

## Characterization of orange oil microcapsules for application in textiles

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**Abstract.** The use of orange oil presents as an ecological alternative to chemicals, attracting the attention of the scientific community to the development of eco-friendly antimicrobials. The microencapsulation technology has been used for the application of orange oil to textiles, being an economically viable, fast and efficient method by combining core and shell materials, desirable perceptual and functional characteristics, responsible for properties related to the nature of the product and provides that the wall materials release the functional substances in a controlled manner, in addition to effectively protecting and isolating the core material from the external environment to prevent its volatilization and deterioration, increasing the stability of the oil, such as non-toxicity. Thus, to better exploit the properties of the orange essential oil applied to textile products this study presents a characterization of microcapsules of Melamine formaldehyde obtained by the interfacial polymerization method with variations of proportions of orange oil (volatile) with fixed oil Medium-Chain Triglycerides (MCT) (non-volatile) to assist in the stability of the orange essential oil. Scanning electron microscope (SEM) was used as visualizing tool to characterize microparticles and surface morphology and thermal characteristics of microcapsules were premeditated by mean Differential scanning calorimetry (DSC).

### 1. Introduction

There are significant scientific advances in the field of microencapsulation, which have been used for the development of innovative textile products, such as the case of fabrics containing long lasting fragrances [1-7], biocide agents [8-11] and insecticide [12-13].

There are already researches that prove the antifungal properties [14-16] and insecticides of orange oil [13], which can be added to cotton textiles, helping to protect the microbiological degradation process that occurs when still in use and can cause serious functional, aesthetic and hygiene problems, cause of textile deterioration, discoloration and odors [11, 17]. The use of orange oil presents an ecological alternative to chemicals, attracting the attention of the scientific community to the development of eco-friendly antimicrobials [13-14, 16, 18-20].

Microencapsulation technology has been used for the application of orange oil to textiles, being an economically viable, fast and efficient method and leaving minimal waste [18]. This technique has also been used to microencapsulate a wide range of active, functional, sensitive or volatile substances



[1]. This method combines core and shell materials, desirable perceptual and functional characteristics, responsible for properties related to the nature of the product [3], [5]. The most interesting feature of essential oils microencapsulation is that the wall materials release the functional substances in a controlled manner, in addition to effectively protecting and isolating the core material from the external environment to prevent its volatilization and deterioration, increasing the stability of the oil, such as non-toxicity [1].

The challenge for encapsulating orange oil is to keep it inside the microcapsules during storage as they are mixtures of low molecular weight and base materials are highly frequently reactive, so that they can disrupt the process of forming the microcapsule or can diffuse easily through its shell. To solve these restrictions, the microencapsulation of oils: captures the fragrance in its original form with minimal change and maximum retention; protects the fragrance from interaction with an uncontrolled environment and from premature release during storage; and completely release the fragrance when desired. Therefore, the storage properties can be greatly improved by microencapsulation.

Melamine-formaldehyde resin (MF) is one of the preferred coating materials for the manufacture of microcapsules, because of its superior performance, including high hardness and mechanical strength, excellent thermal resistance, water resistance, resistance to weathering and free air and increase the durability of the perfume [6]. There is still the unlimited possibility of staining [1]. The cured MF resin is non-toxic and can be used in both wet and dry environments [1].

Thus, to better exploit the properties of the orange essential oil applied to textile products this study presents a characterization of microcapsules of MF obtained by the interfacial polymerization method. Variations of proportions of volatile orange oil has been fixed with non-volatile oil medium-chain triglycerides (MCT) to assist in the stability of the orange essential oil. Scanning electron microscope (SEM) was used as visualizing tool to characterize microparticles and surface morphology. Thermal characteristics of microcapsules were premeditated by mean Differential scanning calorimetry (DSC).

## 2. Materials and methods

Materials: Essential orange oil and surfactant polysorbate 20 (Tween 20) both was purchased from Sigma-Aldrich®; MF; fixed oil medium-chain triglycerides (MCT) was purchased from Delaware®. Preparation of microcapsules: Microcapsules containing essential orange oil combined in different proportions with a fixed oil medium-chain triglycerides (MCT) (1:1, 1:2, 2:1) and surfactant Tween 20 with melamine-formaldehyde (MF) resin as shell material were synthesized by interfacial polymerization technology. To characterize microcapsules: SEM model TM 3000, brand Hitachi®. The DSC test was performed on a Metler Toledo DSC 1 Stare System with Stare software

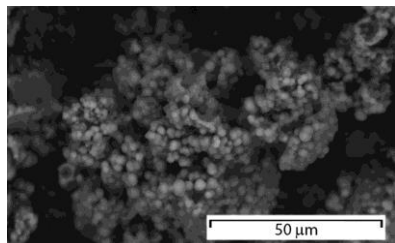
## 3. Results and discussion

The variations in the proportions of orange oil and MCT (1: 1, 1: 2 and 2: 1) were made to improve the stabilization of volatility, finding better efficiency in microencapsulation.

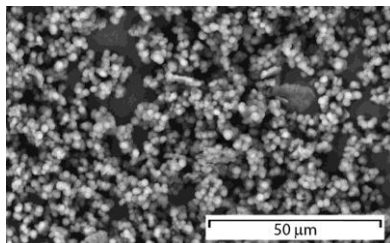
### 3.1. Scanning electron microscope

The variations in the proportions of orange oil and MCT (1: 1, 1: 2 and 2: 1) were made for a better homogenization of the emulsion, stabilizing the volatility, seeking a better efficiency in the encapsulation. In 'Figure 1' it is shown the image obtained by SEM of the microcapsules of orange essential oil and MCT in a ratio of 1: 1. It is observed that there was formation of microcapsules, but they have stick one to another and an agglomerate of microcapsules occurred. 'Figure 2' shows the image obtained from the microcapsules of orange essential oil and MCT in ratio of 1: 2. It is observed that there was also a formation of microcapsules, but still with the formation of agglomerates, although the degree of agglomeration is not as high as the one from microcapsules in ratio 1: 1, 'Figure'1. In 'Figure 3' shows the images of the orange essential oil microcapsule and MCT in ratio of 2: 1, aiming to increase the amount of orange oil to be encapsulated. It is observed by the analysis of the image that there was also formation of microcapsules, but, an improvement in the agglomerate presence was observed in relation to the ratios 1: 1 and 1: 2, 'Figures 1 and 2'.

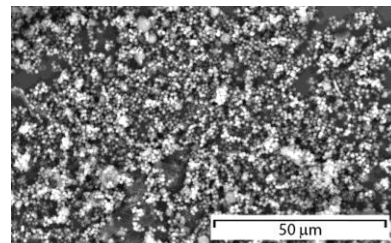
It can be observed that independently of the tested combination microcapsules were formed, but the different proportions of orange oil and MCT represented a variation in a agglomerates presence.



**Figure 1.** Image obtained by Scanning electron microscope (1500x) of the microcapsules of orange essential oil and MCT in a ratio of 1: 1.



**Figure 2.** Image obtained by Scanning electron microscope (1500x) of the microcapsules of orange essential oil and MCT in a ratio of 1: 2.

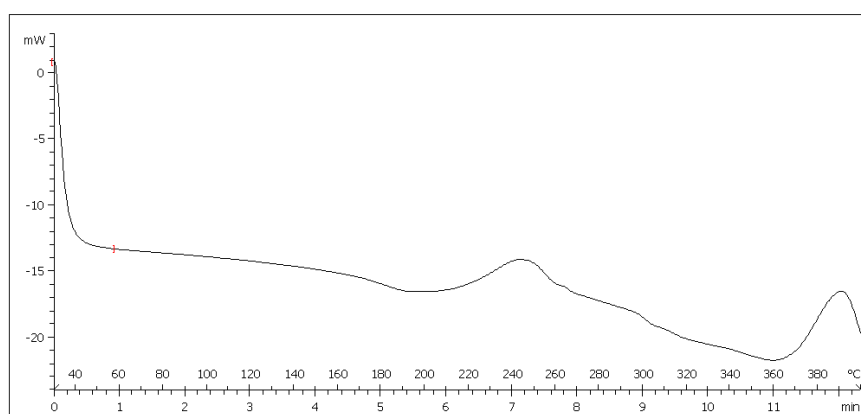


**Figure 3.** Image obtained by Scanning electron microscope (1500x) of the microcapsules of orange essential oil and MCT in a ratio of 2: 1.

### 3.2. Differential scanning calorimetry

Thermodynamics property analysis of microcapsules, essential oil and shell was measured by differential scanning calorimeter. 2 mg microcapsule powders were accurately weighed and placed in an aluminum pan using a sealed empty pan as reference. The scans were carried out at a heating rate of 10 °C/min.

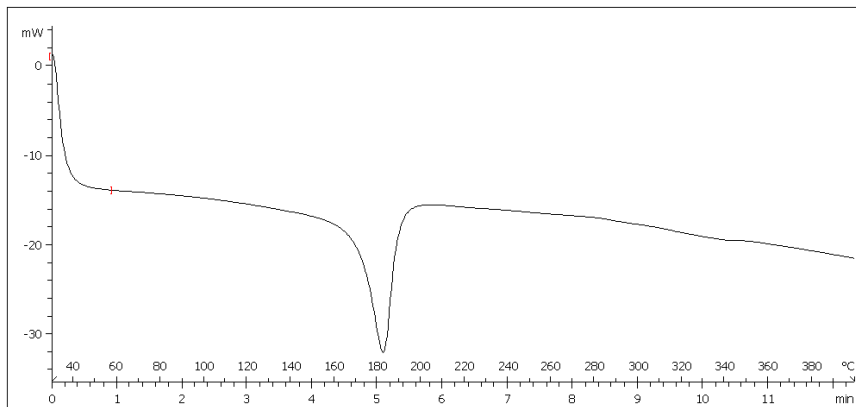
'Figure 4' shows the curve obtained by DSC of the MCT oil. In the DSC curve around 240°C we can see the crystallization point of the MCT and its peak temperature of degradation around 390°C.



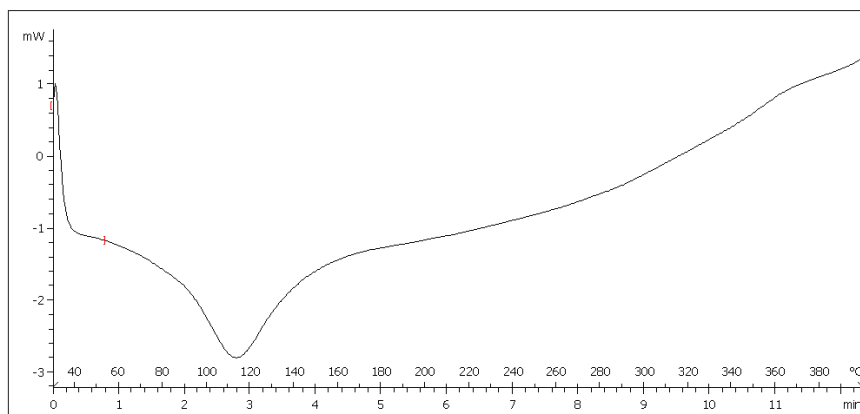
**Figure 4.** Differential scanning calorimetry of the MCT.

'Figure 5' shows the curve obtained by DSC of orange oil. In the DSC curve around 185° C we can see its peak temperature of degradation.

'Figure 6' shows the curve obtained by DSC only of the MF membrane without essential oil. In the DSC curve of the MF membrane we can see its peak temperature of degradation around 110°C.

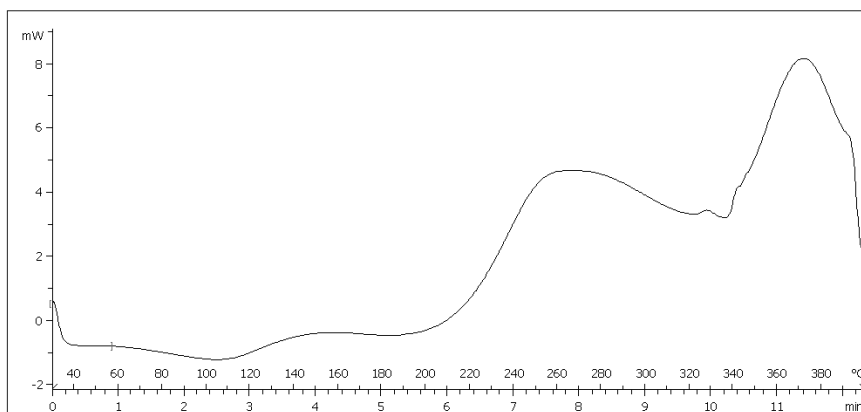


**Figure 5.** Differential scanning calorimetry of the orange oil.



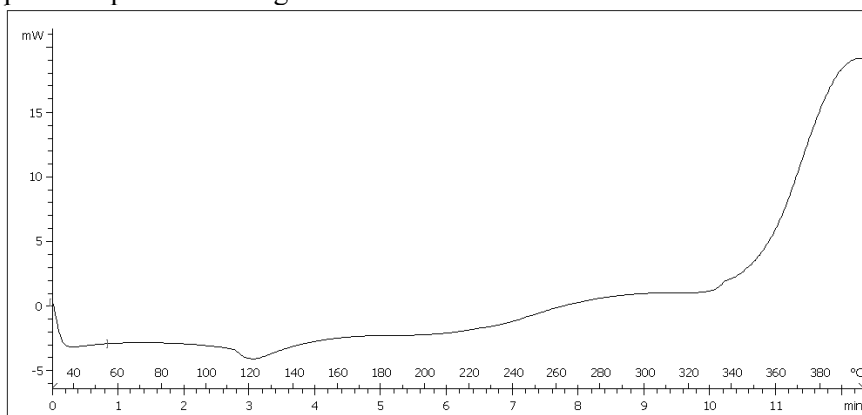
**Figure 6.** Differential scanning calorimetry of the shell MF.

‘Figure 7’ shows the DSC curve of the orange essential oil and MCT microcapsules in the ratio 1:1. In the DSC curve we can observe the peak temperature of degradation of the membrane around 110°C, approximately at 185°C there is a curve that indicates the presence of the orange oil, around 245°C curve that indicates the crystallization point of the MCT and around 370°C its peak temperature of degradation.



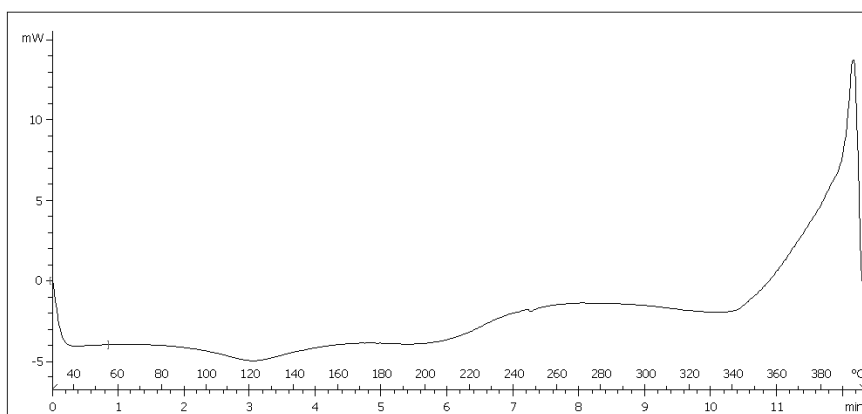
**Figure 7.** Differential scanning calorimetry of the microcapsules of orange essential oil and MCT in a ratio of 1:1.

'Figure 8' shows the DSC curve of the orange essential oil and MCT microcapsules in the ratio 1:2. In the DSC curve we can observe the peak temperature of degradation of the membrane around 120°C and at approximately 390°C the peak temperature of degradation of the MCT. In this analysis it was not possible to identify the presence of orange oil, since it does not present a significant curve around its the peak temperature of degradation.



**Figure 8.** Differential scanning calorimetry of the microcapsules of orange essential oil and MCT in a ratio of 1:2.

In 'Figure 9' it is shown the curve obtained by DSC from the orange essential oil and MCT microcapsules in the ratio 2:1. In the DSC curve we can observe the peak temperature of degradation of the membrane around 120°C, approximately 190°C a curve that indicates the presence of the orange oil, around 250°C curve that indicates the crystallization point of the MCT and around 390°C the its the peak temperature of degradation.



**Figure 9.** Differential scanning calorimetry of the microcapsules of orange essential oil and MCT in a ratio of 2:1.

#### 4. Conclusion

In all experiments the addition of MCT in the microcapsule formulation ensure the microcapsules formation as well as improved the capsules stability.

In the ratio 1: 1 the formation of microcapsules occurred, although they formed agglomerates, as observed in the SEM images. In the ratio 1: 2 the formation of microcapsules occurred, although they still present agglomerates When 2: 1 ratio is used, the formation of microcapsules with little formation of agglomerates occurred.

About, the DSC, regardless the ratio of MCT employed all the curves showed the MCT degradation, however, due to the overlaying of the curves from the different components which comprise the microcapsules, it is not possible to observe the orange oil degradation. Consequently, the DSC technique allows to evidence the presence of MCT but not the orange oil although we can confirm its presence due to the fragrance which is smelled when rubbing the microcapsules. Thus, further testing is required to objectively confirm the effectiveness in the encapsulation of the oil.

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