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RESEARCH ARTICLE

EVALUATION OF FORMATION PROCESSES OF MICROCAPSULES BY THERMAL ANALYSIS

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ABSTRACT

Antioxidants are compounds with large industrial applicationin the pharmaceutical, cosmetic, chemical and food sectors. Anthocyanins are thermosensitive natural pigments with important antioxidant and biofunctional action, and can be better preserved if kept encapsulated. This treatment can be performed by different techniques, among them spray-dying and lyophilization. The objective of this study was to determine the thermal profile of single samples (anthocyanins and gum arabic) and materials obtained by treatment with spray-drying and lyophilization and to evaluate the efficiency of each technique in the encapsulation of anthocyanins. All samples were analyzed by Differential Scanning Calorimetry (DSC) and Scanning Electron Microscopy (SEM). The values of temperature and melting enthalpy for pure compounds and treated materials were also determined. The results showed the microparticles formation containing anthocyanins in the samples treated by spray-drying; and the thermograms obtained by DSC indicated the occurrence of encapsulation for these samples, being this result confirmed by SEM analyzes.

INTRODUCTION

The natural antioxidants found in fruits have important application in the food, pharmaceutical and cosmetic industries. Among the natural antioxidants, flavonoids are found in fruits (red, purple, and citrus), vegetables, vegetables, legumes, teas and red wine. The importance of these compounds is linked to the antioxidant and anti-inflammatory capacities acting in the prevention of several diseases (Kähkönen, 2003; Wang, 1999 and Kong, 2003). Anthocyanins are natural pigments widely used in the food industry. Belonging to the class of flavonoids, these compounds are partially water soluble, and have in their structure glycosides and OH groups. Its phenolic structure is responsible for its antioxidant action. These compounds are thermally sensitive and undergo changes if they are exposed to light and moisture. However, their degradation can be minimized if they are microencapsulated. The microencapsulation process consists of involvement of particles of interest in an edible matrix or microcapsule to protect the material from external factors that affect it physically and chemically (Mahdavi, 2014 and Wilkowska, 2016). Microencapsulation is a technique that aims to protect an active agent from external agents, and can be obtained by different processes, among them, spray-drying, lyophilization, others. Depending on the application, the encapsulation method may be directed according to the desired characteristics for the final product, i.e.: required particle size, physicochemical properties of the whole encapsulated particle (core and wall), use of the final product, mechanisms release, production level and cost (Ré, 1998).

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The analysis of the formation of the microcapsules has been performed through the combination of results obtained by different techniques. The powders formed are usually evaluated by scanning electron microscopy (SEM), particle size and distribution, zeta potential, X-ray powder diffraction (XRD), and thermal analysis (Nguyen, 2004; Silva, 2013; Strasdat, 2013 and Desobry, 1997). The combination of the results of these techniques allows to characterize the obtained material and to demonstrate if the formation of particles perfectly encapsulated, and which were the best conditions. Differential Scanning Calorimetry (DSC) is a thermal analysis technique that measures the change in energy absorbed by the substance and by a reference material (thermally stable) as a function of temperature. Up to now, most of the studies in this area apply DSC as a technique used to characterize formed powder materials, not directly correlating the results to the events with the formation of the encapsulated particles. However, among the responses pointed out by this technique, it is possible to evaluate, through the comparison of the associated events, if the process of formation of microparticles was effective (Wendlandt, 1986). In this sense, the objectives of this study were to obtain thermodynamic data (melting point and enthalpy) of single materials (gum arabic and anthocyanins), and materials containing the mixture of the single materials treated by spray-drying and lyophilization, and to evaluate through these data if the microcapsules were formed in the process.

MATERIALS AND METHODS

Extraction of anthocyanins: The extracts containing anthocyanins (ANT) were obtained from the immersion of the skin of the jambo fruit, also known as malayan apple, in distilled water, during 24 h at 4 ºC, using a rate 1:3 (w/v).

After the extraction the mixture was grinded in a mechanical processor (Philips Walita, RI7625) and it was sifted in sieve (Mesh: 300).

Microencapsulation processes: The microparticles containing anthocyanins were formed using gum arabic donated by NexiraBrasil), two techniques were evaluated: spray-drying and lyophilization. For the first technique, it was used a spray-drying (Labmaq do Brasil, model MSD 1.0) under the following operating conditions: 0.67 L/h of solution flow; 1.65 m³/min of pump flow rate, and 0.35 L/min in the rotameter. For the second treatment, it was used a freeze dryer (model L101, LIOTOP, Brazil) and samples of 35 g of shell extract, with and without addition of gum, previously frozen. The operating conditions were: temperature of -40 ºC, pressure <500mmHg, 1 m/h of speed, during 48 h. The materials treated by spray-drying and by lyophilization were analyzed by DSC and Scanning Electron Microscopy (SEM).

Thermal profile and Particle morphology: DSC thermograms were obtained by a DSC-Q20 Differential Scanning Calorimeter (TA Instruments) using a standard sealed aluminum sample holder. The samples were weighed using a Mettler Toledo Model XS205 Precision Balance. The analyzes were carried out in a nitrogen atmosphere, in a flow rate of 80 mL/min, from 0 to 550 ºC of temperature range, with a heating rate of 10 ºC/min. DSC analyzes were performed for gum, malayan apple extract containing anthocyanins, and for the material (containing gum and anthocyanins) obtained after the treatment by spray-drying and lyophilization. SEM (Hitachi), under a 15 kV, intensity and 1000-fold increments was used to verify the existence of microparticles and to visualize the predominant forms in each formulation.

RESULTS AND DISCUSSION

Figure 1 presents the results obtained by DSC, with the melting point and enthalpy values and degradation temperature highlighted for each material and Table 1 present the values of temperature and enthalpy obtained in each event. Figure 2 shows the results obtained by SEM for each sample evaluated. According to the results presented in Figure 1 and Table 1, it is possible to observe that the thermogram of materials obtained by spray-drying (Figure 1c) showed an endothermic peak at 177.24 °C attributed to the melting point, a melting enthalpy 70.13 kJ/mol and an exothermic peak at 305.80 °C that can be attributed to the overlapping events: degradation of anthocyanins and gum arabic.

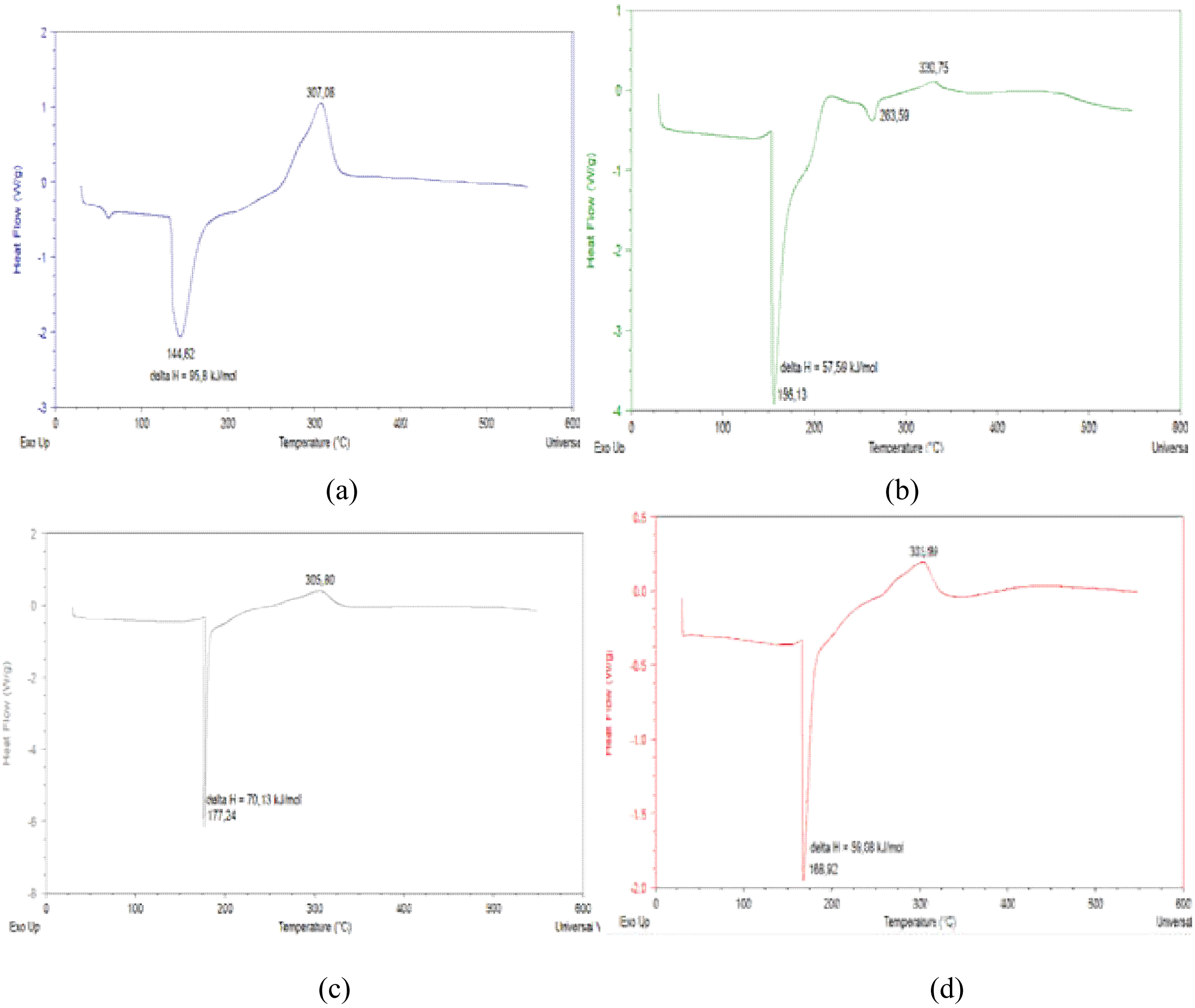
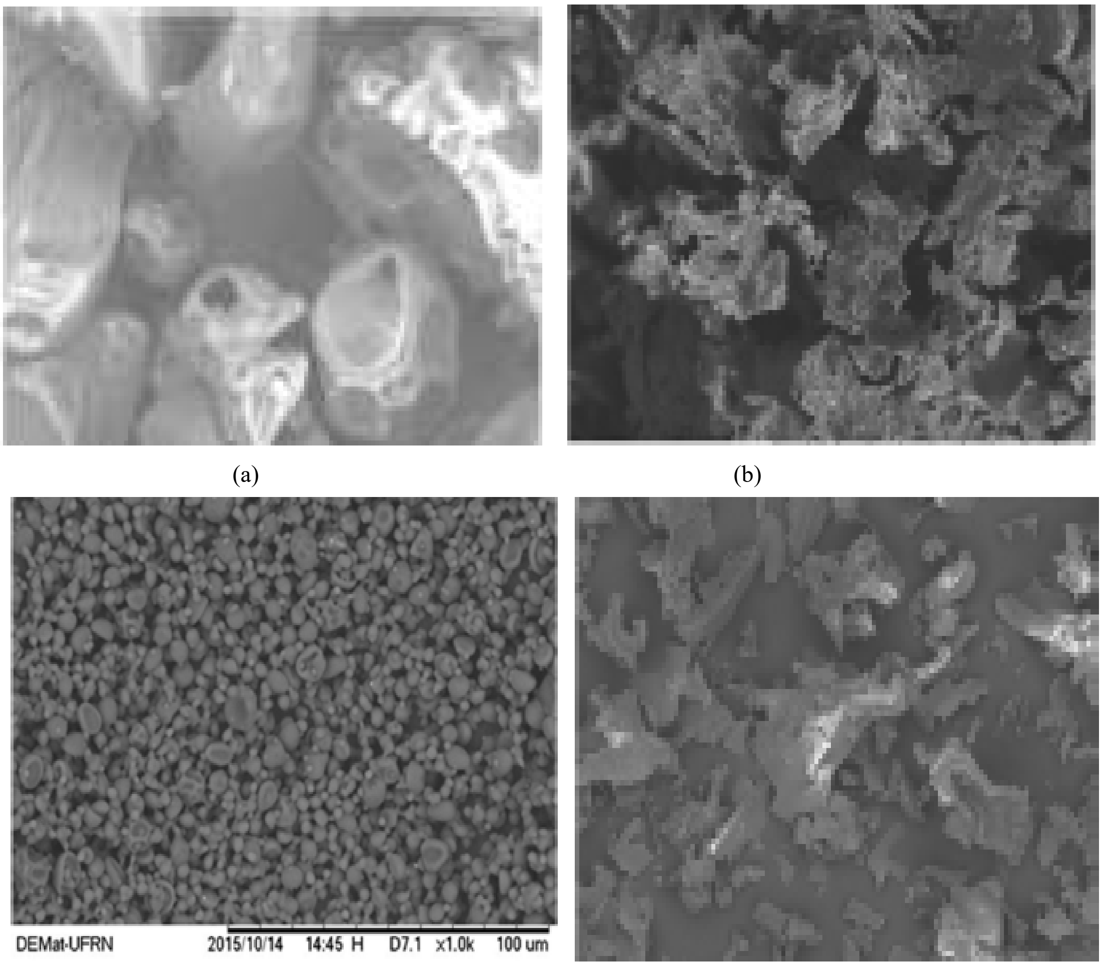


Figure 1. DSC thermograms of the materials: (a) gum arabic (b) anthocyanins, (c) particles obtained by spray-drying, and (d) particles obtained by lyophilization

Table 1. Peak temperature and melting enthalpy data for pure and treated materials

Material	ΔH (kJ/mol)	T_1 (ºC)	T_2 (ºC)	T_3 (ºC)
Gum arabic	95.80	144.62	307.08	-
Anthocyanins	57.59	156.13	263.59	330.75
Particles obtained by spray-drying	70.13	177.24	305.80	-
Particles obtained by lyophilization	59.08	168.92	303.89	-



(c) (d)

Figure 2. Microphotograhs of the materials: (a) gum arabic (b) anthocyanins, (c) particles obtained by spray-drying, and (d) particles obtained by lyophilization, at 1000 fold increase

This thermogram is different from the others obtained from pure gum arabic (Figure 1a), with an endothermic peak at 144.62 ºC, which is related to the moisture present in the sample, a melting enthalpy 95.80 kJ/mol and an exothermic peak at 307.08 ºC, relating to degradation of the polymer; and from anthocyanins (Figure 1b), where an intense endothermic peak at approximately 156.13 ºC, relative to the melting of the substance, a melting enthalpy 57.59 kJ/mol and two exothermic peaks at 263.59 ºC and 330.75 ºC were verified. Sample obtained by lyophilization (Figure 1-d) presented an endothermic peak at 168.92 °C attributed to melting point, a melting enthalpy 59.08 kJ/mol and two exothermic peaks at 254.63 and 303.89 °C. The formation of microparticles is observed when a new exothermic peak occurs, with a new temperature value, different from those related to the pure elements. For the particles obtained by spray-drying, a decrease in temperature was observed, with an exothermic peak at 305.80 ºC, indicating the formation of a new material (the microparticles).

On the other hand, the thermogram of particles obtained by lyophilization presented endo and exothermic peaks close to the values observed for anthocyanins and gum arabic. This last thermogram shows a hybrid behavior between the two single substances, indicating the physical mixture, without formation of a new material. The confirmation of these facts is evidenced in SEM analyzes (Figure 2). The shape of the single materials (anthocyanins and gum arabic) and materials obtained by lyophilization resembles particles without predominant morphology and spongy form (Figures 2-a,b,d). Contrary to the material produced by spray-drying, with predominant spherical shape, characteristic of articles of encapsulated material, indicating that the spray-drying was effective for the microparticle formation (Figure 2-c). The evidence presented by the thermograms, regarding the thermal profile for samples with particle formation, can also be found in other studies

[12,13], although in these, the discussion about the responses of the DSC analyzes does not present this direct correlation. That is, most studies in this area use the DSC technique to characterize the materials produced and not to identify the formation or not of encapsulated particles. This fact can be observed in this work and also when an analysis of the results of already published studies that present profiles of encapsulated and non-encapsulated materials is made.

Conclusion

In this study, anthocyanins from the skin of the jambo fruit were treated in order to promote the formation of microencapsulated particles. This treatment was evaluated by different techniques, and the thermal properties (melting point and enthalpy) of pure and materials treated by spray-drying and lyophilization were determined. The endothermic and exothermic peaks observed in the thermal profile of the thermograms indicated that spray-drying was effective in the microparticles formation, being this fact confirmed by SEM. This study also demonstrated the application of the DSC technique to identify the formation of encapsulated microparticles, being this evidence in accordance with results presented by literature.

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