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Article

Use of a Taguchi Model in *Hibiscus sabdariffa* Extracts Encapsulated by Spray-Drying

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Abstract: Aqueous and ethanolic extracts of *Hibiscus sabdariffa* were spray-drying using maltodextrin (MD) and gum arabic (GA) as carriers agents. An experimental design Taguchi L8 with seven variables was implemented. Physicochemical properties in the encapsulates were evaluated by UV-Vis, XRD, spectroscopy and gravimetric techniques. Treatments with aqueous extracts showed the highest concentration of total soluble polyphenols (TSP) 32.12- 21.23 mg EAG/g DW, and antioxidant capacity (AOX) for ABTS assay. The best treatment for TSP and AOX was T4: 2.5% *Hibiscus* w/w, aqueous extracts, decoction, extract-to-carrier ratio 1:1 (w/w), proportion to carriers (MD:GA) 80:20 (w/w), 10000 rpm, 150°C. Taguchi L8 model is a tool that allows the use of multiple variables with a low number of treatments that indicate the drying conditions that give the best parameters, focusing mainly on TSP and AOX, in addition, is a good alternative for the preservation and stability of the PC in *Hibiscus*.

Keywords: *Hibiscus sabdariffa*; phenolic compounds; spray-drying; antioxidant capacity

1. Introduction

Hibiscus sabdariffa L. is a shrub that belongs to the Malvaceae family, is a good source of dietary fiber (DF) rich in non-starch polysaccharides, and phenolic compounds (PC) as: flavonoids (kaempferol, quercetin, hibiscetin), anthocyanins (cyanidin, delphinidin. hibiscin), and phenolic acids (protocatechuic acid, chlorogenic acid, hibisicus acid) [1, 2]. Some enhanced varieties as 'Cruza Negra' have a high concentration of PC (41.52 mg GAE/g DW) and antioxidant capacity (AOX) [3]. PC extracts from *Hibiscus* can be used in diverse functional applications such as antioxidant, antihypertensive, effectiveness against low-density lipoprotein oxidation, and hyperlipidemia [2], but they are sensitive to UV rays, oxygen, and high temperatures [4]. The spray-drying encapsulation has demonstrated to be a useful and economical technique to preserve chemical stability, and PC structure [4]. The selection of a carrier agent depends upon the physicochemical properties and the final application of the product. The maltodextrin (MD) is one of the most common carrier agents used for encapsulation that shows low viscosity at high solid content, and high solubility similarly to gum arabic (GA) [5]; both carriers are the preferred choice because of their biocompatibility and innocuous nature [5]. This film creates a net with hydrophobic and/or hydrophilic properties that protect PC from external agents [6]. Encapsulation of PC from *Hibiscus* by spray drying has been previously studied but focusing in the effect of spray-drying temperature in *Hibiscus* extracts and the effect on the volatile compound loss; in the evaluation different type of carriers on the release kinetics of *Hibiscus* extract, and using modified achira (*Canna indica L.*) starch as carrier agent for the encapsulation of the *Hibiscus* extract [7-9]. In this regard, it is important to determine the best conditions of multiple variables to optimize the drying conditions. The use of an experimental design allows evaluating multiple variables that influence spray-drying. The Taguchi model provide the best values of variables to ensure quality of the product/process with a few treatments [10]. The aim

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of this study was to evaluate the effect of spray-drying variables, in an aqueous and ethanolic extracts of *Hibiscus sabdariffa*, on the physicochemical properties, PC content, and AOX by the experimental Taguchi L8 model. Seven variables with two levels (extraction solvent, *Hibiscus* concentration, decoction, extract–carriers ratio, proportion of carriers agents, homogenization and dried inlet temperature) and mixtures of MD and GA were utilized as carriers agents.

2. Materials and Methods

2.1 Materials

H. sabdariffa ´Cruza Negra´ calyces were provided by a local producer (Tepic, Nayarit, México, 21° 39′ 15″ N, 106° 32′ 45″ O). The samples were ground (500 μm), mixed to obtain homogenous samples, and preserved in hermetic metallic bags. The carriers used were corn maltodextrin (MD; DE 10) (Tate & Lyle Ingredients, Decatur, Illinois, EE. UU), and gum arabic (GA) (Nexira Food, Rouen, France).

2.2 Experimental design

A Taguchi L8 design was performed to obtain the best extraction conditions in base of PC content. Seven variables with two levels were used (Table 1). The experimental design was composed by eight treatments of the independent variables. The levels were selected on basis of studies reported before (data not shown). To avoid systematic errors, all the experiments were performed in a random order, to minimize the effect of unexplained variability on the responses obtained.

2.3 Sample preparation and spray-drying of Hibiscus extracts

The *Hibiscus* extracts were prepared according to the conditions indicated in Table 1; the extracts were stored in amber flasks at 4 ± 2°C. The solid content of the extracts and –the carriers materials (MD and GA) were dispersed in water and homogenized (Ultra-Turrax T18 with disperser S18N-19G; IKA, Staufen,Germany) (Table 1) during 5 min. The prepared carriers agents solutions were combined with *Hibiscus* extract, and homogenized again at the same conditions. The feed mixtures were spray-drying (B-290, Büchi Labortechnik AG, Flawil, Switzerland) with a main chamber of 165 mm diameter, 600 mm cylindrical height and 1.5 mm nozzle diameter. The pump power was kept at 15% to maintain feed flow rate as 4.5 mL/min, the airflow rate was 38 m^3/h and 3 pulse clean for period. During drying processes, the temperature of the feed mixture was 24°C at constant magnetic stir (Cimarec digital, Thermo Scientific, Waltham, Massachusetts, EE. UU.).

The microencapsulates obtained were evaluated in their physicochemical properties, total soluble polyphenols (TSP) and AOX capacity in order to obtain the best extraction condition.

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Table 1. Experimental design Taguchi L8 and yield calculation of treatments of extracts of *Hibiscus (Hibiscus sabdariffa)* spray-drying[1].

Treatment	*Hibiscus* extracts			Spray-drying conditions				EY[3](%)	EE[4] (%)
	Concentration (%)	Solvent of extraction	Decoction (ºC/min)	Extract:carriers ratio (w/w)	Carriers ratio (MD + GA)[2] (%)	Homogenization (rpm)	Inlet T (°C)		
T1	1	Ethanol 20%	100 / 5	1:1	90:10	10,000	110	61.88 ± 0.93^{b}	86.58± 0.95^{bc}
T2	2.5	Water	100 / 5	1:2	90:10	5,000	110	89.38 ± 1.04^{g}	85.45± 4.47^{bc}
T3	1	Water	NA	1:1	80:20	5,000	110	73.53 ± 1.01^{a}	87.93± 2.94^{c}
T4	2.5	Water	100 / 5	1:1	80:20	10,000	150	86.34 ± 0.05^{f}	86.70± 2.23^{bc}
T5	1	Water	NA	1:2	90:10	10,000	150	69.01 ± 0.42^{c}	88.48± 0.73^{c}
T6	1	Ethanol 20%	100 / 5	1:2	80:20	5,000	150	74.80 ± 0.68^{d}	82.61± 0.18^{ab}
T7	2.5	Ethanol 20%	NA	1:2	80:20	10,000	110	84.40 ± 0.28^{e}	80.95± 1.13^{a}
T8	2.5	Ethanol 20%	NA	1:1	90:10	5,000	150	72.53 ± 0.59^{a}	85.36± 2.02^{abc}

[1] Values are the mean ± standard deviation (n = 3). Different letters in each column indicate significant difference. Mean analyzed by LSD (p<0.05). NA: Not apply

[2] MD: Maltodextrin DE 10, GA: Gum arabic

[3] EY: Encapsulation yield

[4] EE: Encapsulation efficiency, NA, not apply

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2.4 Physicochemical Properties

2.4.1 Encapsulation yield (EY)

EY was evaluated by the material balance of the product recovery given by the perceptual ratio (dry basis) between the total mass of product recovered by the mass of juice fed to the system [11].

2.4.2 Encapsulation efficiency (EE)

The EE is the percentage ratio of encapsulated PC content and total PC content (Eq. 1), it was determined taking the difference of total PC (TPC) and the PC at time 0 when is reconstituting the powder in the solvent which is called surface PC (SPC) following the methodology described in the section 2.8 for total soluble polyphenols (TSP) [12].

$$EE(\%) = \frac{(TPC-SPC)}{TPC} * 100 \quad (1)$$

2.4.3 Moisture, water Activity (Aw), pH, solubility, wettability and bulk density analysis

Partially air-dried samples were kept in a hot-air oven at 110°C until they reached a constant weight, and the moisture content was calculated in terms of the weight loss method 925.10 [13] (AOAC, 2012). An Aw meter (Aqualab 4TEV, Decagon Devices, Pullman, Washington, EE. UU.) was used, 5 g of sample was placed inside the chamber where the Aw was determined by the drop point principle. For pH, dilutions 1:100 (w/v) of the encapsulates were prepared in distilled water and the pH was determined by a potentiometer (Hanna HI 2210, Woonsocket, Rhode Island, EE. UU.) at 25°C [14]. The solubility in encapsulates was evaluated according an existing method [15]. The wettability was evaluated using an existing static method [16], this one was expressed as the time necessary for 1 g of encapsulates to disappear from the water surface. The bulk density was evaluated according to a method reported [17].

2.5 Scanning electron microscopy (SEM)

The morphology of the spray-dried particles was visualized using scanning electron microscopy (SEM) (Philips/XL30-ESEM, Netherlands) at 5 kV with a magnification of 5000×. Samples were mounted on self-adhesive tape and gold coated before imaging (Denton vacuum Desk V operated at 10 mA for 60 s, EE. UU.)

2.6 Absorption spectrum

An UV-Visible spectra 200-900nm was used to evaluated the absorption spectrum in the treatments, MD and GA alone, and the mix of carriers. The distribution was carried out by Dynamic Light Scattering (DLS) using a spectrophotometer (UV 2600, Shimadzu, Kioto, Japan).

2.7 X-ray diffraction (XRD)

Encapsulates were analyzed in a diffractometer (Bruker D8 overtakes Tokyo, Japan) (Kα Cu =1.5460 Å, 40 kV, 30 mA), considering the diffraction intensity as a function of the diffraction angle (2θ) between 10° and 90°, using a step of 0.02° and 0.25s per step.

2.8 Total soluble polyphenols (TSP) content and AOX assays.

The encapsulates were extracted to evaluate the TSP according to an existing method [18]. TSP contents were determined in the supernatants using an existing method [19] in a microplate reader

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(BioTek, Synergy HT, Winooski, VT, EE.UU.). The absorbance was read at 750 nm against a blank, and TSP was calculated using the calibration curve of gallic acid. The results were expressed as gallic acid equivalents (mg GAE/g dry weight (DW)).

The supernatants were used to evaluate the DPPH radical scavenging method, ABTS analysis and, ferric reducing antioxidant power (FRAP) assay. For DPPH assay was analyzed by modifying an existing method [20,21], the absorbance was read at 517 nm, the results are reported in mmol TE (trolox equivalent) /g DW. For the ABTS radical assay based in an existing method [22] with some modifications [21]; the absorbance was read at 734 nm, the results were reported in mmol TE /g DW. For FRAP Assay was analyzed by an existing method [23]; Trolox was used as a standard and methanol acidified as blank, the absorbance was read at 595 nm and the results are reported in mmol TE /g DW.

2.9 Data analysis

The statistical analysis was carried out with the STATISTICA software, version 10.0 (StatSoft Inc. 1984–2007, Tulsa, EE. UU.). The treatments were performed in triplicate; the analytical data were expressed as mean ± standard deviation (SD) for each treatment. Analysis of variance (ANOVA) using a univariate design was performed to determine the differences between the treatments. For means comparison, a Fisher LSD test with a significance level of $\alpha = 0.05$ was applied.

3. Results and Discussion

3.1 Physicochemical properties

The EY values obtained were from 61.88 to 89.39% for T2 (2.5% *Hibiscus*, water, decoction, extract: carriers 1:2, ratio carriers 90:10, 5000 rpm, 110°C) treatment (Table 1). High inlet temperatures cause more proximity of particles to glass transition temperatures, and more adherence, especially if the droplet still with a high moisture content [24]. The extract/carriers ratio is another factor to consider, treatments with a ratio 1:2 (w/w) exist a higher carriers agent content and therefore higher temperature tolerance resulting in higher EY [15]. Other investigations showed similar results in spray-drying with 89 ±3% using MD and GA [16]. The EE was ranged from 80.94 to 88.48% for T5 (1% *Hibiscus,* water, no decoction, extract:carriers 1:2, ratio carriers 90:10, 10000 rpm, 150°C). The relationship between type of carriers, core/coating ratio, ultrasonication time, coating material types, content of PC, and particles size influence in the EE [6,9,12]. Reports indicate that the core (material to be encapsulated) shows physicochemical properties that must be taken into account during the encapsulation process [25]. The decrease in the amount of TSP is attributed to the high inlet temperature, generating the degradation by structural chemical changes of the PC [9], other factors are the ratio extract: carriers, and the extraction conditions (decoction and weight of *Hibiscus*) [5].

The physicochemical properties (Table 2) showed that the highest value in moisture was obtained for T3 (1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C) with 9.55% ± 0.41 and the lowest were between four treatments (all with ethanolic extraction): T1, T6, T7 and T8 ranged between 2.77 – 4.89%; this result can be attributed to the concentration of total soluble solids at the beginning of the spray-drying that generates a decrease in this parameter [24] (Table 2). The decrease in moisture content is a function of the drying temperature; under high temperature, the rate of heat transfer within the particle is high, which leads a faster evaporation of water, thereby the moisture content of the product decreases [5].

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Table 2. Physicochemical analysis to treatments of *Hibiscus* extracts *(Hibiscus sabdariffa*) spray-drying with different proportions of MD and GA[1]

Treatment	Moisture (%)	A_w	pH	Solubility (%)	Wettability (min)	Bulk Density (g/cm^3)
T_1	4.89 ± 0.87^{abc}	0.27 ± 0^{b}	3.09 ± 0.01^{e}	89.22 ± 2.0^{a}	5.14 ± 0.03^{ab}	0.35 ± 0.02^{f}
T_2	5.97 ± 0.86^{c}	0.23 ± 0^{a}	2.75 ± 0.01^{c}	77.11 ± 5.97^{a}	4.76 ± 0.42^{ab}	0.44 ± 0.01^{cde}
T_3	9.55 ± 0.41^{f}	0.38 ± 0^{h}	2.70 ± 0.01^{b}	83.82 ± 3.24^{a}	2.95 ± 0.16^{a}	0.43 ± 0.03^{bcd}
T_4	5.19 ± 0.62^{bc}	0.35 ± 0^{e}	2.65 ± 0.01^{a}	86.06 ± 0.33^{a}	3.08 ± 1.51^{a}	0.48 ± 0.03^{e}
T_5	8.06 ± 0.66^{e}	0.36 ± 0^{f}	2.65 ± 0.01^{a}	82.23 ± 3.05^{a}	3.92 ± 0.46^{a}	0.46 ± 0.03^{de}
T_6	4.49 ± 0.58^{ab}	0.29 ± 0^{d}	3.55 ± 0.01^{g}	90.82 ± 1.42^{a}	6.19 ± 2.97^{ab}	0.42 ± 0.03^{abc}
T_7	3.76 ± 0.35^{ad}	0.28 ± 0^{c}	3.19 ± 0.01^{f}	89.71 ± 2.01^{a}	7.72 ± 0.42^{b}	0.40 ± 0.01^{ab}
T_8	2.77 ± 0.84^{d}	0.37 ± 0^{g}	2.80 ± 0.01^{d}	90.76 ± 0.76^{a}	4.83 ± 2.23^{ab}	0.38 ± 0.02^{af}

[1] Values are the mean ± standard deviation (n = 3). Different letters in each column indicate significant difference. Mean analyzed by LSD ($p<0.05$).

T_1: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:1, ratio carriers 90:10, 10000 rpm, 110°C

T_2: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:2, ratio carriers 90:10, 5000 rpm, 110°C

T_3: 1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C

T_4: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C

T_5: 1% *Hibiscus*, water, no decoction, extract:carriers 1:2, ratio carriers 90:10, 10000 rpm, 150°C

T_6: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:2, ratio carriers 80:20, 5000 rpm, 150°C

T_7: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:2, ratio carriers 80:20, 10000 rpm, 110°C

T_8: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:1, ratio carriers 90:10, 5000 rpm, 150°C

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The Aw showed significant differences (p<0.05) in all the treatments. Aw contents obtained were low, this parameter provides the guideline to predict the stability and shelf life in a product [25]. The values obtained in the pH parameter did show significant differences, the acidity can be due to organic acids from *Hibiscus* such as malic, citric, hibiscus, and hydroxyl citric acids [1]. The solubility (%) did not showed significant differences, this parameter is related to the moisture of the particles [9]. Other factor that influences the solubility is the proportion of the carriers (MD and GA). MD and GA are widely used in the process of spray-drying due to high solubility in water, and for it is emulsification properties [15].

In the wettability parameter, the highest value was obtained for T7 (2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:2, ratio carriers 80:20, 10000 rpm, 110°C) with 7.72 min ± 0.42. Reports indicate that the wettability process corresponds to the phenomenon that allows the penetration of water in a particle [16]. According to reports, wetting times of 2.38 - 1.62 min, have been expressed for a *Hibiscus* extract in spray-drying powder without drying agents [14]. The wettability is directly affected by the molecular interactions between the components [26]. Authors suggest that a higher moisture content is probably the generation of a greater number of hydrophilic groups on the particles, thus reducing time through greater interaction with water [27]. In bulk density, the highest value was T4 (2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C) with 0.46 g/cm^3 ± 0.03; these results are similar to those reported by others authors but lower than those reported for *Hibiscus* extracts without carriers agents (0.73 -0.87 g / cm^3) [14,16]. The bulk density of the encapsulates is related to the molecular weight of the carrier's agents in a heavier material it can fit more easily in the spaces between the particles, occupying less space and getting higher apparent density values [28].

3.2 Scanning electron microscopy (SEM)

In the Figure 1 is shown a SEM of one encapsulate of *Hibiscus* extracts by spray-drying. It shows a spherical particle of different diameters; as well as lightly smooth surfaces attributable to the carriers' agents (MD and GA) with the concavity's formation some with a wrinkled surface. The formation of concavities on the surface of the atomized particles can attribute a contraction during the drying [29]. According to reports, the formation of wrinkle occurs because the slow formation of cover film when droplets are atomized [22].

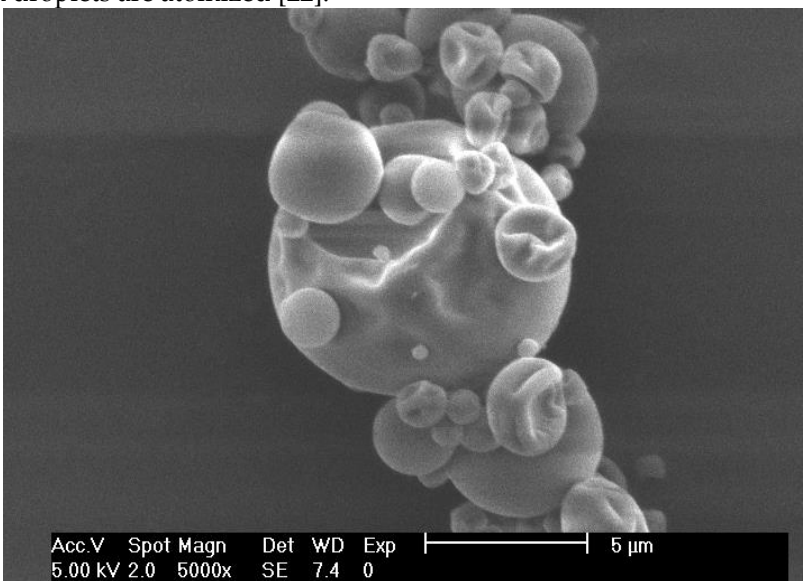

Figure 1. SEM micrography of *Hibiscus sabdariffa* extract encapsulated by spray-drying

3.3 Absorption spectrum

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The Figure 2 are shown the carriers agents spectrum in the mixture and separate, in which it is possible to identify two absorption bands at 211 and 282 nm (Figure 2a). These bands are characteristics of carbonyl groups derived from the conformational structures of MD and GA. The Figure 2b shows all the treatments obtained, it was possible to identify a third weak band at 327 nm. According to reports, this band is attributed to the presence of PC such as catechins, flavonoids and phenolic acids [30]; this phytoconstituents are characterized by the presence of various chromophores and conjugated systems that act as UV-absorbing systems. The presence of anthocyanins from *Hibiscus* was evidenced in the band at 532 nm [1]. These spectrums indicate homogeneous behavior between treatments as well rearrangement between the PC of *Hibiscus* and the carrier agents showing specific bands for each of the signals indicating interaction attributive to hydrogen bond [34].

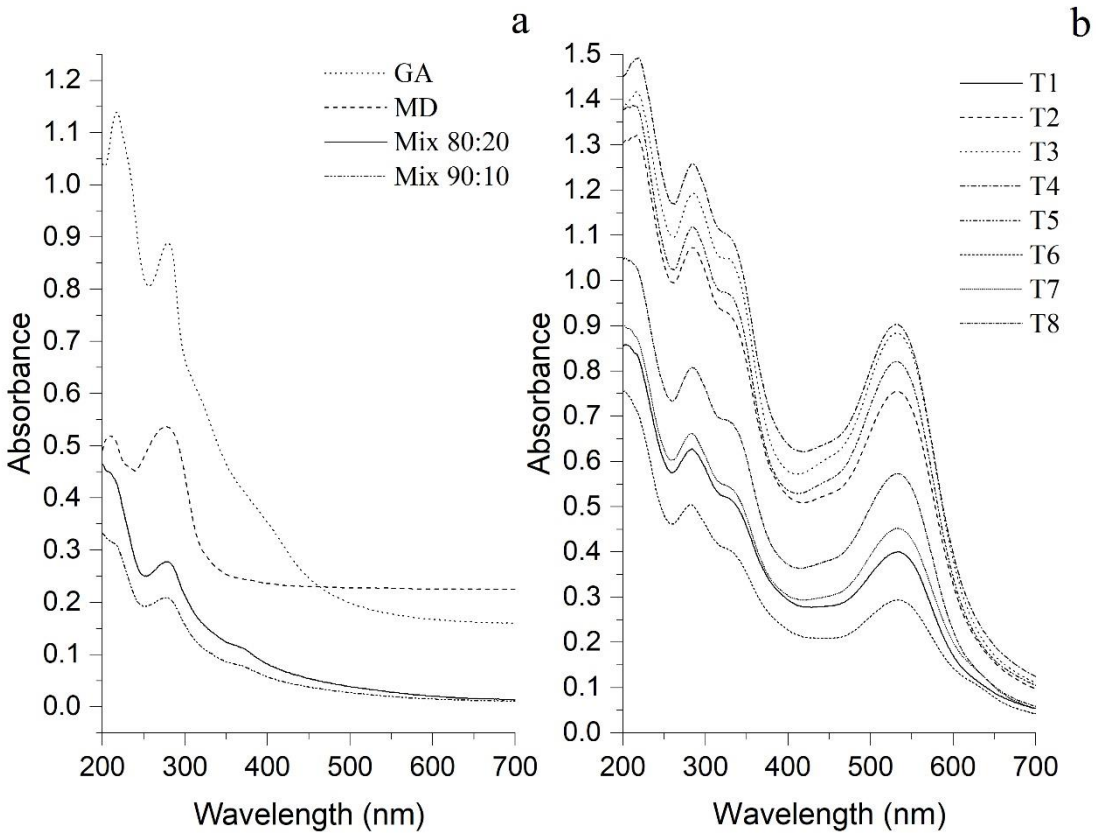

Figure 2. Absorption spectrum. **(a)** Only carriers and mix of carriers 80:20 and 90:10 (MD DE 10: GA). **(b)** Treatments extracts of *Hibiscus (Hibiscus sabdariffa*) spray-drying.

T_1: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:1, ratio carriers 90:10, 10000 rpm, 110°C
T_2: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:2, ratio carriers 90:10, 5000 rpm, 110°C
T_3: 1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C
T_4: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C
T_5: 1% *Hibiscus*, water, no decoction, extract:carriers 1:2, ratio carriers 90:10, 10000 rpm, 150°C
T_6: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:2, ratio carriers 80:20, 5000 rpm, 150°C
T_7: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:2, ratio carriers 80:20, 10000 rpm, 110°C
T_8: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:1, ratio carriers 90:10, 5000 rpm, 150°C

3.4 X-ray diffraction

Using the X-ray diffraction technique (XRD), the MD presents a crystalline structure of 10° to 212 30° (Figure 3), The GA also presents a characteristic peak at 18°, on the 2theta scale, which

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suggests that when the GA and the MD are encapsulated with the different treatments, the intensity of the peaks decreases, which generate molecular interactions between the carriers agents and PC that structurally modify the diffraction profile, resulting in low intensity of the signal and excessive noise, which can describe the characteristic presence of amorphous [25]; similar results reported indicated the presence of amorphous material when mango juice dehydration by spray-drying using different carriers agent [15]. Therefore, according to the XRD patterns in the different treatments, there is a diversity of compounds present in the samples that generate molecular interactions between the carrier's agents that structurally modify the diffraction profile [25].

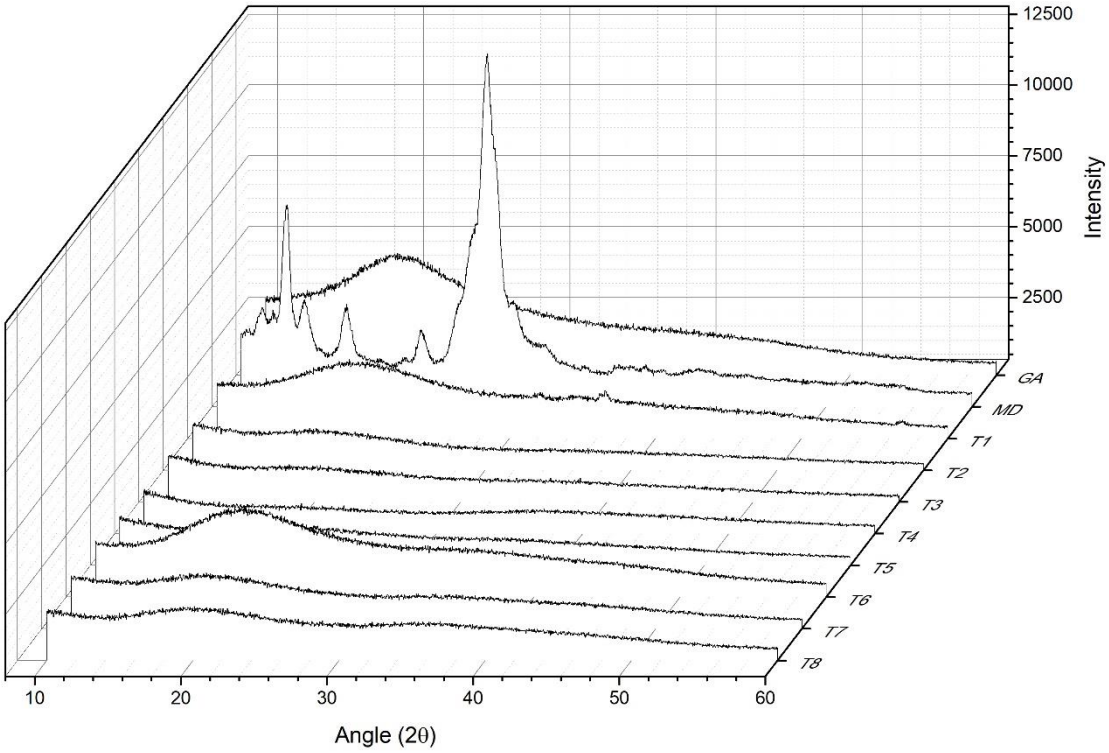

Figure 3. X-ray diffraction for treatments extracts of *Hibiscus (Hibiscus sabdariffa*) spray-drying. MD: maltodextrin DE 10 and GA: gum arabic

T_1: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:1, ratio carriers 90:10, 10000 rpm, 110°C
T_2: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:2, ratio carriers 90:10, 5000 rpm, 110°C
T_3: 1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C
T_4: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C
T_5: 1% *Hibiscus*, water, no decoction, extract:carriers 1:2, ratio carriers 90:10, 10000 rpm, 150°C
T_6: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:2, ratio carriers 80:20, 5000 rpm, 150°C
T_7: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:2, ratio carriers 80:20, 10000 rpm, 110°C
T_8: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:1, ratio carriers 90:10, 5000 rpm, 150°C

3.5 PC content and AOX in Hibiscus encapsulates

Table 3 shows the content of TSP and AOX in encapsulates of *Hibiscus* extracts where significant differences were found between treatments (p<0.05). The highest content in TSP was for T4 (2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C) with a concentration of 32.13 ± 0.06 mg EAG / g DW; and the lowest to T6 (1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:2, ratio carriers 80:20, 5000 rpm, 150°C) treatment with a 7.16 ± 0.05 mg EAG / g DW, this concentration of TSP can be explained due to the low concentration of substrate

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(1%), the extraction solvent (EtOH 20%) and the extract:carriers ratio (1: 2). Values have been reported of 41.52 ± 0.99 mg EAG / g DW for the same 'Cruza Negra' under the same extraction method [3]. The polar solvents have influence in the extraction, because the major PC in *Hibiscus* are flavonoids with glycosidic structures and are more soluble [31]. For that could be consider the extraction of gallic and chlorogenic acid and (+) - catechin present in *Hibiscus* [32].

The AOX, in the aqueous extracts showed two fold higher than ethanolic extracts with a correlation with the results of TSP; Reports indicate that when the polarity of the solvent increases, there are higher EY of total soluble solids and extractable PC [33].

For the evaluation of antiradical activity (ABTS), the treatment with the highest value corresponds to T4 (2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C) 216.28 ± 5.24mmol ET / g DW (Table 3); similar values were reported with values from 221.5 - 363.2mmol ET / g DW in different varieties of *Hibiscus* [31]. This can be explained by the heat treatment to which the calyces are subjected for extraction, and the hydrolysis generated by the acidity gave that the ellagic and gallotannin tannins [32].

The activity for the DPPH radical, attributed to the condensed tannins derived from the heat treatment, since they exhibit a good (DPPH) AOX capacity mainly due to the tannins and flavonoids contain a variety of hydroxyl groups that show stronger AOX and elimination of free radicals of the PC present [35]. The chelating activity (FRAP- Iron reducing antioxidant power), T3 (1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C) showed highest capacity with 271.95 ± 6.63 mmolET/g DW value; previous in vitro studies have demonstrated the ability of flavonoids such as catechins and quercetin to inhibit the formation of free radicals and chelation of metal ions, particularly those of iron and copper [36]. Higher AOX values were presented by the FRAP and ABTS techniques, can be attributed to the presence of gallic acid and chlorogenic acid, as well as the presence of phenolic acids and flavonoids and anthocyanins present in *Hibiscus* (p.e. catechin and quercetin) which, given the position of the hydroxyl groups, are responsible for the high chelating activity and the search for radical power [32].

Table 3. Total soluble polyphenols (TSP), partial identification of polyphenol profile and antioxidant capacity (AOX) in treatments of extracts of *Hibiscus* (*Hibiscus sabdariffa*) spray-drying[1]

Treatment	TSP (mg GAE/g DW)[2]	AOX (mmol TE /g DW)[3]		
		ABTS	DPPH	FRAP
T_1	16.00 ± 0.56^{d}	103.16 ± 3.79^{b}	28.60 ± 3.30^{ab}	220.06 ±2.27^{a}
T_2	21.23 ± 0.17^{e}	139.64 ± 12.13^{c}	57.61 ± 5.19^{e}	258.43 ± 1.13^{f}
T_3	30.51 ± 0.41^{g}	201.80 ± 0.05^{e}	89.15 ± 11.00^{d}	271.95 ± 6.63^{g}
T_4	32.13 ± 0.06^{h}	216.28 ± 5.24^{f}	87.78 ± 2.76^{cd}	231.41 ± 0.13^{e}
T_5	25.69 ± 0.11^{f}	155.16 ± 0.12^{d}	80.12 ± 4.78^{c}	224.45 ±1.29^{a}
T_6	7.16 ± 0.05^{a}	24.27 ± 0.01^{a}	21.29 ± 0.02^{b}	67.97 ±1.29^{b}
T_7	9.14 ± 0.03^{b}	24.42 ± 0.11^{a}	30.67 ± 0.42^{a}	140.56 ± 0.83^{c}
T_8	13.73 ± 0.21^{c}	24.04 ± 0.04^{a}	33.02 ± 0.04^{a}	154.09 ±0.20^{d}

[1] Values are the mean ± standard deviation (n = 3). Different letters in each column indicate significant difference. Mean analyzed by LSD (p<0.05).

[2] GAE, gallic acid equivalent, dry weight (DW); TE, trolox equivalent, dry weight (DW)

T_1: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:1, ratio carriers 90:10, 10000 rpm, 110°C

T_2: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:2, ratio carriers 90:10, 5000 rpm, 110°C

T_3: 1% *Hibiscus*, water, no decoction, extract:carriers 1:1, ratio carriers 80:20, 5000 rpm, 110°C

T_4: 2.5% *Hibiscus*, water, decoction, extract:carriers 1:1, ratio carriers 80:20, 10000 rpm, 150°C

T_5: 1% *Hibiscus*, water, no decoction, extract:carriers 1:2, ratio carriers 90:10, 10000 rpm, 150°C

T_6: 1% *Hibiscus*, ethanol 20%, decoction, extract:carriers 1:2, ratio carriers 80:20, 5000 rpm, 150°C

T_7: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:2, ratio carriers 80:20, 10000 rpm, 110°C

T_8: 2.5% *Hibiscus*, ethanol 20%, no decoction, extract:carriers 1:1, ratio carriers 90:10, 5000 rpm, 150°C

4. Conclusions

Based on the results the Taguchi L8 model allowed to obtain the best values in the studied variables to ensure the quality of the *Hibiscus sabdariffa* extracts encapsulates. The T4 treatment showed the highest concentration of TSP with AOX. A good EE (%) and storage stability showed interactions between the carriers agents and the PC present in the calyces. These new interactions were confirmed by the results of UV-Vis spectroscopy and X-ray diffraction. The use of an experimental model can lead to the use of small number of experiments to evaluate the effect of spray-dryer in *Hibiscus* extracts.

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