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REGULAR ARTICLE

Hygroscopic thermal and chemical properties of cinnamon essential oil microparticles obtainded by spray drying

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ABSTRACT

This study aimed at evaluating the potential of gum arabic in combination with maltodextrin for the microencapsulation of cinnamon essential oil by spray drying to maximize its hygroscopic, thermal and chemical stability. The corresponding isotherm exhibited type II behavior (sigmoidal curve), and the best fit was obtained for the GAB model (E = 4.81%). Differential scanning calorimetry (DSC) analysis showed that the increased moisture content caused a significant reduction of the glass transition temperature (T_g) of the microparticles. Microparticles stored at intermediate humidity exhibited thermal stability and a lower mass loss, while storing at low temperatures led to higher antioxidant capacity and cinnamaldehyde retention.

Keywords: Sorption Isotherms; Glass transition; Cinnamon essential oil; Antioxidant activity; Gordon-Taylor equation

PRACTICAL APPLICATIONS

Knowledge about the stability of encapsulated foods is of great importance for the food industry in which the behavior and physicochemical and sensorial alterations of these microparticles subjected to different conditions of storage and process can be predicted.

INTRODUCTION

Cinnamon essential oil (*Cinnamomum zeylanicum*) is one of the most used products in the pharmaceutical, food, and cosmetic industries due to its chemical and sensory characteristics. The oil is predominantly composed of cinnamaldehyde, which exhibits excellent antioxidant and antimicrobial activity (Cardoso-Ugarte et al., 2016).

Spray drying is the most common dehydration process used for sensitive foods as flavors, essential oils, herbal extracts, enzymes, lipids, oleoresins, fruits, and colorants, pharmaceuticals (Cortés-Rojas & Oliveira 2012, Goula & Adamopoulos 2012), and other substances because of the rapid solvent evaporation from the droplets (Mahdavi et al., 2014). This process involves packaging the core material in a polymer matrix in three steps: atomization, dehydration, and powder collection for increased protection and stability (Fang & Bhandari 2012, Fernandes et al., 2013).

Gum arabic and maltodextrin are the materials most commonly used to ensure good protection of the encapsulated compounds (Akhavan Mahdavi et al. 2016b, Ferrari et al., 2013, Mahdavee Khazaei et al., 2014, Oliveira et al., 2013, Pitalua et al. 2010, Rajabi et al., 2015, Rocha-Parra et al., 2016). Carbohydrate biopolymers exhibit plasticizer characteristics, promoting the formation of spherical and smooth-surface microcapsules and increasing the bonding strength between the wall and core material. This strong interaction inhibits undesirable reactions caused by different storage conditions (Akhavan Mahdavi et al., 2016a,b).

This study aimed to evaluate the stability of cinnamon essential oil microcapsules during spray drying using

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maltodextrin and gum arabic as wall materials. Adsorption isotherms, thermal analysis, antioxidant activity, and oil retention analyses were performed to predict the best storage conditions.

MATERIALS AND METHODS

Materials

Cinnamon essential oil (Ferquima, São Paulo, Brazil) extracted from the leaves was used as the microencapsulation material. The wall materials consisted of gum Arabic (GA; Nexira, São Paulo, Brazil) and maltodextrin Maltogil DE 20 (MD; CARGILL FOODS, São Paulo, Brazil) based wall material which provides a better protection efficiency cinnamaldehyde in previously study (Felix et al., 2016). Maltodextrin DE 20 was chosen because of its short chain, favoring the formation of a more permeable structure (Abd Ghani et al., 2017).

Emulsion preparation

A biopolymer solution (1: 3 biopolymer/water) was prepared and stored for 24 hours for complete dissolution. The ratio of the components used to prepare the wall materials blends was 1:1 (gum arabic: maltodextrin; w/w). Cinnamon essential oil (5% oil/emulsion) was added to the biopolymer solution and subjected to homogenization (Ultraturrax, Tecnal, Picaricaba, Brazil) at 8,000 rpm for 2min. Emulsions containing 30% (w/w) of the wall material were used as a feed liquid for the spray-drying process (Felix et al., 2016).

Spray-drying process

The emulsions were dried using a spray-dryer (model MSD 1.0; Labmaq do Brasil, Ribeirão Preto, Brazil) equipped with a two-fluid nozzle atomizer (nozzle system of diameter 3 mm in a drying chamber of dimensions 670 mm height and 200 mm of diameter). The feed emulsion flow was kept in 0.7 L/h and drying air flow was 40 L/min. The inlet was maintained at 180°C (Martins et al., 2014). The microparticles obtained were sealed aluminum packing and stored under refrigeration (4 to 7°C), in protected from temperature, light penetration and gas permeation until further analysis (Fernandes et al., 2013).

Thermal analysis

Thermogravimetric analysis (TGA, DTG-60H, Shimadzu, Japan) was used to determine the stability of microparticles at high temperatures. Approximately 3-mg samples were prepared in aluminum pans. The thermal program used a temperature range of 25 to 550°C and a heating rate of 10°C/min under a flow nitrogen gas at 100mL/min (Toledo Hijo et al. 2015).

Sorption isotherms

The sorption isotherms of microparticles were determined by a gravimetric static method using a saturated saline solution at 25°C. Seven saturated saline solutions (NaCl, K_2CO_3, $MgCl_2$, LiCl, $Mg(NO_3)_2$, KCl, and $NaNO_3$) were used, with water activity (A_w) varying from 0.11 to 0.84. The moisture adsorption isotherms were correlated to water activity using different models (Table 1). The parameters of these equations were estimated by correlating the mathematical models with experimental data using a quasi-Newton nonlinear regression of the Statistica 8.0 software (Statsoft, Tulsa, USA) at a 5% significance level (Botrel et al., 2014). The lower mean relative percentage deviation modulus ($E(\%)$) is defined in Eq. 1:

$$E(\%) = \frac{100}{n}\sum_{i=1}^{n}\left(\frac{\left|Y - \hat{Y}\right|}{Y}\right) \quad (1)$$

Glass transition temperature

The glass transition temperature (T_g) was determined by differential scanning calorimetry (DSC) using a DSC-60 instrument (Shimadzu, Japan). The detection limit of the apparatus was 0.3W. Approximately 1 mg samples were prepared in aluminum pans, with an empty pan used as reference. Each sample in the relative humidity study was subjected to heating from −60 to 180°C at a rate of 10°C/min under a nitrogen gas flow of 50mL/min (Toledo Hijo et al., 2015). Two runs were performed for each sample, with the second scan reducing the relaxation enthalpy of the amorphous powder that appears in the first scan and thereby enhancing the accuracy of T_g measurement in the DSC thermogram. All relative humidity measurements were done in triplicate. The Gordon-Taylor model (Eq. 2) was used to probe the

Table 1: TGA of cinnamon essential oil microparticles at different equilibrium moisture contents

Aw	Stage 1			Stage 2		
	T_0	T_F	ML (%)	T_0	T_F	ML (%)
0.113	28.81±3.57^{a}	112.37±4.49^{a}	8.18±3.22^{a}	209.58±2.48$^{a.b}$	356.55±2.19^{a}	53.96±0.83^{a}
0.328	26.44±3.22^{a}	102.58±1.42^{a}	7.94±0.35^{a}	212.85±1.31$^{a.b}$	360.03±3.72$^{a.b}$	52.53±0.37^{a}
0.432	29.26±3.85^{a}	135.59±4.92^{b}	10.04±0.27^{a}	215.86±1.99$^{a.b}$	360.28±3.53$^{a.b}$	45.32±1.00^{b}
0.743	30.92±6.11^{a}	143.21±3.53^{b}	8.78±1.96^{a}	207.24±1.54^{a}	368.60±6.41^{b}	51.09±0.08^{a}
0.753	31.60±2.29^{a}	144.90±4.21^{b}	9.92±0.38^{a}	213.11±4.82$^{a.b}$	356.35±6.08^{a}	51.77±0.83^{a}
0.843	28.91±0.94^{a}	186.58±1.87^{c}	13.17±0.77^{b}	218.64±1.45^{b}	344.36±8.48^{c}	54.11±1.28^{a}

T_0: Stage initial temperature; T_F: Stage final temperature; ML (%): Mass loss. ^{a,b,c}: Identical letters in same column represent no significant difference (p>0.05)

plasticizing effect of water in the microcapsules. The glass transition temperature of water was assumed to be −135°C (Tonon et al., 2009):

$$T_g = \frac{w_s T_{gs} + k w_w T_{gw}}{w_s + k w_w} \quad (2)$$

where T_g, T_{gs}, and T_{gw} are the glass transition temperatures of microparticles, anhydrous solids, and amorphous water, respectively; w_s and w_w are the mass fractions of solids and water, respectively, and k is a constant proportional to the plasticizing effect of the diluent, in this case water. The parameters of these equations were estimated by correlating the mathematical models to experimental data using a quasi-Newton nonlinear regression of the Statistica 8.0 software (Statsoft, Tulsa, USA) at a 5% significance level.

Cinnamon essential oil retention and antioxidant activity stability

Essential oil microcapsule samples (~ 1g) were packaged in Petri dishes (60 mm x 10 mm) were subjected to four different storage conditions (25°C in light, 25°C in the dark, 4°C in the dark, and 35°C in the dark with relative humidity of 63%) in a temperature controlled chamber at two-week intervals to evaluate the antioxidant activity stability of microencapsulated cinnamon oil (Cano-Higuita et al., 2015, Mahdavee Khazaei et al., 2014).

Cinnamaldehyde was used as a reference compound to evaluate the retention of essential oil within microparticles, being the major component of the latter oil. To evaluate the oil composition before and after microencapsulation, gas chromatography (GC) analysis was performed on an HP7820A gas chromatograph (Agilent) equipped with a flame ionization detector. The samples (~30mg) were kept in chloroform (500µL) and ultrasonicated for 15min. After centrifugation at 6,500rpm, an aliquot (2µL) of the supernatant was subjected to GC analysis (HP5 column, 30m ×0.32mm ×0.25µm, Agilent). The initial column temperature was 100°C, which was ramped at 5°C/min to 200°C; the injector (splitless) and detector temperatures were 200 and 220°C, respectively. Hydrogen was used as a carrier gas (3mL/min), and the injection volume was 2µL. Data were acquired using the EZChrom Compact Elite software (Agilent). Cinnamaldehyde levels at different concentrations of cinnamon essential oil were recorded and plotted. The data were fitted to a linear regression equation (Eq. 3):

$$y = 0.242\,x + 0.012;\ R^2 = 0.997 \quad (3)$$

where x is the cinnamaldehyde pick area, and y is its concentration in cinnamon essential oil (mg/mL).

Antioxidant activity (AA, %) was evaluated using the DPPH radical (1,1-diphenyl-2-picrylhydrazyl) according to the modified method of Mensor et al.(2001) (Mensor et al., 2001). Oil extraction was performed by adding 0.1g of microparticles to 50mL of ethanol: water solution (1:2 v/v). The mixture was homogenized using an ultrasonic homogenizer (Branson) at 250W for 1min.

The tubes were charged with 2.4mL of absolute ethanol, 1mL of DPPH solution (2mg/50mL), and 0.1mL of the post-extraction sample. To eliminate a possible contribution of staining, samples were analyzed in parallel, utilizing a white test sample volume (0.1mL) and 3.4mL of absolute ethanol. The control was prepared by mixing 1.0mL of DPPH solution with 2.5mL of absolute ethanol. After 1-h incubation in the dark at room temperature, the absorbances were recorded against a blank at 517nm. Tests were performed in triplicate, and the inhibition of free-radical DPPH was calculated using Eq. 4:

$$AA(\%) = 100 - \left[\left(100 \times \frac{A_A - A_B}{A_C}\right)\right] \quad (4)$$

where A_A is sample absorbance, A_B is blank absorbance, and A_C is control absorbance.

Statistical analysis

All experiments were carried out in triplicate. The mean and standard error of each treatment were calculated from replicates. Variance analysis (ANOVA) was carried out to determine any significant differences between treatments ($p<0.05$).

RESULTS AND DISCUSSION

Thermogravimetric Analysis

Fig.1 shows the thermogravimetric analysis results for cinnamon essential oil microparticles stored at different humidity levels. Many authors have stated that the carbohydrate thermograms feature three stages: 1) loss of moisture and volatile material on the surface; 2) degradation of microparticles; 3) degradation of the remaining organic compounds (Silva et al., 2016, Toledo Hijo et al., 2015). All thermograms showed two weight loss stages, which is important for understanding microparticle behavior during heating. Since no differences were observed between stages 2 and 3, they were combined to stage 2 (degradation of wall material).

The first stage (Table 1) shows a significant increase ($p<0.05$) of weight loss (%) with increasing storage humidity, caused by absorption of more water by the microparticles. Klein et al. (Klein et al. 2015) observed

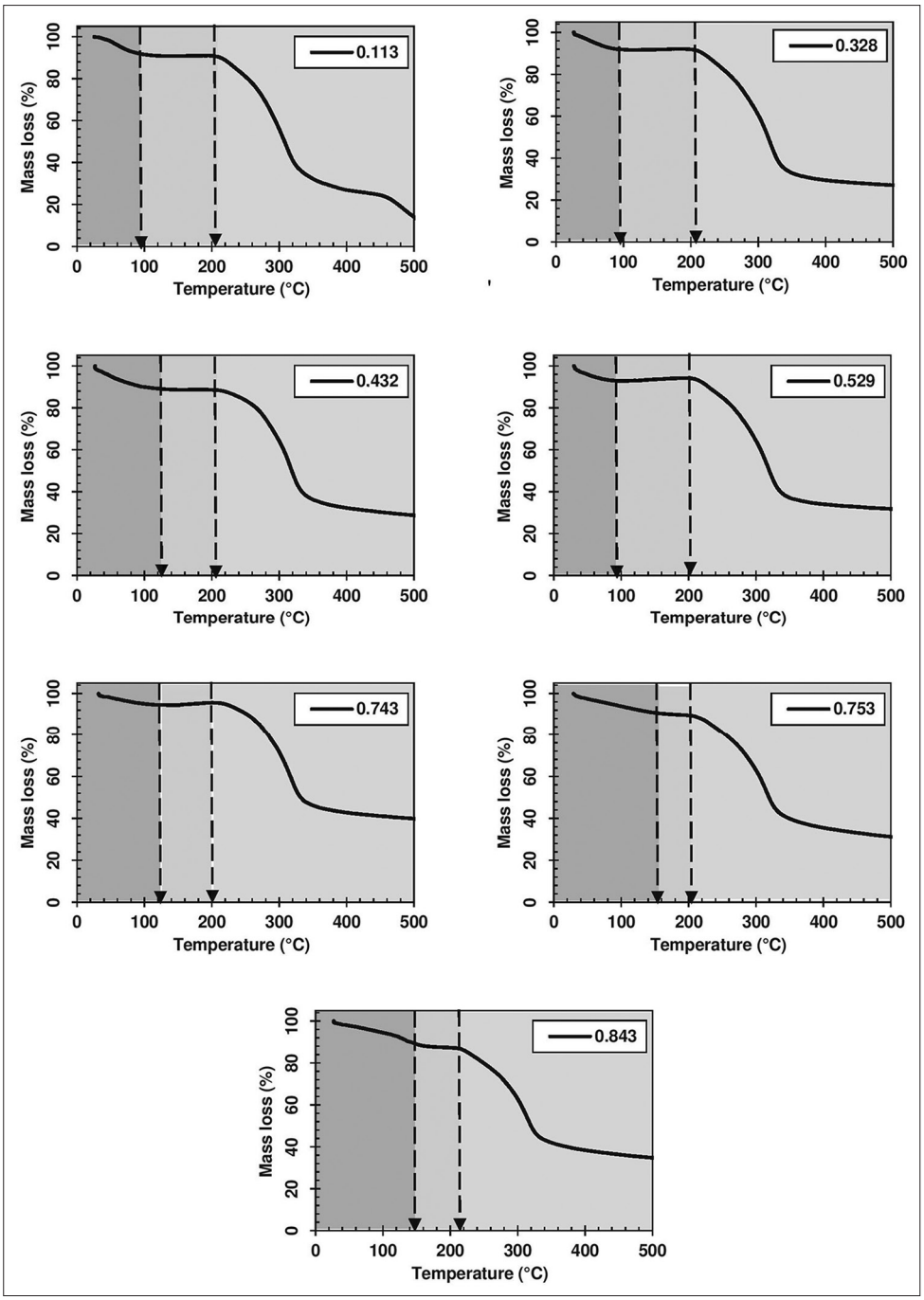

Fig 1. TGA curves of cinnamon essential oil microparticles subjected to storage at different humidity. The different shades of gray represent TGA stages.

no difference in the TGA curves for gum arabic and maltodextrin materials, and gum arabic/maltodextrin microparticle containing guaraná extract obtained by spray-drying technique, excluding any interference of the material in resistance to thermal degradation. The significant weight loss difference in the first stage can also be related to the morphology and particle size of the biopolymer. Another important factor is the hygroscopic nature of gum arabic, favoring the absorption of water by microparticles (Carrillo-Navas et al. 2011, Ferrari et al. 2013, Rodríguez-Hernández et al. 2005).

The second stage (Table 1) is of greatest importance for food microencapsulation studies, since it shows the maximum temperature at which microparticles do not exhibit structural deformation or degradation of the wall material. A comparison of all TGA curves at different relative humidity values showed the least microparticle degradation

at A_w=0.432. Da Costa et al. (Gomes da Costa et al., 2015) also observed greater stability of Swiss cheese bioaroma microcapsules at intermediate relative humidity. The same authors suggested that a gradual temperature increase may change the biopolymer structure, facilitating intermolecular bond formation and improving thermal stability.

Sorption isotherms and glass transition temperature

Water activity and the glass transition temperature are good parameters for predicting the quality of food. To ensure the stability of food products, the water activity needs to be close to the monolayer moisture content, and the process temperature needs to be below T_g (Vega-Gálvez et al., 2014).

The moisture content and glass transition temperature for each studied relative humidity are shown in Table 2. The former is an important parameter for predicting the final quality of powdered foods, since its high values can accelerate undesirable chemical and physical reactions. The amount of water absorbed by food depends on the vapor pressure at which the product is stored (Botrel et al., 2014). We observed that the moisture content increased with increasing relative storage humidity.

Table 3 shows the model isotherm parameters obtained from experimental data. All models presented a good fit, with errors between 4.81 and 7.49. According to Lomauro, Bakshi, & Labuza (1985) *E* was less than 10%, confirming the good fit of experimental data. Fig. 2 shows experimental adsorption isotherms and model fits for cinnamon essential oil microparticles at 25°C. The adsorption isotherms showed an increase in equilibrium moisture content with increasing water activity. The GAB model is one of the most studied, primarily because its coefficients are related to physical and chemical adsorption parameters (Silva et al., 2015). To properly reflect the sigmoid isotherm type (Type II), the values of GAB equation coefficients are confined to the range of $0.24<K<1$ and $5.67<C<\infty$ (Lewicki 1997).

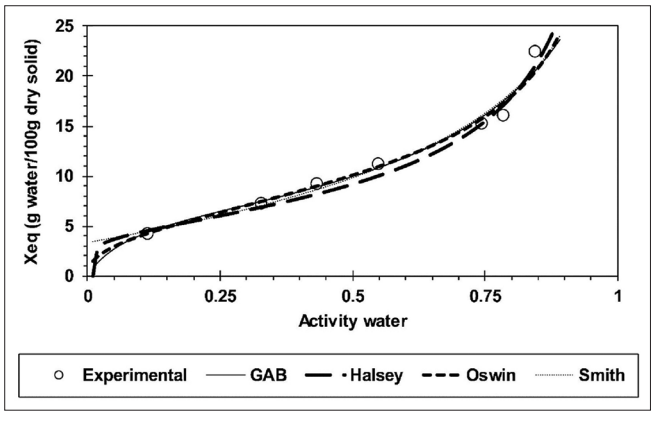

Fig 2. Adsorption isotherm for cinnamon essential oil microencapsulated at 25°C.

The observed monolayer moisture content was 6.274g water/100g dry solid. The importance of knowing the latter quantity is associated with adding a minimum amount of moisture to food in order to reduce its deterioration and increase shelf life (Choudhury et al. 2011). Higher monolayer moisture contents may be related to the structural characteristics of biopolymers used as wall materials.

In their studies of water adsorption in different biopolymers, Pérez-Alonso et al., (2008) and Rascón et al., (2015) observed values of 8.11 and 6.894g water/100g dry solid, respectively, for monolayer moisture content in gum arabic. Using blends of gum arabic and maltodextrin Ferrari et al. (2012) observed a monolayer moisture content of 8.60g water/100g dry solid.

The glass transition temperature is related to stickiness, crispness, collapse, amorphous-to-crystalline transformations, and the rates of non-enzymatic browning in foods (Vega-Gálvez et al., 2014). Due to the amorphous nature of carbohydrates, studying the glass transition

Table 2: Equilibrium moisture content and glass transition temperature at different relative humidity values

Aw	X_{eq} (g water/100g dry solid)	Tg (°C)
0.113	4.32±0.02	83.9±7.3
0.328	7.25±0.01	80.6±4.92
0.432	9.22±0.22	71.02±2.93
0.549	11.2±0.41	49.59±3.85
0.743	15.3±0.03	20.81±5.41
0.783	16.1±0.44	11.96±2.22
0.843	22.5±0.22	–7.6±2.65

Aw: Activity water; X_{eq}: Equilibrium moisture content; Tg: Glass transition temperature

Table 3: Isotherm models and Gordon-Taylor model coefficients

Model	Coefficients	Values
GAB	X_0 (g water/100g dry solid)	6.276
	C	15.929
	K	0.832
	E (%)	4.810
Halsey	A	12.142
	B	1.652
	E (%)	7.487
Oswin	A	10.097
	B	0.420
	E (%)	5.547
Smith	A	3.335
	B	21.586
	E (%)	6.569
GORDON-TAYLOR	k	0.136
	T_{gs} (°C)	84.841
	E (%)	6.820

X_0: monolayer moisture content; A, B, C, K and k: model×s coefficients; T_{gs}: Glass transition temperature of anhydrous biopolymers

temperature is important to understand their behavior in industrial processes (Ruiz-Cabrera & Schmidt 2014). In amorphous systems, the glass transition is a kinetic phenomenon associated with the change in the polymer physical state, which depends on temperature, time, and composition of the material (Collares et al., 2002).

The Gordon-Taylor model can be used to predict the glass transition temperature in binary solid/water systems subjected to storage at different relative humidity levels (Goula et al., 2008, Gustavo V. Barbosa-Cánovas et al. 2008). A plasticizer can be defined as a material which increases the viability of flexibility and extensibility when incorporated into a polymer. At the molecular level, this leads to increased free volume, decreasing viscosity and increasing wettability (Collares et al., 2002).

We observed that T_g decreased at increased water activity (83.90 to −7.6°C). According to Table 3, the *k* coefficient of the Gordon-Taylor model was 0.136, and T_{gs} was equal to 84.84°C. Soottitantawat et al. (2004) studied the effect of humidity on the storage of encapsulated D-limonene and observed T_g values of 79.3, 51.2, and 14.3°C at water activities of 0.33, 0.54, and 0.75, respectively, corroborating the results of our work.

Tonon et al. (2009) and Kurozawa et al. (2009a) obtained T_{gs} values of 88.29 and 79.12°C, and 94.70 and 91.90°C for gum arabic and maltodextrin, respectively. The same authors reported that using these compounds in microencapsulation processes may promote product thermal stability due to the increase of T_g. Mosquera, Moraga, & Martínez-Navarrete (2012) observed improved stability of strawberry powder when using maltodextrin and gum arabic as wall materials.

One way to represent the variation of moisture content and the glass transition temperature as a function of water activity is to plot the two curves on the same graph (Fig. 3) and predict the critical storage conditions of cinnamon essential oil microparticles, as is well documented in literature (Kurozawa et al. 2009; Moraga et al. 2004, 2006; Toledo Hijo et al. 2015, Tonon et al. 2009).

It can be seen that at room temperature (25°C) the storage conditions are critical for higher water activity and equilibrium moisture content of 0.743 for 15.30g water/100g dry solid, respectively. Our results agree with those of Kurozawa et al. (2009a) who studied the use of gum arabic and maltodextrin as carrier agents in hydrolyzed chicken meat proteins.

Retention of cinnamon essential oil

The concentration of cinnamaldehyde in microcapsules at different storage conditions as a measure of cinnamon essential oil retention is shown in Fig. 4, being higher at lower temperatures. The wall material used could also protect the core material during the first 45 days. Subsequently, the amount of essential oil was significantly reduced. Note that better cinnamaldehyde retention values were observed for the refrigeration temperature of 4°C in comparison with other temperatures. This indicates the effect of temperature on the stability of essential oils, since higher temperatures favor their degradation and volatilization by diffusion through the pores of the wall material.

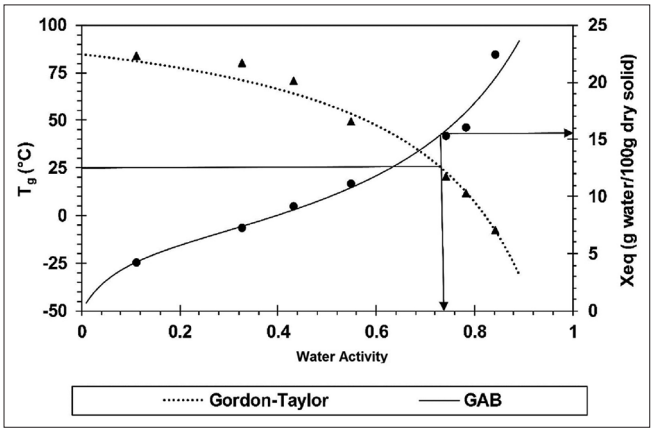

Fig 3. Relationship between the water activity at 25°C, water content, and the glass transition temperature of cinnamon essential oil microparticles.

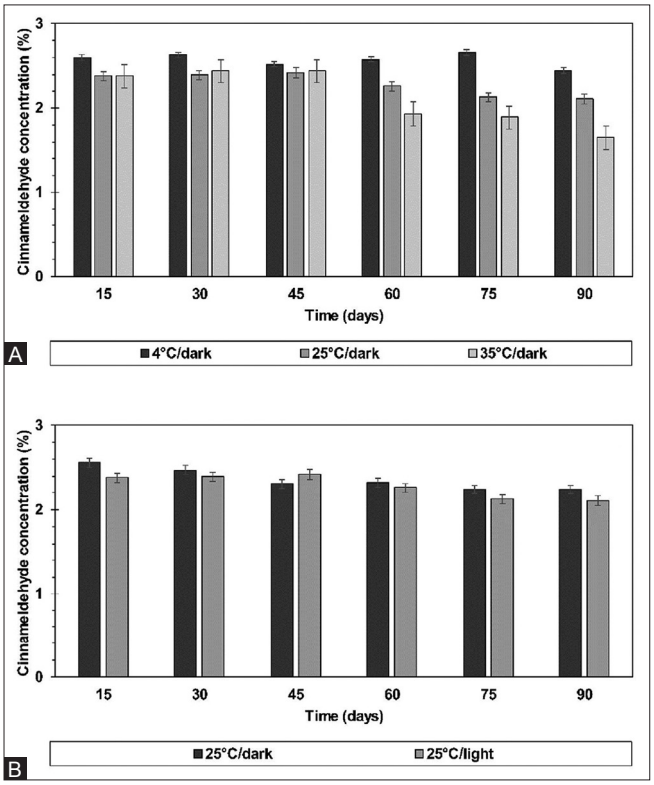

Fig 4. Cinnamaldehyde concentration (%) in cinnamon essential oil microparticles subjected to storage at different (A) temperature and (B) luminosity.

Delfiya et al. (2015) evaluated the effect of temperature on the retention of turmeric oleoresin using maltodextrin and gum arabic as wall materials and noted reduced oil retention at increased temperatures. This reduction could be caused by disruption of the wall material, exposing the core material to degradation and volatilization.

When studying the effect of light (Fig. 4B), we noticed only small differences in the retention of cinnamaldehyde by microcapsules at constant temperature, and can therefore conclude that the wall materials used provided the microparticles with good protection against luminosity. These results agree with those obtained for antioxidant activity in Fig. 5. Delfiya et al. (2015) studied the effect of light on the degradation of turmeric oleoresin microencapsulated in gum arabic and maltodextrin, and observed a high protection degree for 45 days of storage, in agreement with this work. The authors suggested that gum arabic protects the oil due to film formation during the drying process. Matioli and Rodriguez-Amaya (2002) studied the stability of lycopene encapsulated with maltodextrin and gum arabic during storage in light and dark. These authors observed that the synergy between biopolymers improves the stability of the encapsulated material compared to the effect of individual components. According to Bhandari (1992) higher concentration of solids results in greater protection of the core material and hence greater stability.

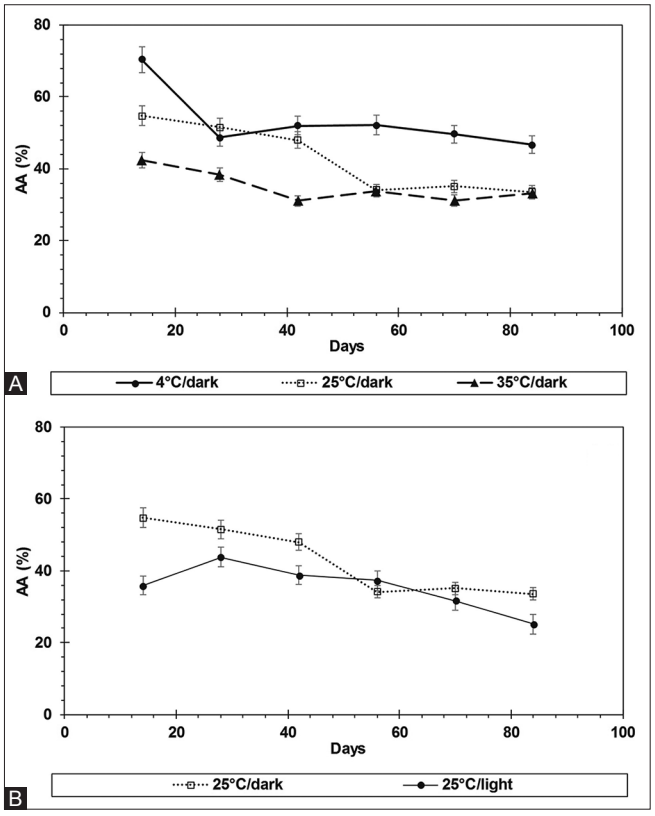

Fig 5. Effect of temperature (A) and light (B) on AA (%) of cinnamon essential oil microparticles.

Antioxidant activity

The antioxidant activity of food can be defined as its ability to defend the human organism against radicals and prevent neurodegenerative disorders caused by persistent oxidative stress (Rocha-Parra et al., 2016). Studies on essential oil microencapsulation necessitate the knowledge of their stability, since these oils are used as natural antioxidant sources (Venkateshwarlu G et al., 2014). The effect of temperature and light exposure on antioxidant activity is shown in Fig. 5. Increased temperature had a significant effect on AA during the storage period. Higher AA values were observed at a refrigeration temperature of 4°C in comparison with 25°C, confirming that temperature may affect the stability of encapsulated essential oils. We further observed that the AA remained practically constant after 56 days for all temperatures studied. When investigating the effect of light on microcapsule stability, the former was found to significantly interfere with the AA of encapsulated cinnamonessential oils.

Guan and Zhong (2015) observed antioxidant activity values above 60% for anthocyanins encapsulated by gum arabic. Ferrari et al., (2013) and Carvalho et al., (2016) observed significant increases in the antioxidant activity of blackberry and jussara extract microparticles in polymer blends of gum arabic and maltodextrin, respectively, compared to gum arabic only. Gum arabic effectively protects sensitive compounds due to its structure, which shows numerous ramifications and a small amount of protein-linked carbohydrate chains, facilitating the formation of films around these compounds and protecting them from adverse storage conditions (Kuck & Noreña 2016).

CONCLUSIONS

TGA curves indicated that the degradation onset temperature of the wall material was close to 200°C, regardless of the relative storage humidity. A high moisture content was observed due to the highly hygroscopic nature of gum arabic. The results confirmed the strong T_g plasticizing effect of water in the wall materials. All sorption isotherm models showed good fits with E<10%. A decrease in antioxidant activity was observed during storage, with the best results obtained for samples stored at 4°C. Samples stored in the light also showed significantly different behavior compared to those stored in the dark. The wall material used showed good oil retention during the first 45 days of storage. After this interval, the retention of cinnamon essential oil was influenced by temperature. Light was also a significant factor for oil retention, as

compared to storage in the dark. Therefore, gum arabic and maltodextrin proved to be good wall materials for the conservation of microencapsulated cinnamon essential oils.

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Author's contributions

P. H. Campelo-Felix and R. V. B. Fernandes wrote the article and corrected it. D. A. Botrel and S. V. Borges designed the study. P. H. Campelo-Felix, J. A. Figueiredo, V. Ferraz and M. I. Yoshida conducted the experimental work.

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