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Microencapsulation by spray drying of laurel infusions (Litsea glaucescens) with maltodextrin

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ABSTRACT

The effect of maltodextrin as an encapsulating agent on spray dried (SD) laurel infusions was studied (inlet temperatures: 140, 160 and 180 °C, and feed rate: 8 and 10 mL/min at fixed flow atomization). In the SD samples, the phenolic content (TPC), antioxidant capacity (DPPH*), morphology (SEM), chemical structure (FTIR), rheology properties and release profiles were studied. The results show that laurel infusion had 42.10 (±0.23) mg gallic acid equivalent/g of laurel and EC_{50} of 0.40 (±0.10) mg laurel/mL of DPPH*, the SD microparticles showed defined morphologies. Encapsulation of laurel infusion was achieved with an efficiency of ~70%. The reconstituted SD powders solutions showed a shear-thinning rheological behavior ($n < 1$). The results evidenced that the best conditions for laurel encapsulation by SD were 160 °C inlet temperature and 8 mL/min feed rate.

1. Introduction

Laurel (*Litsea* spp.) is one of the most used spices in the world. These spices are commonly used as food additives, provide flavors, scents, colors and even help in food preservation. It is commonly known as "bay laurel" and it has been reported to impart antimicrobial and antioxidant properties to food. Laurel has been reported to be used as an aid in gastrointestinal disorders, inflammation problems and atherosclerosis (Shan et al., 2007; Cherrat et al., 2014). The laurel spice has been used since ancient times in traditional Chinese medicine (Xie and Yu, 1996). All these benefits are related to the phytochemicals that compose laurel which include polyphenols such as phenolic acids and flavonoids (Kong et al., 2015; Tsai and Lee, 2011). The phenolic compounds (polyphenols) in Laurel spice provide natural antioxidant capacities to trap free radicals and inhibit oxidative processes in the body (Shan et al., 2005). However, polyphenols are extremely labile at ambient conditions (Ultraviolet, radiation, temperature, oxygen, stomach digestion, etc.) which affects their stability and reduce the antioxidant benefits, so that their protection with encapsulation vectors becomes crucial for the preparation of functional food (D'Archivio et al., 2010; Ersus and Yurdagel, 2007; Jafari et al., 2008). The most common microencapsulation process is spray drying (SD), which has proven to be an effective technology in protecting polyphenolic compounds. SD consists in converting water suspensions into powdered microparticles, which are composed of a wall material (shell) and a core (encapsulated material) (Reineccius, 1988). Carbohydrates, such as maltodextrins are one of the main wall materials used as encapsulating materials to protect polyphenolic compounds (Desai and Park, 2005; Ersus and Yurdagel, 2007; Jafari et al., 2008). Maltodextrins are hydrolyzed starch, they have a low cost and possess high water solubility (>75%) and low viscosity in aqueous solutions. Maltodextrins form a coating film minimizing oxygen contact of the encapsulated material (Pourashouri et al., 2014). Microparticles obtained from SD are able to last for longer periods of time and they have been reported to release the encapsulated materials under simulated conditions of the digestive tract (Medina-Torres et al., 2013) SD is the ideal process to achieve mechanical stability of encapsulated polyphenols particles and preserve their bioactivity (Mahdavi et al., 2014; Khazaei et al., 2014). There have been

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Nomenclature

MD	Maltodextrin, MD10
La	Laurel
SD	Spray drying
LaInf	Laurel infusions
SDLaInf14008	Spray drying laurel infusions at 140 °C and fed at 8 mL/min
SDLaInf16008	Spray drying laurel infusions at 160 °C and fed at 8 mL/min
SDLaInf18008	Spray drying laurel infusions at 180 °C and fed at 8 mL/min
SDLaInf14010	Spray drying laurel infusions at 140 °C and fed at 10 mL/min
SDLaInf16010	Spray drying laurel infusions at 160 °C and fed at 10 mL/min
SDLaInf18010	Spray drying laurel infusions at 180 °C and fed at 10 mL/min
GAE	Gallic acid equivalents
CE	Catechin equivalents
TPC	Total phenolic content
TFC	Total flavonoids content
DPPH*	2,2-diphenyl-1-picrylhydrazyl radical
SEM	Scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
PSD	Particle size distribution
EC_{50}	Efficient concentration

multiple studies of encapsulation of the non-polar fraction of laurel, which is used in perfume applications or in synthesis of natural oils (Martins et al., 2014; Chelaru et al., 2014). However, few studies deal on the aqueous fraction. The aim of this study is to evaluate the use of maltodextrin as a wall material in the SD encapsulation of laurel infusions, searching for the best drying conditions in relation to the preservation of the phenolic content, antioxidant capacity and to characterize the SD powders in micro (FTIR, PSD, SEM) and macro (rheology, controlled release) experimental tests.

2. Materials and methods

2.1. Materials

Laurel was obtained in Durango, Mexico. It was ground and sieved (40 mesh). Maltodextrin DE10 (MD) was also obtained from a domestic supplier (NIFRA Comerciales, S.A. Mexico City, Mexico) and was used as encapsulating material.

2.2. Laurel infusión, (LaInf)

Infusions were prepared with 50 g of laurel, in 500 mL of water at 80 °C with constant stirring during 10 min. The infusion was then filtered, centrifuged and lyophilized (Edwards Freeze-dryer Modulyo, Crowley, Sussex, UK), for analysis of phenolic content and antioxidant activity (Reference sample). The percentage yield extraction (% EY) was determined (Eq. (1)):

$$\%EY = \{[\text{Total solids after lyophylization LaInf (g)}]/[\text{Total weight of raw Laurel used to prepare infusion (g)}]\} \times 100 \quad (1)$$

Table 1
Experimental design of spray drying conditions of laurel infusions.

Treatment	Drying temperature, (°C)	feed rate, (mL/min)
SDLaInf14008	140	8
SDLaInf16008	160	8
SDLaInf18008	180	8
SDLaInf14010	140	10
SDLaInf16010	160	10
SDLaInf18010	180	10

2.3. Preparation of dispersions LaInf with MD

Dispersions were prepared using the laurel infusion and adding 10% (w/v) of maltodextrin. The mixture was homogenized with magnetic stirrer at 300 rpm for 1 h at 25 °C. Dispersions were then spray dried using a Mini Spray Dryer B-290 Buchi (Flawil, Switzerland), with rotor stator in parallel flow. The samples were SD using a fractional factorial design 2^3, (2 feed rates: 8 and 10 mL/min, and 3 inlet temperatures: 140, 160 and 180 °C) at constant pressure of 6.5 bar. These conditions were selected according to related studies with the spray drying using maltodextrin as encapsulanting vector (Janiszewska and Witrowa-Rajchert, 2009; Tonon et al., 2011; Gallegos-Infante et al., 2013). The performance of the drying process was determined according to Eq. (2):

$$\%SDY = [(\text{Weight of microcapsules obtained after spray drying})/(\text{Total weight of initial wall material and LaInf})] \times 100 \quad (2)$$

The nomenclature of the samples and experimental design are shown in Table 1.

2.4. Moisture

Water content was quantified by means of the AOAC 925.10 gravimetric method: a sample of 2 g was dried in a hot air oven at 103 °C for 1 h, and moisture loss was determined by weighting and comparing the sample weight prior and after drying (AOAC International, 2000).

2.5. Volumetric density

Volumetric density was conducted according to the method described by Papadakis et al. (2006): 100 g of sample powder was transferred to a 250 mL graduated cylinder and the cylinder was tapped by hand on a bench 100 times from a height of 10 cm. Then the bulk density was calculated by dividing the mass of the powder by the final volume occupied by the powder in the cylinder.

2.6. Total phenolic content

The total phenolic content (TFC) of LaInf and powders (SDLaInf) was made by using the Folin-Ciocalteu method modified by Heimler et al. (2005). The calculation was determined using a calibration curve of gallic acid content ranging from 10 to 120 μg/mL (R^2 = 0.992) to interpolate results of the TPC.

2.6.1. Total flavonoid content

The determination of total flavonoids was made as proposed by Heimler et al. (2005), by using a calibration curve of catechin content ranging from 10 to 120 μg/mL (R^2 = 0.99) to interpolate the results of the TFC.

Table 2
Results drying performance, moisture, bulk density, total phenol, total flavonoids and DPPH* EC_{50} LaInf and SDLaInf.

Sample	Yield	Moisture [1]	Density[2]	TPC[3]	TFC[4]	DPPH* CE_{50}[5]
LaInf	12.10 (±0.33)	–	–	42.10 (±0.23)^{f}	17.43 (±0.84)^{d}	0.4 (±0.1)^{c}
SDLaInf 14008	47.56 (±0.22)^{b}	4.24 (±0.16)^{b}	0.180 (+0.002)^{a,b}	14.57 (±0.24)^{c}	13.20 (±0.22)^{b}	1.9 (±0.1)^{a,b}
SDLaInf 16008	50.42 (±0.51)^{c}	2.75 (±0.28)^{a}	0.180 (+0.002)^{a,b}	**20.22 (±0.30)**^{e}	**19.50 (±0.25)**^{e}	**1.3 (±0.1)**^{d}
SDLaInf 18008	62.32 (±0.30)^{f}	1.82 (±0.11)^{a}	0.183 (+0.002)^{b}	12.84 (±0.06)^{b}	10.28 (±0.20)^{a}	1.7 (±0.1)^{a}
SDLaInf 14010	53.06 (±0.15)^{d}	4.32 (±0.07)^{b}	0.180 (+0.002)^{a,b}	10.30 (±0.29)^{a}	9.74 (±0.15)^{a}	2.3 (±0.1)^{e}
SDLaInf 16010	60.43 (±0.40)^{e}	2.53 (±0.07)^{a}	0.176 (+0.002)^{a}	10.24 (±0.16)^{a}	9.70 (±0.30)^{a}	3.1 (±0.1)^{f}
SDLaInf 18010	48.59 (±0.25) ^{c}	2.58 (±0.82)^{a}	0.179 (+0.002)^{a,b}	16.71 (±0.27)^{d}	15.10 (±0.27)^{c}	2.0 (±0.1)^{b}

* Different letters indicate significant statistical differences (ANOVA, Tukey test, $p < 0.05$).
[1] percentage content.
[2] in g/cm^3.
[3] total phenol content (mg GAE/g of Laurel).
[4] flavonoid content (mg CE/g of Laurel).
[5] EC_{50} (mg/mL of DPPH*).

2.6.2. Radical-scavenging activity,(2,2-diphenyl-1-picrylhydrazyl, DPPH method)*

The antioxidant capacity of the samples was measured with the DPPH* assay (Brand-Williams et al., 1995) with slight modifications. A methanolic solution of LaInf (0.1 mL) prepared at various concentrations (100–500 μg/mL) was mixed with 3.9 mL of DPPH * dissolved in methanol (6×10^{-5} M). After 30 min, the absorbance was measured at 515 nm at 20 °C. Methanol was used as target. The antioxidant capacity calculated was expressed as EC_{50}, the concentration of antioxidant required to cause 50% inhibition (Eq. (3)):

$$\%, \text{Inhibition} = [(\text{Conc.ofDPPH}*_{t=30'})/(\text{Conc.ofDPPH}*_{t=0})] \times 100 \quad (3)$$

2.7. Analysis by infrared spectrometry, (FTIR)

Analysis of LaInf, MD and SDLaInf was performed. The analysis was performed with a FTIR Nicolet 6700 – diamond point (Thermo Fisher Scientific, USA) using the method of the disc of potassium bromide (KBr). 100 scans were performed at a resolution of 1 cm^{-1}, from 4000 cm^{-1} to 400 cm^{-1}.

2.8. Scanning electron microscopy, (SEM)

Microscopy was conducted according to the method of Quiñones-Muñoz et al. (2011). The sample was fixed with tape into a copper surface and it was vacuum coated with gold at 10 mbar for 90 s (model Desk II, Denton Vacuum, NJ, USA), and examined in a scanning electron microscope (JEOL Mod. JSM6300 Jeol, Japan) at an accelerating voltage of 20 kV, and 1000X magnification.

2.9. Particle size distribution, (PSD)

The PSD was evaluated on a Master-Sizer 2000 with laser diffraction (Malvern Instrument Ltd., UK). Powdered was dispersed using deionized water as dispersing agent (SDLaInf, Refractive index = 1.582; dispersant agent = 1.330).

2.10. Rheological behavior of spray dried laurel infusions

The rheological characterization of SDLaInf powders was made by preparing aqueous solutions at 0.03 g/mL content (reconstituted powders). Simple shear flow and oscillatory tests were made with a controlled stress rheometer (AR-G2, TA Instruments) with concentric cylinder geometry (21.96 mm outside diameter, 20.38 mm inside diameter, 59.50 mm height, and 500 μm gap from the base), at a constant temperature of 25 °C, controlled by a circulating water bath (Cole Parmer Polystat and Peltier AR- G2). Samples were reconstituted in deionized water using a magnetic bar at 300 rpm for 1 h at 25 °C. Aqueous dispersions were analyzed for both viscous behavior [$\eta(\dot{\gamma})$] in simple shear flow in a range of 0.1–200 s^{-1} as well as linear oscillatory shear flow to estimate the behavior of the viscoelastic properties (storage modules G′ and loss G″) in a frequency range of 0.1–200 rad•s^{-1}. Each rheological evaluation was made by duplicate at 25 °C of temperature. Experimental data were obtained and analyzed directly from TA Rheology Advantage Data Analysis V.5.7.0 (TA Instrument Ltd., Crawley, UK) software.

2.11. Controlled release analysis

Controlled release of SDLaInf powders was developed according to Desai and Park, (2005) in a dissolution device (Franz cell) equipped with a permeable membrane of 0.45 μm (Nylon HV, Millipore) in a water bath at 37 °C under constant stirring (300 rpm). The sample was resuspended to 0.2 g/mL in deionized water at pH ~6.0, trying to simulate the release under digestive tract conditions.

2.12. Statistical analysis

A factorial experimental design (3 drying temperatures x 2 feed rate to dryer) was used to make this work. Experimental data were analyzed by ANOVA and Tukey test ($\alpha = 0.05$). Using Statistica 7.0 (StatSoft, Tulsa, Oklahoma, USA).

3. Results and discussion

3.1. Percent-yield from extraction, drying, moisture content and density

The samples of treatments that were fed at 8 mL/min to the dryer had the highest drying performance (Table 2). This behavior is associated with the longer residence time of the sample in the SD chamber. A similar behavior but with a lower yield (~25%) was reported for the encapsulating infusions of *Quercus resinosa* (Gallegos-Infante et al., 2013) using maltodextrin as encapsulating agent. The percentage moisture content ranged from 1.83 (±0.11) to 4.24 (±0.16). The lowest content in the SDLaInfLa16008

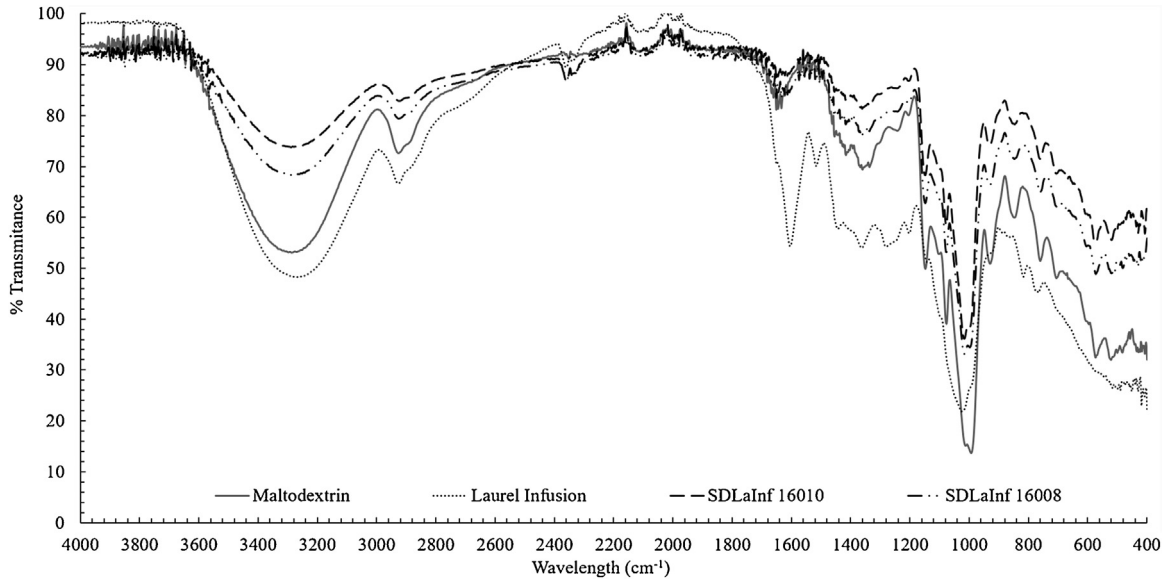

Fig. 1. Comparative FTIR spectrum between MD, LaInf, SDLaInf 16008 and SDLaInf16010.

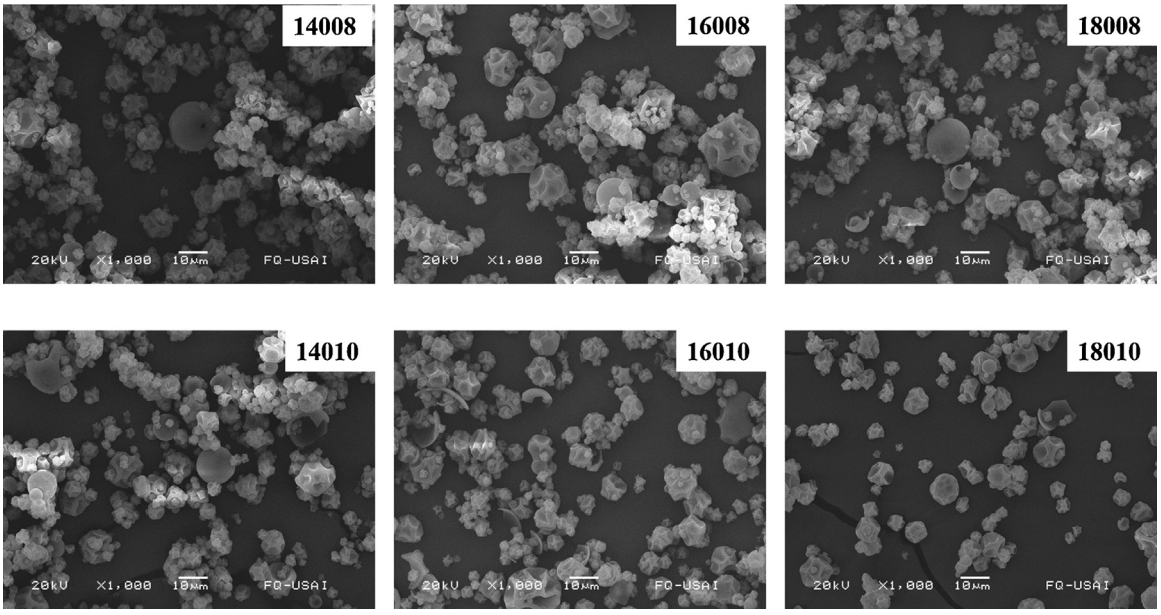

Fig. 2. Microscopy of SDLaInf: T_i: 140, 160 and 180 °C to F_a: 8 and 10 mL/min, increased to 1000×.

shows even less than equivalent studies with extracts of melon and apple mixture with pomegranates (Quek et al., 2007; Ochoa-Martínez et al., 2011). The density values were in the range of 0.30–0.33 g/cm^3, like have been reported in previous studies.

3.2. Total phenolic content, flavonoids and antioxidant activity

The total phenolic content in the lyophilized laurel infusion (reference sample) was 42.10 (±0.23) mg GAE/g of La according to Table 2. Regarding the SD powders, the higher phenolic content was SDLaInf16008 with 20.22 mg GAE/g of laurel, which is comparatively lower than the lyophilized sample (42.10 mg GAE/g of laurel). However, the lyophilized sample is only taken as a reference since the drying process is significantly different. Phenol concentration determined in the SD laurel (10–20%, see Table 2) was higher than that reported for a variety of European laurel (Shan et al., 2005, Muchuweti et al., 2007) and even higher than what is contained in 13 of 15 Brazilian herbal infusions (as chamomile, anise, mint and others) (Moraes-de-Souza et al., 2008). Flavonoid content was found to be higher (sample LaInf 16008 with 19.50 mg CE/g of laurel) than that achieved by lyophilization (17.43 mg CE/g of laurel), this is an important result since SD process is economically more viable that the freeze-drying process (lyophilization).

The presence of phenolic compounds within the laurel results in antioxidant properties in a natural material, which provides the capacity of inhibiting free radical activity. The result of the evaluation of antioxidant capacity of laurel is shown in Table 2. Samples with higher phenolic and flavonoid content were found to have the best antioxidant properties. In the analysis of antioxidant activity, the mean inhibitory concentration (EC_{50}) was measured and reported. EC_{50} is the concentration necessary to inhibit 50% solution of synthetic radical DPPH*, a low EC_{50} indicates potent antioxidant activity, namely the amount necessary to inhibit one oxidizing compound is minimal. Other factor may be affecting the results such as

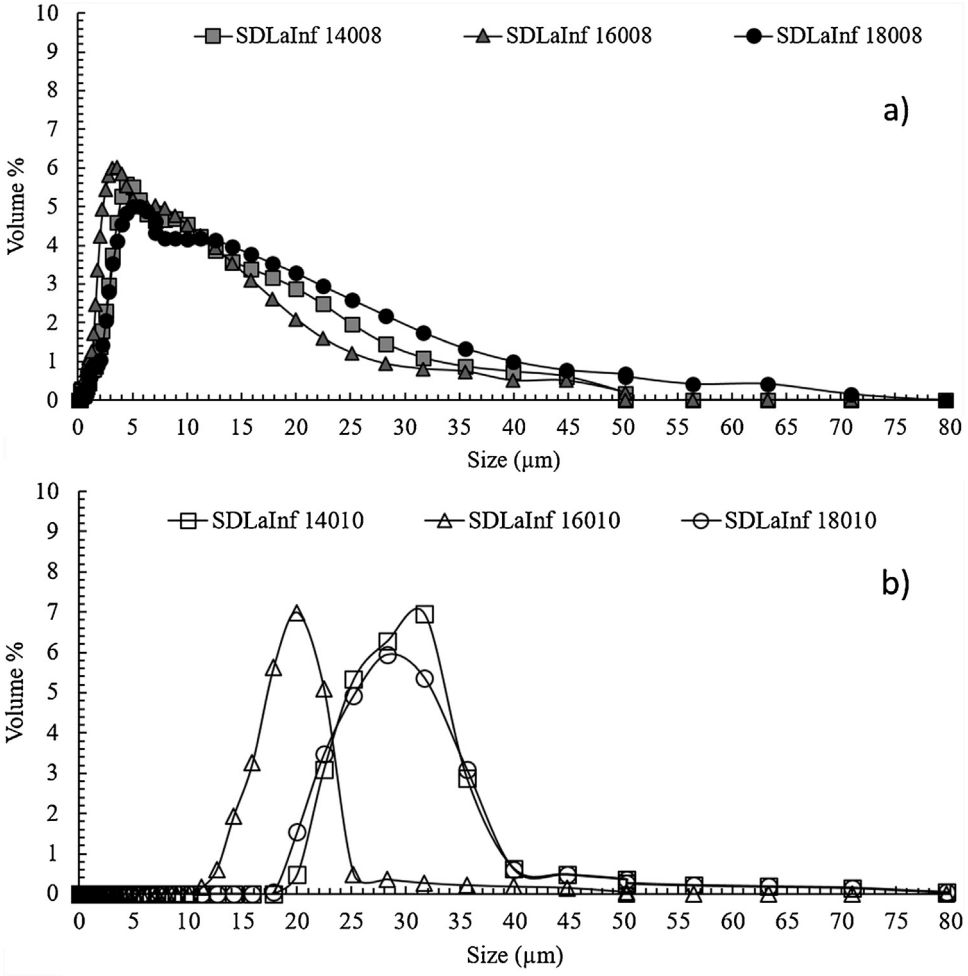

Fig. 3. Particle size distribution of SDLaInf at fed rate of: a) 8 mL/min and b) 10 mL/min.

the outflow spray drying conditions which were not controlled nor evaluated here.

In this study the EC_{50} determined for the LaInf was 0.45 mg/mL (R^2 = 0.998). This value is slightly below that reported for alcoholic extracts of *Cinnamon cassia* (Yang et al., 2012). However, the drying process impacted on the antioxidant capacity of SDLaInf powders. The EC_{50} of powders range from 1.3 (±0.12) to 3.1 (±0.11) mg/mL according to Table 2, where sample SDLaInf16008 showed the best antioxidant potential of the SD samples. This behavior is common during the encapsulation process of compounds with antioxidant capacity using spray drying at temperatures > 65 °C (Krishnaiah et al., 2012; Gallegos-Infante et al., 2013). According to results in total phenolic content, flavonoids and DPPH* radical scavenging; it is possible to state that feed rate is a crucial parameter in SD processing which is related to the contact time with hot air of the material in the chamber. Aim, feeding at 8 mL/min seems to be a favorable conditions to trap laurel, which consequently minimizes damage to the polyphenols and increases the antioxidant potential of the powders. In addition to the above, chemical analyses were performed on the microparticles, the characterization by molecular spectroscopy (FTIR) to identify the composition of a sample, morphology (SEM) and dimensional (PSD) are reported.

3.3. Spectrometry analysis, (FTIR)

The analysis of a compound by FTIR spectrometry reveals the type of molecules that constitute the compound of interest. Samples of LaInf (raw materials) and SDLaInf (SD product) were analyzed in solid form. FTIR-spectra (Fig. 1) showed a typical absorption band at a wavelength of λ= 3300 cm^{-1} characteristic of the hydroxyl group (-OH), the fact that this signal is significantly reduced in the SD samples is taken here as an evidence of the quality of the drying process since this signal is an indication of the water content of the sample. The typical absorption bands for Laurel are clearly in the range of 1100–1900 cm^{-1} and they can be considered as its molecular fingerprint, a comparison with the maltodextrin (MD) spectrum evidences the lack of this absorption bands. These typical Laurel absorption bands are practically lost in the SD samples (SDLaInf) which is considered here as a signal of a good encapsulation process. According to this, the samples dried at 160 °C and 10 mL/min feed rate showed the best laurel entrapment profile when comparing FTIR-spectra of all treatments. In Fig. 1, the characteristics wavelength (chemical bond) are shown, it is evident that the signal wavelenghts 1020 cm^{-1} (C—O; C—O—C), 1440 cm^{-1} (—CH2; –CH; =C—H), 1519 cm^{-1} (R-CO—NH —R), 1600 cm^{-1} (C—C, C=C, H—O—H) and 2387 cm^{-1} (CH2, nCH3) are closely related to aromatic compounds with phenyl bonds similar to those in polyphenolic compounds, such as flavonoids (Schulz and Baranska, 2007; Heneczkowski et al., 2001). Similar absorptions have been reported in studies where the presence of phenolic content from herbs and aromatic plants was studied (Lu et al., 2011; Ragupathi Raja Kannan et al., 2011).

3.4. Scanning electron microscopy, (SEM)

The morphology of the SD powder microparticles was also analyzed. In Fig. 2, the micrographs of the powder SDLaInf with MD are shown. The microparticles have rough surfaces with cavities and structural cracks. These morphological irregularities are possibly due to the process of removing water during the SD. As the drying temperature increases (resulting in a higher rate of evaporation), smoother and more defined surfaces are formed, similar to those reported by Alamilla-Beltrán et al. (2005), who found that lower air inlet temperatures resulted in irregularly shaped microparticles with creased surfaces, while higher air inlet temperatures resulted in more rigid microparticles and porous surfaces. According to micrographs, the drying process at 160 °C and low feed rates results in more defined microparticles with semispherical morphology, with no apparent cracks and without agglomerations by stickiness process. Similar results were reported by Tonon et al. (2009) for açai juice encapsulated with maltodextrin, observing irregular, smooth and spherical- semispherical conformations.

3.5. Particle size distribution of SDLaInf solutions

In Fig. 3a the particle size distribution (PSD) of the samples fed at 8 mL/min, and dried at the three temperatures (140, 160 and 180 °C) are shown. According to particle size analysis, the samples dried at feed rates of 8 mL/min were homogeneous and smaller in size, *i.e.*, around 6 μm (D_{50} 6.32 μm) with a quasi-modal distribution (Fig. 3a). This result is interesting because smaller particles in solution are able to form uniform arrangements and the spaces between particles are minimal, solutions containing this particles are homogeneous and initially stable to mechanical flow. In Fig. 3b, the quasi-modal distributions of the microparticles obtained at high feed rate (10 mL/min) are shown. The microparticles had dimensions from 10 to 40 μm, as a result of the drying process the behavior of these samples is associated with adhesion and agglomeration processes. Larger particles may be formed by agglomeration and interactions between the particles. Similar results were reported using maltodextrin for encapsulation of polyphenolic compounds of *Orthosiphon stamineus* extracts (Pang et al., 2014), and also in reports of encapsulation of rosemary oil with maltodextrin and gum arabic in various proportions (Janiszewska and Witrowa-Rajchert et al., 2009). Note that this analysis is important to determine the dimensional space occupied by microparticles in aqueous dispersions. This information is essential to shed light on the mechanical response (dynamic spectrum and viscosity of dispersions).

3.6. Rheological behavior of spray dried laurel infusions

The flow curves in simple shear regime show, for all samples, a shear thinning behavior ($n < 1$) with a tendency to a newtonian plateau at high shear rate (Fig. 4). The results of SDLaInf conditions 140 and 160 at 10 mL/min, showed higher viscosity, which is associated to larger modal particles promoting flow resistance especially at high shear rates (Hill and Carrington, 2006). However, high drying temperatures (180 °C) seem to adversely affect the viscosity of the dispersion (*i.e.* the thermal effect reduces the viscosity of the solution), which may be associated with thermal degradation and/or oxidation of microparticles during the spray drying process (Tuyen et al., 2010).

Fig. 4b, shows the viscosity for samples dried at low flow rate (8 mL/min). Here, the shear thinning behavior is not as pronounced as the samples dried at high feed rate which is associated here with higher flow stability. Sample dried at 140 °C shows a greater viscous response, here again drying temperature seems to reduce the viscosity of the solutions, although the relation is not direct since the

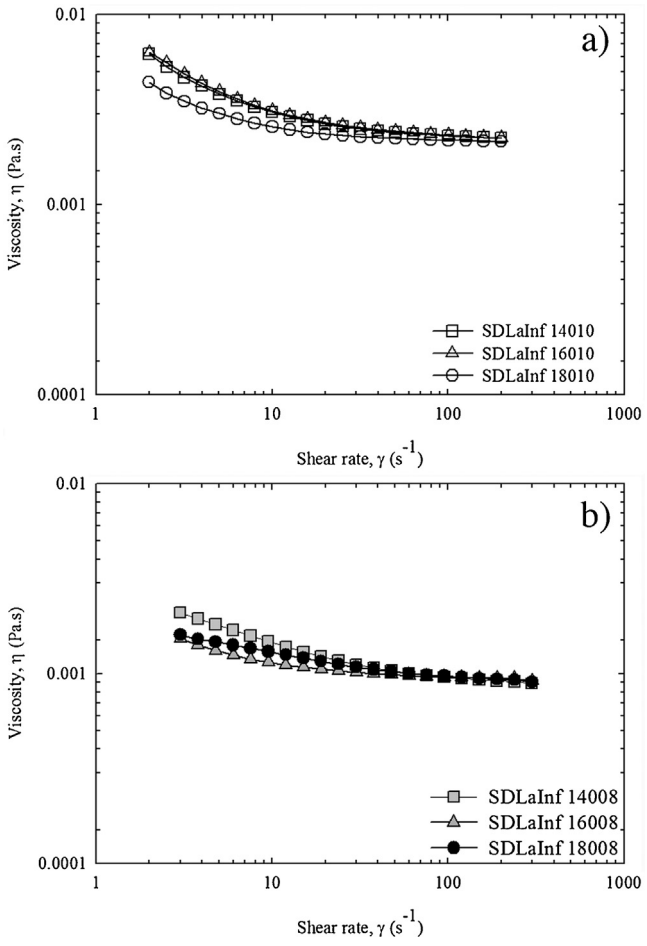

Fig. 4. Viscosity curves of SDLaInf at fed rate of: a) 8 mL/min and b) 10 mL/min.

sample with the lowest viscosity is the one dried at 160 °C and not the one dried at 180 °C. At high shear rates $\dot{\gamma} > 10\ s^{-1}$, all samples overlapped. Similar results have been reported in studies of spray drying (Grabowski et al., 2008; Medina-Torres et al., 2000, 2013).

In addition, the effect of SD parameters in dynamic storage (G') and loss (G″) modulus as a function of the oscillatory flow of SDLaInf reconstituted samples is shown in Fig. 5.

These samples at low feed rate (8 mL/min, see Fig. 5a) show a *solid-like* behavior, with a plateau zone formation at low frequencies yet with dominant viscous behavior (G" > G'). This evidences phase interactions between MD and LaInf at short times due to encapsulation (Medina-Torres et al., 2009, 2013).

On the other hand, samples fed to the dryer at high flow (10 mL/min, see Fig. 5b) showed a completely different behavior. This may be associated with particle agglomeration. It is evident that the feed rate was determinant in this study, similar trends were reported in studies using carbohydrates as an encapsulating agent (Medina-Torres et al., 2013; León-Martínez et al., 2011).

3.7. Controlled release analysis

The release characteristics of microencapsulated systems is very important to estimate stability and the storage periods that microcapsules may possess and the same way determined release profile of the core to produce controlled release systems. In this study it was found that SDLaInf did not present problems of solubilization in aqueous media, this may be demonstrated according to the formation of homogeneous solutions without obvious precipitates during assay development (electric potential, pZ > 30 mV). How-

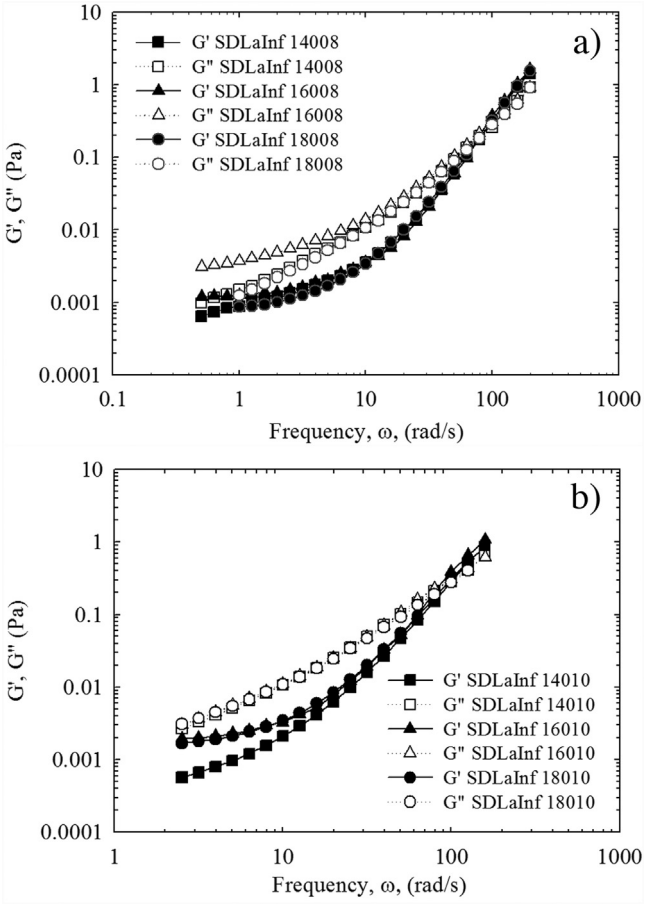

Fig. 5. Oscillatory shear curves of SDLaInf at fed rate of: a) 8 mL/min and b) 10 mL/min.

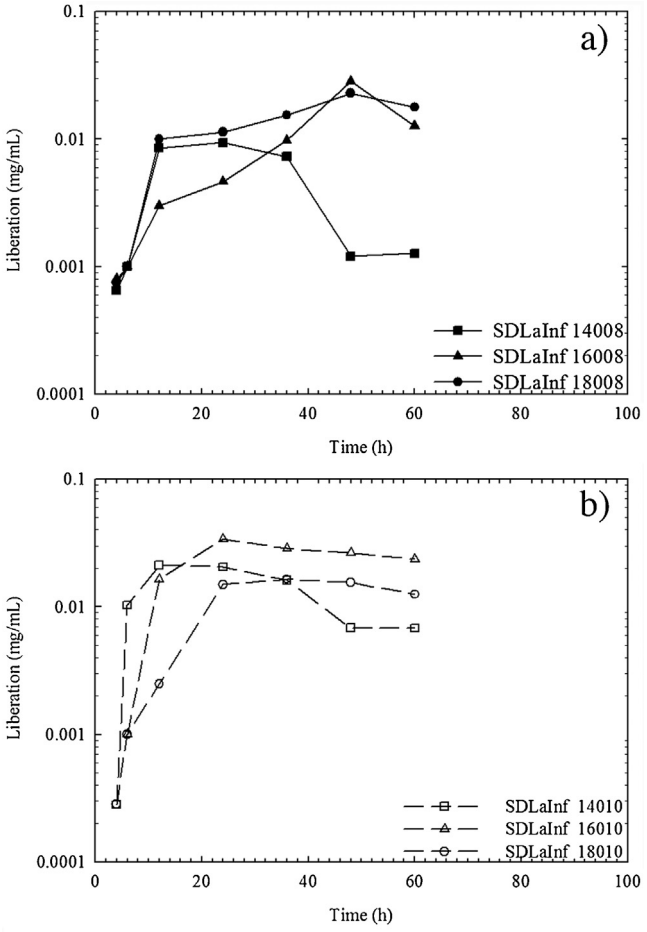

Fig. 6. Release curves of SDLaInf at different feed rate to dryer. a) 8 mL/min and b) 10 mL/min.

ever, the feed rate made a difference in the release profiles. Samples of SDLaInf fed at high feed rate to the dryer (Fig. 6b) have less stability (as it demonstrated in mechanical testing flow in this work) and consequently release faster its content (~24 h). On the other hand, those SDLaInf dried at low feed rate (Fig. 6a) resulted in an extended release of content (48 h), especially in those samples dried at temperatures of 160–180 °C. Liberation profiles reach a maximum after approximately 48 h when ~70% of the encapsulated compounds have been released. This will ensure optimum absorption of the polyphenols in the small intestine.

This release profiles try to mimic conditions such as those inside the digestive tract, where release of the spices is desirable, with an extended release period allowing the antioxidant benefits of this spice to deliver for a longer time in the body. The co-existence of two microstructures was confirmed by FTIR, rheological behavior and controlled release of SDLaInf. This can be attributed to microencapsulation conditions.

4. Conclusions

Spray drying of Laurel (*Litsea glaucescens*) infusions using maltodextrin as a wall material under the temperature and feed rate conditions developed in this study showed that encapsulation and interactions are both possible. The best conditions for stability, particle size, morphology and controlled release of encapsulated laurel infusions were found at a feed rate of 8 mL/min and 160 °C temperature.

The SD samples dried at 160 °C have smaller particle size, quasi-modal particle size distribution, semi-spherical morphology, improved mechanical response to shear, and more prolonged release under similar conditions as that of the digestive tract.

This study conditions may shed some light on the properties and stability of microencapsulated water infusions of Laurel spice, which is currently an area in development.

References

Alamilla-Beltrán, L., Chanona-Pérez, J.J., Jiménez-Aparicio, A.R., Gutiérrez-López, G.F., 2005. Description of morphological changes of particles along spray drying. J. Food Eng. 67 (1), 179–184.

Brand-Williams, W., Cuvelier, M.E., Berset, C., 1995. Use of a free radical method to evaluate antioxidant activity. LWT-Food Sci. Technol. 28 (1), 25–30.

Chelaru, C., Albu, M.G., Kaya, D.A., Meghea, A., Madalina, I., Öztürk, Ş., Giurginca, M., Segvi, N., Turkmen, M., Dinca, L., 2014. Synthesis and characterisation of microcapsules based on natural biopolymers and laurel essential oil. In: The 5th International Conference on Advanced Materials and Systems, Bucharest, pp. 23–25.

Cherrat, L., Espina, L., Bakkali, M., García-Gonzalo, D., Pagán, R., Laglaoui, A., 2014. Chemical composition and antioxidant properties of *Laurus nobilis* L. and *Myrtus communis* L. essential oils from Morocco and evaluation of their antimicrobial activity acting alone or in combined processes for food preservation. J. Sci. Food Agric. 94 (6), 1197–1204.

D'Archivio, M., Filesi, C., Varì, R., Scazzocchio, B., Masella, R., 2010. Bioavailability of the polyphenols: status and controversies. Int. J. Mol. Sci. 11 (4), 1321–1342.

Desai, K.G.H., Park, H.J., 2005. Encapsulation of vitamin C in tripolyphosphate cross-linked chitosan microspheres by spray drying. J. Microencapsulation 22 (2), 179–192.

Ersus, S., Yurdagel, U., 2007. Microencapsulation of anthocyanin pigments of black carrot (Daucus carota L.) by spray drier. J. Food Eng. 80 (3), 805–812.

Gallegos-Infante, J.A., Rocha-Guzmán, N.E., González-Laredo, R.F., Medina-Torres, L., Gomez-Aldapa, C.A., Ochoa-Martínez, L.A., Martínez-Sánchez, E.C., Hernández-Santos, B., Rodríguez-Ramírez, J., 2013. Physicochemical properties and antioxidant capacity of oak (Quercus resinosa) leaf infusions encapsulated by spray-drying. Food Biosci. 2, 31–38.

Grabowski, J.A., Truong, V.D., Daubert, C.R., 2008. Nutritional and rheological characterization of spray dried sweetpotato powder. LWT-Food Sci. Technol. 41 (2), 206–216.
Heimler, D., Vignolini, P., Dini, M., Romani, A., 2005. Rapid tests to assess the antioxidant activity of Phaseolus vulgaris L. Dry beans. J. Agric. Food. Chem. 53 (12), 3053–3056.
Heneczkowski, M.K., Nowak, D., Kuzniar, A., 2001. Infrared spectrum analysis of some flavonoids. Acta Poloniae Pharm. Drug Res. 58 (6), 415–420.
Hill, A., Carrington, S., 2006. Understanding the links between rheology and particle parameters. Am. Lab. 38 (21), 22.
Jafari, S.M., Assadpoor, E., He, Y., Bhandari, B., 2008. Encapsulation efficiency of food flavours and oils during spray drying. Drying Technol. 26 (7), 816–835.
Janiszewska, E., Witrowa-Rajchert, D., 2009. The influence of powder morphology on the effect of rosemary aroma microencapsulation during spray drying. Int. J. Food Sci. Technol. 44 (12), 2438–2444.
Khazaei, K.M., Jafari, S.M., Ghorbani, M., Kakhki, A.H., 2014. Application of maltodextrin and gum Arabic in microencapsulation of saffron petal's anthocyanins and evaluating their storage stability and color. Carbohydr. Polym. 105, 57–62.
Kong, D.G., Zhao, Y., Li, G.H., Chen, B.J., Wang, X.N., Zhou, H.L., Shen, T., 2015. The genus Litsea in traditional Chinese medicine: an ethnomedical: phytochemical and pharmacological review. J. Ethnopharmacol. 164, 256–264.
Krishnaiah, D., Sarbatly, R., Nithyanandam, R., 2012. Microencapsulation of Morinda citrifolia L. extract by spray-drying. Chem. Eng. Res. Des. 90 (5), 622–632.
León-Martínez, F.M., Rodríguez-Ramírez, J., Medina-Torres, L.L., Lagunas, L.M., Bernad-Bernad, M.J., 2011. Effects of drying conditions on the rheological properties of reconstituted mucilage solutions (*Opuntia ficus-indica*). Carbohydr. Polym. 84 (1), 439–445.
Lu, X., Wang, J., Al-Qadiri, H.M., Ross, C.F., Powers, J.R., Tang, J., Rasco, B.A., 2011. Determination of total phenolic content and antioxidant capacity of onion (*Allium cepa*) and shallot (*Allium oschaninii*) using infrared spectroscopy. Food Chem. 129 (2), 637–644.
Mahdavi, S.A., Jafari, S.M., Ghorbani, M., Assadpoor, E., 2014. Spray-drying microencapsulation of anthocyanins by natural biopolymers: a review. Drying Technol. 32 (5), 509–518.
Martins, I.M., Barreiro, M.F., Coelho, M., Rodrigues, A.E., 2014. Microencapsulation of essential oils with biodegradable polymeric carriers for cosmetic applications. Chem. Eng. J. 245, 191–200.
Medina-Torres, L., Brito-De La Fuente, E., Torrestiana-Sanchez, B., Katthain, R., 2000. Rheological properties of the mucilage gum (*Opuntia ficus indica*). Food Hydrocolloids 14 (5), 417–424.
Medina-Torres, L., Calderas, F., Gallegos-Infante, J.A., González-Laredo, R.F., Rocha-Guzmán, N., 2009. Stability of alcoholic emulsions containing different caseinates as a function of temperature and storage time. Coll. Surf. A 352 (1), 38–46.
Medina-Torres, L., García-Cruz, E.E., Calderas, F., Laredo, R.G., Sánchez-Olivares, G., Gallegos-Infante, J.A., Rodríguez-Ramírez, J., 2013. Microencapsulation by spray drying of gallic acid with nopal mucilage (Opuntia ficus indica). LWT-Food Sci. Technol. 50 (2), 642–650.
Moraes-de-Souza, R.A., Oldoni, T.L.C., Regitano-d'Arce, M.A.B., Alencar, S.M., 2008. Antioxidant activity and phenolic composition of herbal infusions consumed In Brazil. CYTA J. Food 6 (1), 41–47.
Muchuweti, M., Kativu, E., Mupure, C.H., Chidewe, C., Ndhlala, A.R., Benhura, M.A.N., 2007. Phenolic composition and antioxidant properties of some spices. Am. J. Food Technol. 2 (5), 414–420.
Ochoa-Martínez, L.A., González-Herrera, S.M., Morales-Castro, J., Rocha-Guzmán, N.E., Trancoso-Reyes, N., Urbina-Martinez, M.J., 2011. Propiedades de rehidratación y funcionales de un producto en polvo a base de jugo de granada y manzana. Ciencia@UAQ 4 (2), 19–25.
Pang, S.F., Yusoff, M.M., Gimbun, J., 2014. Assessment of phenolic compounds stability and retention during spray drying of Orthosiphon stamineus extracts. Food Hydrocolloids 37, 159–165.
Papadakis, E., Gardeli, C., Tzia, C., 2006. Spray drying of juice concentrate. Drying Technol. 24, 173–180.
Pourashouri, P., Shabanpour, B., Razavi, S.H., Jafari, S.M., Shabani, A., Aubourg, S.P., 2014. Oxidative stability of spray-dried microencapsulated fish oils with different wall materials. J. Aquat. Food Prod. Technol. 23 (6), 567–578.
Quek, S.Y., Chok, N.K., Swedlund, P., 2007. The physicochemical properties of spray-dried watermelon powder. Chem. Eng. Process. 46, 386–392.
Quiñones-Muñoz, T., Gallegos-Infante, J.A., Rocha-Guzmán, N.E., Ochoa-Martinez, L.A., Morales-Castro, J., González-Laredo, R.F., 2011. Mixing and tempering effect on the rheological and particle size properties of dark chocolate coatings. CyTA J. Food 9 (2), 109–113.
Ragupathi Raja Kannan, R., Arumugam, R., Anantharaman, P., 2011. Fourier transform infrared spectroscopy analysis of searass polyphenols. Curr. Bioact. Compd. 7 (2), 118–125.
Reineccius, G.A., 1988. Spray-drying of food flavors. In: ACS Symposium Series-American Chemical Society, (USA).
Schulz, H., Baranska, M., 2007. Identification and quantification of valuable plant substances by IR and Raman spectroscopy. Vib. Spectrosc. 43 (1), 13–25.
Shan, B., Cai, Y.Z., Sun, M., Corke, H., 2005. Antioxidant capacity of 26 spice extracts and characterization of their phenolic constituents. J. Agric. Food Chem. 53 (20), 7749–7759.
Shan, B., Cai, Y.Z., Brooks, J.D., Corke, H., 2007. The in vitro antibacterial activity of dietary spice and medicinal herb extracts. Int. J. Food Microbiol. 117 (1), 112–119.
Tonon, R.V., Brabet, C., Pallet, D., Brat, P., Hubinger, M.D., 2009. Physicochemical and morphological characterisation of açai (Euterpe oleraceae Mart.) powder produced with different carrier agents. I. J. Food Science Technol. 44 (10), 1950–1958.
Tonon, R.V., Grosso, C.R., Hubinger, M.D., 2011. Influence of emulsion composition and inlet air temperature on the microencapsulation of flaxseed oil by spray drying. Food Res. Int. 44 (1), 282–289.
Tsai, S.F., Lee, S.S., 2011. Flavonoid composition in the leaves of twelve Litsea and Neolitsea plants. J. Chin. Chem. Soc. 58 (3), 376–383.
Tuyen, C.K., Nguyen, M.H., Roach, P.D., 2010. Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (*Momordica cochinchinensis*) fruit aril powder. J. Food Eng. 98 (3), 385–392.
Xie, Z.-W., Yu, Y.-Q., 1996. The Guide of National Chines Herbal Medicine (I). People's Medical Publishing House Beijing.
Yang, C.H., Li, R.X., Chuang, L.Y., 2012. Antioxidant activity of various parts of Cinnamomum cassia extracted with different extraction methods. Molecules 17 (6), 7294–7304.