerizing a monomer in a dispersed hydrocarbon-monomer mixture which phase-separates during the polymerization. This phase-separated polymer subsequently serves as a locus for polymerization. The morphology of the demixing structure is determined by the type of surfactant chosen, the polarity of the monomer, and the choice of hydrophobe.

In this paper, different monomer/almond oil ratios as well as different amounts of the surfactant (SDS) were applied to obtained nanocapsules and their effects on the morphology of capsules were investigated by different experimental techniques for footwear applications.

### Experimental

Polystyrene nanocapsules containing almond oil were prepared by the convenient one-step direct miniemulsion (O/W) polymerization. Aqueous miniemulsion was obtained by mixing

monomer (styrene), almond oil and initiator (2,2'-azobis(2-methylbutyronitrile)), and then adding the solution to an aqueous solution of surfactant (SDS) and water, followed by ultrasonification. The polymerization was started by heating to 72 °C and kept at this temperature overnight. After polymerization, the samples were freeze-dried.

After that, the miniemulsions and nanocapsules obtained were analysed by different experimental techniques: The solid content was measured gravimetrically and the average particle size by means of dynamic light scattering (DLS). For morphological characterization, transmission electron microscopy (TEM) was carried out. The average molecular weights of the polymers were determined by gel permeation chromatography. Chemical properties were analysed by FTIR spectroscopy and <sup>1</sup>HNMR. Finally, the glass transition temperatures were measured by differential scanning calorimetry (DSC) and the thermal degradation by thermogravimetry.

## Results and discussion

The styrene concentration as monomer in the oil phase as well as the surfactant (SDS) concentration in the aqueous phase, determined the morphology of the nanocapsules containing almond oil. Nanocapsules showed a core/shell structure analysed by transmission electron microscopy (TEM) with average diameter of 75-150 nm depending on the monomer/oil ratio and the surfactant concentration.

According to the results obtained an increase in the surfactant concentration in the aqueous phase, as well as, an increase in the monomer concentration in the oil phase produced a decrease in the nanocapsules average size and an increase in the molecular weight of shell polystyrene. This increase is due to a similar cohesion energy between the polymer and the almond oil and therefore the morphology of the nanocapsules is determined by the parameters that affect the interfacial tension.

PS nanocapsules containing different amounts of almond oil was studied under nitrogen atmosphere by TGA. A degradation process in a two steps process is observed and an increase in the thermal stability of the almond oil occurs when the oil is encapsulated.

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