

# Dark chocolate with a high oleic peanut oil microcapsule content

Silvia AC Agibert<sup>1</sup> and Suzana C da S Lannes<sup>2\*</sup>

## Abstract

**BACKGROUND:** In accordance with the market demand for healthier indulgent food products, the present study aimed to determine the viability of the industrial production of dark chocolate with microcapsules of high oleic peanut oil content. Microcapsules of high oleic peanut oil were added to a control formulation using variations of mixing time.

**RESULTS:** The chocolates presented a rheology characterized by a pseudoplastic behavior adjusted to the Casson model ( $r > 0.98$ ) and calorimetric behavior indicating melting onset (21 °C), peak melting (32 °C) and melting end (41 °C); caramelization peak (183 °C); and carbonization peak (237 °C), being considered thermal stable. The mixing time and the amount of microcapsules added to the control chocolate did not significantly influence the flow limit ( $11.09 \pm 1.73$  Pa) or the physical characteristics of the chocolate: pH ( $6.74 \pm 0.14$ ), maximum particle size ( $0.019 \pm 0.001$  mm), water activity ( $0.358 \pm 0.023$ ) and brittleness ( $18.61 \pm 3.74$  N). However, the addition of microcapsules with a high oleic peanut oil content significantly increased the chocolate whiteness index, thixotropy and Casson's plastic viscosity, although it did not have a significant influence on the mixing time.

**CONCLUSION:** The products obtained have a desirable quality and physical properties, being suitable for industrial production.  
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**Keywords:** thermal analysis; rheology; Casson; thixotropy; texture analysis

## INTRODUCTION

The current growth in demand for healthier food products and the presentation of studies demonstrating that a significant portion of the population has high levels of cholesterol suggests that there is a need to explore feasible alternatives for the use of nutritional oils in the development of healthy food products with nutritional properties of interest for the population. Chocolate is one of the most consumed and appreciated foods in the world. Standing out among the different factors that differentiate it from other foods, as well as influencing its sensorial characteristics and its nutritional and functional profile, is the presence of flavonoids (epicatechin, catechin, and procyanidins), which are antioxidant compounds present in the cocoa pulp with an effect on human vascular health.<sup>1</sup> In addition to dark chocolates with a high cocoa content, premium, gourmet and products containing other intentionally added bioactive compounds are also trending in the world market.

Vegetable oils, comprising nutritional oils, are oils that have a lipid profile with a relevant fraction of polyunsaturated fatty acids (linolenic fatty acid and linoleic fatty acid) and monounsaturated fatty acids (oleic fatty acids), as well as natural antioxidants (tocopherols, carotenoids, phenolic compounds and phytosterols), as minor components that, by protecting the oil from oxidative deterioration and retarding rancification, help to increase its stability. A high oleic peanut oil content results in a higher oxidative stability and more health benefits than conventional peanut oil because it has a higher content of tocopherol and linoleic (polyunsaturated) fatty acids and palmitic (saturated) fatty acids are naturally replaced by oleic fatty acid (monounsaturated), without affecting the allergenicity of the peanut oil.<sup>2</sup>

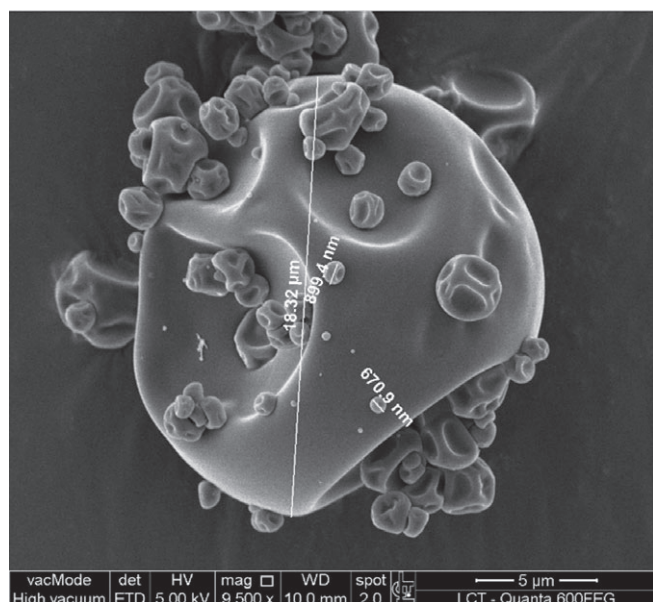
Fortifying foods with bioactive isolates not only leads to health-promoting properties but also results in a considerable decrease in desirable physical properties. Microencapsulation technologies are used to preserve the health-promoting properties of bioactive materials and promote targeted delivery.<sup>3</sup> By modifying the physical characteristics of the original material, microencapsulation can provide adequate chemical and mechanical strength; facilitating its manipulation, storage, transport, delivery and application; converting a liquid material to powder; as well as promoting its stability, compatibility with food matrices and a more uniform distribution in the application system.<sup>3</sup>

In addition to a high stability and low moisture content of the final product, microencapsulation by atomization in spray drying is considered to be a low cost, flexible technique as a result of the great availability of equipment at laboratory, pilot and industrial scales.<sup>3</sup> Polysaccharides, such as maltodextrin and arabic gum, are normally used to microencapsulate essential oils, although interactions among the core, wall material and food matrix have not yet been comprehensively investigated with respect to the use of food products as a vehicle for fortification with encapsulated bioactives.<sup>3</sup>

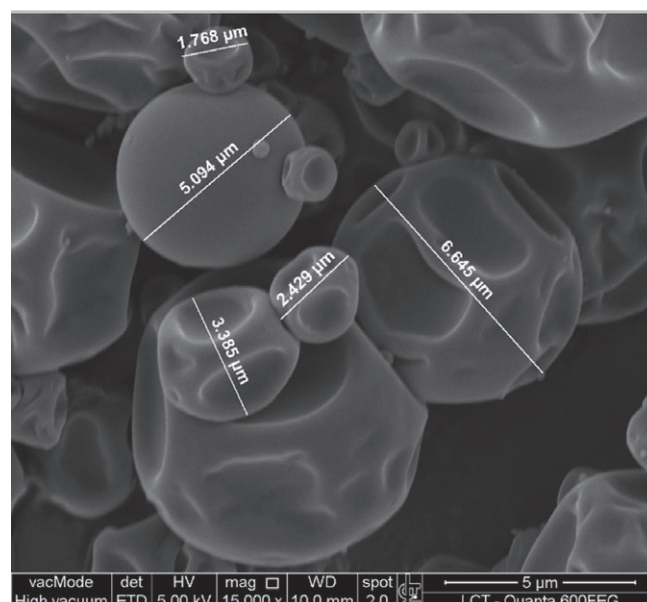
The study of the physical properties of foods is fundamental for evaluating the behavior of the product during production,

\* Correspondence to: SC S Lannes, Pharmaceutical-Biochemical Technology Department, Pharmaceutical Sciences School, University of São Paulo (USP), São Paulo, Brazil. E-mail: scslan@usp.br

Pharmaceutical-Biochemical Technology Department, Pharmaceutical Sciences School, University of São Paulo (USP), São Paulo, Brazil



**Figure 1.** Image obtained by SEM with acceleration of 5.00 kV with a 9500 $\times$  increase in content of microcapsules with high oleic peanut oil produced in a laboratory spray dryer.



**Figure 2.** Image obtained by SEM with acceleration of 5.00 kV with a 15 000 $\times$  increase in content of microcapsules with high oleic peanut oil produced in a laboratory spray dryer.

storage and packaging until the moment of consumption. Control of rheological, calorimetric and texture parameters can ensure the viability of the industrial production of stable and quality food. The rheological properties [Casson's yield stress (Pa), Casson's plastic viscosity (Pa s) and thixotropy (Pa s<sup>-1</sup>)] can be influenced by both moisture and maximum particle size, and are important in the process design with respect to the pump, quality control, storage and food texture prediction. Although other factors can contribute to the brittleness of the final product, these rheological properties can predict about 75% of the variability in the final texture of the temperate chocolates, affecting the sensorial perceptions and, finally, the acceptance of the product by the consumer.<sup>4</sup> The calorimetric properties are important with regard to pH, water activity and color characteristics in the evaluation of product quality because the melting point is a property of the material that guarantees its stability during transportation, storage and final consumption.

Accordingly, the present study aimed to evaluate the physical properties of dark chocolate with a high oleic peanut oil microcapsule content, with a view to industrial production.

## MATERIALS AND METHODS

Peanut oil was obtained from the cold hydraulic pressing of high oleic peanut seeds IAC-505 (donated by the Agronomic Institute of Campinas – IAC, Campinas, Brazil), using equipment with capacity of 15 tons of pressure as maintained on an average sample of 500 g for 300 s on batch samples (FMB SIWA, Faulbach, Miltenburg Bavaria, Germany).

The microencapsulation of the high oleic peanut oil (consisting of 820 g kg<sup>-1</sup> oleic acid, 25 g kg<sup>-1</sup> linoleic acid and 14 g kg<sup>-1</sup> saturated fatty acids) with wall material consisting of maltodextrin (MOR-REX® 1920; Ingredion, Mogi Guaçu, Brazil) and purified and instantaneous arabic gum (Instantgum BA; Nexira, Rouen, France), in accordance with a method adapted from Chatterjee & Bhattacharjee,<sup>5</sup> was conducted in a Mini Spray Dryer B-290 (Büchi, Flawil, Switzerland) and yielded 65%, with the atomizer operating

conditions: 20% pump; spray pressure of -4500 Pa; 100% vacuum cleaner; nitrogen pressure of 400 kPa; flow rate of 0.11 cm<sup>3</sup> min<sup>-1</sup>; and inlet and outlet temperatures of 150 and 86 °C, respectively. The aspirator pressure was maintained between 4.2 and 7.5 kPa during the entire operation. The emulsion was prepared from the blend of 30 g of maltodextrin and 15 g of arabic gum dissolved in 250 cm<sup>3</sup> of distilled water, where 6.25 g of high oleic peanut oil was emulsified using an Ultra-Turrax MA 102 homogenizer (Marconi, Piracicaba, Brazil) at 1567.642  $\times$  g for 60 s. Theoretically, the high oleic peanut oil content in the microcapsule obtained was 117.9 g kg<sup>-1</sup>.

The microcapsules were evaluated using scanning electron microscopy (SEM) with a Quanta 600 FEG scanning electron microscope (FEI, Hillsboro, OR, USA), equipped with energy dispersive X-ray spectrometer Quantax 400 (Technology SDD – Silicon Drift Detector; Ketek GmbH, Munich, Germany) and Sprit software (Bruker Instruments, Inc., Billerica, MA, USA). The samples were distributed in double carbon tape, metallized with platinum and fixed in support for SEM. The collection of secondary electron images was performed by the Technological Characterization Laboratory of the Polytechnic School of the University of São Paulo (São Paulo, Brazil).

The chocolates were formulated in universal equipment WA-FA20 (Mazzetti, Milan, Italy), where the ingredients were sequentially added to the equipment [490 g kg<sup>-1</sup> alkalized cacao liquor (Cargill, São Paulo, Brazil), 420 g kg<sup>-1</sup> refined sugar (União, Sertãozinho, São Paulo, Brazil), 83 g kg<sup>-1</sup> deodorized cocoa butter (Cargill), 5 g kg<sup>-1</sup> soy lecithin (Tovani Benzaquen Ingredients, São Paulo, Brazil), 1 g kg<sup>-1</sup> polyglycerol polyricinoleate-PGPR (Prozyn, São Paulo, Brazil) and 1 g kg<sup>-1</sup> vanillin powder (Daxia Ingredients and Additives, São Paulo, Brazil)], with a working temperature of 45 °C and a total process time of 2 h. The 5% microcapsule blend to the dark chocolate was performed in a planetary mixer (BP5; Erli Máquinas Para Laboratórios Farmacêuticos Ltda, São Paulo, Brazil) at 45 °C for 5 and 11 min. The tempering of the chocolates was performed by hand on a marble table. The determination of the crystallization degree of the chocolate was obtained from the

**Table 1.** Physicochemical characteristics of dark chocolates with and without a high oleic peanut oil microcapsule content

Physicochemical characteristics	Control formulation (CF)	Formulation mixed for 5 min (F5)	Formulation mixed for 11 min (F11)
Water activity	0.357 ± 0.046 a	0.336 ± 0.019 a	0.382 ± 0.031 a
pH	6.76 ± 0.18 ab	6.88 ± 0.08 a	6.59 ± 0.25 b
Maximum particle size (mm)	0.023 ± 0.002 a	0.019 ± 0.003 a	0.020 ± 0.003 a
$L^*$	20.71 ± 0.86 b	22.77 ± 1.23 a	23.32 ± 0.99 a
$a^*$	3.88 ± 0.47 a	3.17 ± 0.75 ab	2.56 ± 0.35 b
$b^*$	9.48 ± 0.47 a	9.08 ± 0.92 ab	8.16 ± 0.51 b
WI	20.05 ± 0.90 b	22.17 ± 1.33 a	22.84 ± 0.94 a

Data are the mean ± SD from triplicate analysis. Values with the same lowercase letter within a line are not significantly different ( $P > 0.05$ ) as determined using Tukey's multiple comparisons.

tempering curve (29–18 °C) in Multitherm TC equipment (Bühler AG, Uzwil, Switzerland) and it was considered suitable for continuing the study of solid product that obtained a temper index of between 3 and 5, as recommended by Bühler AG<sup>6</sup> and Voltz & Beckett.<sup>7</sup> Immediately after the pre-crystallization, chocolate was moulded and cooled at 6 °C by 20 min, then demoulded and dehumidified at 20 °C and 60% relative humidity for 24 h, after which it was packed in aluminum paper and stored at 20 °C and 60% relative humidity. All samples were performed in duplicate.

Water activity analyses were performed on Novasina-AW equipment (Novasina, Lachen, Switzerland). The pH measurements were obtained in a digital potentiometer (Quimis, Diadema, Brazil). The determination of the maximum particle size was carried out using a Digimatic (Mitutoyo, Kawasaki, Japan) 293 series digital micrometer. The color spectrum was determined in a HunterLab/ColorQuest XE (Hunter Associates, Reston, VA, USA) colorimeter calibrated with a white ceramic reference standard, where the readings were performed in the CIELAB system ( $L^*$ ,  $a^*$  and  $b^*$ ) and data were analyzed using Universal Software, version 4.10 (Hunter Associates). The values of  $L^*$ ,  $a^*$  and  $b^*$  were transformed into whiteness index (WI), according to:  $(WI) = 100 - [(100 - L^*)^2 + (a^*)^2 + (b^*)^2]^{0.5}$ , as defined by Lohman and Hartel.<sup>8</sup>

Nutritional composition was measured based on the official method of the AOAC,<sup>9</sup> after acid hydrolysis<sup>10</sup> of the grated samples. The total lipid content was determined by Soxhlet extraction. Fatty acids were methylated<sup>11</sup> and the analyses were performed using a model 430 GC gas chromatograph (Varian Inc., Palo Alto, CA, USA) equipped with automatic injector, flame ionization detector and Galaxie Chromatography software (Varian Inc.). A capillary column of fused silica SP-2560 (Supelco, Bellefonte, PA, USA) was used (length 100 m, internal diameter 0.25 mm) containing 0.2 µm of polyethylene glycol within the column. The conditions were: split injection, 50:1 ratio; column temperature: 140 °C for 300 s, programmed to 240 °C at a ratio of 4 °C min<sup>-1</sup>; drag gas: helium, at 37 psi isobaric pressure; linear velocity of 20 cm s<sup>-1</sup>; gas make-up: helium at 29 cm<sup>3</sup> min<sup>-1</sup>; injector temperature: 250 °C; and detector temperature: 280 °C. The qualitative composition was determined by comparing the peak retention times with the respective fatty acid standards. The quantitative composition was performed by area normalization, expressed as percentage by mass and converted to the form g kg<sup>-1</sup>, in accordance with the official AOCS Ce 1-62 method.<sup>12</sup>

The rheological behavior of molten dark chocolate was characterized by a rotational test carried out on a MARS rheometer

**Table 2.** Fatty acids profile of lipid fraction of dark chocolates with and without a high oleic peanut oil microcapsule content

Measured fatty acids (g kg <sup>-1</sup> )	Control formulation (CF)	Formulation with 5% microcapsules
C16:0	274.21 ± 0.15 a	259.67 ± 0.25 b
C16:1	1.99 ± 0.03 a	1.89 ± 0.01 b
C18:0	370.49 ± 0.36 a	357.84 ± 0.25 b
C18:1n9c	323.75 ± 0.27 a	338.61 ± 0.14 b
C18:2n6c	17.73 ± 0.18 a	29.33 ± 0.15 b
C20:0	11.35 ± 0.03 a	10.99 ± 0.07 b
C18:3n3	0.49 ± 0.00 a	1.67 ± 0.14 b

Data are the mean ± SD from triplicate analysis. Values with the same lowercase letter within a line are not significantly different ( $P > 0.05$ ) as determined using Tukey's multiple comparisons.

(Thermo Scientific, Waltham, MA, USA) using a cone-shaped sensor (C35/1 Ti polished) with a gap of 0.024 mm at 40 °C after 10 min of rest and preconditioning of the sample at 55 °C for 75 min, based on the official method of the IOCCC.<sup>13</sup> The rotational assay was conducted at a controlled rate in three steps: (i): 0.00–65.00 s<sup>-1</sup>,  $t = 180$  s; (ii) 65.00 s<sup>-1</sup>,  $t = 60$  s; and (iii) 65.00–0.00 s<sup>-1</sup>,  $t = 180$  s. The amount of sample was sufficient to fill the space between the plates. Data regarding plastic viscosity and yield stress were adjusted to the Casson equation.<sup>8</sup>

Brittleness of tempered chocolate were conducted on a TA-XT2 texture analyzer (Stable Micro Systems, Godalming, UK) using a three-point bend test, with a HDP/3PB probe and samples (9.4 cm × 1.2 cm × 0.6 cm tempered chocolate bars) conditioned at 25 °C. The brittleness is related to the maximum load (N) necessary to break the bar of tempered chocolate. The parameters used were: pre-test velocities, test and post-test: 2.0, 10.0 and 10.0 mm s<sup>-1</sup>; load cell 25 kg; trigger force: 0.05 N; strength in compression – return to start. Data were collected using the Texture Expert Exceed Program, version 2.64 (Stable Micro Systems).

The calorimetric analyzes were conducted on a differential scanning calorimeter DSCi Series System (Instrument Specialists Incorporated, Twin Lakes, WI, USA). After calibrating the equipment at a scan rate of 20 °C min<sup>-1</sup> using a sealed aluminum pan with a cap as reference, samples of approximately 5 mg were loaded into 40-µL pans, sealed with a lid, and heated at a rate of 20 °C min<sup>-1</sup> from 15 to 250 °C in nitrogen atmosphere (N<sub>2</sub>), in accordance with the method of Afoakwa *et al.*<sup>14</sup> with

**Table 3.** Physical properties of dark chocolates with and without a high oleic peanut oil microcapsule content

Physical properties	Control formulation (CF)	Formulation mixed for 5 min (F5)	Formulation mixed for 11 min (F11)
Casson's yield stress (Pa)	9.55 ± 0.24 a	12.96 ± 1.93 a	10.78 ± 2.08 a
Casson's plastic viscosity (Pa s)	1.78 ± 0.24 a	3.64 ± 0.51 b	4.29 ± 1.14 b
Thixotropy (Pa s <sup>-1</sup> )	1230 ± 203 a	3467 ± 1171 b	3898 ± 1103 b
Texture: brittleness (N)	21.5 ± 5.4 a	14.8 ± 4.2 b	18.8 ± 0.9 ab

Data are the mean ± SD from triplicate analysis. Values with the same lowercase letter within a line are not significantly different ( $P > 0.05$ ) as determined using Tukey's multiple comparisons.

**Table 4.** Calorimetric properties of dark chocolates with and without high oleic peanut oil microcapsules

Calorimetric properties (°C)	Control formulation (CF)	Formulation mixed for 5 min (F5)	Formulation mixed for 11 min (F11)
Peak melting	31.6 ± 0.6 a	32.1 ± 0.7 a	31.5 ± 0.2 a
Peak caramelization	183.6 ± 3.2 a	181.0 ± 4.2 a	184.8 ± 0.4 a
Peak carbonization	237.1 ± 0.3 a	236.8 ± 0.5 a	236.8 ± 0.5 a

Data are the mean ± SD from triplicate analysis. Values with the same lowercase letter within a line are not significantly different ( $P > 0.05$ ) as determined using Tukey's multiple comparisons.

modifications. The temperature flow heat curve was generated using Infinity Pro-Thermal Analysis software (Instrument Specialists Incorporated) based on data acquired by the Acquire Program (Instrument Specialists Incorporated). The calorimetric properties (melting onset, peak melting and melting end, as well as the peaks of the caramelization and carbonization temperatures) were identified on the graphic plotted by using Origin, version 8 (OriginLab Corp., Northampton, MA, USA).

Analysis of variance, followed by Tukey's test, was conducted using Minitab, version 17 (Minitab Inc., State College, PA, USA) to analyze data at the 95% confidence level.

## RESULTS

The microcapsule morphology observed using SEM is shown in Figs 1 and 2. The results of particle size (from 1 to 20 µm) are within the particle size range produced by atomization, which is 5–150 µm according to several studies cited in Santos *et al.*<sup>15</sup> The microcapsules analyzed also presented continuous walls, rounded, without cracks, cracks or ruptures, and with the presence of concavities or flattenings with deep depressions in the surfaces, a typical morphology of microcapsules produced by atomization with arabic gum as an encapsulating agent, indicating the formation of a continuous film that guarantees less permeability to gases and greater protection and retention of the filling, all suggesting a highly successful process.

The physicochemical characteristics of dark chocolates with and without high oleic peanut oil microcapsules are shown in Table 1.

The lipid content of the chocolate without high oleic peanut oil microcapsules ( $35.84 \pm 0.39 \text{ g kg}^{-1}$ ) was higher than the lipid content of the chocolate with a high oleic peanut oil microcapsule content ( $33.95 \pm 1.48 \text{ g kg}^{-1}$ ). The nutritional composition of dark chocolates with and without high oleic peanut oil microcapsules is shown in Table 2.

The physical properties of dark chocolates with and without high oleic peanut oil microcapsules are shown in Table 3.

The three dark chocolates demonstrated melting, caramelization and carbonization peaks, under instrumental heating, as shown in Fig. 1 and Table 4.

The events showed in Fig. 3 correspond to calorimetric properties: melting onset (21 °C), peak melting (32 °C) and melting end (41 °C); endothermic peak (183 °C) as observed shortly after the change of the baseline; and exothermic peak (237 °C). Mass loss was observed at the end of the analysis.

## DISCUSSION

The measurement of the WI is widely used to allow identification of fat bloom appearance because the higher value of this index, the whiter the color of the surface, and therefore the greater the development of fat bloom.<sup>16,17</sup> However, because no fat migration defect was observed in the samples analyzed, it is possible that, despite the significant difference found between the samples, this defect appearance was not identified. According to Son *et al.*,<sup>18</sup> a WI is adequate around  $20 \pm 1$ , immediately after the production of dark chocolate, as obtained in the present study, and only when the visual fat bloom reaches a relevant level (WI up to 35) can the perception of colour of the product affected and the fat bloom be confirmed.<sup>19,20</sup>

It was possible to observe an increase in the WI when adding the microcapsules of high oleic peanut oil content, regardless of the mixing time used in the processing, probably as a result of microcapsules of a white color, or the influence of a microcapsule on the recrystallization of lipids, as reported by Silva *et al.*<sup>17</sup> The same increase in WI was observed by Erdem *et al.*<sup>21</sup> who developed a dark chocolate enriched with maltodextrin and lemon fiber and obtained no negative effects on the color properties of samples.

Results demonstrating pH values close to the neutrality range (low acidity), as obtained with the alkalized cacao liquor used in the formulation, guarantee a greater probability of acceptance by the consumer market.<sup>22</sup>

The addition of microcapsules did not influence the water activity of the samples and, with water activity results below 0.60 (low active water foods), which may contribute to the microbiological safety of the chocolate in that very few microorganisms



can multiply under such conditions,<sup>17</sup> few deterioration reactions occur,<sup>23</sup> demonstrating the chemical and physical stability of the analyzed chocolates when stored in appropriate conditions of humidity and temperature.

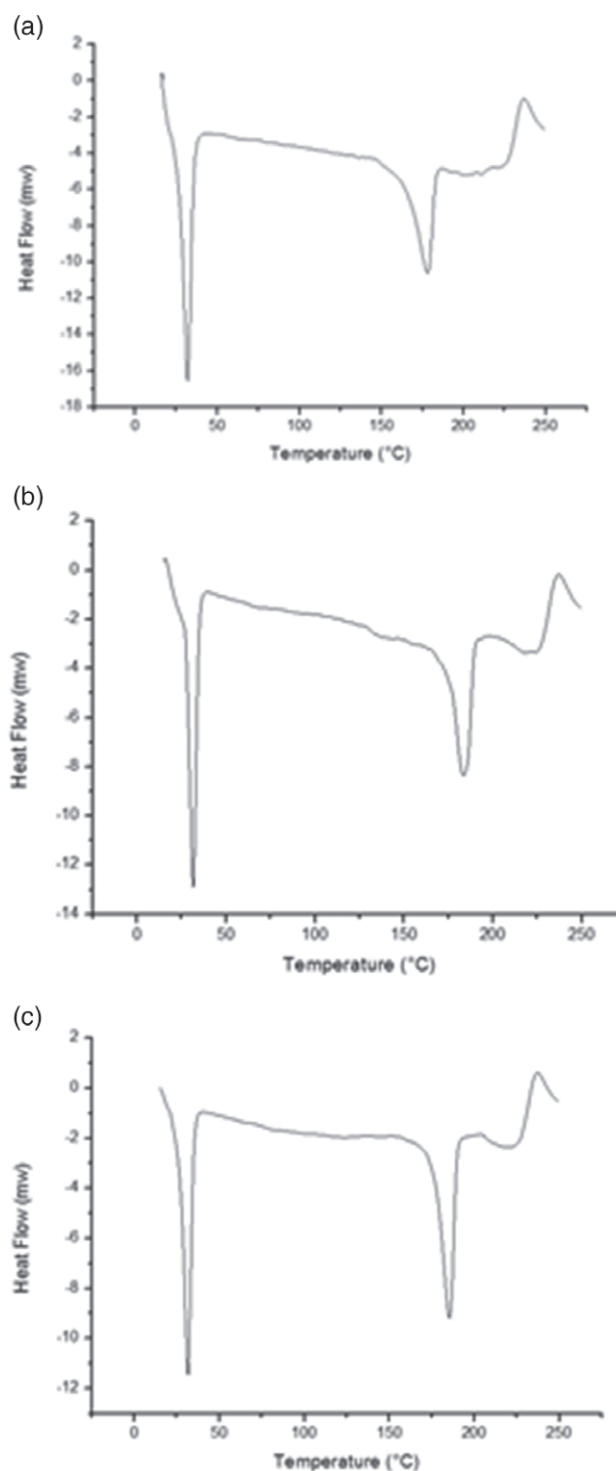
The addition of microcapsules reduced the lipid content of dark chocolate as a result of the presence of amount of wall material in the sample. The addition of microcapsules also influenced the nutritional composition of the samples, increasing the content of unsaturated fatty acids in the lipid fraction of chocolate.

Dark chocolate comprises a suspension of very fine particles (up to 25  $\mu\text{m}$ ) of cocoa solids and sugar, coated by a fatty phase consisting of cocoa butter.<sup>22</sup> The microstructure (maximum particle size) that, in the present study, was not significantly influenced by microcapsule addition is an important fundamental variable for the texture adjustment of dark chocolate and is related to the stage of refining, the composition of fatty base (cocoa butter content and lecithin content) and the physical forces of interaction between the particles, which all influence mechanical properties, transport phenomena, and the physical and rheological properties of foods. Accordingly, this determines the consistency and viscosity of chocolates, as well as quality in terms of mechanical and sensorial attributes.<sup>22,14</sup>

The statistically significant difference ( $P < 0.05$ ) observed for the parameter of brittleness does not represent technological significance because it is within the range of expected results for good dark chocolate, until possible observation of a good snap at room temperature.<sup>1</sup>

Although the mixing time did not influence the rheological parameters of the chocolates, the interaction between cocoa dispersion and the continuous phase influences the microstructural properties of the product, affecting its rheological characteristics in terms of yield stress, apparent viscosity and thixotropy.<sup>24</sup> The Casson's plastic viscosity and thixotropy showed significant differences ( $P < 0.05$ ) between the control formulation and the other formulations with microcapsules of high oleic peanut oil (Table 3). The determined values of Casson viscosity for all the chocolates are in agreement with those reported in the literature.<sup>25–27</sup> However, as studied by Lalicic-Petronijevic *et al.*,<sup>28</sup> in the present study, microcapsules were included in the chocolate mass without concurrently raising the fat content. This means that dark chocolate containing microcapsules showed higher particle–particle interactions as a result of the lower amount of free fat, as well as the presence of polysaccharides in an amorphous state, which, because of its irregular structure, trends to trap fat, promoting more aggregate matrices with less space between particles, and thus impede the flow and increasing the product viscosity.<sup>24,28</sup> The same behavior has been reported by Aidoo *et al.*<sup>29</sup> when they increased inulin concentrations with a simultaneous reduction in polydextrose in dark chocolate, resulting in consistent increases in the Casson plastic viscosity, leading to decreases in Casson yield stress. In the present study, the yield stress showed no significant differences ( $P > 0.05$ ) between the three dark chocolates because the relative standard derivation varied around 19%, which made it difficult to detect significant statistical differences, despite a study stating that a high-fat content coats the solid particles and makes it easy to start a flow (yield stress), as well as continue the flow (plastic viscosity) of chocolate mass.<sup>25</sup>

There are ranges of acceptability for the rheological parameters of chocolate, which depend on the purpose of use for which the product is destined: Casson's plastic viscosity ( $\eta_{CA}$ ) from 1 to 2 Pa s; Casson's yield stress ( $\tau_{CA}$ ) from 5 to 200 Pa.<sup>30</sup> Casson's plastic viscosity above the desired value for formulations with



**Figure 3.** Calorimetric profile of chocolates: (a) control formulation (CF); (b) formulation mixed for 5 min (F5); (c) formulation mixed for 11 min (F11).

microcapsules of high oleic peanut oil content may indicate an increase in the energy required for the flow of chocolate mass; however, the highest values of thixotropy observed in formulations with microcapsules of high oleic peanut oil demonstrated a more aggregated structure of this chocolates compared to the control formulation, which may be unable to recover most of its initial structure,<sup>24</sup> facilitating the displacement of the product in the production line and moulding step.

As shown in Table 4, the calorimetric properties were equivalent ( $P < 0.05$ ) in three dark chocolates, indicating that the thermal stability of the chocolate with microcapsules is compatible with the thermal stability of the control chocolate, independent of the mixing time. The events shown in Fig. 3 correspond to melting properties: melting onset (21 °C), the temperature at which a specific crystal form starts to melt; peak melting (32 °C), the temperature at which melting rate is greatest; and melting end (41 °C), the temperature at which all crystals were completely liquefied. The endothermic transitions were in the range expected for a well tempered dark chocolate melting profile, where the most desirable polymorphic forms were obtained: form V (32–34 °C) because it not only melts just below body temperature, but also gives a good snap and glossy appearance, as well as form VI with a melting range of 34–36 °C,<sup>31,32</sup> confirming the heat resistance of samples and also that there was no migration of oil from the microcapsules during the manufacturing process. Other events shown in Fig. 3 correspond to the endothermic peak (183 °C) observed shortly after the change of the baseline, which is associated with the glass transition of the material, corresponding to the crystallization and/or caramelization of sugars present in the sample, and an exothermic peak (237 °C) that may correspond to the carbonization of the sample because a loss of mass was observed at the end of the analysis.<sup>33</sup>

## CONCLUSIONS

The chocolates developed in the present study showed thermal stability and physical characteristics adequate for industrial production. Therefore, the use of microcapsules that resist the chocolate manufacturing process (tempering, moulding, crystallization and demoulding), represents a viable alternative to protecting the fatty acids and antioxidants present with a high oleic peanut oil content against the factors causing oxidation and consequently the development of an unpleasant taste and odor because of oxidative rancidity. The mixing time did not influence the physical characteristics of the product.

## ACKNOWLEDGEMENTS

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