



## Research article

# Yacon powder mix: Effects of the composition and the process of microencapsulation by spray drying

María Isabel Arango-Torres<sup>a</sup>, Misael Cortés-Rodríguez<sup>a</sup>, Esteban Largo-Ávila<sup>b</sup>,  
Manuela Gallón-Bedoya<sup>a</sup>, Rodrigo Ortega-Toro<sup>c,\*</sup>

<sup>a</sup> Universidad Nacional de Colombia, Sede Medellín - Faculty of Agricultural Sciences, Department of Agricultural and Food Engineering, Functional Food Research Group, Cra. 65 No. 59A -110, Medellín, CP 050034, Colombia

<sup>b</sup> Universidad del Valle, Regional headquarters Caicedonia, Cra. 14 No. 4 - 48, Caicedonia, Valle del Cauca, Colombia

<sup>c</sup> Universidad de Cartagena, Faculty of Engineering, Food Engineering Program, Food Packaging and Shelf Life research group (FP&SL), Cartagena de Indias D.T. y C., Avenida del Consulado, Calle 30 No. 48-152, Colombia

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## ABSTRACT

Yacon is a tuber known as a healthy food due to its effects as an antidiabetic, anti-inflammatory, anticancer, and prebiotic agent; it is rich in fructooligosaccharides (FOS) and antioxidants, and due to its sweet taste and low-calorie content, it is used as a substitute for ordinary sugar. This research aimed to evaluate the influence of the composition of the feed and the microencapsulation process by spray drying (SD) on the properties of a yacon powder mixture (YP). Response surface methodology with a central composite design with a face-centered composition ( $\alpha = 1$ ) was used, considering the independent variables: inulin (IN) (3–5% w/w), maltodextrin (MD) (3–5% w/w), air inlet temperature (AIT) (140–160 °C), air outlet temperature (AOT) (75–85 °C) and atomizer disc speed (ADS) (18000–22000 rpm), and the dependent variables: moisture ( $X_w$ ), water activity ( $a_w$ ), hygroscopicity (Hy), solubility (S), particle size (percentile  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$ ), total phenols (TP), antioxidant capacity (ABTS and DPPH), color (CIE-Lab\*) and yield (Yi). The suspension formulation contained xanthan gum (0.167 %) and a mixture of ascorbic and citric acids (0.3 %). The  $a_w$  and  $X_w$  values of the YP guarantee its microbiological stability; however, the process formulation produces a complex matrix (FOS- sugars- MD - IN) with high affinity for water, which favors adsorption phenomena (hygroscopic material) and high reconstitution (high solubility). The independent variables that best fit the experimental optimization criteria were: IN = 3.0 %, MD = 5.0 %, AIT = 143.7 °C, AOT = 80.1 °C, ADS = 22000 rpm, where  $Y_i = 84.2$  %, and the quality of the YP:  $X_w = 2.4$  %,  $a_w = 0.220$ , Hy = 23.0 %, S = 96.9 %,  $D_{10} = 10.6$   $\mu\text{m}$ ,  $D_{50} = 23.4$   $\mu\text{m}$  and  $D_{90} = 169.3$   $\mu\text{m}$ , TP = 1228.2 mg gallic acid equivalent/100 g, ABTS = 2295.9 mg Trolox equivalent (TE)/100 g, DPPH = 5192.3 mg TE/100 g,  $L^* = 80.5$ ,  $a^* = 5.1$  and  $b^* = 17.4$ . SD is an effective technology that positively impacts the development of new food products. In addition, the YP could have multipurpose applications for the industry, generating value in this agri-chain.

\* Corresponding author.

E-mail addresses: [rortegap1@unicartagena.edu.co](mailto:rortegap1@unicartagena.edu.co), [rodrigo.ortega.toro@gmail.com](mailto:rodrigo.ortega.toro@gmail.com) (R. Ortega-Toro).

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## Abbreviations

Name	Abbreviature	Name	Abbreviature
Air inlet temperature	AIT	Percentile 10, 50 y 90 %	D <sub>10</sub> , D <sub>50</sub> y D <sub>90</sub>
Air outlet temperature	AOT	Red - Green chromaticity	a*
Ascorbic acid equivalents	AAE	Relative mean error	RME
Atomizer disc speed	ADS	Solubility	S
Dry base	db	Spray drying	SD
Equation	Eq.	Temperature differences	ΔT
Fructooligosaccharides	FOS	Total phenols	TP
Gallic acid equivalent	GAE	Trolox equivalent	TE
Glass transition temperature	Tg	Water activity	a <sub>w</sub>
Hygroscopicity	Hy	Xanthan gum	XG
Inulin	IN	Yellow - Blue chromaticity	b*
Luminosity	L*	Yacon powder mix	YP
Maltodextrin	MD	Yield	Yi
Moisture	Xw		

## 1. Introduction

Yacon (*Smallanthus Sonchifolius*) is an Andean root belonging to the Asteraceae family. Unlike most edible roots, it stores carbohydrates as fructooligosaccharides (FOS) rather than starch [1]. FOS are recognized for their beneficial effects on health; they stimulate the immune system [2], reduce the concentration of insulin, cholesterol, triglycerides, and phospholipids in the bloodstream [3], promote calcium reabsorption, and thus contribute to the prevention of osteoporosis [4]. On the other hand, FOS is considered prebiotic fibers because human digestive enzymes do not hydrolyze them; however, they are fermented by *Lactobacillus* and bifidobacteria, promoting their growth over pathogenic bacteria [5]. Additionally, phenolic compounds, mainly chlorogenic acid (caffeine-kinase) and other caffeic acid derivatives, are responsible for their antioxidant properties [6].

Yacon is the plant with the highest FOS content in nature; for this reason, research focused on obtaining these compounds from yacon stimulates the industrialization of this crop. However, there is a significant challenge to increase marketability, which lies in increasing the shelf life of fresh produce by inhibiting the action of polyphenol oxidase and peroxidase enzymes, which promote oxidation, exhibiting a dark green hue that makes it unattractive [7].

Currently, FOS applications in the food industry include fat substitution (mayonnaise and low-calorie cheeses), reduction of caloric content (chocolate substitutes), elevated water retention (pastries, bakery, and sausages), inhibition of crystal formation in ice cream and, in general, they are useful for modifying the texture or creaminess of some foods. FOS is a subgroup of inulin (IN); while the latter contains 2 to 60 molecules, FOS have 2 to 10 molecules in their chain; therefore, their physical properties and applications differ. IN has an incipient sweet taste and low relative solubility in water, which make it an excellent substitute for fat; in contrast, FOS is soluble in water and with a slightly sweet taste (30–50 % of the sweetening power of sugar), which is why they are used as hypocaloric substitutes [8] because they provide only a quarter of the caloric value of sucrose.

Heat-sensitive molecules such as phenols and some with antioxidant activity are susceptible to thermal degradation during processing and storage, compromising their quality and efficacy. Microencapsulation offers a solution by protecting these molecules within encapsulating matrices, providing a physical barrier against heat and other adverse environmental factors [9]. The properties of the powder blends obtained are affected by the characteristics of the feed suspension to the SD (total solids, amount and type of drying additives, particle size and distribution, viscosity, surface tension, and temperature, among others) [10,11], which in turn depend on the nature of the materials that form it. Similarly, these properties are affected by the operating conditions of the SD (feed rate, air inlet temperature (AIT), air outlet temperature (AOT), atomizing disk speed (ADS) or spray nozzle pressure, and residence time, among others). Usually, drying agents are used to improve the properties of powder blends, acting as encapsulants, maltodextrin (MD) being highlighted for its high solubility, mild flavor, low cost, and for contributing to reducing the stickiness and crystallization tendency of powders [12]; however, it has a caloric intake of 3.92 kcal/g and a glycemic index of 85. On the other hand, other drying additives have been effectively used in various products: starches, gum Arabic, cyclodextrin, proteins, and others [13,14,54].

The yacon powder mixture (YP) represents an innovative food that is framed within functional foods and whose productive alternative could favor the population with diabetes problems because it has a reduced caloric intake (maximum 1.5 kcal/g) and a negligible glycemic response [15]. In this context, the objective of the research was to evaluate the influence of the composition and the microencapsulation process by SD on the properties of YP.

## 2. Methods

### 2.1. Materials

Yacon tubers harvested at 9 months of commercial maturity in Santa Elena (Medellín, Antioquia), Xanthan Gum (XG) (Instantgum™ BA, Nexira, France), citric acid and food grade ascorbic acid, MD with Dextrose equivalent 19–20 (019200, China 25) and IN (Fibruline S20, USA) were used.

## 2.2. Formulation of yacon suspensions

The roots were washed with pressurized water, disinfected with HClO solution (50 ppm), peeled, and passed through a juice extractor (Oster) for preliminary structure disintegration. Batches of 5000 g were prepared in a rotor-stator homogenizer Silverson, USA, model L5M-A at 10000 rpm for 10 min. The suspension formulations contain a mixture of acids to control browning (0.15 % citric acid and 0.15 % ascorbic acid) and XG (0.167 %) as a stabilizing agent, previously determined by Arango et al. [16]. Additionally, the drying additives MD and IN were added in the proportions established by the experimental design. The suspensions obtained were processed in a second piston homogenizer ST REGIS, 3DD13-2941, Chicago, USA, operating at 1200 psi during 4.8 min of recirculation [16].

## 2.3. Microencapsulation process by spray drying

A pilot co-current flow spray dryer (Vibrasec, model PASLAB 1.5) operating under vacuum conditions (14.7 Pa) was used. The evaluation of the SD process was performed using the response surface methodology, considering a face-centered central composite experimental design ( $\alpha = 1$ ) with five independent variables: IN (3–5% p/p), MD (3–5% p/p), AIT (140–160 °C), AOT (75–85 °C) and ADS (18000–22000 rpm), and dependent variables: moisture ( $X_w$ ), water activity ( $a_w$ ), hygroscopicity (Hy), solubility (S), particle size ( $D_{10}$ ,  $D_{50}$ , and  $D_{90}$ ), total phenols (TP), antioxidant capacity measured with ABTS and DPPH radicals, color (CIE- $L^*a^*b^*$  coordinates) and yield (YI). The YP dependent variables were characterized on 3 samples taken at 30, 45, and 60 min for each experiment.

$X_w$  was determined by weight loss at 60 °C in a vacuum oven (10 mbar) for 24 h [17].  $a_w$  was determined using a dew point hygrometer at 25 °C (Aqualab 3 TE series, Decagon, Devices, Pullman, WA, USA). S was determined according to the methodology of Eastam and Moore [18] modified by Cano-Chauca et al. [19]; considering 1 g YP dispersed in 50 mL of water, centrifugation at 8000 rpm/5 min at 25 °C and taking a 25 mL aliquot of the supernatant in a previously weighed Petri dish, which was subsequently dried in an oven at 105 °C and 5 h, determining S from the difference in weights. Hy was determined by the gravimetric method of water sorption isotherm construction, according to the methodology described by Martínez-Navarrete et al. [20], using a saturated solution of KI at 25 °C ( $a_w = 0.689$ ) and expressed as a percentage on a dry basis (db). TP content was determined from a gallic acid standard curve, describing the results in mg gallic acid equivalent (GAE)/100 g [21]. Antioxidant capacity was determined from DPPH [22] and ABTS [23] antiradical activity by taking a 0.2 g sample of YP and dissolving it with 25 mL of boiling water. The mixture was vortexed for 1 min, left in a water bath at 90 °C, 10 min, and subsequently centrifuged at 8000 rpm, 10 min, 25 °C, and the supernatant was analyzed, expressing the results as mg Trolox equivalent (TE)/100 g, using the standard curve developed according to the methodology of Miller et al. [24]. Particle size was determined on the Mastersizer 3000 analyzer (Malvern Instruments Ltd., Malvern, Worcestershire, UK), expressed in 10, 50, and 90 % percentiles ( $D_{10}$ ,  $D_{50}$ , and  $D_{90}$ , respectively). The color was determined in CIE-  $L^*a^*b^*$  coordinates using an X-Rite spectrophotometer, model SP62, illuminant  $D_{65}$ ,  $10^\circ$  observer as a reference, and the specular component included. From the reflection spectra, the color coordinates  $L^*$  (luminance),  $a^*$ , and  $b^*$  are the green-red and yellow-blue chromaticity, respectively, were obtained [25]. Finally, Yi was determined as the ratio between the total YP solids obtained and the feed suspension and SD solids.

## 2.4. Data analysis and experimental optimization

The results were analyzed by ANOVA with a confidence level of 95 %, using Design Expert 10.0 software (Stat-Ease, Inc USA). The effect of the independent variables was determined using the multiple regression method to predict the independent variables' linear, quadratic, and interaction coefficients in the response surface models. A polynomial model of order 2 (Equation (1)) was used, where Y is the dependent variable;  $\beta_0$  is the model constant;  $\beta_A$ ,  $\beta_B$ ,  $\beta_C$ ,  $\beta_D$ , and  $\beta_E$  are the linear coefficients;  $\beta_{A2}$ ,  $\beta_{B2}$ ,  $\beta_{C2}$ ,  $\beta_{D2}$ , and  $\beta_{E2}$  are the quadratic coefficients; and  $\beta_{AB}$ ,  $\beta_{AC}$ ,  $\beta_{AD}$ ,  $\beta_{AE}$ ,  $\beta_{BC}$ ,  $\beta_{BD}$ ,  $\beta_{BE}$ ,  $\beta_{CD}$ ,  $\beta_{CE}$ ,  $\beta_{DE}$  are the coefficients of the interactions of the independent variables. The adequacy of the models was performed using the lack of fit test and the regression coefficient ( $R^2$ ).

$$Y = \beta_0 + \beta_A A + \beta_B B + \beta_C C + \beta_D D + \beta_E E + \beta_A^2 A^2 + \beta_B^2 B^2 + \beta_C^2 C^2 + \beta_D^2 D^2 + \beta_E^2 E^2 + \beta_{AB} AB + \beta_{AC} AC + \beta_{AD} AD + \beta_{AE} AE + \beta_{BC} BC + \beta_{BD} BD + \beta_{BE} BE + \beta_{CD} CD + \beta_{CE} CE + \beta_{DE} DE \quad (1)$$

The SD process was experimentally optimized based on the ANOVA results, defining criteria, weights, and impacts for each dependent variable to achieve the desired quality attributes in the powdered product. The mathematical models were validated by comparing the theoretical values with those obtained from three trials at optimum conditions, determining the relative mean error (RME).

## 2.5. Scanning electron microscopy (SEM) and differential scanning calorimetry (DSC)

The microstructure of the YP (optimal conditions of the SD process) was determined with a scanning electron microscope (SEM) (JSM-5910, JEOL Ltd., Tokyo, Japan). The samples were coated with gold and overlaid on carbon tape. The micrograph analysis was performed under a high vacuum (10<sup>-6</sup> torr), 10 kV accelerating voltage, spot size 36, and at magnifications of 100×, 500×, 1000×, and 1500×. On the other hand, the glass transition temperature (Tg) for the YP was determined with differential scanning calorimetry (DSC) T.A. Instrument, Q2000, U.S.A. with the refrigeration unit operating up to -60 °C (T.A. Instrument refrigerated cooling system 90) and with a holding time of 1 min at -60 °C and a heating ramp of 10 °C/min from -60 °C to 160 °C.

**Table 1**  
YP properties according to experimental design.

Run	Independent variables					Dependent variables														
	AIT (°C)	AOT (°C)	ADS (rpm)	IN (%)	MD (%)	Xw (%)	$a_w$	S (%)	Hy (%)	TP (mg GAE/100 g)	DPPH (mg TE/100 g)	ABTS (mg TE/100 g)	D <sub>10</sub> (µm)	D <sub>50</sub> (µm)	D <sub>90</sub> (µm)	L*	a*	b*	Yi (%)	
1	150	80	20000	3	4	3.3	0.278	96.1	22.1	1309.5	2566.6	2389.0	12.5	26.1	93.7	78.8	5.1	16.4	66.9	
2	140	75	22000	5	5	2.8	0.273	90.3	21.5	915.8	1468.8	1012.6	14.5	29.3	112.0	82.6	3.1	19.1	56.0	
3	160	85	18000	3	5	3.2	0.274	97.8	22.0	1027.6	6489.4	2149.3	8.8	18.5	32.8	81.7	2.9	19.3	52.5	
4	140	75	18000	3	3	5.4	0.350	95.5	24.2	929.9	1854.1	1708.3	12.6	34.0	71.6	81.2	2.9	19.8	52.5	
5	160	80	20000	4	4	2.3	0.242	96.3	22.5	1019.9	2356.7	2230.9	16.6	35.9	114.0	77.5	6.8	20.6	51.5	
6	140	80	20000	4	4	2.9	0.256	97.7	20.5	1006.8	2489.3	2249.4	11.9	27.0	85.5	80.0	4.8	18.8	57.5	
7	140	85	22000	3	5	2.9	0.296	93.5	24.4	1486.2	7133.2	2724.9	17.1	28.1	130.3	81.4	2.8	19.1	59.3	
8	150	80	20000	4	4	2.0	0.231	91.6	19.1	1460.5	1972.3	2031.1	20.0	52.2	175.7	73.9	12.3	12.9	60.0	
9	160	85	18000	5	3	3.1	0.166	94.6	26.9	1025.6	6546.9	3320.2	13.7	33.1	82.5	67.4	13.5	23.4	54.4	
10	160	85	22000	3	3	4.3	0.309	95.7	23.5	1290.5	6914.2	2068.5	16.0	29.6	119.7	81.6	3.3	18.6	68.3	
11	150	80	20000	4	3	2.7	0.250	96.1	24.8	1600.7	2477.1	1803.2	18.8	36.8	102.4	76.0	8.6	18.0	77.5	
12	140	85	22000	5	3	4.3	0.368	99.6	27.0	1078.9	6567.9	1964.2	18.3	52.2	180.7	75.7	5.5	24.8	56.5	
13	150	80	20000	4	4	2.7	0.227	94.1	22.3	1424.2	2283.6	2265.2	11.5	26.4	108.4	75.0	8.9	19.5	63.0	
14	150	80	22000	4	4	2.7	0.272	95.4	20.0	1221.5	4441.5	1999.1	18.2	45.7	275.0	72.1	11.3	24.6	54.2	
15	150	80	20000	4	4	3.0	0.239	95.3	24.0	1185.1	2041.3	2037.5	11.9	27.8	118.0	76.1	8.2	20.8	69.9	
16	160	75	22000	3	5	2.9	0.241	95.2	25.4	1202.7	2395.5	2372.3	19.8	35.7	109.3	76.8	7.8	22.4	69.3	
17	150	80	20000	4	5	2.0	0.243	97.4	19.5	1583.5	2186.8	1601.5	18.3	35.3	104.0	80.7	5.1	17.3	63.5	
18	150	85	20000	4	4	3.1	0.282	93.2	23.6	1249.3	2381.1	1171.1	22.3	45.6	138.7	72.8	11.4	23.9	51.3	
19	150	80	20000	4	4	2.8	0.267	95.5	21.9	1401.5	2533.2	2221.0	14.9	34.3	123.3	76.5	6.9	22.5	62.7	
20	150	80	18000	4	4	0.9	0.232	96.0	20.3	1288.0	4567.4	2297.1	21.2	87.5	146.0	66.9	13.6	27.5	46.3	
21	150	75	20000	4	4	2.6	0.268	95.5	22.1	1193.6	2461.6	1836.8	21.2	58.4	122.7	74.8	9.5	24.9	46.2	
22	150	80	20000	5	4	2.6	0.256	96.6	23.9	1096.1	2345.4	1576.8	18.3	38.5	101.0	80.3	3.6	16.9	60.1	
23	160	75	22000	5	3	4.4	0.354	99.9	29.6	1237.7	6990.3	2362.5	15.4	30.7	82.1	77.1	4.9	24.3	42.4	
24	140	85	18000	5	5	2.0	0.230	95.9	20.2	1059.3	2255.9	2544.4	9.7	22.8	71.7	77.0	7.4	19.3	63.5	
25	160	75	18000	5	5	2.3	0.245	95.6	20.6	1108.3	2144.1	1695.8	13.1	27.3	52.2	76.6	7.6	20.2	55.0	
26	150	80	20000	4	4	3.0	0.248	93.7	25.3	1393.4	1707.3	2925.4	12.8	27.4	115.0	75.1	9.6	18.4	66.7	

### 3. Results and discussion

Table 1 presents the mean values of the YP dependent variables as a function of the independent variables, according to the experimental design evaluated. On the other hand, Fig. 1 presents the behavior of the response surface graphs of the dependent variables that showed statistical effects.

The microencapsulation process conferred YP mean values of  $X_w$  in the ranges  $(0.9 \pm 0.1 \%$  -  $5.4 \pm 0.3 \%$ ) and  $a_w$  between  $(0.166 \pm 0.006$ – $0.368 \pm 0.001)$ , being statistically affected ( $p < 0.05$ ) by ADS, and by AOT-AOT and ADS-IN interactions, respectively. These results are characteristic in powdered foods whose water content is strongly bound; moreover, they are microbiologically stable and with low rates of degradative reactions [26]. The incorporation of drying additives such as MD and IN significantly decreases  $a_w$  [27] due to the water binding to hydrophilic polymers; additionally, they contribute to the physicochemical stability of YP by increasing the glass transition temperature ( $T_g$ ).

The response surface plots show an unexpected behavior in the  $X_w$ , being higher with increasing ADS; however, it could be presenting increases in the feed flow to the SD to guarantee the temperature differences ( $\Delta T_{inlet - Outlet}$ ) inside the drying chamber, generating a decrease in the tangential velocity and larger droplets at the disk discharge, which will have a lower temperature profile towards the interior of the droplet during the residence time and lower water evaporation [28]. Significant effects of ADS on  $X_w$  have been reported with constant feed flows, allowing small droplets at high ADS, higher evaporation, and lower  $X_w$ . Additionally, the increase of solids in the feed due to additives reduces the free water content, facilitating drying; this situation is evident in the YP, mainly when the feed contains higher IN (5 %). The behavior of  $a_w$  was like  $X_w$  and dependent on the linear ADS-IN interaction that causes the lowest values to be reached at conditions of 18000 rpm –5% and 22000 rpm-3%, and the highest at 18000 rpm-3% and 22000 rpm-5%. Additionally, the quadratic interaction effect confers a bending behavior that minimizes  $a_w$  at approximately 80 °C OAT and 4.3 % IN. This situation could be attributed to a possible complex formation between MD and IN with native FOS of different degrees of polymerization and sugars present (sucrose, fructose, glucose) [29], which produces changes in the diffusional properties of water from within the droplet and in H2-bridge and dipole-dipole type forces [30].

On the other hand, the difference in molecular weights in the new food matrix regulates the movement of water within it [31]. It produces dry materials in an amorphous (metastable state), especially in SD, where the mass transfer occurs rapidly, not allowing crystallization of the solutes [30]. The  $a_w$  values ranged from 0.166 to 0.368, similar to those obtained by Gangta et al. [7] for dried

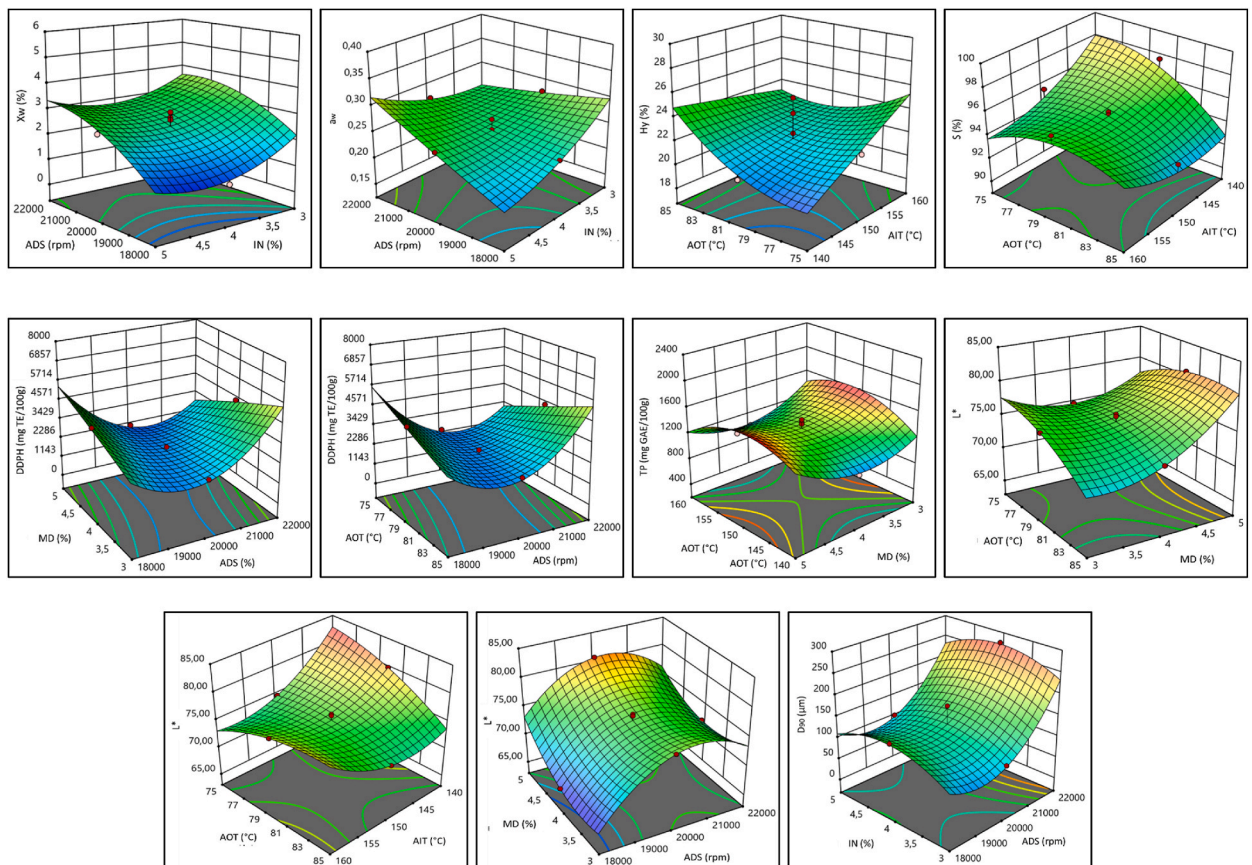


Fig. 1. Response surface plots of YP dependent variables as a function of independent variables.



yacon pulp obtained by different methods and slightly higher than those obtained by Jedlińska et al. [32] for spray-dried kiwifruit.

Hy and S of YP are not statistically affected by the microencapsulation process; however, their mean values ranged between 19.1–29.6 % and 90.3–99.9 %, respectively, characteristics that define microcapsules as a highly hygroscopic product [19] and with high reconstitution capacity, remaining homogeneously mixed with water, even though *IN* is less soluble than MD due to its semi-crystalline structure [12], probably the integration of the mechanical homogenization systems rotor-stator and pistons, influenced in the reduction of particle size of the feed to the SD, favoring the reconstitution of YP. The high affinity of the native components and additives of YP with the surrounding water demonstrates the presence of significant adsorption strength, H<sub>2</sub>-bridging, and dipole-dipole type [33], which could represent flow problems (stickiness between microcapsules) during storage [34], therefore, packaging with the lowest water vapor permeability will be required, to avoid the decrease of T<sub>g</sub> and thus, not to increase the cohesion phenomena and the overall instability of the product [35].

According to the response surface plots, the microencapsulation conditions that favored lower Hy were: AIT (140 °C), AOT (75 °C), ADS (22000 rpm), *IN* (3 %), and MD (5 %), and higher S: AIT (140 °C), AOT (75 °C), ADS (18000 rpm), *IN* (3 %) and MD (5 %). It is observed that low temperatures of AIT and AOT prevent possible hydrolysis of FOS and their consequent production of low molecular mass and low T<sub>g</sub> monomers (31 °C, 5 °C, 62 °C for glucose, fructose, and sucrose, respectively) [36]. Szlapak et al. [37] reported Hy values between 18 and 23 % in YP obtained by foam mat drying; on the other hand, Lin et al. [30] said lower Hy values for spray drying using FOS as wall material (9–11 %), concerning those found in this article. While S was similar to that reported by Lazzare et al. [38] in YP obtained by SD from ultrafiltered yacon permeate, were also identical to those obtained by Landim et al. [31], who used FOS as a spray-dried coating, reaching an S of 99.2 %. On the other hand, Shrivastava et al. [56] obtained higher S values (84 %) in microencapsulation of custard apple with the following spray drying conditions: inlet temperature of 135 °C, outlet temperature of 75 °C, and the maltodextrin content of 15 g/dL. Other works have concluded that S increases when MD is added to mango powder [19] and in amaranthus betacyanin pigments dried by SD (Cai & Corke, 2000) because it has open structures with at least two hydroxyl groups per molecule that allow it to bind water easily [39].

Fresh yacon roots in fresh state presented ABTS or DDPH values of 2.6 mmol TE/100 g, values that are in the range reported by Campos et al. [40]: 0.23–13.6 mmol TE/100 g db and lower than that reported in soursop pulp: 3.5 mmol TE/100 g [41] and in yacon concentrate (5.5–6 mmol TE/100 g) [6]. On the other hand, the mean values of DPPH and ABTS of YP fluctuated between 1468.8 and 7133.2 mg TE/100 g and 1012.6–3320.2 mg TE/100 g, respectively; however, the literature on the antioxidant capacity of dehydrated yacon is incipient; scarcely, Sousa et al. [21] reported in freeze-dried yacon flour values of 222 ± 2 mg ascorbic acid equivalents (AAE)/100 g db.

The DPPH results contrast the ABTS since no significant differences were found in the latter; however, the values obtained are relevant. The DPPH response surface plots showed that the behaviour of DPPH is highly dependent on the conditions imposed in the microencapsulation process, maximizing its values concerning the interactions thus: AIT-OAT (140°C–75 °C and 160°C–85 °C), AIT-MD (160°C–3% and 140°C–5%), MD-*IN* (5%–3% and 3%–5%), ADS-MD (22000 rpm–3% and 18000 rpm–5%) and ADS-AOT (22000 rpm–85 °C and 18000 rpm–75 °C). The ABTS did not show significant differences ( $p < 0.05$ ) concerning the factors evaluated, nor with their interactions. Conversely, the DPPH denotes an important statistical affectation due to the microencapsulation process: quadratic interaction of the ADS, which minimizes its values in the operating range of 20000–21000 rpm; in addition to the linear interactions AIT-OAT, AIT-MD, ADS-AOT, ADS-MD, and *IN*-MD. The last two interactions are mainly highlighted where the trends show the highest DPPH values; on the one hand, the ADS-MD interaction is consistent with the encapsulant mass/drop mass ratio, which offers good MD protection and is enhanced by the presence of *IN* and low temperatures [10]; however, with the ADS-OAT interaction, it is not clear at 22000 rpm–85 °C, since, the low mass/energy ratio of the droplet could favor a higher degradation of the antiradical activity or an increase of the antioxidant activity with increasing temperature, due to non-enzymatic browning reactions that generate compounds able to interact with the method radicals [42].

The mean TP values of YP ranged from 915.8 to 1600.7 mg GAE/100 g, being higher than those reported in yacon dried by different methods (145.10–309.72 mg/GAE/100 g) [7] but lower than those reported in spray-dried yacon (2313 mg GAE/100 g) [15]. The response surface plot showed a curvilinear behavior because of quadratic interactions of AIT and MD, denoting higher TP protection at intermediate temperatures (150 °C) and maximum and minimum MD conditions (5 and 3 %). Some authors have attributed these changes during the transformation processes of fruits or other processed food matrices, on the one hand, to factors associated with biogeographical aspects, soil, variety, primary production, and ripening stage [15], such as well as to those factors associated with the applied process. However, for the microencapsulation process by SD, several authors highlight a technology that preserves critical levels of phenolic components or other components with physiological activity [10].

In general, it is considered that a complex of the added MD-*IN* components is formed in YP with native components of the matrix: FOS-cellulose-hemicellulose-pectin [12,43], which exert a protective effect to different extents on TP and antioxidant capacity (ABTS and DPPH). Rivas et al. [44] reported TP values of 370.21 mg GAE/100 g in a mango and passion fruit juice encapsulated with a mixture of *IN*-MD and that it was even maintained during storage time thanks to the interactions of the matrix with the mix of encapsulants, which managed to protect the bioactive compounds of the product; on the other hand, Santos et al. [45] showed the effect of combining MD with gum Arabic to improve the encapsulation effect of phenols and compounds with antioxidant activity in sapote juice. Other studies have shown the protective effect of fructooligosaccharides and protein-based encapsulants. The addition of fructooligosaccharide reduces the water activity and hygroscopicity of the microcapsules, avoiding the oxidation of the bioactive compounds inside them [14,54]. Another mixture of carbohydrates with proteins showed good encapsulation properties in the study conducted by Castro et al. [55]. This study used an encapsulating agent based on maltodextrin and gelatin in clarified juice from purple cactus pear. This mixture of encapsulants resulted in the protection of bioactive compounds with antioxidant activity.

The colour parameter L\* varied between (66.91–82.58), finding an effect ( $p < 0.05$ ) of the interactions of the factors TSA-MD and

**Table 2**  
Regression coefficients and R<sup>2</sup> of the models for the dependent variables.

Coefficients	X <sub>w</sub>	a <sub>w</sub>	S	Hy	TF	DPPH	ABTS	D <sub>10</sub>	D <sub>50</sub>	D <sub>90</sub>	L*	a*	b*	Yi
β <sub>0</sub>	2.45	0.2469	95.19	21.91	1329.48	1329.48	2055.57	16.78	40.89	133.06	75.38	8.88	19.96	60.63
β <sub>A</sub>	-0.3060	-0.0070	-0.7100	1.01	6.56	6.56	-9.25	2.35	4.47	14.22	-1.26	0.9735	0.9115	-3.01
β <sub>B</sub>	0.2930	0.0065	-1.14	0.7550	27.86	27.86	-332.86	0.5665	-6.40	8.00	-0.9635	0.9880	-0.5135	2.55
β <sub>C</sub>	0.8895	0.0200	-0.2650	-0.1850	-33.25	-33.25	-149.00	-1.47	-20.87	64.50	2.58	-1.13	-1.43	3.95
β <sub>D</sub>	-0.3220	-0.0110	0.2650	0.9000	-106.70	-106.70	-406.08	2.91	6.20	3.63	0.7830	-0.7215	0.2650	-3.43
β <sub>E</sub>	-0.3435	-0.0035	0.6600	-2.68	-8.64	-8.64	-100.87	-0.2665	-0.7665	0.8165	2.37	-1.74	-0.3550	-6.99
β <sub>AB</sub>	0.5225	-0.0103	1.42	-1.84	-131.19	-131.19	16.62	-2.38	-9.15	7.02	3.59	-2.52	-1.52	-2.96
β <sub>AC</sub>	0.1830	0.0150	1.22	0.6045	26.74	26.74	-350.92	2.82	9.00	-30.47	1.27	-1.29	0.3915	-3.17
β <sub>AD</sub>	0.9387	0.0112	0.4905	-0.8380	10.35	10.35	49.98	-3.79	-19.30	6.83	-0.5577	0.7814	-0.6495	-2.83
β <sub>AE</sub>	0.6312	0.0025	-0.2695	0.4895	-95.95	-95.95	-545.50	-0.7661	-14.72	4.02	1.35	-0.1003	-1.53	6.96
β <sub>BC</sub>	-0.4478	0.0187	1.14	-0.0180	36.61	36.61	-38.72	4.68	23.35	5.66	1.77	-2.44	1.13	-7.68
β <sub>BD</sub>	0.2427	-0.0023	0.9180	-1.30	-62.90	-62.90	486.17	-2.11	-4.16	42.59	-1.09	0.8559	0.6715	-2.72
β <sub>BE</sub>	-0.0545	-0.0008	-0.1945	0.4245	-58.65	-58.65	78.39	-0.4811	-6.88	20.38	2.33	-1.48	-1.15	1.02
β <sub>CD</sub>	0.1720	0.0417	-0.0170	0.6270	15.37	15.37	-320.07	1.54	11.30	-3.96	-0.8085	-0.2478	1.76	-9.01
β <sub>CE</sub>	-0.5168	-0.0120	-3.11	2.52	10.38	10.38	-432.66	5.50	13.51	-6.92	-1.05	0.8362	0.5002	1.96
β <sub>DE</sub>	0.3100	0.0037	-2.96	-0.8155	-46.48	-46.48	-389.84	-2.39	-16.98	25.02	-0.3997	1.51	-1.71	6.81
β <sub>A</sub> <sup>2</sup>	0.3482	-0.0007	1.07	-0.0335	-288.95	-288.95	334.87	-4.12	-13.99	-36.39	3.35	-2.89	-1.01	-3.80
β <sub>B</sub> <sup>2</sup>	0.5692	0.0258	-1.50	1.38	-80.84	-80.84	-401.32	3.40	6.61	-5.51	-1.59	1.79	3.69	-9.62
β <sub>C</sub> <sup>2</sup>	-0.4993	0.0023	-0.1827	-1.34	-47.59	-47.59	242.86	1.33	21.17	74.32	-5.91	3.79	5.37	-8.13
β <sub>D</sub> <sup>2</sup>	0.6582	0.0173	0.4673	1.49	-99.48	-99.48	77.65	-2.98	-13.16	-38.84	4.17	-4.32	-4.03	5.15
β <sub>E</sub> <sup>2</sup>	0.0237	-0.0032	0.8623	0.6615	289.81	289.81	-202.89	0.1990	-9.36	-32.99	2.97	-1.88	-3.02	12.17
R <sup>2</sup>	0.9407	0.9739	0.808	0.8418	0.9321	0.9929	0.8281	0.6951	0.8120	0.9414	0.9902	0.9397	0.7563	0.8927
Model p-value	0.0663	0.011	0.528	0.405	0.088	0.0005	0.457	0.832	0.515	0.065	0.001	0.069	0.692	0.214
Lack of fit (p-value)	0.126	0.467	0.097	0.466	0.293	0.155	0.120	0.093	0.114	0.609	0.912	0.678	0.3976	0.052

TEA-TSA and linear effects of MD and ADS, obtaining lighter powders due to the white colour provided by the maltodextrin and low moisture content in the powder. Fig. 1 shows that an increase in TSA leads to an increase in the L\* parameter, due to the reduction in moisture content that exhibits a clearer sample. The encapsulation agent protects the natural pigments (5 % MD) from heat. On the other hand, the lowest L\* values were reached when the MD level was low, and TSA elevated, mainly due to the creation of dark compounds called melanoidins coming from dimer thermolysis; these appear when the higher PD oligomers are subjected to temperatures between 70 and 80 °C [7,38]. The TEA-TSA interaction shows that the lower temperatures evaluated produced the powder with a lighter shade. The main effect of ADS ( $p < 0.05$ ) on the L\* parameter was positive, observed with greater clarity ( $>L$ ) when the system operates at high ADS (22000). This condition probably decreased the residence time of the compounds inside the chamber avoiding their browning [46]. Our results are like those reported by Haas [47], where an increase in the colour parameter L\* is evident as the particle size decreases due to elevated light scattering in the samples. The results were similar to those obtained in blackberry encapsulated with polydextrose (15 %) due to the oxidation of bioactive compounds by the increase in drying temperature (160 °C) [57].

No significant differences ( $p > 0.05$ ) were found in the parameters a\* and b\*; the maximum and minimum values reached were a\* (13.61–2.76) and b\* (27.48–12.87), respectively, both positive parameters indicated a light cream shade. The parameters a\* and b\* were higher than those reported by Ref. [38], who obtained a yellow-greenish YP. The above could indicate that bioactive compounds such as  $\beta$ -carotene responsible for the yellow color in yacon pulp ( $>a^*$ ) were preserved, and chlorogenic acid and L-tryptophan, related to the dark green color that occurs when there is enzymatic oxidation.

YP had a particle size between (8.1–23.0  $\mu\text{m}$ ), (16.5–91.4  $\mu\text{m}$ ) and (31.6–355.0  $\mu\text{m}$ ) for the D10 D50 and D90 percentiles, respectively. YP was coarser compared to orange juice powder (5 and 65  $\mu\text{m}$ ) [46] and finer than coconut powder (46.3  $\pm$  3.0–1153.2  $\pm$  208.3  $\mu\text{m}$ ) [48], both obtained by SD. The D90 percentile presented significant linear effects ( $p < 0.05$ ) concerning the ADS, its average value being 133.08  $\mu\text{m}$ . In the literature, it has been reported that an increase in ADS causes a decrease in particle size due to the production of droplets dispersed with a thinner film [49]; however, in this research, the increase in ADS produced larger particle sizes. The particle size distribution of the YP presented a bimodal behaviour denoting its variability. This effect could be explained by changes in the feed flow (changes in the mass ratio of atomizing air to liquid) to maintain the constant temperatures of the experimental design. Smaller particles adhere strongly to the surface of larger particles, causing agglomerates and flow problems [50]. This defect can be controlled by avoiding the formation of rough and cracked surfaces that occur during particle collapse on drying, i.e. when there is a slow film formation process [38]. The same structure was reported in mango powder encapsulated with 12 % gum Arabic [19]. The MD technique promotes the development of smooth surfaces yielding improved particle distribution [19].

Conversely, an increase in temperature and drying rate elevated the film formation rate on the droplet surface. The drying agent significantly affects the particle size because it increases the viscosity and the feed flow rate, consequently, the average droplet diameter. Likewise, a high inlet temperature contributes to forming a rigid and impermeable layer, followed by forming vapor bubbles that promote droplet expansion [46].

The Yi had no significant differences ( $p > 0.05$ ) because of dry conditions, although it presented values between 42 and 78 %. The values obtained are higher than the yields reported for coconut powder (31.3–52.6 %) [48]. The lowest Yi values were obtained at TEA of 160 °C, probably due to low Tg monosaccharides, which at temperatures above their Tg become sticky and thus adhere to the inner wall of the drying chamber [51]. In a study of IN powder extracted from Jerusalem artichoke tubers, they found that an increase in inlet temperature from 170 °C to 190 °C improved the process yield from 87.23 to 90.59 respectively, probably, with higher AIT powders are produced with higher Tg, larger particle sizes due to higher bulk density leading to better yields [49] due to fewer particles being entrained through the Venturi. Likewise, increasing the total feed solids (10°Brix to 30°Brix) improved Yi (67.8–88.1) [52]. The same findings were reported by Igual et al. [53] on the Yi of luand lo pulp. For its part, Islam, and Chen [9] suggested that Yi should be elevated by the feed rate rather than by increasing ADS; however, this is not applicable in matrices with high sugar content, as stickiness problems could occur.

**Table 3**

Experimental and predicted values by the mathematical models of the YP dependent variables.

Variable	Experimental value	Value predicted by the model	RME (%)
Xw (%)	2.44	2.89	15.6
$a_w$	0.22	0.235	12.8
TP (mg GAE/100 g)	1228.2	1513.9	18.9
DPPH (mg TE/100 g)	5192.3	6450.9	19.5
ABTS (mg TE/100 g)	2295.9	3320.2	30.8
S (%)	96.9	96.9	0.01
Hy (%)	23.0	19.9	15.8
D <sub>10</sub> ( $\mu\text{m}$ )	10.6	10.5	1.1
D <sub>50</sub> ( $\mu\text{m}$ )	23.4	14.5	61.5
D <sub>90</sub> ( $\mu\text{m}$ )	169.3	166.3	1.82
L*	80.54	81.1	0.69
a*	5.15	3.4	51.5
b*	17.4	16.0	8.6
Yi (%)	72.9	70.6	3.2



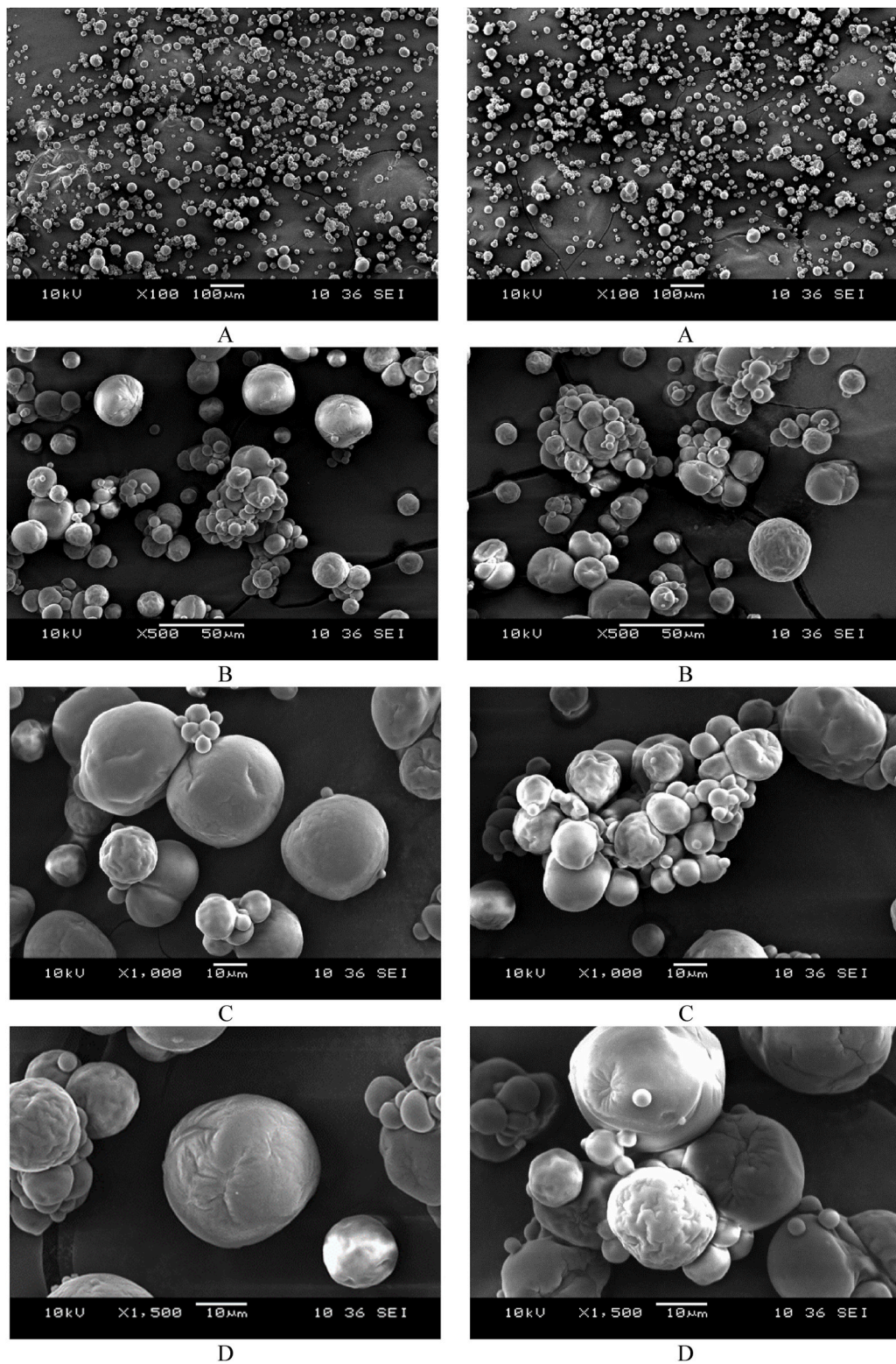


Fig. 2. SEM micrographs of YP under the optimal conditions of the SD process at  $100\times$  (A),  $500\times$  (B),  $1000\times$  (C) and  $1500\times$  (D).

### 3.1. Modeling and experimental optimization of the SD process

ANOVA evaluated the fit of the response surface models, test of lack of fit, and regression coefficients ( $R^2$ ), which are presented in Table 2. The quadratic regression models with  $p < 0.05$  and those with the best regression fit ( $R^2 > 0.9$ ) were  $a_w$ ,  $L^*$ , and DPPH, indicating that with 95 % accuracy, these variables can be explained as a function of the drying conditions evaluated, showing good agreement between the results predicted by the model and those observed within the experimentation. The  $R^2$  fit values for the variables  $b^*$ ,  $D_{10}$ ,  $D_{50}$ , ABTS,  $Y_i$ ,  $S$ , and  $H_y$  were  $< 0.9$ , perhaps due to several factors not controlled for in the research: variation in yacon root shades, particle aggregation during drying, among others, the lack of fit test, resulted in no significant p-value ( $p > 0.05$ ) in terms of the response variables evaluated, indicating adequate models to describe the data behavior.

The experimental optimization was conducted, considering the adjustment models of the statistically significant dependent variables. With  $R^2 > 0.9$  of the SD process, maximizing the color parameter  $L^*$  (lower browning), DPPH (higher antioxidant capacity with benefits in consumer health),  $D_{90}$ , and  $Y_i$  (lower losses); on the other hand,  $a_w$  was minimized (better stability), the other criteria (ABTS, FT,  $a^*$ ,  $b^*$ ,  $D_{10}$ ,  $D_{50}$ ,  $X_w$ ,  $S$ , and  $H_y$  were not fixed, since their fluctuations were not very large and had no statistical differences. Under this context, the optimum operating conditions were: IN = 3 %, MD = 5 %, TEA = 143.7 °C, TSA = 80.1 °C, ADS = 22000, with a desirability of 84.2 %. Table 3 compares the experimental values of the yacon powder response variables at the optimum point with the theoretical values obtained from mathematical models, observing a good approximation between them with RME values  $< 5$  %.

### 3.2. Microstructure and glass transition temperature

Fig. 2 presents the SEM micrographs at different magnifications (100×, 500×, 1000× and 1500×) of the YP, illustrating its microstructure under the optimal conditions of the SD process. Fig. 2A show individual particles with a spherical shape and sizes less than 25 μm, and a variety of groupings of small particles that form conglomerates with sizes less than 100 μm. This microstructure, a result of the YP's cohesive characteristics due to the high particle-particle interactions at the low levels of humidity that reach the outlet of the SD (2.4 %), is a significant finding. This microstructural behavior becomes more pronounced at higher magnifications (Fig. 2B, C and 2D), where individual particles and conglomerates of different sizes (polydisperse) exhibit areas of smooth surface and other areas with microstructural collapse (shrinkage) without fracturing (non-porous). This collapse, primarily caused by the thermal stress during the sudden evaporation of water from the interior, is a key factor in the YP's behavior, and a crucial aspect of our research [58].

On the other hand, this behavior may be due to the low  $T_g$  (Fig. 3) that the YP presented (21.13 °C), which explains a gradual loss of the glassy amorphous state (non-thermodynamic solid) at the same process outlet conditions, which can intensify during storage at medium and high temperatures. Under these conditions, for better conservation of the YP, packaging with low permeability to water vapor is recommended not to promote physicochemical destabilization, stickiness, and agglomeration. On the other hand, it is recommended that its storage be approximately 20 °C below the  $T_g$  [59] to slow its transition to the rubbery state. Therefore, refrigerated conditions would be the most appropriate.

The literature reports little information on the morphology of YP. Szlapak-Franco et al. [60] inform the scanning electron microscopy of particles in yacon juice powder and concentrated yacon juice obtained by drying on a foam mat at different conditions. The samples presented an irregular and porous structure with a large number of cavities.

Cortés et al. [61] report spherical particles with some degree of surface roughness or microstructural collapse in the form of cracks

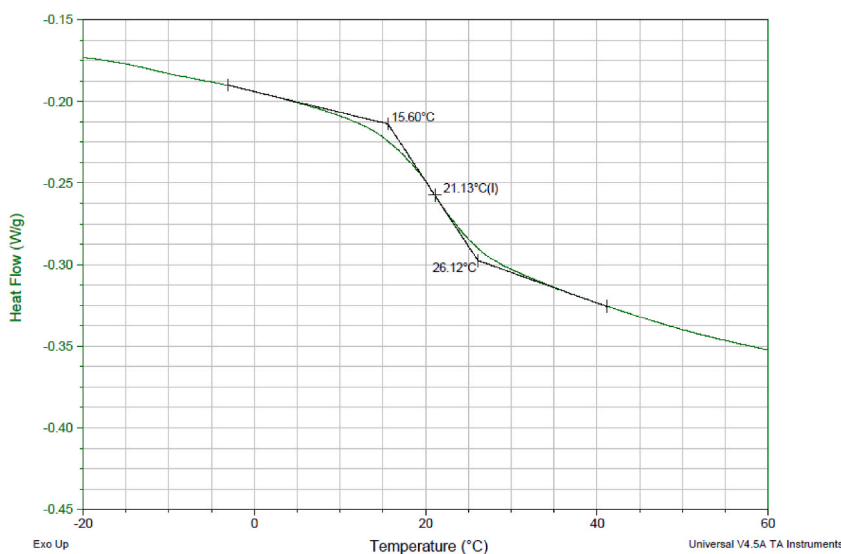


Fig. 3. Glass transition temperature of YP under the optimal conditions of the SD process.

from concentrated sugar cane juice powder previously fermented with kefir granules, obtained by spray drying. Other investigations have reported characteristics similar to those found in the present investigation: Cardona et al. [62] (pineapple powder), Islam et al. [63] (pink guava powder), among others.

#### 4. Conclusions

The SD is a suitable technology for generating value in the yacon agri-chain; however, the YP obtained is a complex matrix with a high affinity for water, which favors adsorption phenomena and could lead to flow problems during storage (stickiness). A reformulation of the colloidal feed suspension to the SD is recommended, for example, the addition of high molecular weight solutes that increase the  $T_g$  and contribute to the formation of amorphous microstructures and low glycemic index. On the other hand, adding proteins that modify the atomized particles' surface properties and using an anti-compaction agent alone or mixed could be favorable. However, the effect of the process on the color of the yacon microcapsules would have to be evaluated in detail because Maillard reactions could occur above 90 °C.

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#### Data availability statement

Data included in article/supp. material/referenced in article.

#### Ethics declarations

Review and/or approval by an ethics committee was not needed for this study because I don't work with humans or animals.

#### CRediT authorship contribution statement

**María Isabel Arango-Torres:** Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Data curation. **Misael Cortés-Rodríguez:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Esteban Largo-Ávila:** Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis, Data curation, Conceptualization. **Manuela Gallón-Bedoya:** Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Rodrigo Ortega-Toro:** Writing – review & editing, Writing – original draft, Validation, Data curation, Conceptualization.

#### Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Misael reports financial support, administrative support, equipment, drugs, or supplies, and travel were provided by Ministerio de Ciencia y Tecnología de Colombia. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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