Preparation and characterization of gelatin/arabic gum microcapsules containing methyl salicylate deposited onto a cotton fabric

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INTRODUCTION

In the last years the micro encapsulation technique has been of particular interest for researchers, due to be an efficient technique for physical capturing of active substances that are sensitive to external agents and thus allowing a sustained release of these substances. On textile industry, the microencapsulation has become an important technique since it allows to develop new finishing and differential properties. The present study evaluated the effects of Methyl Salicylate complexion, using gelatin and Arabic gum as wall membrane components of the microcapsule and impregnating a jersey 100% cotton fabric.

MATERIALS AND METHODS

The materials used for the preparation of the microcapsules were Gelatin (Sigma Aldrich, Germany) and Arabic Gum (Sigma Aldrich, Germany), both as components for the wall, and Methyl Salicylate as the core material. The method used was the complex coacervation, for which three different solutions were prepared separately in order to be further mixed by means of mechanical agitation, using a temperature-controlled system. The adjustment of the pH was necessary to allow the electrostatic interaction between the gelatin’s amino groups and the Arabic Gum’s carboxylic groups. The microcapsule’s wall formation was induced by a controlled cooling of the solution, below the jellification temperature. With the purpose of obtaining a more rigid wall membrane, glutaraldehyde at 25% was added, due to be a powerful reticulation agent. In order to evaluate the formation of the microcapsules the following characterization techniques were used: FTIR, SEM, TGA/DTG and UV-VIS efficiency.
RESULTS AND DISCUSSION

The micro encapsulation process efficiency, measured by UC-Vis method, achieved 68.9%. The interaction of the polymers used in the microcapsule formation was analysed using IR spectroscopy approximately at 500 – 4,000 cm$^{-1}$. Arabic Gum is a polysaccharide which possess in its chemical structure carboxylic groups that are represented at 1.60 cm$^{-1}$ and 1.421 cm$^{-1}$ which are derived from the carboxylic acid (C=O) symmetrical and asymmetrical vibration stretching effect. The gelatin, a biodegradable and biocompatible amphoteric protein, is characterized by having amide and amine groups in its chemical structure. The most sensitive spectral region of the structural components of this protein is the amide I band, at 1700 – 1600 cm$^{-1}$.

It can be observed in the microcapsule spectrum that there was a band displacement of amide I and amide II from de 1.635 cm$^{-1}$ and 1.531 cm$^{-1}$ to 1.676 cm$^{-1}$ and 1.536 cm$^{-1}$, showing that it was generated an electrostatic interaction between the gelatin’s amine groups and the Arabic gum’s carboxylic groups, meaning that the materials from the microcapsule wall participated in the process of microencapsulation by means of electrostatic interaction.

![Figure 1 - FTIR of the microcapsules](image-url)
The thermograms from the biopolymers, gelatin and Arabic gum, presented two decomposition stages. These different stages of thermal degradation can be identified by the peaks observed in the DTG curve, which identify the beginning and end of a thermal degradation event. The first stage corresponds to the water removal associated to the polymers hydrophilic groups, while the second stage is due to the decomposition of the polysaccharide molecular chains.

![Image of thermograms](image_url)

**Figure 2**: Thermograms of the biopolymers, methyl salicylate and microcapsules

The complete degradation of methyl salicylate happens approximately at 240 °C. However, a mass loss has its beginning at 150°C, showing the need for microencapsulation due to the material’s volatility. From the analysis of the microcapsule’s thermogravimetric curve and the first derivative, one can observe that the degradation occurs in three stages. The first stage corresponds to the removal of water; the second stage refers to the liberation of the active principle, since it is in this stage that the initial degradation of the wall polymer chains occurs. The third stage of degradation represents the polymers thermal decomposition. The liberation profile of the microcapsules and the oil on 100% CO jersey fabric confirms the increase in stability and protection of the encapsulated Methyl Salicylate, since the active principle was released in more than 90% in less than 50 minutes.
Figure 3 - Liberation profile for the microcapsules and the free oil on 100% CO fabric

By means of micrographic analysis one can observe that the gelatin and the Arabic gum form spherical microcapsules on the textile fibres and are distributed without a significant agglomeration.

Figure 4 - SEM of the micropcapsules and the impregnation on a 100% cotton jersey fabric.

CONCLUSIONS

The present study allows concluding that there was a formation of microcapsules due to the electrostatic interactions between gelatin and Arabic gum, as seen with the FTIR results. These microcapsules present themselves as thermally stables, which allowed the protection of the encapsulated Methyl Salicylate. Due to this, it was possible to apply the product in cotton fabric and verify its distribution in the latter, thus indicating the possibility of the
development of a finishing with the properties of this particular oil for a controlled release effect. A fabric with the specific properties of metil salicylate is thus possible to produce.

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