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Chitosan microparticles loaded with essential oils having in view leather applications

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Chitosan is a biopolymer attracting considerable attention for diverse applications due to its unique properties like biodegradability, biocompability, non-toxicity and antimicrobial activity [1]. It is obtained by the partial *N*-deacetylation of chitin, which is the second most abundant polysaccharide in nature, next to cellulose. Chitin can be obtained from several natural resources, it can be found in crustacean shells, mollusk and insect exoskeleton, as well as, in the cell wall of some fungi [2]. Chitosan can present various deacetylation degrees (DD), different acetamide group's distributions along the polymer chain and a variety of molecular weights. Chitosan is a weak base insoluble in water and organic solvents but soluble in dilute acidic aqueous solutions (pH < 6.5), which are capable of converting the glucosamine moieties into its soluble form (R-NH_3^+). These functional groups are responsible for chitosan binding ability to materials possessing opposite charged groups. Additionally, it is widely accepted that chitosan antimicrobial activity is related to this polycationic structure [2].

Chitosan is a viable base material for functional coatings development, namely due to its antimicrobial activity. This characteristic is especially useful for footwear applications that constitute products prone to microbial attack. In this context, developing antimicrobial coatings to be used in footwear components in direct contact with the feet are of great interest, both at industrial level (reducing possibility of material deterioration and quality loss), as well as, from the consumer's point of view (decrease of skin infections and minor unpleasant odours). One possible drawback of this application could be addressed to the durability and efficiency of the antimicrobial activity when applied to the end-use product, since it is directly associated with the availability of the positively charged R–NH_3^+ groups that are depleted during use.

Following our previous work, where leather impregnation with chitosan was studied at laboratorial level with quite promising results [3], and having in view the final application (footwear leather components), where the antimicrobial effect must need to be enhanced, we have developed the idea of microencapsulate different essential oils, with recognized antimicrobial activity like lemon, oregano, eucalyptus and pine [6]. These chitosan-based microparticles, loaded with the chosen essential oil are intended to be applied conjunctly with the chitosan coating process previously studied. Microencapsulation will ensure that the loaded core material will be progressively released, reinforcing the antimicrobial durability and effectiveness of the final product. Essential oils are known as natural derivatives with several benefits in addition to antimicrobial activity, providing a way to integrate other value-added properties to the final-product, like aroma and softness.

The objective of this work is the production of chitosan-based microparticles using a straightforward technique comprising an atomization spray to generate the particles that will consolidate upon contact with a coagulation/crosslinking bath. Comparatively to the most used water-in-oil (w/o) emulsion based preparation techniques, this one presents noteworthy advantages since particles are generated in a single step and the washing stage is considerably simplified. The used system is presented in figure 1. The experimental procedure comprises the following stages: (1) Chitosan solution (CS) preparation at a concentration of 2.5 % (w/v) in acidic medium (3%, v/v of acetic acid) This solution is left under stirring overnight and thereafter filtrated; (2) Oil-in-water (o/w) emulsion preparation by emulsifying the essential oil (EO) with the chitosan solution at the desired EO/CS ratio (v/v) and using Tergitol 15-