Encapsulation of waxes

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INTRODUCTION

The use of latent heat as energy storage is a manner for applying different materials as source of renewable energy. A great number of materials was identified for different range of operation temperature facilitating the selection for many applications. This includes fatty acids, inorganic salts and hydrocarbons (Cabeza 2011, Abhat 1987, Suppes 2003). However, the study of waxes is quite limited. In this study, some commercial waxes were characterized and particles with them were produced by complex coacervation. Adequate melting point, similar melting and crystallization points and high latent heat are desirable for a good PCM. These characteristics were evaluated in the particles.

MATERIALS AND METHODS

Materials. Gelatine type-B (LF 21502/04, Gelita, Brazil) and gum Arabic (ref. IRX49345, CNI, Brazil). As core material was used: paraffins waxes (Solven wax 120 e 170 - SOLVEN Solventes e químicos Ltda, Brazil), bee and carnauba waxes (Gmceras) and the modified waxes Syncrowax BB4 and Syncrowax ERLC (Croda do Brasil, Brazil). Pequi and Babassu oils from Croda do Brasil, Brazil.

Microparticles preparation: *S*olutions of gelatine (GE) and gum Arabic (GA) were prepared at 2.5% w/w. The core material (2.5g) was emulsified in the 100 mL gelatin solution at 14000 rpm for 3 minutes (Ultra turrax, IKA, Germany) followed by incorporation into 100 mL GA solution ºC. Slow reduction of pH solution until pH 4.0 and gradual cooling (3 hours) of the system from the melting point of the waxes to 10 ºC.

Thermal assays: Determination of thermal properties of the pure waxes and their dried microcapsules were made using differential scanning calorimetry (2920 Modulated DSC). The samples were heated at a rate of 10 °C/min, from 0 to 100 °C, with an empty pan used as a reference. The melting temperature of the PCM (Tm) was determined using the onset temperature. The percentage of PCM in the microcapsules (EE) was calculated as the ratio between the latent heat of the microcapsules (ΔHmc) and that of the corresponding pure compound (ΔHfree) multiplied by 100.

RESULTS AND DISCUSSION

Table 1 shows the thermal properties obtained from the calorimetric analysis to dehydrated microparticles prepared by complex coacervation between the gelatin and gum Arabic with different waxes, containing only 50% of the active component in relation to the wall material. The profiles obtained in DSC analysis were not shown, but all the materials evaluated showed no subcooling.

Regarding to the melting heat, the waxes presented high capacity of storage heat varying between 127.5 to 162.2. The oils analyzed, i.e. pequi and babassu, presented the lowest values.

Table 1. Characteristics of microparticles produced with waxes.

Particles	T_m(ºC)	ΔH_m free (J/g)	EE (%)	D (µm)
Carnauba wax	71.77	160.4	42.0	143.4
Bee wax	50.04	135.1	39.72	168.4
Paraffin 120	46.47	127.5	24.22	111.6
Paraffin 170	47.43	148.6	24.96	191.2
Babassu oil	8.04	71.94	90.17	167.5
Pequi oil	-	29.37	76.30	136.5
BB4 wax	46.37	162.2	17.7	192.3
ERCL wax	60.67	161.4	35.92	160.7

It is possible to observe that, in general, all of the waxy materials had presented low efficiency of encapsulation, around 20 and 40%. Figure 1 illustrates two microparticles representing the general behavior. One of these has been presented low efficiency of encapsulation (42% - Figure 1A) and other a high (90.17% - Figure 1B). This difference is attributed to the composition of the active component. Waxes usually are solids in the environment temperature which difficult their entrapment by the coacervate wall. In this case, the slow solidification of the matrix contributes with the output of the apolar waxes crystals.

Particularly, the carnauba wax is a carboxylic acid ester with long chain alcohols which results in a large hydrophobic molecule. Already babassu oil is a natural oil rich in fatty acids mainly lauric and myristic. Saturated fatty acids have strong polarity due to the presence of carboxyl groups and the improvement in the stability emulsified droplets should also due to opposite position of the terminal methyl and carboxylic groups.

The mixture of lauric and myristic acid was studied by

Sari (2005) and showed melting point ranging from 28 to 52 ºC, which indicates that this oil contains other substances that reduce its melting point to 8 ºC.

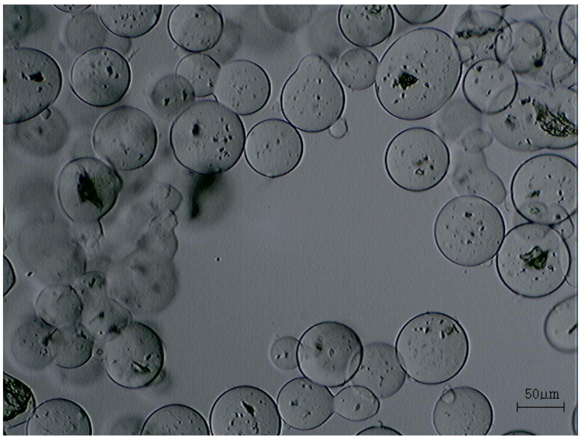

Figure 1. Coacervates microparticles containing A:Carnauba wax, B:Babassu oil

The efficiency of encapsulation can be significantly increased using strategies for microparticles production as increasing the amount of active in the formulation (Onder 2008; Fagundes 2013), or incorporation of a surfactant (Salaun 2009). By this way, Salaun (2009) successfully incorporated 89.8% of carnauba wax in microparticles with two coatings using a core/wall mass ratio of 4 and span 85 as surfactant. Deveci (2009) has obtained a maximum of 62% of encapsulation with eicosane in chitosan/fibroin microparticles by the increasing of the core/wall ratio and using span 20 as emulsifier. Onder (2008) ranged from 40 to 80% of the amount of core material added in relation to the wall material. The lowest efficiencies of encapsulation was obtained with 40% core/wall ratio and ranged of 23% for hexadecane and 8% for octadecane. These values were increased when this ratio was 80%, and the particles produced with hexadecane reached 78% and those with octadecane 70% of efficiency of encapsulation.

Nevertheless, in the literature some microparticles produced by coacervation and containing different kinds of waxes showed similar energy capabilities, indicating that the particles produced in this study are suitable for use in the heat storage.

The phase transition temperatures were identical to those of the free compounds indicating that the wall material does not influence the thermal capacity of the samples.

The average diameter of the microcapsules were about 150 um. Complex coacervation between gum Arabic and gelatin has produced particles with sizes ranging from 50-100 µm with paraffin wax (Hawlader 2000) or natural coco fatty acid mixture (Özonur 2006).

Yet is essential to evaluate the thermal stability of these materials to repeated cycles of heating and cooling to ensure its long-term performance. Some authors suggest that heat of fusion constant after about 30 repetitions indicates good stability (Liu 2001).

CONCLUSIONS

The core material tested in this study are promising for PCM applications, except the pequi oil, that has presented low heat capacity and low efficiency of encapsulation. Although babassu oil is not be among the best candidates to serving as PCM, it was friendly with the encapsulation process by complex coacervation and its particles showed thermal capacity identical to those showed by the carnauba wax microparticles.

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