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Understanding the Influence of Encapsulating Matrix on the Physical and Thermal Properties of Oregano Essential Oil Powder

Ariel Antonio Campos Toledo Hijo[1], Joyce Maria Gomes da Costa1, Eric Keven Silva[1*],Viviane Machado Azevedo[1], Maria Irene Yoshida[2], Soraia Vilela Borges[1]

[1]Food Science Department, Federal University of Lavras, 37200-000, Lavras, MG, Brazil

[2]Federal University of Minas Gerais, 37200-000, Belo Horizonte, MG, Brazil

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***Corresponding author:** Eric Keven Silva, Food Science Department, Federal University of Lavras, 37200-000, Lavras, MG, Brazil, Tel: (+55 19 982833762); E-mail: engerickeven@gmail.com*

Abstract

In this study, the influence of blends of carbohydrates on the physical and thermal properties of oregano essential oil micro particles was evaluated. Powders were produced by spray drying using different wall materials: gum Arabic (GA), modified starch (MS) and maltodextrin (MD). The micro particles were characterized by morphology, moisture content, water activity, particle size distribution, particle mean diameter, poly dispersity index, glass transition temperature (Tg) and thermal stability. The partial replacement of GA by MS or a mixture of MS and MD resulted in micro particles with lower moisture content. The results showed that micro particles with GA had higher values of Tg (97.02 °C), indicating more physical stability. However, the use of MS and MD results in a slight reduction of Tg. TGA results showed that the nature of wall materials have considerable effect on the thermal degradation of micro particles. Thermal analysis revealed the potential of application of these carbohydrates for substitution of GA.

Keywords: glass transition; gum Arabic; modified starch; microcapsules; thermal stability;

Introduction

The microencapsulation method via spray drying has been widely employed for encapsulation of essential oils and other flavors, due to the efficiency in the retention of volatile compounds and protection of the active compound against external agents, such as light and oxygen(Adamiec and Kalemba, 2006; Kha et al., 2014; Liu et al., 2001). In addition to their technical advantages (Liu et al., 2015), this technique allows the combination of encapsulating agents, including those with low cost, to produce higher quality products with lower production cost, which increases the industrial interest(Vaidya et al., 2006).

Essential oils are complex mixtures of fatty acids with long alkyl chain length and organic compounds with low molecular mass, such as esters, alcohols, phenols and others. They are commonly extracted from plants and spices through hydro distillation, mechanical process or by using solvents. These substances can have antioxidant and antimicrobial activity due to the high content of active compounds in their composition, such as terpenic (α-terpinene, limonene and terpinen-4-ol) and phenolic compounds (carvacrol and thymol), present in the oregano essential oil(Dambolena et al., 2010).The encapsulation techniques, such as spray drying are employed in order to produce stable products containing essential oils, because these substances exhibit chemical instability to temperature and humidity changes and contact with oxygen(Adamiec and Kalemba, 2006).

The study of the best combination of wall materials is of interesting order to maximize the retention of the active agent, as well asto obtain more stable particles. The properties of the encapsulating materials are the main factors that affect the microencapsulation efficiency (Gharsallaoui et al., 2007; Reineccius, 2004).Carbohydrates, including hydrolyzed and modified starches, gums, cellulose derivates and cyclodextrins, are commonly used in spray drying. Gum arabichas been the most common wall material used in the encapsulation of oils and flavors, since it has emulsifying properties and is excellent in the retention of volatiles(Da Costa et al., 2013; Fang et al., 2005; Fernandes et al., 2014; Toledo Hijo et al., 2014). Despite its excellent characteristics, in recent years, the gum Arabic has increased in cost, has limited availability and impurities (Gharsallaoui et al., 2007; Tonon et al., 2012).Thus, the study of encapsulants or combination of ingredients to replace the gum Arabic for microencapsulation has been encouraged.

In this context, the study of the effect of different encapsulating materials on the micro particle properties is an important subject. Modified starches and maltodextrins have shown to have excellent encapsulating abilities when combined with other materials, such as gum Arabic and proteins (Vaidya et al., 2006; Yoshii et al., 2001). Maltodextrins are partially hydrolyzed starch products formed by chains of D-glicose connected byα-

(1,4) bonds(Shahidi and Han, 1993). They are produced by acid or enzymatic hydrolysis of starches, or even a combination of both processes(Chronakis, 1998). The use of maltodextrin offers several advantages, such as low cost, high solubility, low viscosity and excellent protection of flavors against oxidation(Cano-Chauca et al., 2005; Goubet et al., 1998; Reineccius, 1991). Also, maltodextrins with high molarmass and low value of dextrose equivalent (DE) provide high physical stability to the wall matrix system(Bae and Lee, 2008).The modified starch is an encapsulating agent commonly used for their encapsulation abilities, such as excellent retention of volatiles (above 93%), the stabilization of the emulsion and low viscosity(Arancibia et al., 2011). Capsul®, also known as octenylsuccinate starch, is a starch derivative obtained by starch esterification with octenylsuccinate anhydrous acid, resulting in a hydro phobically modified starch (Hui et al., 2009; Wang et al., 2011). Through this modification, the hydro phobicity of the octenylsuccinate is introduced and the hydro philicity of the starch is maintained. As a result, this starch derivative has been reported as an effective emulsifier(Wang et al., 2011) and is thus preferably used as an encapsulating agent in the microencapsulation process of foods.

The polymer association has become an alternative way for producing micro particles and it is highly demanded in many foods and pharmaceutical applications (Dalmoro et al., 2012; Vaidya et al., 2006).In fact, for ensuring and expanding the applicability of new particles in such fields, the study of their physical and thermal stability is required. However, these properties have not been fully evaluated. Therefore, the aim of this study was to evaluate blends of gum Arabic with maltodextrin and modified starch as a matrix system for microencapsulation of oregano essential oil by spray drying, in order to obtain a dried product with a high technological standard. The micro particles were characterized by moisture content, water activity, particle size distribution, particle mean diameter, poly dispersity index, morphology, glass transition temperature and thermal stability.

Material and Methods

Material

Oregano (*Origanum vulgare L.*) essential oil was purchased from Laszlo Aromatherapy Ltd (Belo Horizonte, MG, and Brazil) and used as the core material for the production of micro particles. Modified starch (Capsul – Snow Flake® E6131), donated by Corn Products (Mogi, SP, Brazil), maltodextrin (Globe® 1905 DE 20) and gum Arabic (Colloides Naturels SP, Brazil) were used as wall materials. In order to increase the capacity and stability of the emulsions or suspensions, we used modified starch, which modification consists of adding a lipophilic component (octenyl succinate) to the starch molecules.

Experimental design

The proportion of the wall materials were defined from the optimization reported by Da Costa et al. (2013)in order to obtain blends of carbohydrates with different concentration of GA (Table 1).

Table 1: Proportions of wall materials for each treatment used as feed solution for the spray drying process

Proportions of wall materials (g/100 g)				
Assay	**Microparticles**	**Gum Arabic (GA)**	**Modified Starch (MS)**	**Maltodextrin (MD)**
1	GA100	100	0	0
2	GA50:MS:MD	50	25	25
3	GA25:MS	25	75	0
4	GA25:MD	25	0	75
5	GA25:MS:MD	25	37.5	37.5

Production of oregano essential oil micro particles

The emulsion was prepared by adding 90 g of wall materials and 10 ml of oregano essential oil in 900 mL of distilled water. Maltodextrin and gum Arabic were hydrated in distilled water for 12 hours at 10 to 12 °C. The respective wall materials were dissolved in distilled water at 60-70 °C using a homogenizer at a speed of 20,000 g-force for 30 min. Then, modified starch was added at 82 °C, maintaining homogeneity until complete dissolution of the wall materials. After complete dissolution of the encapsulants below 10 °C, the oregano essential oil was added with full rotation of 20,000 g-force for 5 minutes.

The emulsion was submitted to drying using a LABMAQ Brazil spray dryer, Model MSD 1.0 (RibeirãoPreto, São Paulo, Brazil) equipped with a dual-fluid nozzle spraying system and an opening of 1.2×10-3 m. The inlet and outlet air temperatures were 180 ± 2 °C and 105 ± 2 °C, respectively. The feed flow rate was adjusted to 2.97×10-7 m3 s-1, the air flow inlet was maintained at 5.8×10-4 m3 s-1 and the compressed air pressure to the spray flow was 5 bars.

The powder was collected and stored under refrigeration (4-7 °C) in glass flasks protected from light and water vapor to avoid possible alterations in the material as well as agglomeration and oxidation until further analysis.

Moisture content and water activity

The moisture content was determined by gravimetric method according toAOAC (2000). The water activity (a_w) was measured directly using a digital AQUALAB device (CX-2 model, Decagon Devices Inc., Pullman, WA) with controlled temperature (25 ± 0.5 °C). Each measurement was performed in triplicate.

Morphology

The particle morphology was evaluated by scanning electron microscopy (SEM). The powders were fixed on a piece of double-sided adhesive tape, mounted on SEM stubs with a diameter of 1 cm and a height of 1 cm, coated with gold under vacuum and examined with a scanning electron microscope SEM 1430VP - LEO (Electron Microscopy Ltd., Cambridge , UK). The microscope was operated at 20 kV with magnifications from 900-1200.

Average diameter and particle size distribution

The average diameter and particle size distribution were determined using a Master size 2000 laser light diffraction instrument (model Hydro 2000 MU, Malvern Instruments, Malvern, UK). A small sample of the powder was suspended in isopropyl alcohol p.a. (Synth) as a dispersing medium while stirring. The particle size distribution was monitored during each successive measurement until the measurements were constant. The average volumetric diameter (D4, 3) was measured, and the poly dispersity index (PDI) was calculated according to Equation (3) below:

$$PDI = \left(\frac{d_{90} - d_{10}}{d_{50}} \right)$$

Where, d_{90} d_{50} and d_{10} correspond to the average diameter equivalent to 90%, 50% and 10% of the cumulative volume, respectively.

Thermal analysis

Differential scanning calorimetry (DSC)

The micro particles were analyzed using a DSC calorimeter model 60-SHIMADZU (Shimadzu Corporation, Kyoto, Japan) to determine the glass transition temperature (Tg). Approximately 4-6 mg of sample was prepared in aluminum pans, while an empty pan was used as reference. The curve were obtained according to the follow heating program: samples were cooled from 25 °C to -70 °C and then heated from -70 °C to 120 °C at 10 °C min-1. The glass transition temperature (Tg) was obtained in the heating curve.

Thermo gravimetric analysis (TGA)

The thermal stability of the oregano essential oil micro particles was evaluated by thermo gravimetric analysis in a Shimadzu TG-DTA 50 H (Shimadzu Corporation, Kyoto, Japan) to quantify the mass loss of the powders. The analyses were performed under nitrogen at a flow rate of 50 mL min-1. The samples were heated at 10 °C min-1 from 25 °C to 500 °C (Lavorgna et al., 2010).

Results and Discussion

Moisture content and water activity

Moisture content and water activity are important parameters for the characterization of particles, because they are directly related to the shelf life of foods. Through these parameters, it is possible to estimate the microbiological stability and storage conditions of dried foods. Micro particles of oregano essential oil presented values of water activity and moisture content between 0.1-0.18 and1.1-3%, respectively (Table 2).The values of moisture content of micro particles are according to the minimum specification for most dried powders (3-4%) and similar values were also found in studies on spray drying. Alvarenga Botrel et al. (2012)reported values of moisture content between 1.3 and 3.65% of microcapsules of oregano essential oil using gum Arabic, modified starch and maltodextrin as wall materials. According toBotrel et al. (2014), the drying conditions are the main factors affecting the moisture content of micro particles, although different encapsulating material can have significant effect. It was observed that micro particles containing higher concentration of modified starch (GA25:MS and GA25:MS:MD) showed lower values of water activity (0.10).This result can be related to the structure of the modified starch, because it has fewer hydrophilic groups compared with GA and MD. However, samples containing only GA (GA100) and their mixture with MD (GA25: MD) showed higher moisture content of3%. The hydrophilic branches present in the GA and MD structures contribute to the water adsorption, and consequently, to the increase in the moisture content.

Table 2: Mean values for water activity and moisture content of the oregano essential oil micro particles

Microparticles	Water activity	Moisture Content (%)
GA100	0.180 ± 0.002	3 ± 1
GA50:MS:MD	0.12 ± 0.02	1.3 ± 0.3
GA25:MS	0.10 ± 0.01	1.1 ± 0.4
GA25:MD	0.18 ± 0.02	3 ± 1
GA25:MS:MD	0.10 ± 0.02	1.3 ± 0.7

Fonte: Da Costa, et al. (2013)

Morphology and particle size distribution

The scanning electron microscopic (SEM) images (Figure 1) showed that micro particles, for all samples, had an external topography characterized by spherical shapes with several sizes, no apparent pores, invaginations and continuous walls. Additionally, the micro particle structures had irregularities (depressions) on the surface. The formation of hollow particles is frequently observed in dried products by spray drying(Ré, 1998), and can be explained by the vacuoles produced inside the particles by the shrinking process that occurs after the development of the outer surface and the expansion of air trapped within the particles(Nijdam and Langrish, 2006; Ré, 1998). These imperfections on the surface of the particles have been reported by other authors and can be associated with the characteristics of the encapsulating agent and the conditions of the process(Alvarenga Botrel et al., 2012; Bertolini et al., 2001; Tonon et al., 2011; Trindade and Grosso, 2000).The mixture of different wall materials influences the morphology of the micro particles. However, no apparent differences in the surface characteristic of micro particles of oregano essential oil were observed. Janiszewska and Witrowa-Rajchert (2009) did not observe any differences in shapes of rosemary oil micro particles using different proportions of gum Arabic and maltodextrin as wall materials.

The integrality of the wall was evident for all samples, except for GA25: MS that showed cracks. This drawback does not ensure the protection of the core material and promotes permeability to oxygen and water vapor. Micro particles of essential oil that present cracks are susceptible to lose volatile compounds.

Table 3: Diameters of micro particles produced with different wall materials. [a-d] Letters represent a significant difference of 5% for the samples evaluated from different wall materials.

Microparticle	$D_{(0.1)}$ (µm)	$D_{(0.5)}$ (µm)	$D_{(0.9)}$ (µm)	$D_{(4,3)}$ (µm)	PDI
GA100	2.4 ± 0.2	8.1 ± 0.3	15.7 ± 0.3	8.5 ± 0.2 b	1.67 ± 0.04
GA50:MS:MD	2.29 ± 0.03	8.7 ± 0.1	17.2 ± 0.1	9.3 ± 0.1 c	1.72 ± 0.01
GA25:MS	2.3 ± 0.1	8.3 ± 0.3	15.7 ± 0.5	7.8 ± 0.3 a	1.63 ± 0.01
GA25:MD	3.0 ± 0.1	10.5 ± 0.1	19.7 ± 0.2	11.0 ± 0.1 d	1.60 ± 0.01
GA25:MS:MD	2.49 ± 0.02	9.1 ± 0.1	16.9 ± 0.1	9.0 ± 0.1 c	1.627 ± 0.003

Micro particles GA25: MD showed higher mean diameter, 11.0 µm, expressed by D [4, 3] (Brouckere mean diameter).When modified starch was used as wall material in micro particles GA25:MS, the size of the particles decreased and the mean diameter was 7.8 µm (Table3).

The size of spray dried particles is affected by several factors, such as viscosity, concentration of encapsulating material and drying conditions(Jafari et al., 2008). According toJafari et al.(2008) high inlet temperatures in the drying process produces larger particles than those dried under conditions that result in slow drying. In addition, larger particles have higher encapsulation efficiency(Jafari et al., 2008).

The diameters of 8.5 µm were obtained using only gum Arabic as an encapsulating material. These values were lower than those observed by Fernandes et al. (2014). The authors evaluated the effect of encapsulating materials in the microencapsulation of rosemary essential oil (Rosmarinus officinalis L.), and obtained higher average diameter values of 13.5 µm with gum Arabic as an encapsulating agent and13.4 µm using starch in the process.

Values of D_{10}, D_{50} and D_{90} ,which indicate diameters of 10%, 50% and 90%, respectively, of the volume of the group of micro particles, were determined (Table 3).The PDI of micro particles, calculated using the equation 3, were low (1.60–1.72), which indicates a homogeneous distribution.

The histogram showed in (Figure 1) shows a bimodal behavior with two different peaks, each of which is the predominant diameter. This behavior is interesting considering the powder storage, since the population of smallest particles can penetrate into the spaces between the larger particles, occupying a smaller space.

Differential scanning calorimetry (DSC)

The transition from the solid glassy state to the semi-liquid gummy state of a micro particles occurs at the glass transition temperature (Tg)(Costa et al., 2015). The Tg is specific to each material and is affected by many factors, such as chemical structure, molecular weight and moisture content of the micro particles (Fernandes et al., 2014; Toledo Hijo et al., 2014).By knowing the Tg of amorphous materials, it is possible to predict storage and processing conditions of dried products. This is because above this temperature, physicochemical and structure changes, such as collapse, stickiness and micro particle rupture, can occur, and consequently, release of the active material (Botrel et al., 2014; Netto et al., 1998).

The DSC curves are shown in (Figure 2), representing the Tg of micro particles obtained from the midpoint of the glass transition range. The Tg determined by DSC analysis were 97.02 °C, 85.25 °C, 96.58 °C, 94.61 °C and 84.55 °C for micro particles GA100, GA50:MS:MD, GA25:MS, GA25:MD and GA25:MS:MD, respectively. The micro particles with gum Arabic as wall material (GA100) showed higher values of Tg (97.02 °C). According toBhandari and Howes (1999), among the solid components of amorphous dried foods, carbohydrates have more influence in the glass transition temperature. Wall materials that have high molar mass show high glass transition temperature. Thus, considering the materials studied in this work, GA has a higher molar mass(47,000–3,000,000 g/mol)(Anderson, 1977), and consequently, a higher Tg compared with MD (DE 20) and MS, which have molar masses of900 g/mol(Roos and Karel, 1991) and2,800,000 g/mol(Nilsson et al., 2006), respectively. The presence of GA in the encapsulating matrix of essential oil micro particles contributes for obtaining more stable products. This means that the higher the Tg of dried products, the higher their thermal stability, i.e., at temperatures above 97.02 °C, the micro particles GA100 changed from the glassy state to the gummy state and changes in their structure occurred.

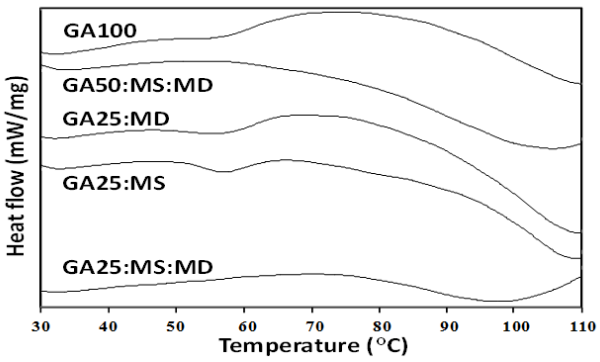

Figure 2: DSC curves obtained at heating temperature rate of 10°C min^{-1} for micro particles of oregano essential oil

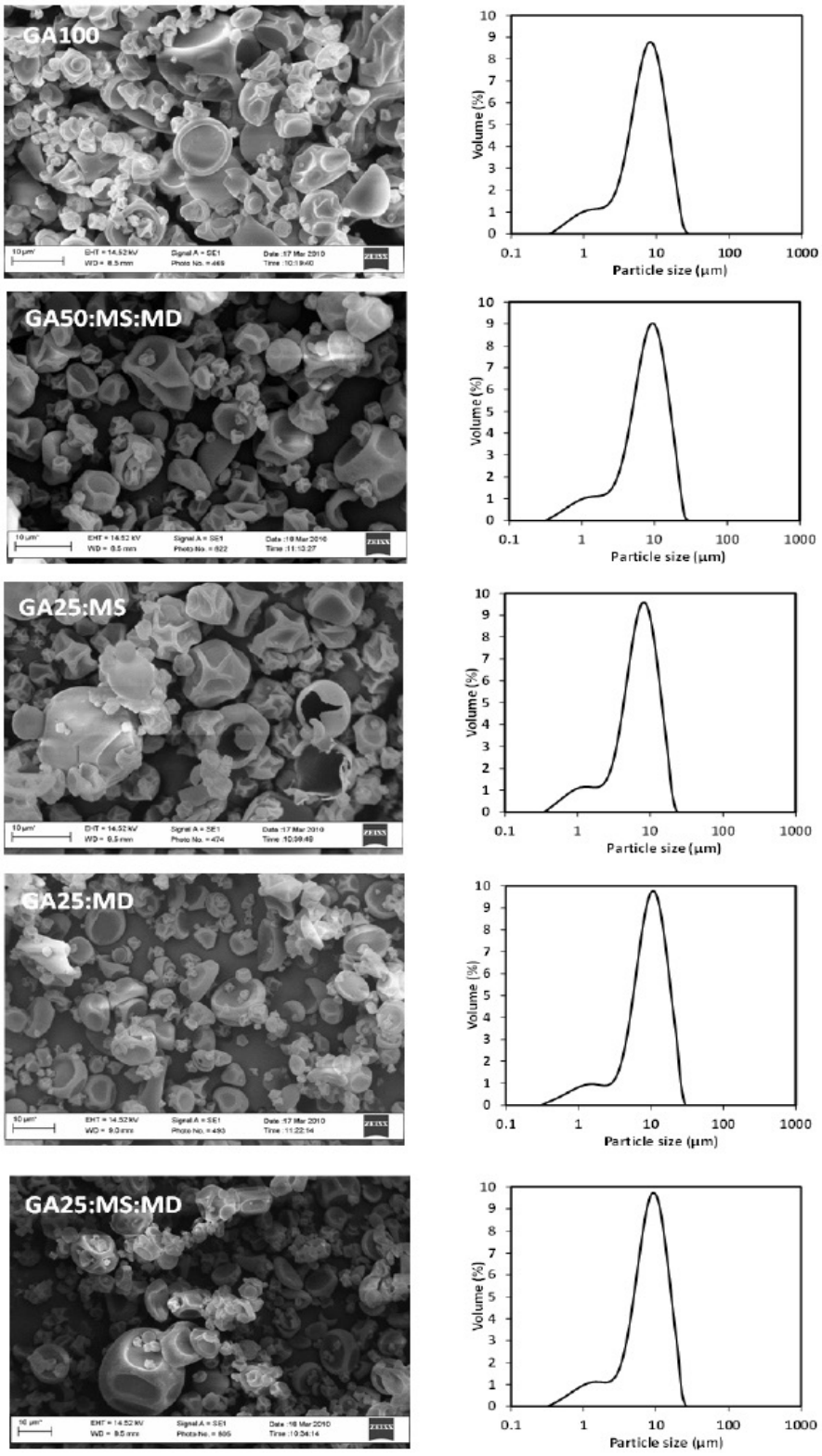

Figure 1: Morphology and particle size distribution of micro particles produced with different wall materials

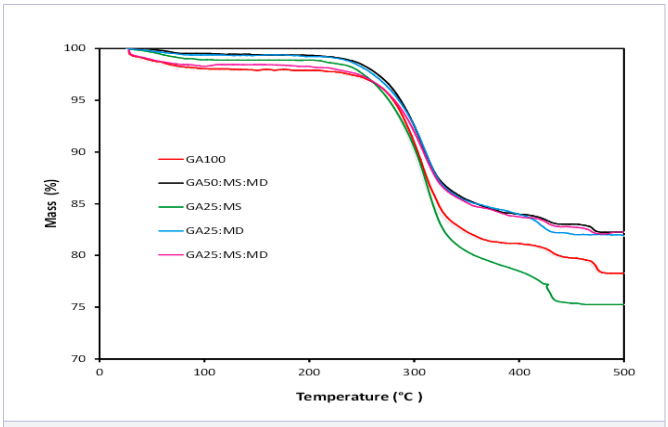

Figure 3: TGA curves for thermal decomposition of oregano essential oil micro particles

The use of MS and MD in the formulation of micro particles of oregano essential oil for the substitution of GA resulted in a slight reduction of Tg. However, GA25: MS and GA25: MD showed Tg values close to the Tg of micro particles containing only GA (GA100). This reveals that there is a potential for the application of MS and MD as wall materials as a substitution of GA in these concentrations, taking into account physical and thermal stability aspects. On the other hand, the use of MD and GA (GA25: MD) resulted in micro particles with lower Tg than those with MS and GA (GA25: MS) as wall materials. This difference is related to the lower molar mass of MD compared with MS, causing the opposite effect of GA(Fernandes et al., 2014).Toledo Hijo et al. (2014) studied the influence of water acting as plasticizer in the thermal properties of oregano essential oil micro particles stored at different relative humidity, containing GA and MS as wall materials and found Tg values between 91.29 and 164.47 °C. However, studies evaluating the effect of wall materials on the thermal properties of micro particles of oregano essential oil were not found in the literature. Micro particles with MS, MD and GA (GA50: MS: MD and GA25:MS: MD)in their formulation showed the lowest values of Tg, and as the concentration of GA decreased, their Tg also decreased, presenting a lower physical stability.

Considering the Tg as an indicator of micro particle stability in storage conditions(Bhandari and Howes, 1999; Ferrari et al., 2013), the micro particles of oregano essential oil were stable in storage at 25 °C, i.e., they remained in the glassy state (amorphous) at this temperature, which is lower than the glass transition temperatures of the micro particles studied in this work (between 84.55 °C and 97.02 °C).

Thermo gravimetric analysis (TGA)

The TGA thermo gram of the micro particles is shown in Figure 3. The thermal degradation of the samples showed three steps of 97.45% of mass loss in the 25-500 °C temperature range. Thermal decomposition of three steps of systems containing polymers similar than those used in this work has been reported by several authors(Janković, 2013; Liu et al., 2008; Mudgil et al., 2012). The results of TGA showed that the nature of the wall materials have considerable effect in the thermal degradation steps of micro particles. The first stage represents the evaporation or dehydration process from the start of heating until at temperature of 164 °C with 1.31% of average mass loss. At this stage, loss of water occurred corresponding to the critical moisture content, adsorbed volatile compounds or unencapsulated oil of micro particles. The micro particles GA25: MD and GA50: MS: MD showed higher initial degradation temperatures (T_{onset})of 64.64 and 63.03 °C and lower percentage of mass loss of 0.62 and 0.63% (M_{loss}), respectively. Micro particles that have a lower percentage of mass loss variation result in more stable products to thermal degradation(CANEVAROLO, 2003), indicating that the micro particles GA25:MD and GA50:MS:MD had higher thermal stability in the first step of thermal decomposition.

The second step of thermal decomposition occurred at the average temperature of 283.36 °C, showing the highest percentage of Mloss of 79.57%.At temperatures between 194.71 and 372.00 °C, the depolymerization process and thermal degradation of the wall material of micro particles occurred. Mudgil et al. (2012) reported thermal decomposition of guar gum at approximately 280 °C. The thermal degradation temperature of starches has been reported by several authors to be approximately 300 °C(Guinesi et al., 2006; Liu et al., 2008; Soares et al., 2005). At this temperature a release of gases, such as CO_2, CO and water occurred, due to the pyrolysis of the starch.

The third step of thermal decomposition corresponds to the oxidation of the organic matter or inert carbonaceous residues with an average loss of mass of 2.26% approximately at temperatures closed to 425.33 °C.

Conclusion

Lower water activity was obtained in micro particles containing higher concentration of modified starch (GA25: MS and GA25: MS: MD). The micro particles showed spherical shapes with irregularities on the surface. The DSC results showed that powders containing only GA as wall material presented higher physical stability, i.e., higher Tg (97.02 °C). However, MS (GA25:MS) and MD (GA25:MD) used as wall materials also presented high physical stability with Tg values of 96.58 °C and 94.61 °C, respectively. TGA results presented three steps of thermal decomposition, commonly for carbohydrate polymers. Also, TGA thermo gram showed that the nature of biopolymers have considerable effect on the thermal degradation of oregano essential oil micro particles. The replacement of GA by MS or MD or the mixture of MS and MD is feasible. Thermal analysis revealed the potential of application of these biopolymers for substitution of GA.

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