



Microencapsulation by spray drying of *Lanena microcarpa* extract: Technological characteristics and antioxidant activity

[Microencapsulación mediante secado por pulverización del extracto de *Lanena microcarpa*: Características tecnológicas y la actividad antioxidante]

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Abstract

Context: A functional extract from *Lanena microcarpa* (Lm), possess interesting antioxidant and anti-inflammatory properties. However, the unprocessed dried extract occurs as sticky and low-water-soluble material showing critical properties for industrial applications. The unprocessed dried extract is not always enough stable to preserve its functional properties, also giving practical difficulties for the manufacturing.

Aims: This research aimed to produce Lm extract microparticles with enhanced functional stability and technological characteristics by spray-drying.

Methods: Lm extract was microencapsulated by spray-drying using a sodium-carboxymethylcellulose (NaCMC) based matrix. Physicochemical and technological characteristics (determined by UV, HPLC, LLS, SEM, DSC, and in vitro dissolution tests), as well as antioxidant properties (DPPH-test) of the resulting powder (LmC) were examined.

Results: The produced spray dried microparticles showed satisfying encapsulation efficiency, good functional stability and enhanced technological properties.

The selected carrier and process conditions led to a stable and handling microencapsulated powder form with improved water dissolution rate. Moreover, the matrix was also able to preserve the antioxidant activity of the phenolic compounds-rich extract.

Conclusions: The made-up powder resulted in a functional component that can be used with great potential in cosmetics, foods or nutraceutical products.

Keywords: Antioxidant; functional stability; in vitro dissolution test; *Lanena microcarpa*; sodium-carboxymethylcellulose matrix; spray-drying.

Resumen

Contexto: Un extracto funcional a partir de *Lanena microcarpa* (Lm), posee propiedades antioxidante y anti-inflamatorias interesantes. Sin embargo, el extracto seco no procesado es pegajoso y el poco soluble en agua, por lo que muestra propiedades críticas para aplicaciones industriales. El extracto seco no procesado no siempre es lo suficientemente estable como para preservar sus propiedades funcionales, dando también dificultades prácticas en el proceso de fabricación.

Objetivos: Esta investigación tuvo como objetivo producir microparticulas del extracto de Lm con estabilidad funcional y características tecnológicas mejoradas mediante secado por aspersión.

Métodos: Este documento informa sobre la encapsulación del extracto de Lm por secado por aspersión en una matriz basada en carboximetilcelulosa sódica (NaCMC). Se examinaron las características físico-químicas y tecnológicas (determinadas por UV, HPLC, LLS, SEM, DSC, y en las pruebas de disolución in vitro), así como las propiedades antioxidantes (prueba de DPPH) del polvo resultante (LMC).

Resultados: Las microparticulas secadas por pulverización mostraron eficacia de encapsulación satisfactoria, buena estabilidad funcional y propiedades tecnológicas mejoradas.

Los vehículos y condiciones de proceso seleccionados condujeron a una forma estable y manejable del polvo microencapsulado con una mejorada velocidad de disolución de agua. Por otra parte, la matriz también fue capaz de preservar la actividad antioxidante del extracto rico en compuestos fenólicos.

Conclusiones: El polvo obtenido resultó en un componente funcional que se puede utilizar con un gran potencial en cosméticos, alimentos o productos nutracéuticos.

Palabras Clave: Antioxidante; estabilidad funcional; *Lanena microcarpa*; matriz de carboximetilcelulosa; prueba de disolución in vitro; secado por pulverización.

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INTRODUCTION

Lannea microcarpa Engl and K. Krause (Anacardiaceae), trivial name African or Wild Grapes, is a tropical tree distributed in different African regions known for its economic value and medicinal properties. Traditional remedies prepared from its leaves, bark, roots, and fruits are used to treat several human diseases such as mouth blisters, rheumatism, sore throats, dysentery, conjunctivitis, stomatitis, skin eruptions and ulcers (Bazongo et al., 2014).

The grape-like fruits, as a source of vitamins, are eaten fresh or squeezed and drunk as juice whereas the leaves are used for food and fodder (Haarmeyer et al., 2013). Oil from the seeds is a component of hair-care products (Picerno et al, 2006).

In our previous study, the chemical composition, anti-inflammatory activity and the lack of skin irritation of a polar extract from *L. microcarpa* has been reported (Picerno, et al., 2006). Furthermore, Bationo et al. (2012), showed the antioxidant and antibacterial properties of the aerial parts of this specie. Despite all the interesting nutritional and biological properties of *Lannea*, the poor solubility of the unprocessed extract occurring as sticky material involves practical difficulties for an industrial use. In addition, constituents release and degradation/oxidation process taking place during the storage period (Shu et al., 2006) may reduce the active ingredient content and, consequently, the health beneficial values.

A convenient way to increase the shelf-life of a plant derivative is to transform it into a stable dry powder form by spray-drying with appropriate polymers (Laine et al., 2008; Picerno et al., 2011b; Sansone et al., 2011a; 2011b).

Food and cosmetic ingredients in spray-dried powder form have reduced bulk weight and size, long-lasting biological stability, and easier transportation and handling (Bhavesh et al., 2014; Sansone et al., 2014).

In a previous work, a new sodium carboxymethylcellulose (NaCMC) coating matrix was developed to encapsulate a soy extract by spray-drying to improve both the water

dissolution rate and permeation properties of the final product (Sansone et al., 2013). The aim of the present contribution was to apply the NaCMC spray dried matrix to Lm extract to verify the versatility of the microencapsulation method as well as enhancing functional stability and technological characteristics of Lm extract. The influence of the microencapsulation method on characteristics of *Lannea*/NaCMC powder (LmC) has been investigated with respect to the unprocessed extract (Lm). Solid state, dissolution rate and functional stability under harsh storage conditions have been studied.

MATERIALS AND METHODS

Chemicals

Sodium carboxy methyl cellulose (NaCMC, medium viscosity, E466) and myricetin 3-O- α -L-rhamnopyranoside were supplied by Sigma Aldrich (Milan, Italy). All other chemicals used were of reagent grade.

Plant material and preparation

Leaves of *L. microcarpa* Engl et K. Krause, were collected near Bamako, Mali, in May-July 2002 and identified by Dr. Rokia Sanogo (University of Bamako, Mali). A voucher sample (LM, 2002) was deposited at the Herbarium of the Faculte de Medicine, Pharmacie et D'Odontostomatologie, University of Bamako, Mali.

Leaves extract of Lm was prepared and characterized as reported by Picerno et al. (2006). Briefly, dried leaves of *L. microcarpa* were defatted with n-hexane and chloroform and then extracted with methanol. The dried extract was partitioned between n-butanol (n-BuOH) and water to afford an n-BuOH-soluble portion. This was suspended in water and lyophilized to give Lm extract. A portion of this was purified by column chromatography and isolated compounds were identified by spectroscopic methods.

Microencapsulated powders production

Liquid feed preparation and spray drying conditions

A slightly modified method previously reported elsewhere (Sansone et al., 2013) to prepare the liquid feed has been used. Briefly: a liquid solution (200 mL) containing 1:1 w/w NaCMC/Lm (2 g) was prepared using a H₂O/EtOH 1/1 solvent ratio. NaCMC was dissolved in water; then, the ethanol was slowly introduced into the solution and, finally, the polymeric solution was added to a glass bubble containing the Lm dried extract under continuous magnetic stirring. A total final concentration (1% w/v) was kept. The homogeneous suspension was sonicated for 30 minutes before further processing. The liquid feeds were spray dried in a Büchi B-191 Mini Spray Dryer (Büchi Laboratoriums-Technik, Flawil, Switzerland) under the following experimental conditions: inlet/outlet temperatures 100/65°C ; spray flow feed rate 5 mL/min; nozzle diameter 0.7 mm; drying air flow 600 L/h, air pressure 6 bar, aspirator 100%. In order to keep homogeneity, while feed was pumped into the spray dryer, the suspensions were gently stirred using a magnetic stirring. Each preparation was carried out in triplicate. All spray-dried NaCMC-Lm powders were collected and stored under vacuum for 48 h at room temperature before characterization. As a reference, NaCMC powder (Blank) was prepared by spray-drying under the same experimental conditions from an aqueous feed containing NaCMC 0.5% w/v.

Powders characterization

Quantitative analysis

UV method: The concentration of myricetin 3-O- α -L-rhamnopyranoside, chosen as the marker of the Lm extract, was evaluated by measuring absorbance (UV/Vis spectrometer Lambda 25, Perkin Elmer Instruments, MA, USA) at λ 257 nm in 1 mm cell; (Spectracomp 602, Advanced Products srl, Milan, Italy). Calibration curves were previously worked out using MeOH and distilled water. Proportionality between

absorbance and concentration was verified in the range 50 - 300 mg/L ($R^2 > 0.999$) for MeOH and 5-25 mg/L ($R^2 > 0.999$) for water. Samples (40 mg) of both Lm and LmC produced powders were dissolved in 40 mL methanol, shaken and centrifuged for 15 min at 3000 rpm. The supernatants filtered with 0.45 μ m filters were analysed. Each analysis was made in triplicate. Results expressed in terms of average values and compared to those obtained by HPLC analyses. Moreover, as control, the extract, dissolved in MeOH, was analyzed for its total phenolic content according to the Folin-Ciocalteu colorimetric method (Picerno et al., 2011a; Aquino et al., 2014). Total phenols were expressed as of myricetin 3-O- α -L-rhamnopyranoside equivalent (μ g/mg extract).

HPLC method: Myricetin 3-O- α -L-rhamnopyranoside concentration was also evaluated by a HPLC apparatus [Agilent 1100 series system equipped with a Model G-1312 pump, a Rheodyne Model G-1322A loop (20 μ L), a DAD G-1315 detector, and a 150 x 3.9 mm i.d. C-18 μ -Bondapak column]. Peaks areas were calculated with an Agilent integrator. The eluent was TFA 0.1% in water (solvent A) and methanol (solvent B). The elution gradient used was as follows: 0 \rightarrow 10 min, 20 \rightarrow 30% B; 10 \rightarrow 40 min, 30 \rightarrow 40% B; 40 \rightarrow 50 min, 40 \rightarrow 50% B; 50 \rightarrow 60 min, 50 \rightarrow 100% B, flow rate of 0.8 mL/min, DAD detector set at λ 257 nm.

Linearity: Reference standard solutions of myricetin 3-O- α -L-rhamnopyranoside were prepared at three concentration levels (0.6-2.4 mg/mL) and were injected (20 μ L) three times. The standard curve was analyzed using the linear least-squares regression equation derived from the peak area (regression equation $y = 2027.3 x - 317.12$, $R^2 = 0.999$, where y is the peak area and x the concentration).

Specificity: Peak associated with the marker was identified by its retention time ($t_r = 33.6$ min) and confirmed by co-injection and UV spectrum compared with standard.

Yield and loading efficiency

Production yields were gravimetrically determined (balance Crystal 100 CAL – Gibertini (max 110 g, d = 0.1 mg; +15°C/30°C) and expressed as the weight percentage of the final product compared to the total amount of the materials sprayed.

The theoretical extract content (TEC) was calculated as percentage of Lm compared to the initial total content of components (Lm plus NaCMC) in the feed before spray-drying.

Actual polyphenol content of the unprocessed extract Lm (APC_{Lm}), and spray-dried powders (APC_{LmC}) was determined by UV and HPLC methods as previously described and expressed as myricetin 3-O- α -L-rhamnopyranoside equivalents in percentage to 100 mg of powder.

The actual extract content (AEC) was derived by APC and calculated as

$$\text{AEC\%} = \text{APC}_{\text{LmC}} / \text{APC}_{\text{Lm}} \times 100$$

The extract-encapsulation efficiency (EE%) was the ratio of the actual to the theoretical extract content $\text{EE\%} = \text{AEC}/\text{TEC} \times 100$

Each analysis was made in triplicate and results expressed in terms of average values.

Particle size analyses

Sizes and dimensional distributions of both Lm and LmC were carried out with a Laser Light Scattering (LLS) granulometer (Beckman Counter LS 230, Particle Volume Module Plus, U.K.). An excess of each powder sample was added to 50 mL distilled water under continuous stirring until reaching oversaturation conditions. About 200 μ L of each suspension were poured into the small volume cell to obtain an obscuration between 8 and 12%. Particle size distributions were calculated using the Fraunhofer model. The analyses were made in triplicate. Results were expressed as d_{50} indicating the volume diameter at the 50th percentile of the particle size distribution and span value calculated as $[(d_{90} - d_{10})/d_{50}]$.

Morphology

Morphology of the particles was examined by scanning electron microscope (SEM, Zeiss EVO MA10, Carl Zeiss SMT AG, Munchen-Hallbergmoos, Germany) operating at 14 kV; the powders were coated with Au/Pd and eventually observed at different extensions. The fluorescent microscopy assays (FM) were performed observing the samples with a Zeiss Axiophot fluorescence microscope, with 63 x 1.4 NA plan Achromat oil immersion objectives (Carl Zeiss Vision, München-Hallbergmoos, Germany) using standard DAPI (4', 6-diamidino-2-phenylindole) optics that adsorbs violet radiation (max 372 nm) and emits a blue fluorescence (max 456 nm).

Differential scanning calorimetry (DSC)

Samples of NaCMC raw material, Lm and processed powder LmC were analyzed by Differential Scanning Calorimetry on an indium calibrated Mettler Toledo DSC 822e (Mettler Toledo, OH, USA). Thermograms were recorded by placing accurately weighed quantities (8-10 mg weighed with a microbalance MTS Mettler Toledo, OH, USA) of each sample in a 40 μ L aluminium pan, which was sealed and pierced. The samples underwent one dynamic thermal cycle; they were heated from 25°C to 350°C at a heating rate of 10°C/min.

In vitro dissolution tests

In vitro dissolution/release tests of Lm and LmC powders were carried out, according with the Farmacopea Ufficiale Italiana (F.U.I. XII, 2009). Briefly, samples of 500 mg were dissolved in 1000 mL of distilled water on a dissolution test apparatus n. 2 (SOTAX AT Smart Apparatus (Basel, CH) on line with a spectrophotometer UV/Vis spectrometer Lambda 25, Perkin Elmer Instruments, MA, USA). The paddle velocity and temperature were 100 rpm and 25°C. All the dissolution/release tests were made in triplicate; only the mean values are reported (standard deviations < 5%). Amount of the extract dissolved was measured as APC (actual total polyphenol content), so as reported in the previous paragraphs.

Stability studies

Accelerated stability studies

Evaluation of the physicochemical stability was performed according to the method for accelerated stability assessment reported by the ICH guide lines (International Conference on Harmonization, 2003). Glass vials containing 1.5 g of each powder were stored for 6 months at 40 ± 2 °C - 75 ± 5 % of RH in a climatic chamber (Climatic and Thermostatic Chamber, Mod.CCP37, AMT srl, Milan, Italy). At given times (0, 1, 2, 3, 4, 5 and 6 months) samples of each batch were collected. The first analysis (t_0) has been conducted after 48 h from the formulation. The myricetin 3-O- α -L-rhamnopyranoside, content was evaluated by HPLC method. All measurements were performed in triplicate.

Functional stability

The free-radical scavenging activity of Lm and LmC powder was tested, over 6 months of storage, using the stable 1,1-diphenyl-2-picrylhydrazyl radical (DPPH test) according to the procedure previously reported (Picerno et al., 2011a; Sansone et al., 2011a; 2011b). Briefly, Lm (3–15 μ g/mL) or LmC (10–50 μ g/mL), underwent to the same storage conditions, were dissolved in MeOH and centrifuged for 20 minutes. A part (37.5 μ L) of the supernatant was added to 1.5 mL of daily prepared DPPH solution (0.025 g/L in MeOH). An equal volume (37.5 μ L) of the vehicle alone was added to control tubes. Absorbance at 517 nm was measured 10 min after starting the reaction on a Thermo Evolution 201 UV-visible spectrophotometer. The DPPH concentration in the reaction medium was calculated from a calibration curve analyzed by linear regression and EC_{50} (mean effective scavenging concentration) was calculated using the Litchfield and Wilcoxon test (Tallarida and Murray, 1984) as the concentration in μ g/mL of sample necessary to decrease the initial DPPH concentration by 50%.

Statistical analysis

The experimental data in this study are presented as means \pm SD (standard deviation) and were obtained from at least three independent experiments. The resulted data were assessed by analysis of variance; one-way ANOVA tests have been performed to determine the overall significance of variations. A p value of 0.03 was used to verify the significance of all tests.

RESULTS AND DISCUSSION

The obtained results in term of yield, polyphenol/extract content, encapsulation efficiency and dimensional distribution are reported in Table 1 and discussed below.

Microencapsulation process

As shown in Table 1, the process yields (46.7 – 63.2%) resulted not so high; this behaviour was probably due to the small volume liquid feed (200 mL) and low solid content sprayed as well as to the loss of the smallest and lightest particles with the exhaust of the spray dryer. Theoretically, the higher solid content, the larger yield of powder (Fernandez-Perez et al., 2004), but, a too high solids content would result in a low amount of active material being encapsulated into the powder. As a result, the encapsulation efficiency obtained for LmC was fairly high (91.8%). The functionality of Lm is correlated to the polyphenol content (Picerno et al., 2006), the higher is the APC in the produced encapsulated powder form, the greater should be the final formulation functional activity, and thus, the obtained encapsulation efficiency is a very interesting and promising characteristic.

Dimensional analysis and morphology

LLS analysis indicated that spray dried CMC-based microparticles had very narrow size distribution (Blank d_{50} 6.47 μ m) with respect to the parent CMC raw material (d_{50} 21.09 μ m) (Table 1). Moreover, the particles dimensions seem affected by the interaction between polymer and extract. In fact, for LmC it was

Table 1. Composition and characteristics such as theoretical extract content (TEC), theoretical polyphenols content (TPC), actual polyphenols content (APC), actual extract content (AEC), encapsulation efficiency (EE) of unprocessed and processed materials.

Samples	NaCMC g/100 mL	Lm g/100 mL	NaCMC/Lm ratio	Yield %	TEC %	TPC %	APC % w/w	AEC %	EE %	d ₅₀ µm (span)
NaCMC	-	-	-	-	-	-	-	-	-	21.09 (1.06)
Lm	-	-	-	-	-	-	10.7 ± 0.8	-	-	-
Blank	0.5	-	-	63.2 ± 1.7*	-	-	-	-	-	6.47 (0.41)
LmC	0.5	0.5	1:1	46.7 ± 3.4*	50	5.3	4.9 ± 0.1	45.9	91.8	4.75 (1.29)

Data are mean ± SD. *P < 0.003. Statistical comparison between groups was made using ANOVA followed by the Bonferroni parametric test. Particle size distributions were calculated using the Fraunhofer model and was expressed as d₅₀ indicating the volume diameter at the 50th percentile of the particle size distribution and span value calculated as $[d_{90} - d_{10}]/d_{50}$.

observed a reduction of microparticles dimensional distribution (d₅₀ 4.75 µm) with respect to the Blank (d₅₀ 6.47 µm) (Table 1). This behaviour is probably due to a physical interaction between NaCMC and Lm components in forming matrix during the spray drying process. The reduced particles mean size generally improves the flowability and the dissolution rate of the powder

which are desirable technological characteristics in manufacturing and production stages (Da Silva-Junior et al., 2009).

The morphological studies, conducted by microscopy analysis, showed similar results, in term of shape and matrix distribution, of all microparticles produced (Fig. 1).

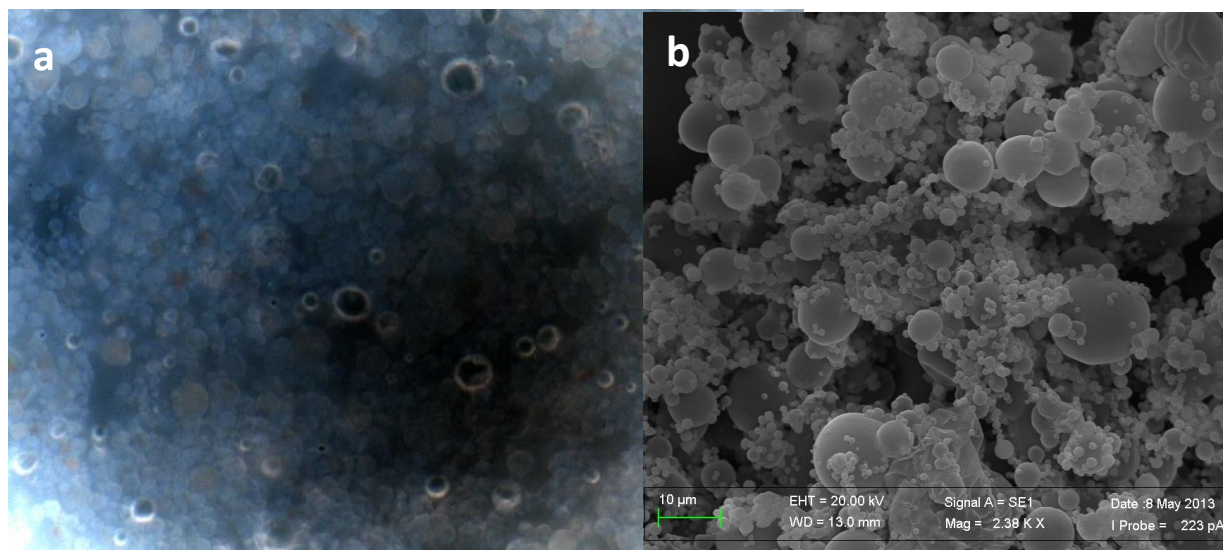


Figure 1. Fluorescence Microscopy (a) and Scanning Electron Microscopy (b) of *Lannea microcarpa*/NaCMC powder (LmC) produced microparticles. The images show the powder in amorphous state with well formed, small and trendy spherical microparticles with a smooth surface.

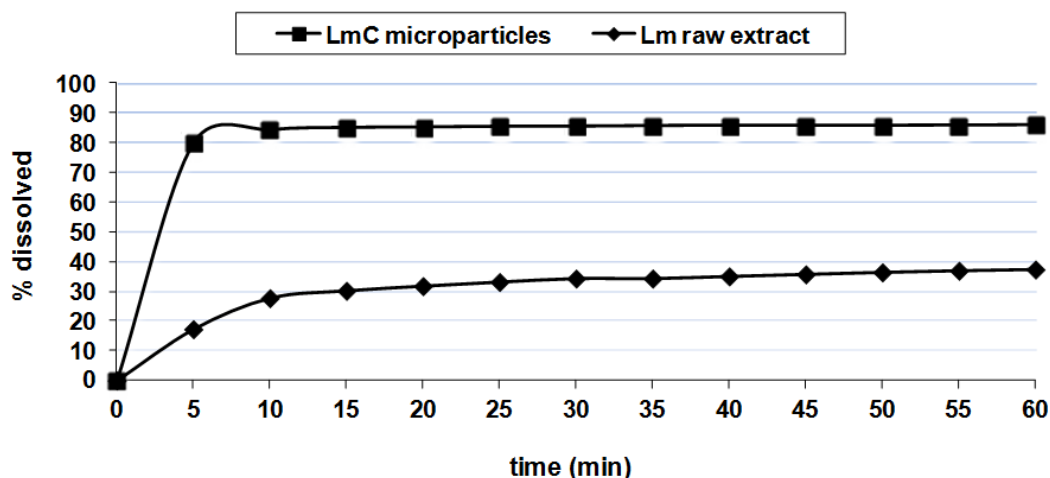


Figure 3. In vitro dissolution profile in water of *Lannea microcarpa*/NaCMC (LmC) microparticles compared to *Lannea microcarpa* unprocessed extract (Lm). In 5 min a release/dissolution (80%) of the extract, evaluated as APC, was obtained from LmC with respect 18% dissolution rate of unprocessed one (Lm).

Stability studies

The extract of *L. microcarpa* is a rich source of polyphenols (Picerno et al., 2006), which are generally known to undergo to oxidation/degradation process. Their reactivity may prevent from the use as functional raw materials for food and pharmaceutical purposes (Laine et al., 2008). In this research, accelerated stability test was used to examine the shelf-life of the product in conditions of storage more harsh than environmental ones (higher temperature and increased humidity) and in a shorter time period

than “real time” stability. The results obtained for LmC produced powder (Table 2) showed that the myricetin 3-O- α -L-rhamnopyranoside remained unaltered. In fact, no decrease of concentration which can be considered significant in terms of stability (< 1%) was recorded by HPLC method (International Conference on Harmonization, 2003). As expected, the stability was strongly dependent on polymeric matrix and its encapsulation efficiency. The whole results suggest that the use of NaCMC was beneficial for its coating and embedding properties as well as for core retention ability.

Table 2. Actual polyphenol content (APC) and free-radical scavenging activity (DPPH test) expressed as the concentration in $\mu\text{g/mL}$ of sample necessary to decrease the initial DPPH concentration by 50% (EC_{50}) of the extract before *Lannea microcarpa* extract (Lm) and after *Lannea microcarpa*/NaCMC powder (LmC) microencapsulation process.

Treatment	APC (%)		EC_{50} ($\mu\text{g/mL}$)	
	t_0	t_6 months	t_0	t_6 months
Lm extract	10.7 ± 0.8	$6.8 \pm 0.4^*$	6.5 ± 0.4	$11.1 \pm 0.3^{**}$
LmC	4.9 ± 0.1	4.9 ± 0.5	7.4 ± 0.2	7.6 ± 0.4
α -tocopherol	-	-	10.1 ± 1.3	10.1 ± 1.6

The test was performed until 6 months of storage. At the time point 3 withdrawals for 3 different samples of each batch were carried out. α -Tocopherol was used as positive control of the DPPH assay. Data are mean \pm SD. * $P < 0.003$, (vs APC at t_0), ** $P < 0.002$ (vs EC_{50} at t_0).

Functional stability

The functional stability of the extract, before and after the spray-drying process, and until six months of study, was evaluated as free-radical scavenging activity using the DPPH test. The strong antioxidant activity of Lm, expressed as EC₅₀, was even than α -tocopherol, used as positive control, and it remained unaltered after the spray-drying process (Table 2). No decrease of EC₅₀, significant in term of stability (less than 5%), was recorded after six months of accelerated storage conditions for LMC respect to the extract raw material. During the same time, the value of inhibition of Lm unencapsulated varied from EC₅₀ 6.5 (0 month) to 11.1 (6 months) (Table 2). Our results are in agreement to those reported by Munin et al. (2010) about the study of stability on formulation containing natural polyphenolic compound. As a result, the selected carrier and spray-drying process are able to preserve the antioxidant activity of the phenolic compounds-rich extract.

CONCLUSIONS

A NaCMC matrix by spray drying was used to encapsulate a functional extract of *L. microcarpa* via spray-drying. The use of a 1:1 NaCMC/extract weight ratio (1% w/v) led to encapsulate high quantity of extract forming stable powders made up of well-formed and micronized particles suitable for storage and handling. Even under harsh storage conditions the bioactive polyphenols and the antioxidant activity did not seem significantly modified. The matrix was also able to rapidly dissolve in water releasing about 80% of the bioactive extract in few minutes, and the final products showed improved technological characteristics with respect the unprocessed extract.

Therefore, the obtained stable powder could be use as ingredient in topical formulations, food supplements and nutraceutical products because of its high polyphenol content which can award a good functionality. The method may be generally applied to encapsulate and delivery poor soluble and not stable extracts in powder form with

extended shelf-life, optimized technological and functional characteristics.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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