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Research Article

Physicochemical, flow and antioxidant properties of microencapsulated powder fractions of *Hibiscus sabdariffa* L. calyxes and *Dichrostachys glomerata* Forssk. fruits

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ABSTRACT

Regardless of the employed extraction methods, the effectiveness of the health properties of plant bioactive ingredients post-extraction depends on preserving their stability, bioactivity and bioavailability of biomolecules when consumed. This study aimed to evaluate the influence of encapsulation on the physicochemical, flow and antioxidant properties of *Dichrostachys glomerata* and *Hibiscus sabdariffa* powder fractions. *Dichrostachys glomerata* powder fractions φ < 212 µm and *H. sabdariffa* powder fractions φ < 315 µm were microencapsulated with maltodextrin, and their characteristics were compared to those of non-microencapsulated powders. A significant effect of encapsulation was evidenced in the physicochemical, flow and antioxidant properties of studied powders (P<0.05). The encapsulation yields were 42.3% and 38.5% for *D. glomerata* and *H. sabdariffa*, respectively. Plant powder microcapsules exhibited cohesive properties and lower particle sizes and total colour while non-microencapsulated powders flowed easily. Total phenolic, flavonoid, condensed tannins and anthocyanin contents of microcapsules were 2 – 3 times less than that of their non-microencapsulated powders. These phenolic compounds were relatively correlated to the antioxidant properties. Microencapsulation of plant fraction powders with potential as functional food ingredients may be an option to preserve the best quality, and on the storage effects towards the quality properties to enhance the shelf life of the product.

Keywords: Plant powder fractions; encapsulation; physicochemical properties; antioxidant activity.

RESUME

Quelles que soient les méthodes d'extraction utilisées, l'efficacité des propriétés sanitaires des ingrédients bioactifs végétaux après extraction dépend de la préservation de leur stabilité, de leur bioactivité et de la biodisponibilité des biomolécules lors de leur consommation. Cette étude visait à évaluer l'influence de l'encapsulation sur les propriétés physicochimiques, d'écoulement et antioxydantes des fractions de poudres de *Dichrostachys glomerata* et *d'Hibiscus sabdariffa*. Les fractions de poudre φ < 212 µm de *D. glomerata* et les fractions de poudre φ < 315 µm de *H. sabdariffa* ont été microencapsulées avec de la maltodextrine et leurs caractéristiques ont été comparées à celles de poudres non microencapsulées. Un effet significatif (P<0,05) de l'encapsulation a été mis en évidence sur les propriétés physicochimiques, d'écoulement et antioxydantes des poudres étudiées. Les rendements d'encapsulation étaient de 42,3 % et 38,5 %, respectivement pour *D. glomerata* et *H. sabdariffa*. Les microcapsules de poudre de plantes présentaient des propriétés de cohésion, une faible taille de particule et une couleur totale inférieure, tandis que les poudres non microencapsulées coulaient facilement. La teneur totale en phénols, flavonoïdes, tanins condensés et anthocyanes des microcapsules était 2 à 3 fois inférieure à celle de leurs poudres non microencapsulées. Ces composés phénoliques étaient relativement corrélés aux propriétés antioxydantes. La microencapsulation de fractions de poudres de plante ayant un potentiel en tant qu'ingrédients alimentaires fonctionnels peut être une option pour préserver la meilleure qualité et pour améliorer les effets du stockage sur les propriétés de qualité afin d'améliorer la durée de conservation du produit.

Mots clés: Fractions de poudre de plantes; encapsulation; propriétés physico-chimiques; activité antioxydante.

1. INTRODUCTION

Nowadays, due to increased interest in the consumption of food-source bioactive compounds, a growing number of research works have been conducted on plant-derived bioactive ingredients. In the meantime, the demand for suitable and advanced extraction processes with an enhanced overall yield of bioactive compounds, also known as

"green technique" is gaining attention. In this respect, a novel green method with economic advantages, called Alternation of Drying and Grinding (ADG), and Controlled Differential Sieving process (CDSp) was proposed (Baudelaire 2013). Various research studies have explored the application of plant grinding followed by CDSp, and supporting evidence was found for its potential use and efficiency to concentrate a wide variety of valuable plant bioactive compounds and to improve the bioactivities of various experimental plant matrices (Becker et al. 2017; Deli et al. 2019ab, 2020ab; Noumi et al. 2020; Mbassi et al. 2021). Remarkably, grinding the dried plant materials (herb, plant, or plant parts) to an appropriate powder particle size followed by CDSp to discriminate powder fractions of homogeneous particle sizes of plant materials, enhances the physicochemical and functional properties, as resulting in the improvement of food powder quality (Jiang et al. 2017). In this respect, our previous research work on *Dichrostachys glomerata* fruits and *Hibiscus sabdariffa* calyx, explored an innovative extraction process based on ADG and CDSp. It was found that the powder fractions of $\varphi < 212$ µm of *D. glomerata* and powder fraction $\varphi < 315$ µm of *H. sabdariffa* concentrated more bioactive ingredients (phenolic molecules, terpenes, vitamins, minerals, carotenoids) with similar antioxidant properties as that of ethanolic extract powder (Deli et al. 2019a; 2020ab; Mbassi et al. 2021; Noumi et al. 2021). Additionally, dried plant grinding followed by sieve fractionation or CDSp has the advantage of enhancing the extraction yield of bioactive ingredients and alleviating the deficiencies of conventional extraction techniques. Inversely, most conventional extraction techniques use mild/high temperatures that can cause thermal degradation and are dependent on the mass transfer rate, being reflected in long extraction times, high costs, and low extraction efficiency, with consequent low extraction yields.

While many have been done to test the efficiency of CDSp to concentrate bioactive ingredients, many still need to be done to preserve its activities. Regardless of the employed extraction methods, the effectiveness of plant bioactive ingredients post-extraction depends on preserving their stability, bioactivity and bioavailability when consumed. Indeed, after isolation, the activity progressively diminished or even completely lost, as the bioactive compounds are highly sensitive to environmental exposure including moisture, light, oxygen and high temperature (Fang & Bhandari 2011). In this sense, encapsulation of plant materials rich in bioactive compounds has been an approach to overcome the issues, as increased research and excellent reviews on encapsulation of plant phenolic compounds and vitamins are found in the literature (Edris et al. 2016; Ribeiro et al. 2020; Reza & Razie 2022). Among various kinds of encapsulation techniques, spray-drying is regarded as an economical and effective process in the food industry and, practically, it is the most commonly used technique for microencapsulation of natural components and heat-sensitive food ingredients (Edris et al. 2016). Spray-drying has been successfully used for the encapsulation of a number of polyphenol-rich plant materials, including *H. sabdariffa* L. extract and anthocyanins (Chiou & Langrish 2007; Sílvia et al. 2018). With spray-drying, a micro/nano environment is created, incorporating unstable ingredients within a carrier matrix and providing protection against the external environment, degradation reactions and preventing the loss of flavour, nutrients, and/or bioactive compounds such as polyphenols, flavonoids, anthocyanins (Fang et al. 2010). Among the numerous biopolymer carriers, maltodextrin (MD) and gum Arabic (GA) are the most popular and common wall materials for encapsulating bioactive compounds. However, the application of GA as a wall material has been curtailed by its high price and impurities (Jafari et al. 2008). In addition, MD has well-defined physical properties that facilitate its use as an additive in the industry (Fazaeli et al. 2012). Nevertheless, the choice of suitable biopolymers as wall material is critical to the success of microencapsulation by spray-drying because the type of wall material determines the physicochemical, morphological, flow and functional properties of the produced microcapsules (Ribeiro et al. 2020; Toyosi et al. 2021). Although the activity of food ingredients is stabilized by encapsulation, it affects the physical properties of powder microcapsules which need to be investigated. In fact, for the use and inclusion of microcapsules of fruits *D. glomerata* and *H. sabdariffa* powders in food matrices, the understanding of their physicochemical and bioactivity properties is essential. To the best of our knowledge, this is the first study on the encapsulation of plant powder fractions in maltodextrin using spray-drying.

Therefore, the underlying objective of the current work was to compare the physicochemical, flow characteristics and antioxidant properties of spray-dried encapsulated and non-encapsulated powder fractions of *D. glomerata* fruits and *H. sabdariffa* calyx.

2. MATERIALS AND METHODS

2.1. Reagents, plant materials and sample preparation

All the chemicals and solvents used in this study were of standard analytical purity and purchased from Sigma-Aldrich (Saint Louis, USA). Dried fruits of *Dichrostachys glomerata* and dried calyxes of *Hibiscus sabdariffa* were

purchased in May 2018 from local markets, respectively in the Center and Adamawa regions of Cameroon. Samples were hand-cleaned of foreign bodies (inorganic materials, dirt, and dust particles) before being used for powder production. Then, each plant material was milled using an electric Ultra-Centrifugal Mill ZM 200 (Haan, Germany) operating at 12000 rpm with a mesh sieve of 1 mm. Dried plant grinding and CDSp procedures followed to obtain the powder fractions were the same as reported in our previous studies (Deli et al. 2019ab; Deli et al. 2020ab). CDSp is based on the separation of particles from powdered plant materials by making them pass through suitably selected sieves of decreasing mesh size (Baudelaire 2013). Thereafter, *D. glomerata* powder fractions φ < 212 µm and *H. sabdariffa* powder fractions φ < 315 µm were retained and used for encapsulation in MD.

2.2. Encapsulation of plant powders in maltodextrin by spray drying method

2.2.1. Preparation of spray drying infeed solution

Hibiscus sabdariffa calyx and *D. glomerata* fruits powders were microencapsulated with MD (DE20) according to the used procedures by Choi et al. (2019). MD solution as wall material was mixed using a high-speed homogenizer (Silverson L4RT high shear mixer) while stirring at 3500 rpm for 15 min. Then, the powder sample was added directly to the encapsulating agent stock solution to give the desired ratio of each powder fraction to the MD on a wet weight basis 1:2 (w/w), to give the total solid content of the final solution of 30% (w/w). The mixture was homogenized continuously for at least 12 h at 4°C to ensure complete hydration of the polymer molecule and to promote the encapsulation process.

2.2.2. Spray-drying of powder-maltodextrin mixture

The previously prepared mixture was filtered through 250 µm size. The filtrated mixture was loaded into the spray dryer unit (SCHULZ INFROPROD, FRA1388/12-078, EU) and spray-dried by a nozzle of φ 1.2 mm. The spray dryer was adjusted under the following operating conditions: 100% suction rate (50 mL/min), drying air inlet temperature 150 ± 1°C, while the pump flow rate was adjusted to maintain an outlet temperature of 70 ± 1°C. The air inlet temperature fell below 40 ± 2°C. The suspension was continuously stirred as the spray drying process continued. The solvent, which is water, was evaporated and a powder formed. Obtained microencapsulated plant powder (microcapsule) was collected at the bottom of the drier and weighed. The yield or powder recovery (%) was calculated by dividing the dry mass of the produced powder by the dry mass of the spray-drying mixture (maltodextrin + plant powder) on a dry weight basis (Equation 1).

$$Yield\ (\%) = \frac{\text{Dry mass of produced powder}}{\text{Dry mass of spray dray drying mixture}} \times 100 \quad \text{(Equation 1)}$$

Each non-microencapsulated plant powder was taken as control, and all powder samples were stored at 10°C in vacuum-sealed plastic boxes. Images of microencapsulated and non-microencapsulated plant powders are presented in **Figure 1**.

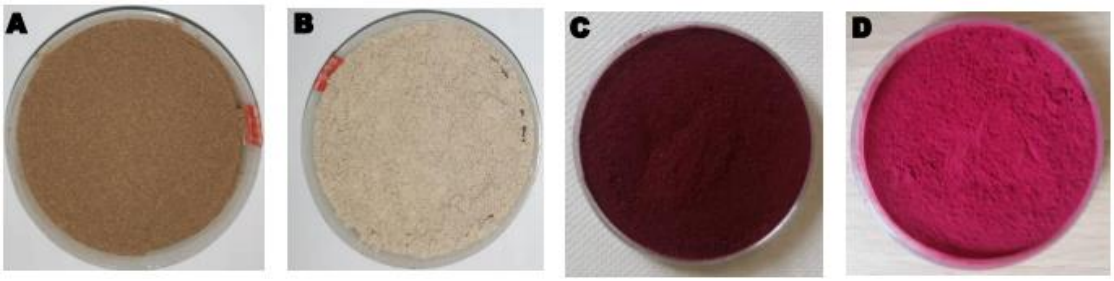

Figure 1. Images of microencapsulated *D. glomerata* (**B**) and *H. sabdariffa* (**D**) powders and non-microencapsulated *D. glomerata* (**A**) and *H. sabdariffa* (**C**) powders.

2.3. Determination of physicochemical properties

2.3.1. Particle size measurements

Particle size distribution of the powders was measured using a Mastersizer 3000 (Malvern Instruments, Orsay, France) which employed laser diffraction. Each powder was placed in the Aero S dry dispersion unit with a 2.5 mm hopper length and 30% feed rate to enable a laser obscuration level of circa 2%. The chosen size estimator was the particle size in volume and granulometric parameters (D_{10}, D_{50} and D_{90}) were determined. D_X means that x% of powder particles had diameters inferior to D_X. Mean particle size was obtained on the basis of volume median

diameter. The polydispersity, i.e. the width of the particle size distribution was determined by span values, that provide information on the degree of homogeneity of powder particles.

$$\text{Span}\ (-) = \frac{D_{90} - D_{50}}{D_{50}} \times 100 \ \text{(Equation 2)}$$

2.3.2. Moisture content and water activity

The moisture content of powder samples was determined by the weight difference of the powders after vacuum oven drying at 105 ± 3°C for 24 h (AOAC, 2005), using equation 3. Water activity (A_W) was measured at 20.0 ± 2°C using an A_W-meter instrument (Rotonic Hygropalm). Briefly, 15 g of powder were poured into a measurement vessel and the A_W-Quick method was employed, leading to water activity stabilization in about 4–5 min (Deli et al. 2019b).

$$\text{Moisture content}\ (\%) = \frac{\text{Loss in moisture (g)}}{\text{Initial weight ofsample (g)}} \times 100 \ \text{(Equation 3)}$$

Where, Loss in moisture = initial weight (g) - final weight (g)

Initial weight: wet or original weight of the sample before drying

Final weight: weight of sample after drying

2.3.3. Colour determination

In each case, powder colour was measured using a colourimeter (Datacolor International Microflash 2078S, Montreuil, France), following the guidelines as reported by Deli et al. (2019b). Concisely, the powder was placed separately in a Petri dish. The colourimeter was calibrated with a standard calibration plate having white and black areas. Subsequently, the measurements were performed at six locations of the powder layer and the colour coordinates L* (whiteness or brightness/darkness), a* (redness/greenness) and b* (yellowness/blueness) of the CIE colour system, were determined. From a* and b* values the chromaticity (C*), Hue angle (H*), and colour intensity were calculated as follows:

$$\text{Chroma}\ (C^*) = [a^{*2} + b^{*2}]^{1/2} \ \text{(Equation 4)}$$

$$\text{Hue angle}\ (H^*) = \tan^{-1}(b^*/a^*) \ \text{(Equation 5)}$$

$$\text{Total color} = [L^{*2} + a^{*2} + b^{*2}]^{1/2} \ \text{(Equation 6)}$$

2.3.4. Flow evaluation

By using the FT4 powder Rheometer (Freeman Technology, Malvern) device, stability and shear cell tests were performed to assess powder flowability in a low-stress environment, compressibility under normal stress and powder flowability in a high-stress environment, respectively.

2.3.4.1. Stability test

The powder was placed in a cylindrical vessel (25 mm × 25 mL split vessel) and underwent a conditioning step at 100 mm s^{-1} blade tip speed. Then, seven test cycles (conditioning + test) were performed to achieve stabilized flow energy. Basic flowability energy (BFE, mJ), corresponding to the stabilized flow energy, was evaluated as the energy needed to displace a conditioned powder sample during the downward movement of the blade in the seventh test cycle (Freeman 2007). Generally, BFE was used to evaluate the flow properties of powder under free-surface conditions. It was calculated from the work of the normal force and torque of the blade when moving downward through the powder bed. The conditioned bulk density (CBD) was also obtained in the stability test and calculated as the ratio of powder mass to vessel volume.

2.3.4.2. Shear cell test

The rotational shear cell accessory of the FT4 powder rheometer was used. This test consisted of a vessel (25 mm × 10 mL split vessel) containing the powder and a shear head to apply both normal stress and rotational shear to the powder bed. Firstly, the powder was conditioned using the helical blade and then slowly pre-compacted under a determined normal load (9 kPa) with a vented piston (Quintanilla et al. 2001). Then, the vessel containing the powder was split, the vented piston was changed for the shear cell head and the powder was recompressed to remove any disturbances caused by the split and ensure that the surface of the powder was properly consolidated. Then, the powder was pre-sheared at 9 kPa to achieve a critically consolidated state. The normal stress σ was

then lowered and the shear stress τ necessary to cause powder bed failure and initiate flow was measured. The pre-shear/shear sequence was repeated five times at decreasing normal stresses σ from 7 to 3 kPa by 1 kPa steps. The curve representing the evolution of τ as a function of σ is thus the so-called yield locus. Shear cell parameters, mainly unconfined yield stress (UYS), major principal stress (MPS), cohesion and flow factor, were then determined by the software by analysis of the Mohr circle. The cohesion is evaluated as the intercept of the linear regression of the shear stress vs. applied normal stress curve, whereas the flow factor coefficient (*ffc*) is defined as the ratio between MPS and UYS. Regarding *ffc*, the flow behaviour was classified as proposed by Jenike (1980): *ffc* < 1 not flowing, 1 < *ffc* < 2 very cohesive, 2 < *ffc* < 4 cohesive, 4 < *ffc* < 10 easy-flowing and 10 < *ffc* free-flowing. Compressibility was deduced from the density values of CBD and density after compaction (bulk density, BD) at 9 kPa, and is indicative of the cohesion between particles.

$$\text{Compressibility}\ (\%) = 1 - \frac{\text{CBD}}{\text{BD}} \times 100 \quad \text{(Equation 7)}$$

2.4. Extraction and determination of phenolic bioactive compounds

2.4.1. Extraction of phenolic bioactive compounds

Extraction was first achieved according to the used method by Deli et al. (2019b) in which maceration of powders in solvent was chosen as an extraction method to minimize possible sample degradation. Besides, the extraction at ambient temperature was also a compromise between extraction efficiency and limitation of thermal alteration of extracted biomolecules (Ćujíc et al. 2015). Practically, 2 g plant powder was mixed with 20 ml methanol/water (70/30 (v/v)). The mixture was subjected to maceration by stirring at 300 rpm for 24 hours at room temperature (18±2°C) and then filtered through with a Wattman filter paper (GE Healthcare companies, China) of 2-3 µm pore size. Thereafter, the supernatant was brought to 15 ml by the addition of extraction solvent and stored at 4 °C until analysis.

2.4.2. Determination of total phenolic content

The method of Wafa et al. (2014) was used with slight modifications. 20 µL of hydromethanolic extract was mixed with 2,980 µL distilled water. Then, 500 µL of 10% (v/v) Folin–Ciocalteu reagent and 400 µL of saturated solution of sodium carbonate (20%, w/v) were added. The mixture was mixed well and incubated at room temperature for 30 min in the dark. The absorbance was measured at 760 nm using a spectrophotometer (Shimadzu UV-VIS 1605, Tokyo, Japan). A calibration curve (R^2 = 0.98) was prepared using standard solutions of gallic acid at different concentrations (40 to 280 g/L). Results were expressed as milligram gallic acid equivalents per gram dry weight (mg GAE/g DW).

2.4.3. Determination of flavonoid content

The method of Dewanto et al. (2002) was used. 0.1 mL of filtered hydromethanolic extract was added to 2.4 mL of distilled water followed by the addition of 0.15 mL of 5% (w/v) sodium nitrite solution (Na_2NO_2). After 6 min, 0.3 mL of 10% aluminium chloride (w/v) was added. The mixture was kept at room temperature for 5 min, and 1 mL 4% (w/v) of 1 M sodium hydroxide was added. The absorbance of the solution was measured at 510 nm against the extraction solvent as blank. A calibration curve (R^2 = 0.99) was plotted from different concentrations of rutin as standards (20 to 140 g/L) and results were expressed in milligrams rutin-equivalent per gram (mg RE/g) of dry weight.

2.4.4. Determination of condensed tannins content

Condensed tannin content was evaluated by Sun et al. (2008) method. A volume of 0.05 µL of filtered hydromethanolic extract was mixed with 3 mL of 4% vanillin (w/v), and 1.5 mL concentrated sulfuric acid was added. The mixture was stirred and kept at room temperature for 30 min. The absorbance was measured at 500 nm by UV/visible spectrophotometry (Sun, Ricardo-Da-Silva, Spranger, 2008) against the hydromethanolic solvent (70/30) as blank. A calibration curve (R^2 = 0.99) was prepared using standard solutions of catechin (100 to 600 µg/mL). Condensed tannin content was expressed as milligrams catechin-equivalent per gram (mg CE/g) of dry weight.

2.4.5. Extraction and determination of anthocyanin content

Firstly, extraction was performed according to the Tony et al. (1994) method. To 1 g of powder, 10 mL of solvent mixtures (ethanol/ HCl, 85/15, v/v) was added. Then, the mixture was stirred at 3000 rpm (Variomag Poly Stirrer)

for 2 h, and centrifuged (Thermo Scientific, Heraeus Megafuge 8R Centrifuge, Osterode am Harz, Germany) at 3000 rpm for 10 min at 4°C. Thereafter, the supernatant was recovered.

The determination of anthocyanin was performed using the pH-differential method (Giusti and Wrolstad, 2001). One hundred (100) µL of extract were pipetted into the tubes containing respectively 4900 µL of 0.025 M potassium chloride buffer at pH 1.0 and 4900 µL of 0.4 M sodium acetate buffer at pH 4.5. The mixtures were stirred and incubated for 15 min at room temperature. Absorbance was measured both at 520 nm and 700 nm, respectively, using a UV–Vis spectrophotometer against a blank containing distilled water. Anthocyanin content was expressed as microgram of cyanidin 3-glucoside equivalent (CGE) per 100 g dry weight of powder as follows:

$$\text{Anthocyanins} = \frac{(\Delta\text{Abs} \times \text{MW} \times \text{F} \times 1000 \times 100)}{\varepsilon \times \lambda \times \text{m} \times \text{DW}} \qquad \text{(Equation 8)}$$

Where ΔAbs (variation of absorbance) = $(\text{Abs}_{520\text{ nm}} - \text{Abs}_{700\text{ nm}})_{\text{pH 1}} - (\text{Abs}_{520\text{ nm}} - \text{Abs}_{700\text{ nm}})_{\text{pH 4.5}}$; MW: molecular weight for cyanidin 3-glucoside (449.2 g/mol), F: dilution factor (-), ε: molar extinction coefficient (29,600 L/mol/cm), λ: path length (1 cm) and m: mass of test sample (g).

2.5. Determination of antioxidant activity

2.5.1. 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging

The method of Athamena et al. (2010) was used. 0.5 mL hydromethanolic extract of plant sample or ascorbic acid (reference) at different concentrations (0.025, 0.05, 0.1, 0.5, 1, 5, 10 and 100 mg/mL) were allowed to react with 2 mL of 0.1 mM DPPH methanolic solution in the dark for 1 h at room temperature and absorbance was taken at 517 nm. DPPH radical scavenging was calculated as follows:

$$\text{Radical scavenging activity (\%)} = \frac{(\text{Control Abs} - \text{Sample Abs}}{\text{Control Abs}}\ x\ 100 \qquad \text{(Equation 9)}$$

The IC_{50} value was estimated from the plot of % free radical scavenged versus log concentrations of plant extracts used to construct the curve from which the value of IC_{50} (µg/mL) was evaluated. The ascorbic acid standard showed the IC_{50} value of 12.69 ± 1.96 µg/mL. The lower IC_{50} means the higher radical scavenging capacity.

2.5.2. Ferric reducing antioxidant power (FRAP)

FRAP was measured according to Oyaizu (1986) method. 1 mL of the extract of each powder was mixed with 2.5 mL of phosphate buffer (200 mM, pH 6.6) and 2.5 mL of potassium ferricyanide solution (1%, w/v). The mixture was incubated in a water bath at 50°C for 30 min, cooled, mixed with 2.5 mL of trichloroacetic acid solution (10%, w/v) and centrifuged at 3000 rpm for 10 min. 2.5 mL of supernatant was removed and mixed with 2.5 mL of distilled water and 0.5 mL of iron chloride (1%, w/v), allowed to react for 10 min at room temperature. The absorbance was measured at 700 nm using a blank containing the constituents mentioned above except the extract which was replaced by distilled water. The reducing power was determined by referring to the calibration curve of ascorbic acid solution and expressed in terms of mg ascorbic acid equivalent per g (mg AAE/g) of dry weight.

2.6. Statistical analysis

The assays were carried out in triplicate and the results were expressed as means ± standard deviation. Results of phenolic contents, physical, flow and antioxidant properties were each subjected to analysis of variance (ANOVA) to determine the occurrence of statistically significant differences among them ($P<0.05$). Duncan's multiple range test was used to determine the degree of significance of the difference between two means. Correlations and Principal Components Analysis (PCA) were equally performed. Stat Graphics Centurion software version 16.1 was used for this purpose. Sigma plot 11.0 was used to plot graphs.

3. RESULTS AND DISCUSSION

3.1. Product yield

The product yield is defined as the ratio of the mass of powder dry matter collected after spray-drying to the weight of total solids in the feed. Results denoted that the plant nature, as well as plant powder particle sizes, have significantly ($P<0.05$) affected the product yields. *D. glomerata* microcapsule showed the highest product yield (42.32%), while *H. sabdariffa* microcapsule led to the lowest product recovery (38.47%). Indeed, *D. glomerata* and *H. sabdariffa* powders used for the encapsulation were different in particle sizes, < 212 µm and < 315 µm, respectively. The difference in the yield of microencapsulated powders may be due to the difference in the particle size distribution of both plant powders. Studies have reported a close relationship between the size of

the particles and losses in the exhaust air, with smaller particle sizes being hardly recovered with the cyclone (Gaiani 2006; Cuq et al. 2013). It is admitted that successful spray-drying must have a product yield higher than 50% (Bhandari et al. 1997). However, the yields in this study were lower than 50% for both plant powders. Many reported studies on microencapsulation of plant products have focused on solvent extracts. However, the present study focuses on the encapsulation of plant fraction powders composed of coarse particle size. This would justify the low obtained yield. In addition, the low yield may result from losses during spray-drying due to particles adhering to the spray-dryer walls, or the retention of very small particles by the cyclone system. Indeed, the processing conditions of the spray-drying process can produce stick products, especially when the composition of the feed is rich in sugars and acids, such as *H. sabdariffa* powder. This could generate an adherence of particles to the internal wall of the drying chamber, creating agglomeration and lower product yield (Bhandari & Howes 2005).

3.2. Physical properties

3.2.1. Particle diameter

The physical characteristics of *H. sabdariffa* and *D. glomerata* powders are presented in **Table 1**. The median particle diameter (D_{50}) of non-microencapsulated *H. sabdariffa* and *D. glomerata* powders were 164.4 and 106.3 µm, respectively, while that of their respective microencapsulated powders were 16 and 17 µm. During the CDSp, *D. glomerata* and *H. sabdariffa* powders were sieved to pass mesh sizes 212 and 315 µm, respectively. The median particle size of the non-microencapsulated, as well as the microencapsulated powders, were in the same trend as the sieve size used for CDSp. Importantly, encapsulation with MD had led to particle size reduction, irrespective of the powder origin. In addition, the polydispersibility (span) of the powders was inversely proportional to D_{50}. The span values of the microencapsulated *H. sabdariffa* and *D. glomerata* powders were 14.20 and 14.14, while the equivalent values for non-microencapsulated powders were 1.30 and 2.60. Lower span values mean narrow distributions of the powders with values higher than 3 indicating broader size distribution. Based on our results, it appeared that encapsulation with MD reduced the size of the particles and increased the broadness of the powder particle size. Choi et al. (2019) reported similar trends of the influence of the encapsulation for *Angelica gigas* powder with MD on particle size characteristics. While the reduction of the size with encapsulation appears not understandable, this may be a result of an indirect action of the encapsulation through limitation of aggregation. In fact, in solution the powders may aggregate, thus leading to apparently large mean particle and small polydispersibility values. On the opposite, in the microencapsulated plant powders, the hydrophobic portions of the MD surrounding the surface of the particles seemed to provide good electrostatic and/or stability between particles, and also interactions between particles and water molecules (Choi et al. 2019).

3.2.2. Colour

Table 1 also shows that colour parameters were significantly ($P<0.05$) affected by plant powder nature and encapsulation with MD. *H. sabdariffa* powder appearing dark and red-purple, the total colour was 63.28, while chroma and hue angle were 20.07 and 14.36, respectively. *H. sabdariffa* microcapsule denoted an increase in chroma and hue angle values, indicating a decrease in total colour or red-purple colour. Similarly, the native dark colour of *D. glomerata* powder was highly affected by encapsulation with MD, the chroma (19.31 to 20.88) and hue angle values (75.63 to 87.97) increased, while the total colour (35.67 to 24.64) decreased. The change in the colour of powder following encapsulation in MD could be related to the distinctive white colour of the carrier material, maltodextrin. This revealed, in concordance with others, that carrier material has a great impact on the colour of the microencapsulated product (Tontul et al. 2017). This is possible through the dilution effect of the carrier on the coloured molecules such as anthocyanins, the powder being encapsulated representing in all cases a total proportion less than 50% (Moura et al. 2018). In this respect, the content of antioxidant molecules is expected to be reduced by more than 50% in microencapsulated *H. sabdariffa* and *D. glomerata* powders. In addition, pigments could be lost due to oxidation at a higher inlet temperature (Sousa et al. 2008).

3.2.3. Moisture content

The moisture content of non-microencapsulated and microencapsulated *H. sabdariffa* and *D. glomerata* powders is shown in **Table 1**. The moisture content ranged from 5.90 - 9.08% and was higher in non-microencapsulated *H. sabdariffa* (8.47) and *D. glomerata* (9.08) powders than in their respective microcapsules (5.90 and 6.24, respectively). In addition, the moisture content was significantly higher in *D glomerata* than in *H sabdariffa*. It is often observed that the moisture of produced microcapsules is lower than 5%, a value which makes them microbiologically stable (Fang 2010; Schuck et al. 2012; Tontul et al. 2017). Indeed, higher moisture content (generally over 10%) induces the development of microorganisms and product deterioration (Kaur et al. 2011).

Also, the moisture content varied with the plant powder nature and encapsulation process. Generally, when higher inlet air temperature is used, it appears a higher temperature gradient between the atomized feed and the drying air, causing rapid water vaporization with a greater rate of heat transfer, and ultimately generating powders with lower moisture content (Kha et al. 2010). In all cases, the moisture content of the flour was low. The lower moisture means better preservation and stability, and limited ability of the water to act as a plasticizer (Tontul et al. 2017; Santana et al. 2017). The moisture content is critical to microcapsule properties such as stickiness, flowability, water activity, drying efficiency, microbial growth and oxidation of their bioactive components and storage stability (Velasco et al. 2003).

3.2.3. Water activity

Encapsulation significantly influenced ($P<0.05$) the water activity (Aw) of *H. sabdariffa* and *D. glomerata* powders, as the microcapsules have a slightly lower Aw (0.29 and 0.26, respectively) than non-microencapsulated powders (0.38 and 0.37, respectively). Aw is an important index for powders due to its effect on the shelf-life of the product. Increasing Aw indicates an increasing amount of free water available for deteriorative reactions, thus shortening the shelf-life (Vardin & Yasar, 2012). Powders with Aw lower than 0.30 are regarded as microbiologically and chemically safe. It indicates that the higher temperature (atmospheric condition) during the atomization has contributed to producing a dry powder with lower water activity. A good positive correlation existed between the Aw and moisture content of the *H. sabdariffa* and *D. glomerata* powders, revealing that the reduction of Aw with the encapsulation process is a consequence of the reduction of the moisture content due to spray drying. However, this association does not occur at all times (Bicudo et al. 2015).

3.3. Flow characteristics

Flowability is an important parameter used in predicting and deciding the condition of processing, formulation, packaging, and transportation of products in the food industry (Tolun et al. 2016). It was found that plant nature and encapsulation significantly ($P<0.05$) influenced the flow characteristics of powders (**Table 2**). Basic flowability energy (BFE) was significantly ($P<0.05$) higher for *H. sabdariffa* than for *D. glomerata* as far as non-microencapsulated powders are concerned, but a reverse trend was observed for microencapsulated powders. This revealed the high interaction that exists between the plant materials and the encapsulation probably due to the difference in composition between powders, hence the reaction with MD. However, *H. sabdariffa* and *D. glomerata* microcapsules exhibited significantly high basic flowability energy (150.45 and 126.86 mJ, respectively) compared to non-microencapsulated powders which had low values of BFE (83.34 and 94.5 mJ, respectively). This may arise from the deleterious influence of shape irregularity and surface roughness of the non-encapsulated powders. A previous study reported that spray drying generates rounded, smooth and spherical shapes (Ribeiro et al. 2020). These authors also reported that encapsulation of cacao pulp with MD produced powders with smoother surfaces. Yet, BFE is dependent on the size, shape, and surface properties of particles and powder particles with smooth and uniform surfaces generating lower BFE (Bharadwaj et al. 2010). However, irregular particles may require more flow energy than spherical particles, due to the influence of particle orientation which involves interlocking of the powder particles (Nan et al. 2017). In irregular particles, interparticle interactions by Van der Walls forces and mechanical linkage may increase with the impairment of powder flowability (Fatah 2009). Inversely, some studies reported an increase in BFE and powder flowability following encapsulation with MD (Ribeiro et al. 2020). In this case, encapsulation with MD may enhance the formation of bridges between particles and agglomeration due to increased hygroscopicity.

Compared to BFE, conditioned bulk density (CBD) was significantly high for *H. sabdariffa*, irrespective of the encapsulation while encapsulation systematically decreased the CBD from 0.61 – 0.54 g/mL to 0.52 – 0.47 g/mL. The effect of the plant origin and encapsulation reflected the difference in particle size as shown above, with encapsulated powders having lower particle size. Relatively high CBD of obtained microcapsules translates to ease the storage with less space requirement. According to Edris et al. (2016) and Chuyen et al. (2019), high density indicates low amounts of air in the microcapsules, thereby decreasing the chances of air-related degradation such as oxidation. In fact, low particles are prompted to improve compaction, and, in this respect, a negative significant correlation was observed between CBD and compressibility ($r= -0,87$; $P<0.01$); similarly, improved compression enhanced the cohesion, as confirmed by the significant correlation between compression and cohesion ($r=0.80$; $P=0.02$). Compressibility was rather high for *H. sabdariffa* and *D. glomerata* microcapsules (71.63% and 73.43%, respectively) in comparison with their non-microencapsulated powders with 66.42% and 70.42%, respectively. A similar trend was observed for cohesion, being higher for *H. sabdariffa* or *D. glomerata* microcapsules (1.09 and 0.86, respectively) in comparison with their non-microencapsulated samples (0.27 and 0.43).

Table 1. Moisture, water activity, particle size and colour characteristics of microencapsulated and non-microencapsulated *H. sabdarifa* and *D. glomerata* powders.

Plants	Treatments	Moisture	A_W	D_{50} (µm)	Span (-)	Chroma	Hue (°)	Total color (-)
H. sabdariffa	Microencapsulated	5.90 ± 0.06^{a}	0.29 ± 0.01^{b}	16.2 ± 0.1^{a}	14.2 ± 0.1^{c}	40.3 ± 0.2^{c}	14.4 ± 0.12^{b}	47.6 ± 0.1^{c}
	Non-microencapsulated	8.47 ± 0.43^{b}	0.38 ± 0.01^{d}	164.4 ± 0.3^{d}	1.3 ± 0.1^{a}	20.1 ± 0.6^{b}	10.4 ± 0.02^{a}	63.1 ± 1.5^{d}
D. glomerata	Microencapsulated	6.24 ± 0.82^{a}	0.26 ± 0.01^{a}	17.5 ± 0.4^{b}	14.1 ± 0.1^{c}	20.9 ± 0.4^{b}	88.0 ± 0.3^{d}	24.6 ± 1.0^{a}
	Non-microencapsulated	9.08 ± 0.03^{c}	0.37 ± 0.01^{c}	106.3 ± 0.4^{c}	2.6 ± 0.2^{b}	19.3 ± 0.5^{a}	75.6 ± 0.6^{c}	35.7 ± 1.6^{b}

Data are presented as mean ± standard deviation, n = 3; the different letters within the same column indicate significant differences at $P < 0.05$ using Duncan's test

Table 2. Flow characteristics of *H. sabdariffa* and *D. glomerata* powders as affected by encapsulation

Plants	Powder samples	Basic flowability energy (mJ)	Conditioned bulk density (g/mL)	Compressibility (%)	Cohesion (kPa)	Flow factor (-)	Flow specification
H. sabdariffa	Microencapsulated	83.34 ± 4.13^{a}	0.52 ± 0.02^{b}	71.63 ± 1.04^{bc}	1.09 ± 0.05^{d}	2.13± 0.27^{a}	cohesive
	Non-microencapsulated	150.45 ± 3.20^{d}	0.61 ± 0.01^{d}	66.42 ± 0.10^{a}	0.27 ± 0.02^{a}	6.08 ± 0.71^{c}	Easy flow
D. glomerata	Microencapsulated	94.55 ± 2.72^{b}	0.47 ± 0.01^{a}	73.43 ± 0.89^{c}	0.86 ± 0.08^{c}	1.96 ± 0.08^{a}	cohesive
	Non-microencapsulated	126.86 ± 1.33^{c}	0.54 ± 0.01^{c}	70.42 ± 0.28^{b}	0.43 ± 0.05^{b}	5.17 ± 0.85^{bc}	Easy flow

Data are presented as mean ± standard deviation, the different letters within the same column indicate significant differences at $p < 0.05$ using Duncan's test.

Table 3. Total phenolic (TPC), flavonoid (FLC), condensed tannin (CTC) and anthocyanin (ATC) contents of non-microencapsulated and microencapsulated *H. sabdariffa* and *D. glomerata* powders

Plants	Powder samples	Polyphenols (mg GAE/g DW)	Flavonoids (mg RE/g DW)	Tanins (mg CE/g DW)	Anthocyans (mg GCE/g DW)
H. sabdariffa	Microencapsulated	55.99 ± 3.12^{a}	42.24 ± 2.04^{a}	22.54 ± 2.49^{a}	98.93 ± 4.12^{a}
	Non-microencapsulated	98.20 ± 6.95^{b}	107.62 ± 2.83^{b}	95.55 ± 10.33^{b}	453.46 ± 5.18^{b}
D. glomerata	Microencapsulated	106.61 ± 4.80^{a}	35.55 ± 4.16^{a}	65.85 ± 6.44^{a}	ND
	Non-microencapsulated	230.11 ± 6.13^{b}	81.55 ± 5.20^{b}	150.82 ± 10.83^{b}	ND

Data are presented as mean ± standard deviation, the different letters within the same column indicate significant differences at $p < 0.05$ using Duncan's test. ND: not determined.

Flow function coefficient had a similar trend to BFE (r=0.95; P<0.001) and CBD (r=0.89; P<0.01), based on their positive higher significant linear correlation. In addition, we found a negative linear correlation between the flow function coefficient and the compressibility (r= -0.79; p=0.02) in conformity with reported data (Fayed and Skocier, 1997). Non-microencapsulated *H. sabdariffa* and *D. glomerata* powders presented rather high values over 4 (6.08 and 5.17, respectively), making them classified as easy flow powder, while microcapsules with *ffc* values below 4 (2.13 and 1.96, respectively), were classified as cohesive powders. The cohesive property of encapsulated powders could not only be due to the particle size, but also to the presence of maltodextrin which may act by increasing hygroscopicity and frictional forces among the microcapsules (Dadi et al. 2020). The influence of powder particle size on flowability may result from an increased specific surface in lower particle size, hence increasing the number of contact points between particles (Nan et al. 2017). The effect of particle size on the flowability has been evidenced in corn and cocoa powders, wheat and soy flours (Fitzpatrick et al. 2005).

3.4. Total phenolic, flavonoid, condensed tannin and anthocyanin contents

Total phenolic (TPC), flavonoid (FLC), condensed tannin (CTC) and anthocyanin (ATC) contents of non-microencapsulated *H. sabdariffa* and *D. glomerata* powders were 98.20 – 230.11 mg GAE/g, 107.62 – 81.55 mg RE/g, 95.55 – 150.82 mg CE/g and 453.46 – 0 mg GCE/g DM, respectively (**Table 3**). It denoted that *D. glomerata* powder samples showed higher (P <0.05) TPC, FLC and CTC than *H. sabdariffa* powders. ATC was not detected in *D. glomerata* powders. Anthocyanins are a group of phenolic compounds found in a wide variety of flowers and fruits presenting a reddish-purple colour. On the other hand, it can be noted that TPC, FLC, CTC and ATC contents (P<0.05) were considerably higher in non-encapsulated powders. The contents in TPC, FLC, CTC and ATC of the microcapsules for the two plants were 2 – 3 times less than that of non-microencapsulated *H. sabdariffa* and *D. glomerata* powders. The reduction of TPC, FLC, CTC and ATC contents of the microcapsules more likely resulted from the dilution effect of the plant powder with maltodextrin (Moura et al. 2018).

3.5. Antioxidant activity

Figures 2 A and **B** present the radical scavenging properties and reducing power of *D. glomerata* and *H. sabdariffa* powders. The highest DPPH radical scavenging activity and reducing power were observed for *D. glomerata* powder samples. In addition, the antioxidant values were significantly low for microcapsules. The IC_{50} of *D. glomerata* and *H. sabdariffa* powders ranged from 45.2 – 157.8 µg/mL and 266.1–929.8 µg/mL, respectively. *D. glomerata* powders also exhibited higher ferric reducing power than *H. sabdariffa* powders. FRAP of non-encapsulated and microencapsulated *D. glomerata* and *H. sabdariffa* powders ranged from 278.3 – 148.3 mg AAE/g and 233.4 – 67.3 mg AAE/g of sample, respectively. The effect of encapsulation on the antioxidant activities (DPPH and FRAP) could be relatively ascribed to their difference in phenolic compounds. Indeed, from the above results, it was denoted that the phenolic compounds were lesser in the encapsulated plant powders. The role of phenolics on antioxidation is mainly due to their redox properties, which allow them to act as reducing agents, hydrogen donors, singlet oxygen quenchers and metal chelators (Deli et al. 2019a).

3.6. Correlation between properties of encapsulated and non-encapsulated powders

Figure 3 is the correlation circle of the different variables, and the individual powders used in the study according to the PCA. The principal component axes F1 and F2 expressed 96.69% variation among the four powders. F1×F2 plot denoted a separation of powders according to plant type and encapsulation effect. The First axe (F1, 76.73%) separated microencapsulated and non-microencapsulated powders with encapsulated individuals positioned on the right side of F1, and non-microencapsulated powders on the left side of the F1. Variables were equally separated along the F1 axe with compressibility, IC_{50} DPPH, cohesion, and span oriented at the right while the others are oriented left. In this respect, the second axe (F2, 19.85%) separated the according to the plant origin. In this respect, *D. glomerata* was positioned on the upper side of the F1 axe while *H. sabdariffa* is found on the downside of the F1 axe. Variables were equally separated along the F1 axe. Consequently, non-microencapsulated *D. glomerata* and *H. sabdariffa* powders were characterized by higher TPC, FLC, CTC, and ATC contents and higher antioxidant activities (DPPH and FRAP) in comparison to microencapsulated plant powders. In addition, non-microencapsulated *D. glomerata* and *H. sabdariffa* powders were characterized by high Aw, D_{50}, *ffc*, CBD, and BFE. This denoted that non-microencapsulated powders had better flowability than obtained microcapsules. Indeed, *glomerata* and *H. sabdariffa* microcapsules were correlated to high compressibility, span, cohesion and DPPH values. Thus, *D. glomerata* and *H. sabdariffa* microcapsules were less rich in phenolics and exhibited lower antioxidant activities and flowability.

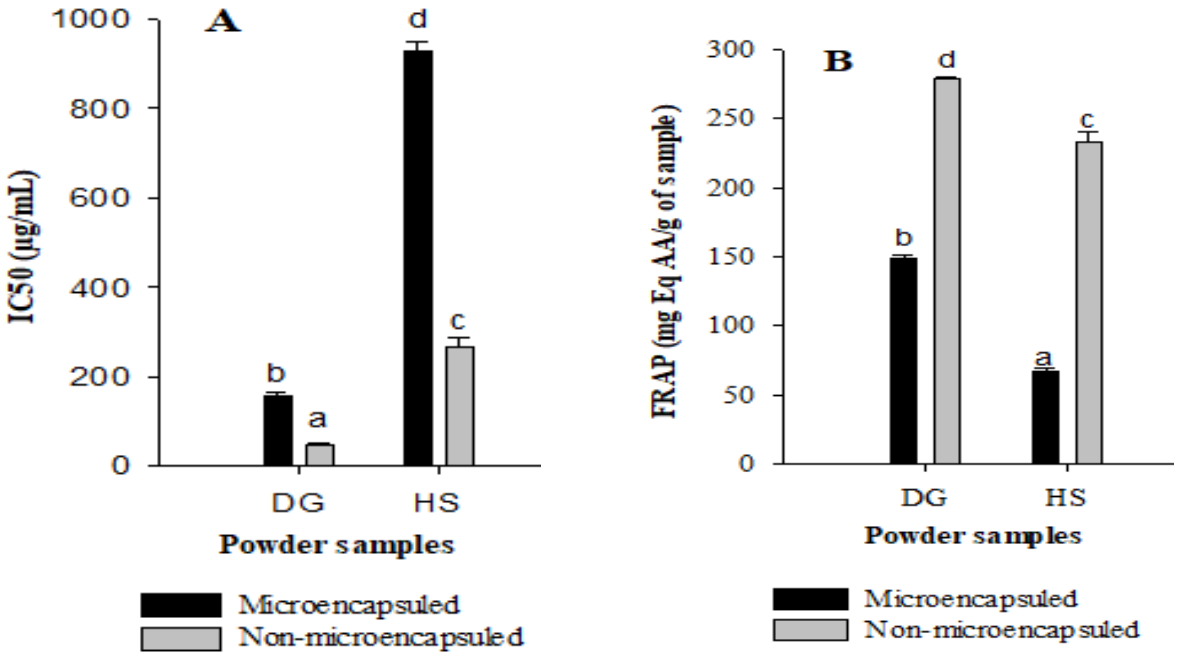

Figure 2. The DPPH radical scavenging activity (A) and ferric reducing power (B) of hydromethanolic extracts of the non-microencapsulated and microencapsulated *D. glomerata* and *H. sabdariffa* powders. Bars topped with different letters indicate statistically different results ($p < 0.05$).

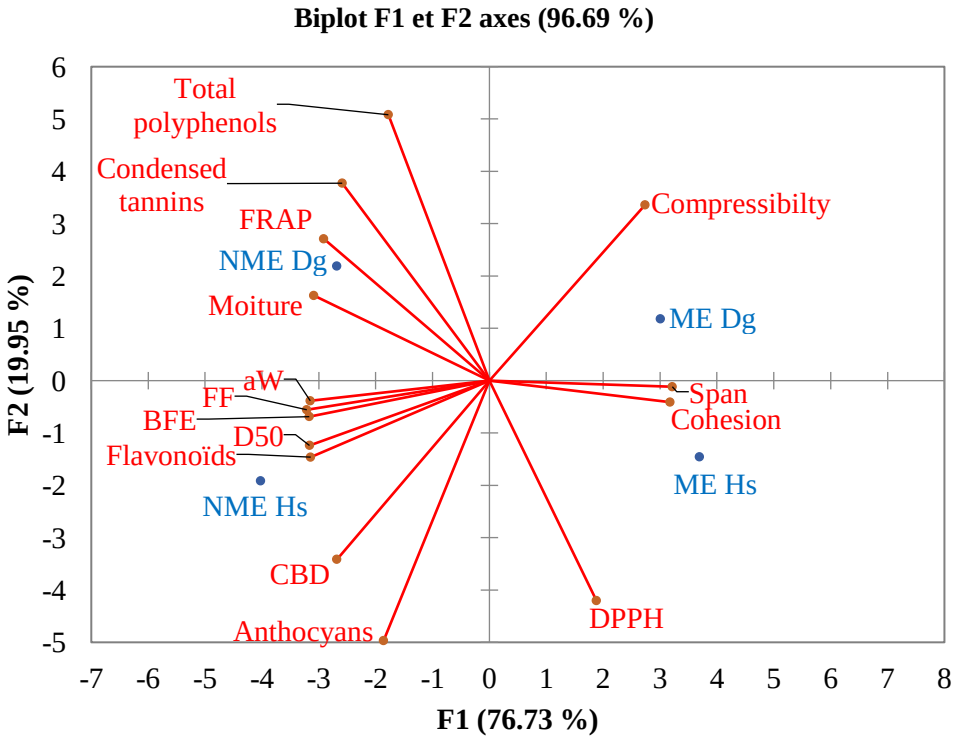

Figure 3. Principal components analysis of physicochemical, flow, and antioxidant properties of investigated non- and microencapsulated plant powders. ME: Microencapsulated, NME: Non-microencapsulated, Dg: *Dichrostachys glomerata*, Hs: *Hibiscus sabdariffa*

4. CONCLUSION

This study permitted to evaluate the influence of encapsulation on the physicochemical, flow and antioxidant properties of *D. glomerata* and *H. sabdariffa* powder fractions. The yields of encapsulation of powders in maltodextrin on spray-dryer are generally lower than 50%. Encapsulation yields microcapsules of powders characterized by low flowability, high cohesion, 2-3 times low phenolics and antioxidant properties.

Microencapsulation of plant fraction powders may be an option to preserve the best quality, and on the storage effects towards the quality properties to enhance the shelf life of the product.

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STATEMENT OF HUMAN AND ANIMAL RIGHTS. This article does not contain any studies with human or animal subjects performed by any of the authors.

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REFERENCES

AOAC (Association of Official Analytical Chemists). In G. Horwitz, & Latimer (Eds.). Official methods of analysis of AOAC international (18th ed.). Gaithersburg, MD: AOAC International. (2005)

Athamena S, Chalghem I, Kassah-laouar A, Laroui S and Khebri S (2010). Activite anti-oxydante et antimicrobienne d'extraits de *Cuminum cyminum* L. Lebanese Science Journal. 11, 72–73

Becker L, Zaiter A, Petit J, Karam M-C, Sudol M, Baudelaire E and Dicko A (2017). How do grinding and sieving impact on physicochemical properties, polyphenol content, and antioxidant activity of *Hieracium pilosella* L. powders? Journal Functional Foods 35, 666–672.

Bhandari B and Howes T (2005). Relating the stickiness properties of foods undergoing drying and dried products to their surface energies. Drying Technology 23, 671–684.

Bhandari B R, Datta N and Howes T (1997). Problems associated with spray drying of sugar-rich foods. Drying Technology 15, 671–684.

Bharadwaj R, Ketterhagen W R and Hancock B C (2010). Discrete element simulation study of a Freeman powder rheometer. Chemical Engineering Sciences 65, 5747–5756.

Bicuda M O P, Jo J, Alves de Oliveira G, Chaimsohn F P, Sierakowski M R, Freitas R A and Ribani R H (2015). Microencapsulation of Juçara (*Euterpe edulis* M.) pulp by spray drying using different carriers and drying temperatures. Drying Technology. An International Journal 33, 153–161.

Chiou D and Langrish T A G (2007). Development and characterisation of novel nutraceuticals with spray drying technology. Journal of Food Engineering. 82: 84–91.

Choi K O, Kim D, Lim J D, Koa S, Hong G-P and Lee S (2019). Functional enhancement of ultrafine *Angelica gigas* powder by spray-drying microencapsulation. LWT - Food Sciences and Technology 101, 161–166.

Chuyen H V, Roach P D, Golding J, Parks S E and Nguyen M H (2019). Encapsulation of carotenoid-rich oil from Gac peel: Optimisation of the encapsulating process using a spray drier and the storage stability of encapsulated powder. Powder Technology 344, 373–379.

Ćujić N, Šavikin K, Janković T, Pljevljakušić D, Zdunić Gand Ibrić S (2015). Optimization of polyphenols extraction from dried chokeberry using maceration as traditional technique. Food Chemistry, *194*, 135–142.

Cuq B, Gaiani C, Turchiuli C, Galet L, Scher J, Jeantet R, Mandato S, Petit J, Murrieta-Pazos I, Barkouti A, Schuck P, Rondet E, Delalonde M, Dumoulin E, Delaplace G and Ruiz T (2013). Advances in food powder agglomeration engineering, in: Jeyakumar Henry, Advances in Food and Nutrition Research, Burlington: Academic Press, pp. 41-103

Dadi D W, Emire S A, Hagos A D and Eun J-B (2020). Physical and Functional Properties, Digestibility, and Storage Stability of Spray and Freeze-Dried Microencapsulated Bioactive Products from *Moringa stenopetala* Leaves Extract. Industrial Crops Products 156(5), 112891.

Deli M, Baudelaire E D, Nguimbou R M, Njintang Y N and Scher J (2020a). Micronutrients and *in vivo* antioxidant properties of powder fractions and ethanolic extract of *Dichrostachys glomerata* Forssk. fruits. Food Sciences and Nutrition 8, 3287–3297.

Deli M, Nguimbou R M, Baudelaire E J, Njintang Y N, Scher J and Mbofung C M (2020). Effect of controlled differential sieving processing on micronutrient contents and in vivo antioxidant activities of *Hibiscus sabdariffa* L. calyxes powder. Food Sciences and Biotechnology 29, 1741–1753.

Deli M, Petit J, Nguimbou R M, Djantou E B, Njintang Y N and Scher J (2019a). Effect of sieved fractionation on the physical, flow and hydration properties of *Boscia senegalensis* Lam., *Dichostachys glomerata* Forssk. and *Hibiscus sabdariffa* L. powders. Food Sciences and Biotechnology 28, 1375–1389.

Deli M., Ndjantou E B, Ngatchic M J T, Petit J, Njintang YN and Scher J (2019b). Successive grinding and sieving as a new tool to fractionate polyphenols and antioxidants of plants powders: Application to *Boscia senegalensis* seeds, *Dichrostachys glomerata* fruits, and *Hibiscus sabdariffa* calyx powders. Food Sciences and Nutrition 7, 1795–1806.

Dewanto V, Wu X, Adom K K and Liu R H (2002). Thermal processing enhances the nutritional value of tomatoes by increasing total antioxidant activity. Journal of Agricultural and Food Chemistry 50, 3010–3014.

Edris A E, Kalemba D, Adamiec J and Piątkowski M (2016). Microencapsulation of *Nigella sativa* oleoresin by spray drying for food and nutraceutical applications. Food Chemistry 204, 326–333.

Fang Z and Bhandari B (2010). Encapsulation of polyphenols e a review. Trends Food Sciences and Technology 21, 510–523.

Fang Z and Bhandari B (2011). Effect of spray drying and storage on the stability of bayberry polyphenols, Food Chemistry 129,1139–1147.

Fatah N (2009). Study and comparison of micronic and namometric powders: analysis of physical, flow and interparticle properties of powders. Powder Technology190, 41–47.

Fayed M E and Skocir T S (1997). Mechanical conveyors: Selection and Operation. Technomic Publishing, Lancaster, PA.

Fazaeli M Z, Emam-Djomeh A K, Ashtari and Omid M (2012). Effect of process conditions and carrier material concentration for improving drying yield and other quality attributes of spay dried black mulberry (Morus nigra) juice. International Journal of Food Engineering 8, 1–20.

Fitzpatrick J, Barry K, Delaney C and Keogh K (2005). Assessment of the flowability of spray dried milk powders for chocolate manufacture. Le Lait 85, 269–277.

Freeman R (2007). Measuring the flow properties of consolidated, conditioned and aerated powders A comparative study using a powder rheometer and a rotational shear cell. Powder Technology 174, 25–33.

Gaiani C. Etude des mécanismes de réhydratation des poudres laitières: influence de la structure et de la composition des poudres, PhD Thesis, Ecole Nationale Supérieure d'Agronomie et des Industries Alimentaires, France (2006)

Giusti M M and Wrolstad R E (2001). Characterization and measurement of anthocyanins by UV–visible spectroscopy, pp. F1.2.1-F1.2.13. In: Current Protocols in Food Analytical Chemistry. Wrolstad RE (ed). Wiley, New York, USA.

Jafari S M, Assadpoor E, He Y and Bhandari B (2008). Encapsulation efficiency of food flavours and oils during spray drying. Drying Technology 26, 816–835.

Jenike A W. Storage and flow of solids. Bull. No. 123, 20th Printing, revised 1980. Engng. Exp. Station, Univ. of Utah, Salt Lake City (1964/1980).

Jiang L, Xu Q-Z, Qiao M, Ma F-F, Thakur K and Wei Z-J (2021). Effect of superfine grinding on properties of *Vaccinium bracteatum* Thunb leaves powder. Food Sciences and Biotechnology 26, 1571–1578.

Kaur M, Kaushal P and Sandhu S K (2011). Studies on physicochemical and pasting properties of Taro (*Colocasia esculenta* L.) flour in comparison with a cereal, tuber and legume flour. Journal of Food Science and Technology 50(1), 94–100.

Kha T C, Nguyen M N, Roach P D (2010). Effects of Spray Drying Conditions on the Physicochemical and Antioxidant Properties of the Gac (*Momordica cochinchinensis*) Fruit Aril Powder. Journal of Food Engineering 98, 385–392.

Mbassi L S, Baudelaire E N, Tsague M V, Nguimbou R M, Rup-Jacques S, Deli M, Fomekong G C, Amadou D and Njintang NY (2021). Sieving fractionation, phenolics profile and *in vivo* antioxidant activities of *Dichrostachys glomerata* Forssk. American Journal of Food Sciences 16(7), 184-191.

Moura S C S R, Berling C L, Germer S P M and Alvim I D and Hubinger M D (2018). Encapsulating anthocyanins from Hibiscus sabdariffa L. calyces by ionic gelation: Pigment stability during storage of microparticles. Food Chemistry 241, 317–327.

Nan M Z, Ghadiri M and Wang Y (2017). Analysis of powder rheometry of FT4: Effect of particle shape. Chemical Engineering Science 173, 374–383.

Noumi V D, Nguimbou R M, Tsague M V, Deli M, Rup-Jacques S., Amadou D, Sokeng S and Njintang Y N (2021). Phytochemical Profile and *In Vitro* Antioxidant Properties of Essential Oils from Powder Fractions of *Eucalyptus camaldulensis* Leaves. American Journal of Plant Sciences 12, 329–346.

Oyaizu M (1986). Studies on products of browning reaction: antioxidative activity of products of browning reaction. Japanese Journal of Nutrition 40, 307 – 315.

Patent WO2013057379 A1 Baudelaire E. Comminution and controlled differential screening method for the dry extraction of natural active principles. Google Patents https://encrypted.google.com/patents/WO2013057379A1?cl=en. (2013)

Quintanilla M A S, Castellanos A and Valverde J M (2001). Correlation between bulk stresses and interparticle contact forces in fine powders. Phys. Rev. E Stat. Nonlinear Soft Matter Phys 64, 51–54.

Reza E K and Razie R (2022). Phenolic profile and antioxidant activity of free/bund phenolic compounds of sesame and properties of encapsulated nanoparticles in different wall materials. Food Sciences and Nutrition 10, 525–535.

Ribeiro L C, Costa J M C and Afonso M R A (2020). Flow behavior of cocoa pulp powder containing maltodextrin. Brazilian Journal of Food Technology 23, e2020034.

Santana A A, Martin L G P, de Oliveira R A, Kurozawa L E and Park K J (2017). Spray drying of babassu coconut milk using different carrier agents. Drying Technology 35, 76–87.

Schuck P, Jeantet R and Dolivet A. Analytical methods for food and dairy powders. John Wiley and Sons (2012).

Sílvia C S R M, Carolina L B, Sílvia P M G, Izabela D A and Míriam D H (2018). Encapsulating anthocyanins from *Hibiscus sabdariffa* L. calyces by ionic gelation: Pigment stability during storage of microparticles. Food Chemistry 241, 317–327.

Sousa A S D, Borges S V, Magalhaes N F, Ricardo H V and Azavedo A D (2008). Spray Dried Tomato Powder: Reconstitution Properties and Color. Brazilian Archives Biology and Technology 51, 807–817.

Sun B S, Ricardo-Da-Silva J M and Spranger M I (2008). Critical factors of vanillin assay for catechins and proanthocyanidins. Journal of Agricultural and Food Chemistry 107(10), 4267–4274.

Tchabo W, Ma Y, Kaptso G K, Kwaw E, Cheno R W, Xiao L, Osae R, Meng Wu M and Farooq M (2018). Process Analysis of Mulberry (*Morus alba*) Leaf Extract Encapsulation: Effects of Spray Drying Conditions on Bioactive Encapsulated Powder Quality. Food and Bioprocess Technology 12, 122–146.

Tontul I and Topuz A (2017). Spray-drying of fruit and vegetable juices: Effect of drying conditions on the product yield and physical properties. Trends Food Sciences and Technology 63, 91–102.

Tony H-P, Shen B, Reusch P and Sebald W (1994). Design of human IL-4 antagonists inhibiting IL-4 and IL-13 dependent responses in T and B-cells with high efficiency. European Journal of Biochemistry 225, 659–665.

Vardin H and Yasar M (2012). Optimisation of pomegranate (*Punica Granatum* L.) juice spray-drying as affected by temperature and maltodextrin content. International Journal of Food Sciences and Technology 47, 167–176.

Velasco J, Dobarganes C, Márquez-Ruiz G (2003). Variables affecting lipid oxidation in dried microencapsulated oils. Grasas y Aceites 54, 304–314.

Wafa G, Amadou D, Larbi KM and Héla E F O (2014). Larvicidal activity, phytochemical composition, and antioxidant properties of different parts of five populations of *Ricinus communis* L. Industrial Crops Products 6, 43–51.