Oil nanocapsules with antimicrobial activity

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Nanoencapsulation technology presents a new option for the shoe industry as its application can transform traditionally used materials or products into smart ones capable of interacting with feet. For instance, they can improve quality of life by incorporating products for foot care such as properly dosed essential oils. The nanoencapsulation 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.

Aromatics plants have traditionally been used in folk medicine as well as to extend the shelf life of foods, showing inhibition against bacteria, fungi and yeasts. Most of their properties are due to essential oils produced by their secondary metabolism. Some essential oils and extracts from several plant species are able to control microorganisms related to skin, dental caries, and food spoilage, including Gram-negative and Gram-positive bacteria. In recent years, interest in natural medicinal products, essential oils and other botanicals, has grown in response to the ever increasing incidence of adverse side effects associated with conventional drugs, and the emergence of resistance to antibiotics, synthetic disinfectants and germicides. Among the wide offer of essential oils, there are some that have recognized their antimicrobial activity such as tea tree oil, lemon or salvia essential oil, for example.

The *in situ* polymerization allows the formation of micro and nanocapsules containing a water-immiscible dispersed phase, with improved mechanical properties and thermal stability. The properties of the membrane depend not only on its chemical structure but also on all the synthesis conditions. The polycondensation of the amino resin occurs in the continuous phase, and the phase separation is linked to the pH and the melamine-formaldehyde resin molar ratio.

In this study a series of melamine-formaldehyde (MF) nanocapsules containing essential oils was prepared by *in situ* polymerization (O/W) method to be applied to footwear materials (lining, insoles, etc…) as antimicrobial agent and their physicochemical properties have been characterized by different experimental techniques.

Experimental

Synthesis of melamine-formaldehyde microcapsules containing antimicrobial oil. First of all, a melamine-formaldehyde (MF) resin to be used as microcapsules shell was prepared from the monomers, melamine and formaldehyde. After that, an O/W emulsion was prepared. The oil phase was composed of the active substance, and the aqueous phase was constituted of distilled water and sodium dodecyl sulfate (SDS) as surfactant. Next is added the above prepared prepolymer and adjust the pH, which causes coagulation of the polymeric shell around the oil droplets. Subsequently, hardening of the polymeric membrane by raising the temperature occurs. Finally, it was allowed to cool down to room temperature and the pH was adjusted to basic. In this study, microcapsules with four different MF/oil ratios were synthesized and characterized.

***Characterization of microcapsules*.** Physicochemical properties of the synthesized nanocapsules containing antimicrobial oils have been characterized by different experimental techniques. The size of the nanocapsules was determined using a particle size meter Coulter LS 230, the chemical structure of the oils and the nanocapsules obtained was analyzed by Fourier transform infrared spectroscopy, (FTIR). Finally, the incorporation of nanocapsules to footwear materials was analyzed by scanning electron microscopy (SEM).

Results and Discussion

Oil antimicrobial activity study.

The antimicrobial activity of TTO against different microorganisms typically found in used footwear (*E. coli, B. subtilis, K. pneumoniae, S. aureus*) was analyzed by in vitro test. Agar Petri dishes were prepared in order to evaluate the antimicrobial activity by measuring the inhibitory halo. The evaluation was carried out in two media: growth in liquid media for 24 h and measurement of inhibition diffusion halos in agar for 24 h.

Nanocapsules characterization.

Figure 1 shows the average particle size distribution in number for the different nanocapsules emulsion synthesized. Nanocapsules containing antimicrobial oil showed a narrow particle size distribution in number with a medium particle size around 100 nm.

FTIR spectra of the nanocapsules containing oils obtained after freeze-dried proved to be the combination of the characteristic bands of the MF resin and the oils. So the intensity of the bands corresponding to the resin decreases as the resin/oil ratio in the nanocapsules decreases. Therefore, there was no evidence of the chemical interactions between the core oil and the polymeric shell of the nanocapsules.

Finally, nanocapsules containing the active substances were incorporated to some materials used as footwear components such as linen and foams to evaluate the feasibility of this technology. Figure 2 shows SEM image of the nanocapsules anchored to the fabric fibers of a lining material.

Conclusions

Some essential oils have shown adequate antimicrobial activity against four microorganisms typically found in used footwear (*E. coli, B. subtilis, K. pneumoniae, S. aureus*).in order to be applied in footwear materials they have been encapsulated for a controlled release

The nanocapsules obtained containing antimicrobial essential oils, the relationship between the resin mass that forms the shell and the active substance contained in the core largely determines the efficiency of the nanoencapsulation process, the morphology of the nanocapsules and the particle size distribution thereof. The results obtained from the incorporation of microcapsules in fabrics and foams demonstrated the feasibility of this technology to achieve the concept of active shoe for footcare.

References

[1] Kunicka-Styczynska A. et al. J. Appl. Microbiol., **107** (6) (2009) 1903.
[2] Reuter J. et al. Am. J. Clin. Dermatol **11**(4) (2010) 247
[3] Luo W et al Polym. Degrad. Stabil. **92**(7): (2007). 1359.
[4] Fabien Salaün et al.. European Polymer Journal **44** (2008) 849.
[5] Li Wei et al. Materials Chemistry and Physics **106** (2007)437.

Figures:

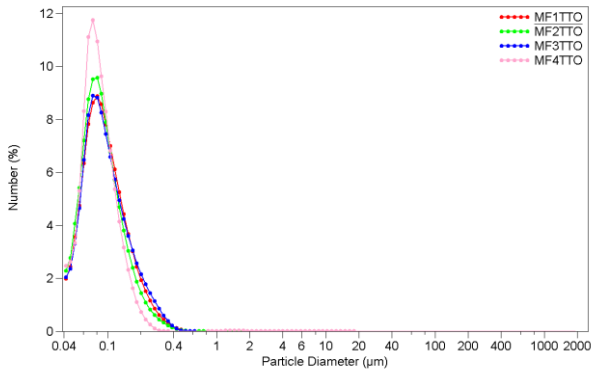

Figure 1: *Antimicrobial oil microcapsules size distributions*.

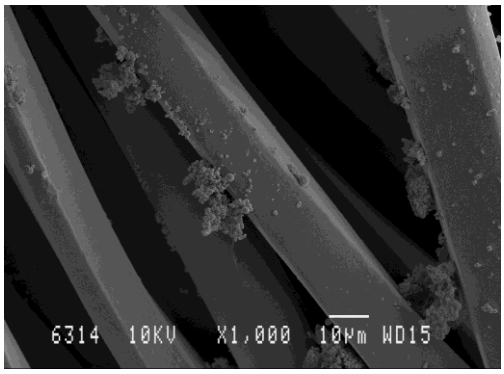

Figure 2: *Essential oil microcapsules incorporated to a fabric used as a footwear component.*

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