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CROP SCIENCE

Recovery of bioactive compounds from an agro-industrial waste: extraction, microencapsulation, and characterization of jaboticaba (*Myrciaria cauliflora Berg*) pomace as a source of antioxidant

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Abstract: This study aimed to evaluate the extraction of bioactive compounds from jaboticaba pomace, produce microcapsules by spray dryer technique, and characterize antioxidant compounds. A factorial experimental design was used in the extraction step. Maltodextrin (DE 10) was used as an encapsulating agent, in a ratio of 1: 1 (w/w), in the microencapsulation process. It was observed the increase of all bioactive compounds analyses comparing jaboticaba pomace with the extract. ATR-FTIR spectroscopy showed a vibrational stretching aromatic ring (1718 – 1731 cm^{-1}) typical for anthocyanins. The Gaussian deconvolution presented extract peak area 7.56% higher than pomace. The encapsulating agent protected anthocyanins during the drying process. Microencapsulation of bioactive compounds from jaboticaba pomace can be useful for food applications whereas they are a rich source of antioxidant compounds. Moreover, the use of agro-industrial waste is promising linked to the use of clean technology as water as an antioxidant extractor.

Key words: natural antioxidants, encapsulation, spray drying, ATR-FTIR.

INTRODUCTION

Jaboticaba *(Myrciaria cauliflora* Berg) is a native and popular fruit from Brazil. Mainly, jaboticaba peel has been reported to contain a great number of phenolic compounds, such as ellagic acid and gallic acid, cyanidin-3-glucoside and delphinidin 3-glucoside, flavonoids and tannins (Batista et al. 2018, Lamas et al. 2018, Plaza et al. 2016).

It has been estimated that the fruit processing generates around 20-60% waste. Several studies highlight bioactive compounds extraction from fruits pomace as a way to reuse the amount of waste produced by the agro-industry. Thus the use of agroindustrial residues as jaboticaba pomace can be feasible for the development of functional foods (Amaya-Cruz et al. 2015, Kowalska et al. 2017, Machado et al. 2018).

Water is a clean solvent, known as economical and environmentally safe to extract bioactive compounds from jaboticaba pomace (Ivanovic et al. 2014, Reátegui et al. 2014, Santos et al. 2017b).

The microencapsulation of functional compounds can be an applicable process to improve the stability of bioactive compounds. The process consist of cover the main compounds with food-grade, safe and biodegradable wall materials (Ye et al. 2018).

Maltodextrin has been used for microencapsulation using spray drying technique, mainly hydrosoluble compounds for food application (Ramakrishnan et al. 2018). The spray drying technique transforms a liquid solution, suspension or emulsion into a dried particle (Ramos et al. 2019).

In this context, the objective of this study was to evaluate, through experimental design, the best conditions for extraction of bioactive compounds from jaboticaba pomace, afterward to produce microcapsules by using a spray dryer and characterize antioxidant compounds.

MATERIALS AND METHODS

Materials

Jaboticaba (*Myrciaria cauliflora* Berg) pomace was purchased from one batch from a producer of Paraibuna-SP, Brazil, and kept frozen (-18ºC) until use. Maltodextrin (M) DE10 was provided by Cargil® (Campinas-SP). The other reagents used were of analytical grade from SigmaAldrich®.

Experimental design

The ultrasound-assisted extraction (UAE) was conducted in an ultrasonic cleaner (Ultracleaner 1650 Unique, 40 kHz frequency, 120 Watts RMS power). The conventional extraction (CE) was conducted in a conventional bath (Nova Orgânica).

A factorial experimental design (2^2) including four points and three repetitions at the central point, totaling twelve experiments was used for optimization of extraction of the compounds from jaboticaba pomace. The ratio used in both extractions was 1:2 (w/v), therefore, jaboticaba pomace (JP) was diluted in water at a concentration of 500 mg mL^{-1}. The extraction variables included, extraction time (X_1= 15 or 45 min) and ultrasound absence or presence (X_2= 0 or 100 %), with temperature fixed at 60°C (Santos et al. 2017b). The response parameters included the content of phenolic compounds, anthocyanins, flavonoids and antioxidant activity.

The experimental data were fitted to the second-order polynomial model to obtain the regression coefficients (β). The generalized second-order polynomial model (equation 1) was used in the response surface analysis.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X + \sum_{i=1}^{k} \beta_{ii} X_{ii}^2 + \sum_{i}^{k-1} \sum_{j}^{k} \beta_{ij} X_i X_j \quad (1)$$

Where, Y is the response variable, X_i and X_j are the independent variables, and k is the number of tested variables (k=2). The regression coefficient is defined as β_0 for intercept, β_i for linear, β_{ii} for quadratic and β_{ij} for cross product term.

The analysis of variance (ANOVA) was used to determine individual linear and interaction regression coefficient using the statistical program STATISTICA version 7.0. Response surface graphs were applied to visualize the simultaneous effect of each variable on each response parameter, the significance of all the terms of the polynomial equation was analyzed statistically ($p<0.05$).

Samples and encapsulation of bioactive compounds

Initially, the pomace was defrosted. Considering the results of experimental design, jaboticaba pomace (JP) was diluted in water at a concentration of 500 mg mL^{-1} and the conventional extraction was conducted in a conventional bath at 60 °C for 45 min.

In order to produce the microcapsule (JM) maltodextrin (M) was mixed to the extracts 1: 1 (w/w), by using mechanical agitation (Ferrari et al. 2012). The samples JE and JM were dried in a spray dryer using the conditions: inlet drying air temperature 175 °C and outlet 105 °C;

Atomization pressure: 4 bar; Average drying air flow: 3.5 $m^3.h^{-1}$; Average feed rate: 0.5 $L.h^{-1}$ in Buchi B-191 Mini Spray-dryer equipment (Valduga et al. 2008).

JP was frozen for 48 h at -10°C and subsequently submitted to freeze drying for 2 days to ensure complete drying (freeze L108, Liobras). The dried samples were stored in plastic containers and kept freezing (-18°C) for future analysis.

Total phenolic compounds (TPC)

The analyses of TPC was realized using a spectrophotometric assay (Pierpoint 2004, Singleton & Rossi 1965). The absorbance was measured at 725 nm. The calibration curve was performed with gallic acid. Results were presented in µg of gallic acid equivalent (GAE) mg^{-1} of product.

Total monomeric anthocyanins (TMA)

The total monomeric anthocyanins was determined using the differential pH method (Lee et al. 2005). The absorbance was verified at 520 and 700 nm. Results were expressed in µg cyanidin-3-glucoside mg^{-1} product, according to equations 2 and 3.

$$AT = (ABS520nm - ABS\ 700nm)pH\ 1.0 - (ABS\ 520nm - ABS\ 700nm)pH\ 4.5 \quad (2)$$

$$\text{Anthocyanins (TMA)} = [(AT \times PM \times 10^3)/ \varepsilon \times \lambda] / C \quad (3)$$

Where: MW = 449.2 g. mol^{-1} (molar mass of cyanidin-3-glucoside); 10^3 = conversion factor from g to mg; ε = 26900L. mol^{-1} (molar absorptivity of cyanidin-3-glucoside); λ = 1 cm (optical length of the cuvette); C= sample concentration.

Total flavonoids (TF)

The determination of total flavonoids (TF) was performed using a spectrophotometric assay, which uses aluminum chloride ($AlCl_3$), sodium nitrite ($NaNO_2$) and sodium hydroxide (NaOH) (Alothman et al. 2009). The absorbance was measured at 510 nm. Quercetin was used to the calibration curve. Results were presented in µg quercetin equivalent (QE) mg^{-1} product.

Antioxidant activity by the radical sequestration method DPPH (2,2-diphenyl-1-picrylhydrazine)

The reduction of the stable radical DPPH was measured by spectrophotometric assay (Thaipong et al. 2006). The absorbance was verified at 515 nm. The efficiency of the sequestering activity was calculated using equation 4. Trolox was used as the standard for the calibration curve, the results were expressed in µM Trolox equivalent (TE) mg^{-1} product.

$$\text{Efficiency of free radical sequestration (\%)} = [(A_{control} - A\ s_{ample})/ A_{control}] \times 100 \quad (4)$$

Where: A Control: Absorbance of negative control; A Sample: sample absorbance average.

Antioxidant activity by ABTS method

The antioxidant activity on the ABTS method was performed using a colorimetric assay. ABTS (2,2' – AZINO - BIS (3-ethylbenzo - thiazoline -6-sulfonic acid) diammonium salt and potassium persulfate ($K_2S_2O_8$) reagents was used (Nenadis et al. 2004). The absorbance was measured at 734 nm. A calibration curve was prepared using a standard solution of trolox. The results were presented in µM Trolox equivalent (TE mg^{-1} product.

Ferric Reducing Antioxidant Power (FRAP) Assay

The antioxidant analysis using FRAP method was performed mixing the samples directly with distilled water and FRAP reagent (Pulido et al. 2000). The absorbance was measured at 595nm after 30

min of incubation at 37°C. Results were presented in μM Trolox equivalent (TE) mg^{-1} product.

Color analysis

The color was measured by using a portable Minolta® CR400 colorimeter, with an integration sphere and angle of view of 3°, that is, d/3 illumination and D65 illuminant. The system used was CIELAB (L*, a*, b*, C and H°).

ATR-FTIR analysis

A Fourier transform infrared spectrometer (Vertex 70v, Bruker, Germany) equipped with an attenuated total reflectance accessory (Platinum, Bruker, Germany) (ATR-FTIR) was used to determine the phenolic compounds and anthocyanins present in the samples. The samples were placed on the ATR crystal, maintaining contact with the crystal throughout the measurement. Each spectrum was an average of 128 scans, with a spectral resolution of 4 cm^{-1}. The spectral measurement range was 4000 to 400 cm^{-1}. Gaussian deconvolution was applied in spectra to obtain the vibrational modes overlapped in the typical anthocyanins bands using OriginPro 8 software.

Morphology by scanning electron microscopy (SEM)

The particle morphology was realized using a scanning electron microscope (JEOL model JSM-6060 LV). Metal support with a double-faced tape of carbon was used to fix the samples, which was covered with gold. Visualization was realized in increases of 250 to 10000 times, with an excitation voltage of 12.5 kV.

Statistical analysis

All analysis were submitted to variance and Tukey's test for the minimum significant difference ($p<0.05$) between averages using the statistical program Sisvar 5.6. The calibration curves for the antioxidant analyses were plotted in Graph Pad Prism 5 software, and the experimental design by using the statistical program STATISTICA version 7.0.

RESULTS AND DISCUSSION

Experimental design

Figure 1 shows the response surface for bioactive compounds extraction and Table I presents ANOVA estimates effect to factorial experimental design (2^2) from jaboticaba pomace. As shown in Table I and Figure 1, time (X_1) was significant ($p<0.05$) for all response variables. Ultrasound presence (X_2) was not significant. The interaction between time and ultrasound (X_1X_2) were significant for TPC, TF and antioxidant activity by DPPH method.

The results suggested that the condition of 45 min and ultrasound absence with temperature fixed in 60°C, could be considered suitable to obtain the optimized extraction of bioactive compounds from jaboticaba pomace, whereas ultrasound presence (X_2) was not significant ($p<0.05$) for all response variables.

It was observed that the ultrasound-assisted extraction (in the used conditions) without another extraction procedure combined was not efficient to extract phenolic compounds and anthocyanins from jaboticaba pomace. Probably, because a low ultrasonic frequency equipment (40 kHz) was not feasible to extract the compounds. Literature suggested degradation of certain anthocyanins due to the ultrasonic frequency (40 kHz) because these compounds are highly sensitive (Santos et al. 2010). In another study, it was observed that some bioactive compounds, as ellagic acid, was not significantly affected by ultrasound-assisted extraction from jaboticaba pomace in an ultrasonic cleaner bath (25 kHz) to 60 min (Rodrigues et al. 2015).

Table I. ANOVA effect estimates to factorial experimental design (2^2) response surface methodology to jaboticaba pomace.

Factor	TPC		TMA		TF		DPPH		ABTS	
	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value	Coefficient	p-Value
Intercept										
0	3.7345	<0.0000*	0.0577	<0.0000*	4.9649	<0.0000*	2.6843	<0.0000*	50.5432	<0.0000*
Linear										
X_1	0.4949	<0.0000*	0.0050	0.0281*	0.7644	<0.0000*	0.6884	<0.0000*	10.1956	<0.0000*
X_2	-0.0167	0.8139	0.0035	0.1062	0.1258	0.3610	0.1105	0.1386	2.0440	0.1872
Interaction										
X_1X_2	0.1999	0.0098*	0.0029	0.1828	0.3020	0.0363*	0.1637	0.0333*	2.7473	0.0813

X_1 and X_2 presents extraction time and ultrasound absence or presence, respectively. *Significant at p<0.05. TPC: total phenolic compounds; TMA: total monomeric anthocyanins; TF: total flavonoids.

In another research the best condition for the extraction of phenolic compounds and monomeric anthocyanins from jaboticaba pomace was found from medium to high extraction times (40 to 60 min) (Rodrigues et al. 2015). A recent study showed that the best condition to extract phenolic compounds from jaboticaba pomace was 80°C and 45 min extraction time in a conventional method (Rodrigues et al. 2018a).

Antioxidant and color analyses

Table II shows the analyses of total phenolic compounds, total monomeric anthocyanins, antioxidant and color of JP, JE and JM samples. It was observed the increase of TPC, TMA, TF, DPPH, ABTS and FRAP by comparing jaboticaba pomace (JP) with extract (JE). This relation was founded in other studies (Rodrigues et al. 2018b, Santos et al. 2017b), the use of spray dryer with high temperature improve the antioxidant activity due to the formation of phenolic compounds resulting from degradation of others compounds (Pitalua et al. 2010).

There are few reports about this fruit, in this case, our discussion was to comparing jaboticaba pomace with another fruit pomace. Analyzing data of JP and comparing TPC with other fruit pomace, in this present work it was found 11.41 ± 0.91 μg GAE mg^{-1} of jaboticaba pomace, literature has shown around 7.5 mg GAE g^{-1} to apple pomace, 8.0 mg GAE g^{-1} to blueberry pomace, 24.0 mg GAE g^{-1} to raspberry pomace, and mg GAE g^{-1} to cranberry pomace (Gouw et al. 2017).

By comparing jaboticaba pomace with blackberry pomace, it can be observed that jaboticaba has higher TPC (11.41 ± 0.91 μg GAE mg^{-1}) than blackberry pomace (8.36 μg GAE mg^{-1}) (Santos et al. 2017b). On the other hand, jaboticaba pomace has lower TMA (0.16 ± 0.03 μg cyanidin-3-glucoside mg^{-1}) than blackberry

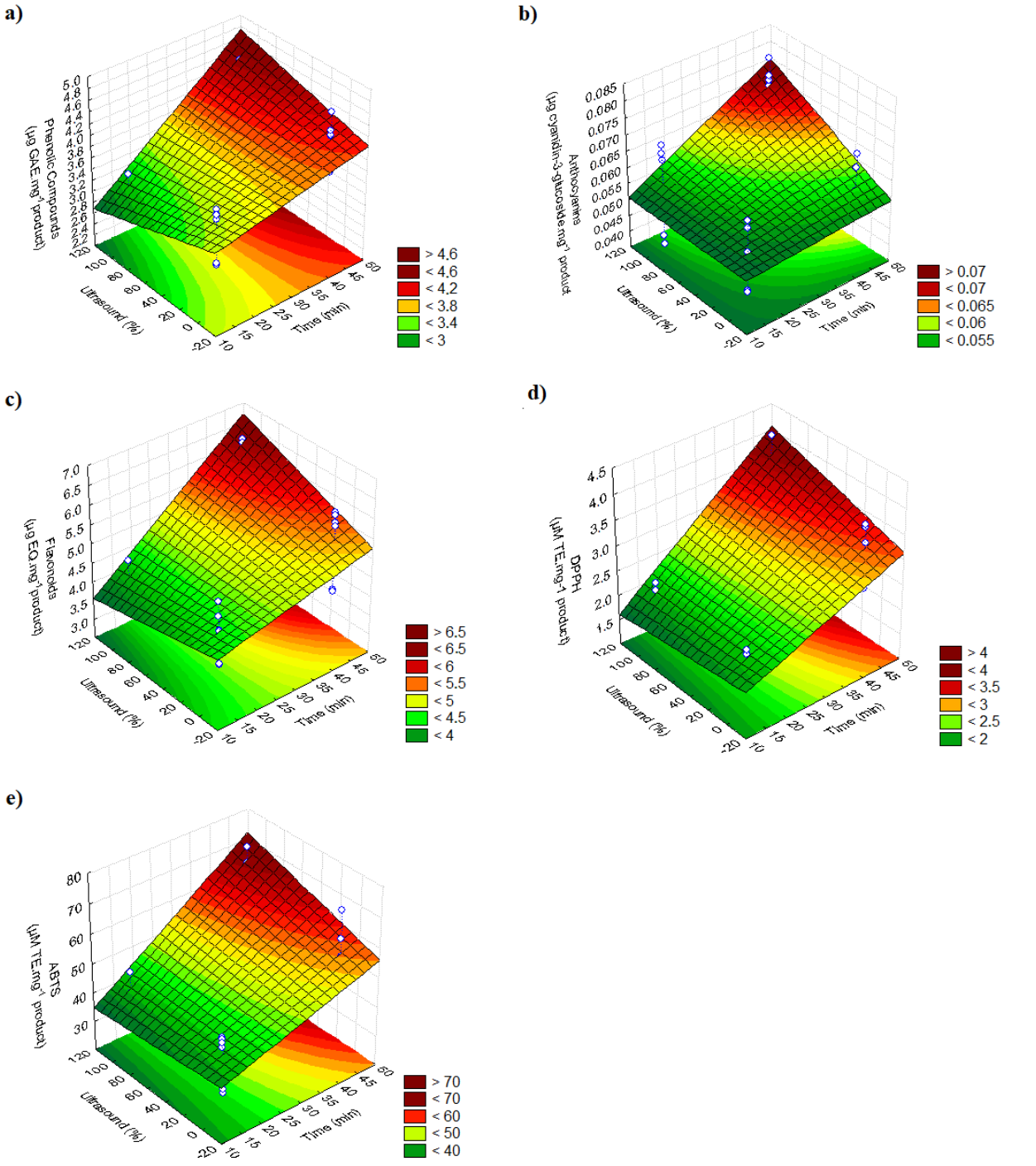

Figure 1. Response surface for bioactive compounds extraction from jaboticaba pomace. (a) TPC: $Y = 0.4949X_1 - 0.0167X_2 + 0.1999X_1X_2$; $R^2 = 0.9443$; (b) TMA: $Y = 0.0050X_1 + 0.0035X_2 + 0.0029X_1X_2$; $R^2 = 0.9761$; (c) TF: $Y = 0.7644X_1 + 0.1258X_2 + 0.3020X_1X_2$; $R^2 = 0.9678$; (d) DPPH: $Y = 0.6884X_1 + 0.1105X_2 + 0.1637X_1X_2$; $R^2 = 0.8084$; (e) ABTS: $Y = 10.1956X_1 + 2.0440X_2 + 2.7473X_1X_2$; $R^2 = 0.8985$.

pomace (0.37± 0.01 µg cyanidin-3-glucoside mg^{-1}) (Santos et al. 2017b).

The high amount of TPC and TF could be the reason for the higher antioxidant activity of jaboticaba pomace. A study with camu-camu found 7.791^{b}± 0.029 mg GAE g^{-1} for TPC, 7.295 ± 0.897 mg QE g^{-1} for TF, 74.145 ± 0.750 mmol TE g^{-1} for ABTS and, 222.000 ± 0.562 mmol TE g^{-1} for FRAP (Rodrigues et al. 2020). All these values were lower than jaboticaba pomace (JP - Table II).

Evaluating JM and JE in relation to TMA (Table II) it was observed that the encapsulating agent protected anthocyanins during the drying process, presenting the same values, since JM present only 50 % of the extract. Microcapsules samples presented around 50% of value in analyses of TPC, TF, DPPH, ABTS and FRAP, as expected. The literature showed than microencapsulation with maltodextrin is efficient for the protection of the bioactive compounds against light, temperature and pH (Ozkan et al. 2019, Rodrigues et al. 2018a, b). Also, the spray drying technique has a great impact on the encapsulation characteristics including stability (Santos et al. 2019, 2017a).

Another study showed higher values of TMA and TPC from jaboticaba pomace extract and microcapsules, it was found in TMA 3.22 µg cyanidin-3-glucoside mg^{-1} and 2.73 µg cyanidin-3-glucoside mg^{-1} for JE and JM, respectively. Besides 115.4 µg GAE mg^{-1} and 52.44 µg GAE mg^{-1} for JE and JM, respectively. This can be explained by the lyophilization method, that did not use high temperatures to dry, and preserve the bioactive compounds from temperature degradation (Rodrigues et al. 2018b).

Regarding color analysis, it was observed JM are lighter than JE, probably due to the use of maltodextrin that provided a significant increase in the value of L*. The same tendency was observed in chromaticity (C). It was observed that when comparing a*, that represents red intensity, the JM values are higher than JE, demonstrating that maltodextrin encapsulation protected the coloring compounds of samples during the drying process.

Table II. Analyses of total phenolic compounds, total monomeric anthocyanins, antioxidant and instrumental color of BP, BE, BM, JP, JE and JM samples.

	JP	JE	JM
TPC[I]	11.41^{e}±0.91	76.77^{a}±5.33	36.39^{c}±2.07
TMA[II]	0.16^{c}±0.03	0.46^{b}±0.07	0.56^{b}±0.01
TF[III]	29.32^{d}±8.34	151.56^{b}±6.10	68.56^{c}±3.39
DPPH[IV]	5.09^{e}±0.11	38.93^{a}±0.29	19.57^{c}±0.56
ABTS[V]	119.00^{e}±16.31	840.56^{a}±10.60	390.06^{c}±15.72
FRAP[VI]	333.98^{e}±5.87	2616.54^{a}±13.68	1398.21^{c}±20.71
L*	29.69^{c}±2.66	19.88^{de}±1.36	54.25^{a}±3.19
a*	14.42^{d}±0.15	13.02^{d}±0.31	28.42^{b}±0.49
b*	9.50^{e}±0.35	13.12^{b}±0.39	11.41^{cd}±0.19
C	17.27^{d}±0.14	18.49^{d}±0.16	30.62^{b}±0.53
H°	33.37^{b} ±1.18	45.20^{a}±1.47	21.88^{d}±0.13

Means followed by the same letters on line did not differ among themselves by Tukey's test (p<0.05). JP: jaboticaba pomace; JE: jaboticaba pomace extract (100% extract); JM: jaboticaba pomace microcapsule (50% maltodextrin + 50% extract). Iµg GAE mg-1product; IIµg cyanidin-3-glucoside mg-1 product; IIIµg QE mg-1product; IV, V and VIµM TE mg-1 product. TPC: total phenolic compounds; TMA: total monomeric anthocyanins; TF: total flavonoids. * indicates the color system CIE.

ATR-FTIR analysis

The JP, JE, JM and M samples were characterized by ATR-FTIR spectroscopy (Figure 2-a) in order to determine possible bioactive compounds. Table III shows the main functional groups assigned to the different vibrations present in the ATR-FTIR analysis. In the ATR-FTIR spectra of JP, JE and JM was observed peaks between 1718 cm^{-1} and 1725 cm^{-1}, which could be assigned to stretching vibrations of the aromatic ring, typical for anthocyanins (Zeng et al. 2018). The peak 1640 cm^{-1} was found in maltodextrin (M) assigned to O-H bending vibration, belonging to saccharides (Ahmad et al. 2018).

The absorption peaks between 1603 cm^{-1} and 1613 cm^{-1} correspond to aromatic compounds with phenyl bonds, such as flavonoids (Heredia-Guerrero et al. 2014, Santiago-adame et al. 2015). Peak at 1440 cm^{-1} in JP sample corresponds to stretching vibration (C-C) aromatic ring conjugated with C=C, the other samples did not present these absorption peaks, probably due to a degradation of these compounds in the extraction process (Heredia-Guerrero et al. 2014).

JM and M have maltodextrin and presented an absorption peak between 929 cm^{-1} and 931 cm^{-1} attributed to C-O stretching of C-O-C groups in the anhydroglucose ring (Wu et al. 2018). Figure 2-b shows the Gaussian deconvolution of JE wich presented a peak area of anthocyanins 7.56% higher than JP. To the relation between JM (Figure 2-c) and JE, JM presented peak area 38.96% lower than JE, which may be related with the maltodextrin a ratio 1: 1 (w/w) used to microencapsulation.

Morphology by scanning electron microscopy (SEM)

Figure 3 shows the morphology of jaboticaba samples analyzed by scanning electronic microscopy. Samples dried by lyophilization (Figures 3-a and 3-b) presented amorphous particles of different sizes, similar to broken glass, with porosity due to the structural rigidity, characteristics of that kind of drying process (Kuck & Noreña 2016).

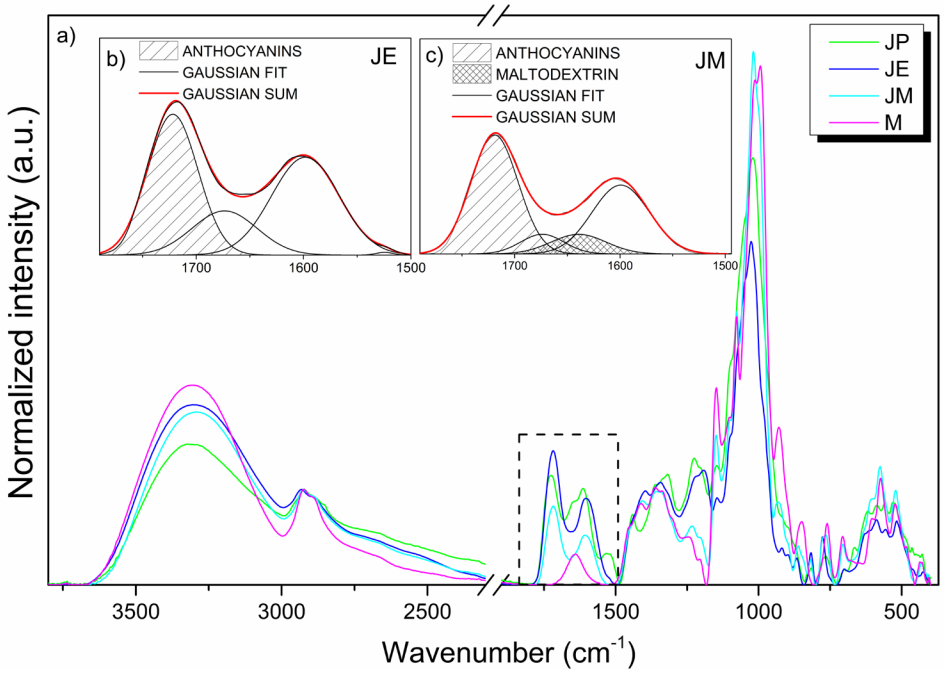

Figure 2. (a) ATR-FTIR spectra of jaboticaba samples. JP: jaboticaba pomace; JE: jaboticaba pomace extract; JM: jaboticaba pomace microcapsule; M: maltodextrin; (b) and (c) Gaussian deconvolutions of JE and JM, respectively, in the spectral range dashed.

Table III. Main vibrational modes assignments observed in the ATR-FTIR spectra.

Assignment *	Wavenumber (cm^{-1})				
	JP	JE	JM	M	Literature reference
ν (O-H)	3316	3303	3292	3306	3300[1]
ν_a (C-H)	2923	2932	2926	2924	2928[2]
ν (C=O)	1725	1718	1718	-	1720[3]
δ (O-H)	-	-	-	1640	1637[6]
ν (C-C)	1613	1603	1606	-	1600[1];1606[4]
ν(C-C) (conjugated with C=C)	1440	-	-	-	1440[4]
ν (C-H)	1225	1219	1232	1240	1233[5]
ν (C-O...H)	1146	1146	1148	1148	1149[2]
ν (C-O)	-	-	1076	1077	1079[2]
ν (C-O-C)	1020	1026	1017	1013	1020[1]
ν (C-O-C) anydroglucose	-	-	931	929	924[2]

***ν, stretching; δ, bending; a, asymmetric.**
JP: jaboticaba pomace; JE: jaboticaba pomace extract; JM: jaboticaba pomace microcapsule; M: maltodextrin.
[1](Santiago-adame et al. 2015); [2](Wu et al. 2018); [3](Zeng et al. 2018); [4](Heredia-Guerrero et al. 2014); [5](Pereira Jr et al. 2015); [6](Ahmad et al. 2018).

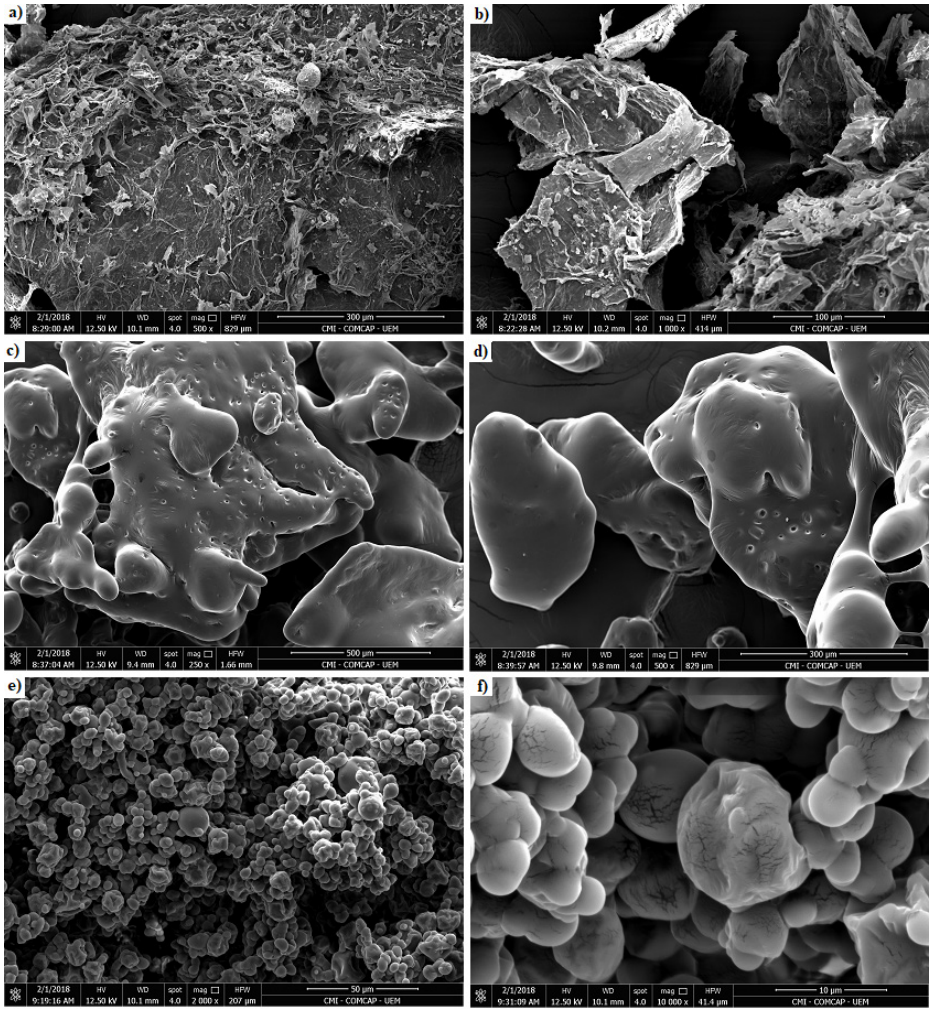

Figure 3. Samples (a) JP: jaboticaba pomace with an increase of 500x and (b) 1000x; (c) JE: jaboticaba pomace extract with an increase of 250x and (d) 500x; (e) JM: jaboticaba pomace microcapsule with an increase of 2000x and (f) 10000x.

In the other hand, the dried extracts obtained by spray drying (Figures 3-c and 3-d) showed amorphous and irregular shape when compared to microcapsules (Figures 3-e and 3-f). It was observed then microcapsules presented a rounded external structure and different sizes, which are characteristics of the samples obtained by this drying method, as a roughness and cracking formed due to the fast evaporation of water (Ferrari et al. 2012).

CONCLUSIONS

The experimental design presented that the best condition for extraction of bioactive compounds from jaboticaba pomace was 45 min in a conventional bath. The use of maltodextrin in microencapsulation was efficient to protect antioxidant compounds under the drying technique, especially for anthocyanins. In order to reuse an agro-industrial waste, it was observed that the antioxidant extraction from jaboticaba pomace is feasible due to bioactive compounds content. In addition, the encapsulation of these compounds is appropriate for technological applications.

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REFERENCES

AHMAD M, ASHRAF B, GANI A & GANI A. 2018. Microencapsulation of saffron anthocyanins using β glucan and β cyclodextrin: Microcapsule characterization, release behaviour & antioxidant potential during in-vitro digestion. Int J Biol Macromol 109: 435-442.

ALOTHMAN M, BHAT R & KARIM AA. 2009. Antioxidant capacity and phenolic content of selected tropical fruits from Malaysia, extracted with different solvents. Food Chem 115: 785-788.

AMAYA-CRUZ DM, RODRÍGUEZ-GONZÁLEZ S, PÉREZ-RAMÍREZ IF, LOARCA-PIÑA G, AMAYA-LLANO S, GALLEGOS-CORONA MA & REYNOSO-CAMACHO R. 2015. Juice by-products as a source of dietary fibre and antioxidants and their effect on hepatic steatosis. J Funct Foods 17: 93-102.

BATISTA ÂG, DA SILVA-MAIA JK, MENDONÇA MCP, SOARES ES, LIMA GC, BOGUSZ JUNIOR S, DA CRUZ-HÖFLING MA & MARÓSTICA JÚNIOR MR. 2018. Jaboticaba berry peel intake increases short chain fatty acids production and prevent hepatic steatosis in mice fed high-fat diet. J Funct Foods 48: 266-274.

FERRARI CC, GERMER SPM, ALVIM ID, VISSOTTO FZ & DE AGUIRRE JM. 2012. Influence of carrier agents on the physicochemical properties of blackberry powder produced by spray drying. Int J Food Sci Technol 47: 1237-1245.

GOUW VP, JUNG J & ZHAO Y. 2017. Functional properties, bioactive compounds, and in vitro gastrointestinal digestion study of dried fruit pomace powders as functional food ingredients. LWT - Food Sci Technol 80: 136-144.

HEREDIA-GUERRERO JA, BENÍTEZ JJ, DOMÍNGUEZ E, BAYER IS, CINGOLANI R, ATHANASSIOU A & HEREDIA A. 2014. Infrared and Raman spectroscopic features of plant cuticles: a review. Front Plant Sci 5: 1-15.

IVANOVIC J, TADIC V, DIMITRIJEVIC S, STAMENIC M, PETROVIC S & ZIZOVIC I. 2014. Antioxidant properties of the anthocyanin-containing ultrasonic extract from blackberry cultivar "Čačanska Bestrna." Ind Crops Prod 53: 274-281.

KOWALSKA H, CZAJKOWSKA K, CICHOWSKA J & LENART A. 2017. What's new in biopotential of fruit and vegetable by-products applied in the food processing industry. Trends Food Sci Technol 67: 150-159.

KUCK LS & NOREÑA CPZ. 2016. Microencapsulation of grape (Vitis labrusca var. Bordo) skin phenolic extract using gum Arabic, polydextrose, and partially hydrolyzed guar gum as encapsulating agents. Food Chem 194: 569-576.

LAMAS CA, LENQUISTE SA, BASEGGIO AM, CUQUETTO-LEITE L, KIDO LA, AGUIAR AC, ERBELIN MN, COLLARES-BUZATO CB, MARÓSTICA MR & CAGNON VHA. 2018. Jaboticaba extract prevents prediabetes and liver steatosis in high-fat-fed aging mice. J Funct Foods 47: 434-446.

LEE J, DURST RW & WROLSTAD RE. 2005. Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: Collaborative study. J AOAC Int 88: 1269-1278.

MACHADO APDF, RUEDA M, BARBERO GF, MARTÍN Á, COCERO MJ & MARTÍNEZ J. 2018. Co-precipitation of anthocyanins of the

extract obtained from blackberry residues by pressurized antisolvent process. J Supercrit Fluids 137: 81-92.

NENADIS N, WANG LF, TSIMIDOU M & ZHANG HY. 2004. Estimation of Scavenging Activity of Phenolic Compounds Using the ABTS+ Assay. J Agric Food Chem 52: 4669-4674.

OZKAN G, FRANCO P, DE MARCO I, XIAO J & CAPANOGLU E. 2019. A review of microencapsulation methods for food antioxidants: Principles, advantages, drawbacks and applications. Food Chem 272: 494-506.

PEREIRA JR VA, DE ARRUDA INQ & STEFANI R. 2015. Active chitosan/PVA films with anthocyanins from Brassica oleraceae (Red Cabbage) as Time–Temperature Indicators for application in intelligent food packaging. Food Hydrocoll 43: 180-188.

PIERPOINT WS. 2004. The extraction of enzymes from plant tissues rich in phenolic compounds. Methods Mol Biol 244: 65-74.

PITALUA E, JIMENEZ M, VERNON-CARTER EJ & BERISTAIN CI. 2010. Antioxidative activity of microcapsules with beetroot juice using gum Arabic as wall material. Food Bioprod Process 88: 253-258.

PLAZA M, BATISTA ÂG, CAZARIN CBB, SANDAHL M, TURNER C, ÖSTMAN E & MARÓSTICA JÚNIOR MR. 2016. Characterization of antioxidant polyphenols from Myrciaria jaboticaba peel and their effects on glucose metabolism and antioxidant status: A pilot clinical study. Food Chem 211: 185-197.

PULIDO R, BRAVO L & SAURA-CALIXTO F. 2000. Antioxidant activity of dietary polyphenols as determined by a modified ferric reducing/ antioxidant power assay. Journal of Agricultural and. Food Chem 48: 3396-3402.

RAMAKRISHNAN Y, ADZAHAN NM, YUSOF YA & MUHAMMAD K. 2018. Effect of wall materials on the spray drying efficiency, powder properties and stability of bioactive compounds in tamarillo juice microencapsulation. Powder Technol 328: 406-414.

RAMOS FM, UBBINK J, SILVEIRA JÚNIOR V & PRATA AS. 2019. Drying of maltodextrin solution in a vaccum spray dryer. Chem Eng Res Des 146: 78-86.

REÁTEGUI JLP, MACHADO APDF, BARBERO GF, REZENDE CA & MARTÍNEZ J. 2014. Extraction of antioxidant compounds from blackberry (Rubus sp.) bagasse using supercritical CO2 assisted by ultrasound. J Supercrit Fluids 94: 223-233.

RODRIGUES LM, DOS SANTOS SS, BERGAMASCO RC & MADRONA GS. 2018a. Jaboticaba byproduct encapsulation by lyophilization: pH and food application stability. J Food Process Eng 41: e12639.

RODRIGUES LM, JANUÁRIO JGB, SANTOS SS, BERGAMASCO R & MADRONA GS. 2018b. Microcapsules of 'jabuticaba' byproduct: Storage stability and application in gelatin. Rev Bras Eng Agrícola e Ambient 22: 424-429.

RODRIGUES LM, ROMANINI EB, SILVA E, PILAU EJ, DA COSTA SC & MADRONA GS. 2020. Camu-camu bioactive compounds extraction by ecofriendly sequential processes (ultrasound assisted extraction and reverse osmosis). Ultrason Sonochem 64: 105017.

RODRIGUES S, FERNANDES FAN, DE BRITO ES, SOUSA AD & NARAIN N. 2015. Ultrasound extraction of phenolics and anthocyanins from jabuticaba peel. Ind Crops Prod 69: 400-407.

SANTIAGO-ADAME R, MEDINA-TORRES L, GALLEGOS-INFANTE JA & CALDERAS F. 2015. Spray drying-microencapsulation of cinnamon infusions (Cinnamomum zeylanicum) with maltodextrin. LWT - Food Sci Technol 64: 571-577.

SANTOS DT, VEGGI PC & MEIRELES MAA. 2010. Extraction of antioxidant compounds from Jabuticaba (Myrciaria cauliflora) skins: Yield, composition and economical evaluation. J Food Eng 101: 23-31.

SANTOS SS, RODRIGUES LM, COSTA SC, BERGAMASCO RC & MADRONA GS. 2017a. Microcapsules of Blackberry Pomace (Rubus fruticosus): Light and Temperature Stability. Chem Eng Trans 57: 1837-1842.

SANTOS SS, RODRIGUES LM, COSTA SC, BERGAMASCO RC & MADRONA GS. 2017b. Microencapsulation of Bioactive Compounds from Blackberry Pomace (Rubus fruticosus) by Spray Drying Technique. Int J Food Eng 13.

SANTOS SS, RODRIGUES LM, COSTA SC & MADRONA GS. 2019. Antioxidant compounds from blackberry (Rubus fruticosus) pomace: microencapsulation by spray-dryer and pH stability evaluation. Food Packag Shelf Life 20: 100177-100182.

SINGLETON VL & ROSSI JA. 1965. Colorimetry of Total Phenolics with Phosphomolybdic-Phosphotungstic Acid Reagents. Am J Enol Vitic 16: 144-158.

THAIPONG K, BOONPRAKOB U, CROSBY K, CISNEROS-ZEVALLOS L & HAWKINS BYRNE D. 2006. Comparison of ABTS, DPPH, FRAP, and ORAC assays for estimating antioxidant activity from guava fruit extracts. J Food Compos Anal 19: 669-675.

VALDUGA E, LIMA L, DO PRADO R, FERREIRA PF & TREICHEL H. 2008. Extração, secagem por atomização e microencapsulamento de antocianinas do bagaço da uva isabel ISABEL (Vitis labrusca). Ciênc Agrotec Lavras 32: 1568-1574.

WU DD, TAN Y, CAO ZW, HAN LJ, ZHANG HL & DONG LS. 2018. Preparation and characterization of maltodextrin-based polyurethane. Carbohydr Polym 194: 236-244.

YE Q, GEORGES N & SELOMULYA C. 2018. Microencapsulation of active ingredients in functional foods: From research stage to commercial food products. Trends Food Sci Technol 78: 167-179.

ZENG YJ, XU P, YANG HR, ZONG MH & LOU WY. 2018. Purification of anthocyanins from saskatoon berries and their microencapsulation in deep eutectic solvents. LWT - Food Sci Technol 95: 316-325.

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