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ORIGINAL ARTICLE

*Impact of drying method as pretreatment for extraction of bioactive compounds from jambolan (*Syzygium cumini *(L.) Skeels)*

*Impacto do método de secagem como pré-tratamento para extração de compostos bioativos do jambolão (*Syzygium cumini *(L.) Skeels)*

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Abstract

Jambolan (*Syzygium cumini* (L.) Skeels) is an under-explored fruit rich in polyphenols, which are associated with health benefits, such as increasing resistance to oxidative stress, inflammatory processes and cardiovascular, and platelet functions. These polyphenols can be obtained by extraction, but an efficient standard method remains a challenge. In this context, this work evaluated the impact of different pretreatments on jambolans to obtain bioactive compounds by aqueous extraction. An Air Circulation Oven (ACO) and Lyophilization (LYO) were used as pretreatments. In addition, the influence of mass, temperature, cycle, and time parameters were studied in the extraction methods used: Percolated Solid-Liquid (PSL), Conventional Solid-Liquid (CSL), and solid-liquid assisted by ultrasound (USL). The extraction yield was from 7.3% (ACO) to 46.3% (LYO), both using the PSL method. In addition, eleven phenolic compounds and six anthocyanins were detected by High Performance Liquid Chromatography-Diode Array Detector (HPLC-DAD), in expressive amounts of catechin and cyaniding in the LYO sample and, these extracts showed higher concentrations of bioactive compounds. The CSL method was more efficient on ACO samples and PSL on LYO samples. LYO extracts showed higher concentrations of bioactive compounds. Therefore, the use of a drying pretreatment results in extracts with a high antioxidant potential for application in the food, cosmetic, and pharmaceutical markets.

Keywords: Aqueous extract; Jambolan; Pretreatment; Bioactive compounds; Polyphenolic compounds; Solvent free.

Resumo

O jambolão é uma fruta pouco explorada e rica em polifenóis, que estão associados a benefícios para a saúde, como aumento da resistência ao estresse oxidativo e aos processos inflamatórios, bem como mostram-se benéficos para as funções cardiovasculares e plaquetárias. Para obtê-los, pode-se fazer por extração, mas um método padrão eficiente permanece um desafio. Nesse contexto, este trabalho avalia o impacto de diferentes pré-tratamentos no jambolão para a obtenção de compostos bioativos por extração aquosa. Estufa com circulação de ar (ACO) e liofilização (LYO) foram usados como pré-tratamentos. Além disso, estudou-se a influência dos parâmetros de massa, temperatura, ciclo e tempo nos métodos de extração utilizados: sólido-líquido percolado (PSL), sólido-líquido convencional (CSL) e sólido-líquido assistido por ultrassom (USL). O rendimento de extração foi de 7,3% (ACO) a 46,3% (LYO), ambos pelo método sólido-líquido percolado (PSL). Além disso, 11 compostos fenólicos e seis antocianinas foram detectados por HPLC-DAD, em quantidades expressivas de catequina e cianidina, na amostra LYO, e esses extratos apresentaram maiores concentrações de compostos bioativos. O método CSL foi mais eficiente em amostras de ACO e o método PSL, em amostras de LYO. Os extratos LYO apresentaram maiores concentrações de compostos bioativos. Portanto, a utilização de um pré-tratamento de secagem resulta em extratos com alto potencial antioxidante para aplicação nos mercados alimentício, cosmético e farmacêutico.

Palavras-chave: Extrato aquoso; Jambolão; Pré-tratamento; Compostos bioativos; Compostos polifenólicos; Livre de solvente.

1 Introduction

The *Syzygium cumini* species, known as jambolan, is an exotic fruit from the Myrtaceae family native to India, which stands out for its high content of bioactive compounds found in the pulp, peel, seeds and leaves. The intensity of the purple color of its peel is directly related to the high anthocyanin content. These compounds are able to preserve food due to their antimicrobial action, in addition to having antioxidant, antiallergic, anti-inflammatory and anti-cancer effects in the human body (Barh & Viswanathan, 2008; Hossain et al., 2017; Singh et al., 2016). For this reason, jambolan is widely used in popular medicine to treat diabetes and various gastrointestinal disorders (Faria et al., 2011; Seraglio et al., 2018) and the extract of jambolan also shows a great potential application in the food, cosmetic and pharmaceutical market.

Obtaining bioactive compounds from exotic fruits presents several challenges, such as the lack of crop production and high perishability, as well as the sensitivity of these compounds to external factors such as light, oxygen and temperature (Joana Gil-Chávez et al., 2013). Traditional extraction methods, including organic solvent and steam distillation, are widely used in the extraction of bioactive compounds and essential oils from various plant matrices (Kapasakalidis et al., 2006; Rufino et al., 2010). However, these techniques usually involve high temperatures and the use of toxic solvents, which can chemically alter and destroy thermosensitive compounds, and subsequent processes for eliminating the residual solvent.

Alternatively, there are the “green extraction” processes that relate to the optimization of raw material, energy, and solvent consumption. The advantages are related to the reduction in the use of solvents and decrease in the extraction time, witch reduces the processing costs (Ameer et al., 2017). Studies using different solvents and/or processes to obtain bioactive compounds from plant matrices showed the strong influence of these factors in the concentration of the compounds. When extracting phenolics from the jambolan by percolation using ethanol:water, Migliato et al. (2009) found that with the right ratio of solute:solvent in the equipment, both consisting of low cost, showed speed and direct responses to the extraction of compounds. The extract obtained from freeze-dried jambolan pulp followed by ethanolic extraction and subsequent concentration by rotary evaporator showed positive effect on antioxidant and antimicrobial activities of fruit polyphenols against reference pathogenic strains (Singh et al., 2016). The extraction of phenols from blackcurrant bagasse and blackcurrant residue (BPR) with 3% of formic acid in methanol and with methanol:water:acetic acid, concluded that acid hydrolysis released a much higher

concentration of bagasse phenols than did of gooseberry press residue (Kapasakalidis et al., 2006). Paul & Das (2018) investigated the influence of the application of different drying techniques on the preservation of the functional properties of jambolan pulp and concluded that Total Phenolic Compounds (TPC) and antioxidant activity of the jambolan pulp are significantly affected by drying. However, the influence of the sample pretreatment on the extraction process to obtain bioactive compounds from jambolans has not yet been addressed.

Processing of jambolan prior to extraction or consumption has a great influence on the chemical structure, affecting the bioavailability of the compound and the content of phytochemicals. In this context, the purpose of this work was to evaluate the influence of drying methods as a pretreatment in the "green" extraction process: Percolated Solid-Liquid (PSL); Conventional Solid-Liquid (CSL); and solid-liquid assisted by ultrasound (USL) of the pulp and peel of a jambolan, aiming at obtaining extracts rich in bioactive compounds with high antioxidant activity.

2 Material and methods

2.1 Material

The fruits of the jambolan (*Syzygium cumini* (L.) Skeels) were collected on the campus of the Federal University of Santa Catarina (*Universidade Federal de Santa Catarina* (UFSC)) in Florianópolis, in the state of Santa Catarina (SC). The collection was done manually in the early hours of the morning, the material was identified and registered in the Flower Herbarium of the UFSC, under the EXSICATA registration number FLOR 31.270. Analytical standards and reagents were purchased from Sigma-Aldrich (St. Louis, MO, USA) while the solvents used in chromatography (High Performance Liquid Chromatography (HLPC) grade) were purchased from Merck (Darmstadt, Germany). Distilled and deionized water with the resistivity of 18.2 MΩ cm was generated by a Milli-Q Plus system from Millipore (Bedford, USA).

2.2 Pretreatment of raw material

For the study in which pulp and peel of the jambolan were used, the seeds were discarded from the process to avoid contamination by interfering components as reported in the studies of Tavares et al. (2016). Two drying methods were performed as pretreatment: air circulation oven (ACO); and freeze-drying (LYO). In the drying air circulation process, the pulp and peel of the jambolan were placed in trays with the following dimensions: 40 cm in length and 33 cm in width; distributed with an approximate 1 cm layer thickness; and followed by a drying process at 55 °C in an air circulation furnace (De Leo, Porto Alegre RS, Brazil) for 8 hours without interruption. The conditions of time and temperature were defined from the stability conditions of the compounds present in fruits and also from conditions cited by Poirot et al. (2007). In the LYO process, the pulp and peel were distributed in trays of an equipment with 18 cm in diameter, and layer thickness of 2.5 cm, and then dried in a lyophilizer (L 101, LIOBRAS Ind. Com. E Serv. Ltda., São Carlos, SP, Brazil) for 48 hours. The time was determined according to the thickness of the layer in the tray.

2.3 Grinding and characterization of the raw material

Samples were ground using a Willey-type knife mill, collected in a plastic bag and stored in a freezer at -18 °C until further analysis. The determination of the moisture content and volatile substances for the jambolan samples was carried out according to Instituto Adolfo Lutz (2008) with adaptations. The *in natura* sample had 79.33 ± 0.30% of humidity, whereas the ACO sample had 29.64 ± 1.07%, and the LYO sample had 4.02 ± 0.23%.

The particles of the ACO and LYO samples were submitted to microstructure analysis. Then, the samples were mounted on aluminum stumps with a carbon tape and sprinkled with a thin layer of gold. The reading

was performed using Scanning Electron Microscope (SEM) (SEM - JEOL JSM - 6390LV, United States) with visualization carried out in increments of 50 to 350 times, at a voltage of 10 kV.

The determination of the particle diameter mean was performed using the Sizer Meter software, version 1.1 (developed by Luiz Henrique Castelan Carlson). The sample dried in the oven had a diameter equal to/greater than 0.850 mm and the freeze-dried sample 0.225 mm.

2.4 Extration of bioactive compounds

The bioactive compounds of jambolans were extracted by three different methods: CSL; PSL; and USL. The solvent used was the distilled water, making the solvent disposal process environmentally safe since it does not use organic solvent, thus eliminating the risks involved in the disposal of unacceptable waste (Ameer et al., 2017). At the end of the extractions, the systems were vacuum filtered (825T, FISATOM, Brazil) with filter paper to separate the solid sample from the solution with the extract. These solutions went through a process of elimination of solvent (distilled water) in lyophilizer (L 101, LIOBRAS Ind. Com. E Serv. Ltda., São Carlos/SP, Brazil) for 48 hours. The extracts packed in amber bottles were stored in a freezer at -18 °C.

2.4.1 Solid-liquid percolation extraction

For the PSL, the solvent was contacted with the sample by washing with and without solvent recirculation through the vacuum filtration process (825T, FISATOM, Brazil) with filter paper under the conditions determined by a Central Composite Design (CCD). The volume of solvent used was 25 mL, according to studies in the literature (Wong Paz et al., 2015).

2.4.2 Conventional solid-liquid extraction

The extractions were performed with adaptations of the methodology described by Castro-López et al. (2017). For this, the sample was kept in contact with the solvent (distilled water) following conditions determined by the CCD. The extractions were performed with temperature control by the use of a water bath (ALB 250C, Piracicaba - SP, Brazil), with the following temperatures: 30; 40; and 50 °C.

2.4.3 Ultrasound-assisted solid-liquid extraction

The method was performed with the parameters described in CCD according to Rodríguez-Pérez et al. (2015) with some modifications. Then, the solution was packed in 60 mL amber flasks and submitted to an ultrasound bath using an ultrasonic washer (Unique, USC-1880 A, Brazil, power: 132w, frequency: 37kHz). In the end, the system was filtered for the times described in the CCD.

2.4.4 Central Composite Design

The extraction runs were determined from a CCD with three factors and three levels for each factor, constituting a total of 11 runs, eight factorials (the combination of levels -1 and +1), and three repetitions in the central point (the three variables at level zero). The independent variables were mass (g), temperature (°C) and cycle for percolated solid-liquid extraction (washing cycles: 0 = without recirculation; 2 = with one recirculation and 4 = with three recirculations) and mass (g), temperature (°C), and time (h) for CSL and USL extractions. The maximum and minimum conditions were determined according to the sensitivity of the bioactive compounds. The evaluated responses were extraction yield, TPC, Total Flavonoids (TF), Total Anthocyanins (TA), and antioxidant activity by 2.2-diphenyl-1-picrylhydrazyl (DPPH) Free Radical Scavenging and Ferric Reducing Antioxidant Power (FRAP). The complete planning matrix is provided as Table 1.

Table 1. Central Composite Design for the variables mass, temperature, cycle, and time.

RUN	Coded Levels			Decoded Levels			
	X_1	X_2	X_3	m (g)	T (°C)	C (adm)	t (h)
1	-1	-1	-1	3	30	0	1
2	1	-1	-1	9	30	0	1
3	-1	1	-1	3	50	0	1
4	1	1	-1	9	50	0	1
5	-1	-1	1	3	30	4	5
6	1	-1	1	9	30	4	5
7	-1	1	1	3	50	4	5
8	1	1	1	9	50	4	5
9	0	0	0	6	40	2	3
10	0	0	0	6	40	2	3
11	0	0	0	6	40	2	3

m (mass); T (temperature); C (cycle); t (time); X_1 = mass variable (g); X_2 = temperature variable (°C) and X_3 = washing cycles variable (admission) for PSL and X3 = time variable (h) for the CSL and USL.

2.5 Analysis of extracts

2.5.1 Determination of Total Phenolic Compounds (TPC)

The determination of the TPC content present in the jambolan extracts was performed by the Folin-Ciocalteu according to Rossi & Singleton (1965) spectrophotometric method adapted by Castro-López et al. (2017). Then, the reaction mixture was composed of 0.1 mL of extract (concentration between 3000 and 4000 mg·L^{-1}), 6 mL of distilled water, 0.5 mL of Folin - Ciocalteu reagent (a mixture of phosphorolysis and phosphotungstate), and 1.5 mL of 20% sodium carbonate, placed in opaque flasks. The vials were shaken and allowed to stand for 2 hours, and the absorbance was measured at 765 nm in a spectrophotometer (Femto, 800XI, Brazil). TPC was calculated according to a standard curve ($y = 0.0012x + 0.008$, $R^2 = 0.9963$), previously prepared with gallic acid. The analysis was performed in triplicate and the results expressed in mg GAE·100 g^{-1} extract.

2.5.2 Determination of flavonoids and anthocyanins

The TF and TA were determined according to the method described by Francis (1982). The analysis was performed using 1 ± 0.1 g of sample and 10 mL of ethanol/hydrochloric acid mixture (85:15). The preparation was stored in the absence of light at 4 °C for 24 h, followed by filtration with cotton and completed the volume to 10 mL with the Et/HCl mixture. The absorbance reading was performed in a UV-visible spectrophotometer (FEMTO, 800 XI, São Paulo, SP, Brazil) at 374 nm for flavonoids and 535 nm for anthocyanins, and the results were expressed as mg·100 g^{-1} of the sample, calculated according to Equations 1a, 1b and 1c:

$$\text{Total Flavonoids} = \frac{\text{Fd} \cdot \text{Abs}}{76.6} \quad (1a)$$

$$\text{Total Anthocyanins} = \frac{\text{Fd} \cdot \text{Abs}}{98.2} \quad (1b)$$

$$\text{Fd} = \frac{100 \cdot \text{Abs}}{\text{m}} \quad (1c)$$

where Fd = dilution factor, Abs = Absorbance (nm), m = mass (g) and v = volume (mL).

2.5.3 Determination of 2.2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging capacity

The free radical scavenging ability was tested by the DPPH according to Khan et al. (2016) with DPPH solution (0.3 mM), and extract concentrations ranging from 10 to 900 $\mu g \cdot mL^{-1}$. The reaction mixture was Vortexed and left in the dark for 30 min at 37 °C. Absorbance was measured on a Ultraviolet-Visible (UV/VIS) spectrophotometer (Femto, model 800 XI) at 517 nm. The percentage of DPPH radical scavenging activity was calculated according to the following Equation 2:

$$DPPH = 100 - \left[\frac{\left(Abs_{sample} - Abs_{white} \right) \cdot 100}{Abs_{control}} \right] \quad (2)$$

Then, the percentage of inhibition was plotted against the concentration, and the IC50 graph was calculated according to Mensor et al. (2001).

2.5.4 Ferric Reducing Ability of Plasma (FRAP)

The antioxidant capacity was also estimated by the FRAP method, following the procedure described by Benzie & Strain (1996) with due modifications. Then, 200 µL of the 2 mM Trolox solution plus 200 µL of $FeCl^3$ were mixed in test tubes, shaking and placed in a water bath for 30 min at 37 °C. In addition, 3600 µL of the TPTZ solution was added, homogenized and placed in an ice bath for 10 min. The absorbance was measured on a spectrophotometer (FEMTO, 800 XI, São Paulo, SP, Brazil) at 620 nm. Equations 3a and 3b:

$$C = \frac{\left(Abs - Absb \right) - A}{B} \quad (3a)$$

where C ($\mu mol \cdot L^{-1}$), A, and B are the coefficients of the standard curve, A_{bs} is the absorbance of sample reading, and A_{bsb} the absorbance of the blank. Calculation of antioxidant activity:

$$C = \frac{C \cdot D \cdot 100}{m} \quad (3b)$$

where **C** ($\mu mol \cdot 100\ g^{-1}$ sample), C ($\mu mol \cdot L^{-1}$), D is the dilution used, and m the sample mass.

2.6 Determination of the chemical profile of extracts

2.6.1 Headspace gas chromatography coupled to mass spectrometry (Headspace GC/MS)

For each experiment, 1000 µL of the vapor phase of the dried and freeze-dryed jambolan extracts were collected with a Headspace-Type Syringe Plunger Syringe of 2500 µL (HAMILTON, USA) and injected into the Gas Chromatograph coupled to the Mass Spectrometer (GC-MS). The conditions used were selected by evaluating the amount and relative area of the peaks in the chromatogram for the extracted compounds. All experiments were performed in triplicates. The volatile compounds were separated and identified using a GS from Agilent Technologies (GC 7890A) coupled to a MS (MS 5975C) equipped with auto sampler Agilent GC Sampler 80, HP-5MS capillary column (Agilent Technologies), stationary phase composed of 5% phenyl and 95% dimethyl siloxane (30 m x 250 µm d.i. x 0.25 µm film thickness). Helium was used as the carrier gas at a flow rate of 1 $mL \cdot min^{-1}$. Chromatographic conditions were as follows: injector temperature 250 °C operating in splitless mode. The column initially at 60 °C, was subsequently heated at a rate of 3 °C min to 260 °C, keeping that temperature for 5 min. The interface transfer line temperature was 250 °C. The quadrupole detector operating in el mode at 70 eV and the mass scan ranged from 50 to 550 m/z. The compounds were identified by comparing their mass spectra. The operating conditions are found in Table 2.

Table 2. Operating conditions Headspace gas chromatography coupled to mass spectrometry (Headspace GC/MS).

Headspace Conditions	
Injection Volume (µL)	1000
Headspace Syringe Size (µL)	2500
Incubation Temperature (°C)	100
Incubation Time (s)	300
Syringe Temperature (°C)	100
Agitator Speed (rpm)	250

2.6.2 High-Performance Liquid Chromatography (HPLC)

The determination of the phenolic profile of jambolan extracts was carried out on a Shimadzu liquid chromatography (Kyoto, Japan) equipped with a quaternary pump, degasser, and Diode Array Detector (DAD). The analytical separation was performed on a Shim-pack C18-ODS reverse phase column (250 x 4.6 mm, 5 µm), equipped with a G-ODS (10 x 4 mm, 5 µm) guard column (Shimadzu, Kyoto, Japan). Samples were diluted in 1% HCl acidified water, filtered through 0.45 µm PTFE membrane filter (Millipore, Massachusetts, USA), and 20 µL were directly injected for the analysis. The quantification of anthocyanins was performed according to Revilla & Ryan (2000). The binary mobile phase consisted of (A) ultrapure water 10% of formic acid (v/v) and (B) methanol 50% solvent A (v/v) in gradient elution mode with a flow rate of 0.8 mL·min^{-1}. The detection of anthocyanins was set at 520 nm. Other phenolic compounds were determined according to Cadahía et al. (2009) using a mobile phase consisting of (A) ultrapure water 2% of acetic acid (v/v) and (B) ultrapure water: acetonitrile (60:40 v/v) with 2% of acetic acid. Gradient elution was performed with the flow rate set at 1.0 mL·min^{-1}. The detection was set at 280 nm for flavan-3-ols and tyrosol, 320 nm for cinnamic acids, and at 360 and 306 nm for flavanols and trans-resveratrol, respectively. All chromatographic analyzes were performed in triplicate.

Additional data can be found in the supplementary material.

3 Results and discussion

3.1 Characteristics of the pre-treated material

The pulp and peel of the jambolan pre-treated by ACO showed 555.56 ± 23.15 mg GAE·100 g^{-1} for TPC; TF of 247.90 ± 0.38 mg·100 g^{-1} and 857.79 ± 4.67 mg·100 g^{-1} for the TA; its antioxidant activity by DPPH and FRAP presented respectively IC50 of 530.56 ± 10.78 µg·mL^{-1}, and 4041.11 ± 170.52 µmol·100 g^{-1}. For pretreatment by LYO, the values were: TPC of 945.95 ± 22.52 mg GAE·100 g^{-1}; TF of 407.80 ± 0.46 mg·100 g^{-1} and for TA 1560.79 ± 3.98 mg·100 g^{-1}; the antioxidant activity was IC50 of 189.82 ± 4.19 µg·mL^{-1} by DPPH, and by FRAP 10533.40 ± 245.98 µmol·100 g^{-1}.

Figure 1 shows the scanning electron microscopy of the particles from ACO (Figure 1a) and LYO processes (Figure 1b), with magnification according to the size of the particles, making clear the difference in shape and size between them. The LYO particles showed a diameter of 225 µm, a slightly rounded shape and high porosity when compared to ACO, which was considerably larger and has an undefined shape. This can be attributed to the phenomenon that occurs during LYO drying, where the sample undergoes slow freezing, forming large water crystals and greater separation of water from the other constituents of the plant matrix (Ezhilarasi et al., 2013), and subsequent drying by sublimation; the drying kinetics of these crystals may have caused the cell wall to rupture (visualized in SEM – Figure 1), thus increasing the porosity of the particles of the sample pre-treated by LYO.

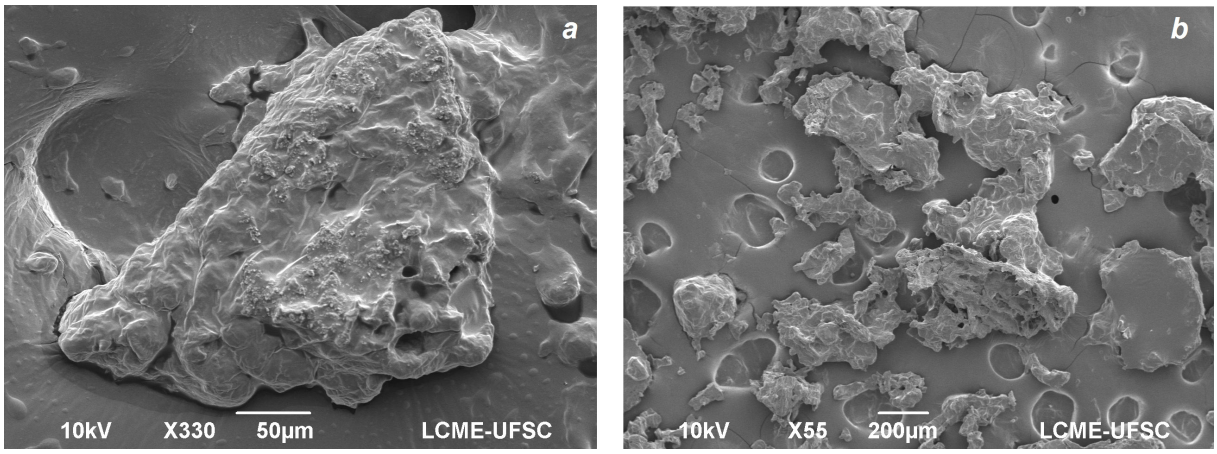

Figure 1. Scanning electron microscopy of the samples of jambolan. *a) dried in an oven and b) freeze-drying.*

3.2 Analysis of the extracts

The effects of the variable's mass, temperature, washing cycle, and extraction time and their interactions were studied to identify the best method for extracting bioactive compounds: PSL; CSL; and USL, also considering the type ofACO and LYO samples. This study was presented in two parts: characterization of the extracts from the ACO sample; and the LYO sample.

3.2.1 Global yield of extractions

According to Ameer et al. (2017), the overall extraction yield is defined as the total amount of compounds present in the solid matrix that can be extracted by the solvent. The global yields of the jambolan extracts are presented in Table 3 and Table 4, for ACO and LYO, respectively.

The PSL extraction yield showed a difference between the ACO and LYO treatments, ranging from 7.32 ± 0.28% to 46.27 ± 0.44% (run 1, lower mass, temperature and cycle). This can be attributed to the characteristics of the particles, the smaller diameter and greater porosity resulting from lyophilization, which leaves the compounds more exposed for extraction, increasing the transfer rate of the compounds present in the plant matrix compatible with the solvent, and also increasing the interaction between them (Migliato et al., 2009).

Authors report that exhaustive washing increases the amount of extracted compounds, thus increasing the total yield of extraction (Joana Gil-Chávez et al., 2013; Migliato et al., 2009). However, evaluating the CSL extraction in run 7 (lower mass and, higher temperature and cycle) we could see that the diameter and morphology of the particles represented a greater influence on the extraction yield, being 20.97 ± 0.65% and 70.39 ± 0.12% for ACO and LYO, respectively. USL extractions for ACO and LYO reaffirmed the influence of particle characteristics, showing extraction yields of 29.17 ± 0.11% and 52.86 ± 0.07% (run 3, higher temperature and lower mass and time).

The Analysis of Variance (ANOVA) of the total extraction yield showed a non-significant adjustment ($p > 0.05$) for the ACO samples in all the extraction methods; the LYO sample showed that there was an influence of the input variables but it was not statistically significant.

3.2.2 Sample dried in an air circulation oven

3.2.2.1 Total phenolic compounds

Phenolic compounds are mainly responsible for the antioxidant activity characteristic of natural products. However, the Folin-Ciocalteu method does not fully characterize the antioxidant activity,as it expresses the

values in terms of the content of gallic acid present in the sample, representing only a good estimate of this property (Roginsky & Lissi, 2005). The TPC determined by Folin-Ciocalteu for the extracts of the different methods for sample ACO are shown in Table 3.

Table 3. Global yield extractions, bioactive compounds and evaluation of the antioxidant activity present of the extracts for different extraction methods in drying in Air Circulation Oven (ACO) samples.

RUN	Coded Levels			Yield[2]	TPC[3]	TF[3]	TA[3]	DPPH[4]	FRAP[4]
						PSL[1]			
1	-1	-1	-1	7.32 ± 0.28^{h}	446.875 ± 11.46^{e}	96.02 ± 0.34^{b}	466.94 ± 2.02^{e}	539.53 ± 5.44^{e}	7903.57 ± 126.27^{c}
2	1	-1	-1	7.62 ± 0.55^{h}	435.417 ± 0.00^{ef}	61.99 ± 1.20^{g}	329.11 ± 0.64^{g}	626.25 ± 12.10^{bc}	12028.57 ± 404.06^{a}
3	-1	1	-1	26.94 ± 0.12^{b}	672.872 ± 48.76^{a}	91.37 ± 0.84^{c}	625.39 ± 1.42^{b}	565.75 ± 5.75^{d}	10778.57 ± 555.58^{b}
4	1	1	-1	11.05 ± 0.35^{g}	391.260 ± 11.18^{f}	87.97 ± 1.32^{d}	556.95 ± 1.36^{d}	554.16 ± 7.22^{e}	11975.00 ± 328.30^{a}
5	-1	-1	1	43.25 ± 0.50^{a}	428.442 ± 9.96^{ef}	80.14 ± 0.23^{e}	567.40 ± 0.97^{c}	653.00 ± 15.48^{b}	3992.86 ± 252.54^{g}
6	1	-1	1	25.68 ± 0.36^{bc}	553.819 ± 19.10^{bc}	59.96 ± 1.23^{g}	253.14 ± 1.35^{i}	713.11 ± 4.14^{a}	6885.71 ± 101.02^{d}
7	-1	1	1	25.64 ± 0.67^{c}	477.837 ± 9.75^{de}	104.67 ± 1.52^{a}	641.51 ± 1.40^{a}	459.72 ± 12.54^{e}	4296.43 ± 25.25^{g}
8	1	1	1	22.26 ± 0.10^{d}	580.556 ± 10.19^{b}	56.89 ± 1.37^{h}	229.43 ± 1.19^{k}	601.31 ± 8.92^{c}	6100.00 ± 0.00^{e}
9	0	0	0	18.46 ± 0.10^{ef}	536.585 ± 0.00^{bc}	51.79 ± 1.91^{i}	297.82 ± 1.90^{h}	468.63 ± 10.77^{e}	5546.43 ± 25.25^{f}
10	0	0	0	17.99 ± 0.83^{f}	458.333 ± 20.83^{de}	68.66 ± 1.67^{f}	347.63 ± 2.12^{f}	605.67 ± 6.72^{c}	5385.71 ± 202.03^{f}
11	0	0	0	19.72 ± 0.13^{f}	509.259 ± 0.00^{cd}	45.32 ± 2.35^{j}	248.48 ± 2.12^{j}	557.07 ± 10.48^{e}	7100.00 ± 50.51^{d}
						CSL[1]			
1	-1	-1	-1	14.12 ± 0.26^{g}	906.48 ± 10.19^{b}	180.33 ± 0.50^{g}	982.75 ± 2.41^{h}	563.12 ± 5.05^{d}	3857.76 ± 283.02^{cd}
2	1	-1	-1	22.06 ± 0.33^{f}	507.09 ± 0.00^{g}	180.15 ± 0.24^{g}	970.68 ± 2.50^{hi}	486.35 ± 14.91^{e}	4046.58 ± 21.96^{c}
3	-1	1	-1	31.35 ± 0.04^{c}	437.50 ± 0.00^{h}	256.58 ± 0.85^{b}	1606.70 ± 6.08^{a}	392.92 ± 10.71^{g}	4332.71 ± 66.46^{c}
4	1	1	-1	30.30 ± 0.49^{c}	697.92 ± 10.42^{d}	355.12 ± 0.40^{a}	1401.47 ± 4.54^{c}	612.90 ± 9.18^{c}	3853.74 ± 264.56^{cd}
5	-1	-1	1	10.38 ± 0.69^{h}	767.44 ± 0.00^{c}	243.44 ± 1.05^{c}	1486.12 ± 4.40^{b}	431.78 ± 8.41^{f}	3549.88 ± 65.31^{de}
6	1	-1	1	24.26 ± 0.27^{e}	630.21 ± 0.00^{e}	179.13 ± 0.38^{g}	920.08 ± 1.92^{j}	667.49 ± 10.86^{b}	2598.30 ± 115.84^{g}
7	-1	1	1	20.97 ± 0.65^{f}	1010.42 ± 10.42^{a}	187.06 ± 0.36^{f}	1211.12 ± 2.49^{e}	535.71 ± 11.10^{d}	3131.068 ± 171.63^{ef}
8	1	1	1	27.45 ± 0.43^{d}	693.09 ± 0.00^{d}	204.22 ± 1.39^{e}	1186.14 ± 3.33^{f}	467.50 ± 7.15^{e}	2820.77 ± 303.98^{fg}
9	0	0	0	42.43 ± 0.08^{a}	607.56 ± 10.66^{e}	167.31 ± 1.74^{h}	958.14 ± 2.85^{i}	479.44 ± 14.34^{e}	5805.26 ± 452.66^{a}
10	0	0	0	40.71 ± 0.20^{b}	536.59 ± 0.00^{f}	186.36 ± 1.70^{f}	1050.95 ± 29.63^{g}	606.46 ± 15.91^{c}	6064.63 ± 312.66^{a}
11	0	0	0	40.93 ± 0.09^{b}	777.17 ± 19.93^{c}	210.83 ± 2.26^{d}	1366.85 ± 21.00^{d}	733.03 ± 9.99^{a}	5210.08 ± 99.03^{b}
						USL[1]			
1	-1	-1	-1	14.28 ± 0.04^{g}	523.81 ± 0.00^{ef}	275.34 ± 1.50^{a}	1322.06 ± 9.05^{c}	446.51 ± 14.28^{e}	3843.38 ± 393.87^{cd}
2	1	-1	-1	28.59 ± 0.55^{e}	895.83 ± 0.00^{a}	241.06 ± 1.53^{b}	1127.77 ± 12.15^{f}	656.94 ± 20.73^{a}	4189.89 ± 20.53^{bc}
3	-1	1	-1	29.17 ± 0.11^{e}	550.00 ± 20.37^{de}	211.06 ± 1.15^{d}	1228.84 ± 2.12^{d}	480.13 ± 0.55^{d}	4204.41 ± 0.00^{bc}
4	1	1	-1	31.07 ± 0.08^{d}	585.65 ± 25.46^{d}	194.33 ± 0.65^{f}	1141.85 ± 2.83^{f}	562.49 ± 4.22^{c}	3996.30 ± 130.62^{cd}
5	-1	-1	1	24.53 ± 0.47^{f}	528.08 ± 9.96^{ef}	230.03 ± 1.48^{c}	1481.21 ± 3.54^{a}	571.21 ± 18.48^{c}	4627.46 ± 21.77^{ab}
6	1	-1	1	30.63 ± 0.16^{d}	665.67 ± 10.91^{c}	164.70 ± 0.59^{i}	1073.84 ± 21.21^{g}	367.87 ± 3.02^{f}	4134.01 ± 134.09^{cd}
7	-1	1	1	45.82 ± 0.37^{a}	721.88 ± 34.38^{b}	229.37 ± 0.77^{c}	1410.99 ± 3.79^{b}	619.94 ± 13.75^{b}	3737.86 ± 294.22^{de}
8	1	1	1	32.81 ± 0.34^{c}	586.24 ± 10.66^{d}	210.79 ± 1.05^{d}	1176.39 ± 4.17^{e}	567.71 ± 7.51^{c}	4941.02 ± 370.70^{a}
9	0	0	0	38.99 ± 0.16^{b}	402.44 ± 22.36^{g}	204.05 ± 0.56^{e}	1209.78 ± 14.14^{d}	418.82 ± 1.75^{e}	3935.01 ± 22.75^{cd}
10	0	0	0	39.13 ± 0.09^{b}	499.07 ± 10.19^{f}	183.26 ± 0.89^{h}	942.51 ± 10.47^{h}	496.33 ± 4.75^{d}	3292.98 ± 85.61^{e}
11	0	0	0	39.31 ± 0.00^{b}	500.97 ± 10.66^{f}	186.48 ± 1.53^{g}	1132.97 ± 14.85^{f}	227.33 ± 3.83^{g}	3716.71 ± 299.62^{de}

[1]Extraction methods: PSL = Percolated Solid-Liquid, CSL = Conventional Solid-Liquid, USL = Ultrasonic-assisted Solid-Liquid. [2] Global yield extractions in %. [3]Bioactive compounds: TPC (Total Phenolic Compounds) in mg GAE/100 g extract; TF (Total Flavonoids) in mg/100 g extract; TA (Total Anthocyanins) in mg/100 g extract. [4]Antioxidant activity: DPPH (2,2-diphenyl-1-picrylhydrazyl) resulting in IC50 value $\mu g \cdot mL^{-1}$; FRAP (Ferric Reducing Ability of Plasma) resulting in µmol/100 g extract.

Results expressed as mean ± standard deviation of three replicates. Means followed by different letters in the same column are significantly different by Tukey's test ($p < 0.05$)

The TPC of the extracts by PSL had an increase of almost 100% with a decrease in mass (run 4 to 3), caused by the principle of mass transfer resulting from the decrease in mass in relation to the extractor solvent,

thus increasing the diffusion between solid and liquid, resulting in increasing the concentration gradient (Cacace & Mazza, 2003), as well as a result of the vacuum pressure that may have destroyed the matrix to a higher degree, causing the release of the phenolic compounds (Paul & Das, 2018).

The CSL increase in time (run 3 to 7) resulted in an increase in the extraction of TPC. The use of USL did not change significantly in relation to the CSL and PSL methods. The ANOVA of the TPC showed a non-significant adjustment ($p > 0.05$) in the CSL and USL methods (Table SM1 contained in supplementary material). For PSL extraction, it influenced the interaction of independent factors, with a ratio coefficient $R^2 = 0.95$. The model that describes the behavior of the TPC (mg GAE·100 g^{-1}) is represented by Equation 4.

$$TPC = 499.2051 - 36.6016 \cdot m \cdot T + 65.1458 \cdot m \cdot c \quad (4)$$

where: m = mass (g), T = temperature (°C), and c = cycle (adm).

Considering the studied variables, it can be concluded that there is a positive influence on the TPC response when the mass is minimum and the cycle increases. The response surface, the Pareto graph and the adjustment of the model in the graph observed x predicted values are shown in the supplementary material Figure SM1, and Figure SM2a.

The determination of the chemical profile was performed in the extracts that presented better results of TPC, runs: 3-PSL, 7-CSL and 2-USL for ACO.

3.2.2.2 Total flavonoids

The determinations of TF in the ACO sample are shown in Table 3. In the three extraction methods, the amount of TF presented results superior to those observed by Faria et al. (2011) in jambolan extracts with 5% ethanol of H_3PO_4. The results indicated that the temperature and the extraction time have a negative effect when increased together, and although the flavonoids are thermosensitive, the temperature has been less harmful than the contact time. It is commonly known that longer extraction times, with higher temperatures, promote the degradation of bioactive compounds (Migliato et al., 2009; Rufino et al., 2010; Tavares et al., 2016).

3.2.2.3 Total anthocyanins

The TA content by PSL (Table 3) showed that the increase in mass and decrease in temperature reflected negatively, the same was observed by Plaza & Turner (2015). In the minimum condition of mass and temperature, the USL and CSL methods showed close values (run 5), representing that the increase in mass influenced negatively, which can be explained by the saturation of the solvent. Even the maximum temperature plotted in this study did not degrade anthocyanins, and the time of contact with the sample may have had a positive influence.

3.2.2.4 Antioxidant activity by DPPH and FRAP

In studies on antioxidant capacity, many researchers evaluated compounds in isolation (Banerjee et al., 2005; Rufino et al., 2010; Tavares et al., 2016), so this assessment does not correlate directly with a single analysis of antioxidant activity. Table 3 shows the results of the antioxidant activity in the ACO extracts, determined by the DPPH and FRAP methods.

Equivalent values of IC50 by DPPH were found by USL and CSL, and although it was evaluated in the different methods, it can be verified that the proportion “minimum mass/maximum temperature” or vice versa, were the reference factors for the highest antioxidant activity. In the USL, the possible increase in temperature caused by agitation creates the need for an adequate control of the experimentation conditions. In general, the extracts that showed higher concentrations of TPC, consequently showed better results of antioxidant activity.

3.2.3 Analysis of the extracts of freeze-drying samples

3.2.3.1 Total phenolic compound

The TPC content for the extract pre-treated by LYO is shown in Table 4. The values presented here were higher than those found by Santos et al. (2020), 1.56 ± 0.01 mg GAE·100 g^{-1} in the study of extraction of pulp and jambolan peel obtained with a mixture of organic solvents using rotation evaporation for elimination solvent, evidencing that additional processes for solvent elimination can negatively interface in the extraction of TPC.

Table 4. Global yield extractions, bioactive compounds and evaluation of the antioxidant activity present of the extracts for different extraction methods in sample drying in Freeze-Drying samples.

RUN	Coded Levels			Yield[2]	TPC[3]	TF[3]	TA[3]	DPPH[4]	FRAP[4]
						PSL[1]			
1	-1	-1	-1	46.27 ± 0.44^{a}	3465.57 ± 94.78^{c}	340.47 ± 1.28^{a}	1441.41 ± 4.05^{i}	59.06 ± 8.73^{f}	16100.00 ± 959.65^{bc}
2	1	-1	-1	10.70 ± 0.32^{i}	4811.88 ± 30.12^{a}	298.81 ± 1.33^{d}	966.69 ± 2.93^{j}	94.17 ± 1.12^{d}	17189.29 ± 126.27^{a}
3	-1	1	-1	33.37 ± 0.79^{e}	3226.96 ± 34.15^{d}	292.97 ±0.21^{e}	1749.21 ± 3.42^{f}	84.21 ± 4.18^{de}	15653.57 ± 25.25^{cd}
4	1	1	-1	7.49 ± 0.26^{j}	4622.51 ± 75.56^{b}	297.93 ± 0.39^{d}	1745.69 ± 2.04^{f}	42.77 ± 0.66^{g}	15600.00 ± 50.51^{cd}
5	-1	-1	1	41.64 ± 0.69^{b}	2046.51 ± 0.00^{f}	216.47 ± 0.70^{i}	1671.47 ± 2.86^{h}	231.26 ± 2.74^{a}	13278.57 ± 151.52^{f}
6	1	-1	1	17.37 ± 0.16^{h}	2566.67 ± 0.00^{e}	318.95 ± 0.42^{c}	1861.05 ± 6.98^{d}	80.72 ± 1.16^{e}	15958.27 ± 475.07^{bc}
7	-1	1	1	29.84 ± 0.18^{f}	3235.14 ± 44.29^{d}	227.49 ± 0.56^{h}	1682.70 ± 3.30^{g}	79.07 ± 0.34^{e}	16983.73 ± 375.06^{a}
8	1	1	1	24.94 ± 0.11^{g}	3316.11 ± 63.69^{cd}	329.30 ± 0.77^{b}	2083.19 ± 2.49^{a}	77.23 ± 1.21^{e}	16554.62 ± 247.59^{ab}
9	0	0	0	31.65 ± 0.10^{e}	2652.78 ± 24.06^{e}	238.13 ± 1.26^{g}	1778.73 ± 2.21^{e}	141.14 ± 4.43^{b}	14807.69 ± 138.76^{e}
10	0	0	0	31.83 ± 0.41^{de}	3167.59 ± 61.27^{d}	298.19 ± 1.46^{d}	2010.78 ± 7.78^{b}	148.45 ± 1.06^{b}	12709.36 ± 304.79^{f}
11	0	0	0	32.95 ± 0.19^{cd}	2710.14 ± 69.03^{e}	281.75 ± 1.02^{f}	1873.39 ± 7.58^{c}	106.64 ± 6.38^{c}	15110.96 ± 0.00^{de}
						CSL[1]			
1	-1	-1	-1	58.17 ± 0.05^{b}	3612.10 ± 10.91^{d}	299.88 ± 1.39^{i}	1974.17 ± 2.51^{g}	177.94 ± 1.74^{c}	17122.05 ± 392.29^{c}
2	1	-1	-1	26.72 ± 0.02^{i}	4054.49 ± 11.75^{b}	378.40 ± 1.72^{b}	2162.29 ± 4.24^{d}	93.55 ± 1.58^{h}	15871.85 ± 965.59^{d}
3	-1	1	-1	48.31 ± 0.04^{d}	3357.29 ± 57.29^{e}	339.92 ± 1.33^{d}	2770.80 ± 2.21^{a}	153.36 ± 1.16^{d}	10542.33 ± 93.53^{h}
4	1	1	-1	36.24 ± 0.02^{f}	2229.17 ± 83.33^{g}	325.98 ± 1.33^{e}	2192.24 ± 3.54^{c}	140.56 ± 5.15^{f}	19773.35 ± 0.00^{a}
5	-1	-1	1	53.99 ± 0.14^{c}	2862.04 ± 10.19^{f}	360.12 ± 1.05^{c}	2484.15 ± 6.36^{b}	147.86 ± 0.44^{de}	14380.95 ± 144.31^{ef}
6	1	-1	1	35.00 ± 0.06^{g}	5640.05 ± 63.66^{a}	314.18 ± 0.62^{g}	1907.17 ± 7.07^{h}	99.97 ± 2.20^{h}	18643.58 ± 714.25^{b}
7	-1	1	1	70.39 ± 0.12^{a}	2073.41 ± 65.48^{h}	260.09 ± 1.13^{k}	1826.61 ± 3.54^{i}	280.72 ± 1.88^{a}	9333.90 ± 20.04^{i}
8	1	1	1	22.29 ± 0.08^{j}	3437.50 ± 9.96^{e}	309.89 ± 0.94^{h}	2100.17 ± 7.04^{e}	149.00 ± 1.27^{de}	13205.21 ± 363.83^{g}
9	0	0	0	34.42 ± 0.08^{h}	3819.44 ± 21.83^{c}	262.96 ± 1.45^{j}	1966.91 ± 2.87^{g}	190.22 ± 3.60^{b}	14636.65 ± 153.72^{e}
10	0	0	0	36.21 ± 0.49^{f}	4021.88 ± 11.46^{b}	319.55 ± 1.40^{f}	2100.22 ± 7.07^{e}	116.74 ± 0.98^{g}	13758.67 ± 147.11^{fg}
11	0	0	0	41.25 ± 0.01^{e}	3372.86 ± 35.26^{e}	324.43 ± 2.18^{e}	2027.82 ± 7.07^{f}	144.15 ± 1.02^{ef}	13094.13 ± 259.62^{g}
						USL[1]			
1	-1	-1	-1	43.84 ± 0.13^{d}	3230.16 ± 65.48^{c}	335.44 ± 1.59^{c}	2220.44 ± 1.66^{c}	162.27 ± 2.18^{b}	15642.86 ± 137.75^{a}
2	1	-1	-1	18.72 ± 0.04^{j}	4583.33 ± 20.83^{a}	266.16 ± 0.72^{h}	1773.86 ± 4.04^{g}	97.04 ± 1.07^{h}	15269.48 ± 68.87^{b}
3	-1	1	-1	52.86 ± 0.07^{a}	1014.88 ± 10.91^{f}	294.00 ± 1.11^{f}	2458.37 ± 3.13^{a}	149.51 ± 1.72^{c}	8439.29 ± 378.81^{h}
4	1	1	-1	23.31 ± 0.02^{h}	3250.00 ± 20.83^{c}	356.01 ± 1.21^{b}	2408.94 ± 4.95^{b}	131.15 ± 1.44^{d}	11645.32 ± 174.16^{g}
5	-1	-1	1	52.27 ± 0.02^{b}	2715.63 ± 57.29^{de}	332.50 ± 2.08^{d}	1719.34 ± 3.54^{h}	134.18 ± 0.06^{d}	12136.90 ± 168.36^{f}
6	1	-1	1	41.10 ± 0.07^{g}	4055.25 ± 49.82^{b}	298.97 ± 0.87^{e}	1705.02 ± 5.66^{h}	101.31 ± 0.34^{g}	14845.47 ± 72.85^{j}
7	-1	1	1	48.21 ± 0.03^{c}	1042.71 ± 11.46^{f}	281.12 ± 0.60^{g}	2113.32 ± 4.24^{d}	209.40 ± 0.82^{a}	7776.79 ± 63.13^{i}
8	1	1	1	21.38 ± 0.03^{i}	2808.76 ± 58.76^{d}	243.94 ± 1.13^{j}	1629.75 ± 2.83^{i}	70.22 ± 0.52^{i}	12247.71 ± 46.34^{f}
9	0	0	0	41.86 ± 0.26^{e}	3139.58 ± 91.67^{c}	256.68 ± 0.88^{i}	1827.51 ± 4.24^{f}	121.05 ± 2.27^{e}	13018.21 ± 0.00^{e}
10	0	0	0	41.45 ± 0.06^{f}	2640.87 ± 21.83^{e}	293.16 ± 1.51^{f}	1870.15 ± 11.35^{e}	109.24 ± 1.04^{f}	13775.97 ± 22.96^{d}
11	0	0	0	41.51 ± 0.05^{f}	2627.78 ± 20.37^{e}	257.14 ± 0.96^{i}	1776.68 ± 21.21^{g}	131.34 ± 1.27^{d}	12781.48 ± 64.20^{e}

[1]Extraction methods: PSL = Percolated Solid-Liquid, CSL = Conventional Solid-Liquid, USL = Ultrasonic-assisted Solid-Liquid. [2]Global yield extractions in %. [3]Bioactive compounds: TPC (Total Phenolic Compounds) in mg GAE/100 g extract; TF (Total Flavonoids) in mg/100 g extract; TA (Total Anthocyanins) in mg/100 g extract. [4]Antioxidant activity: DPPH (2,2-diphenyl-1-picrylhydrazyl) resulting in IC50 value μg·mL^{-1}; FRAP (Ferric Reducing Ability of Plasma) resulting in μmol/100 g extract.

Results expressed as mean ± standard deviation of three replicates. Means followed by different letters in the same column are significantly different by Tukey's test ($p < 0.05$).

ANOVA showed the linear effects ($p < 0.05$) in the reduction of the washing cycle during extraction of TPC by PSL, showing a positive influence. In spite of the well-adjusted R^2, ANOVA and Fisher test showed that the factors studied did not have statistical significance, Table SM2 contained in supplementary material. The three-dimensional graphs of the response surface and Pareto based on the experimental data are presented in Figure 2ab.

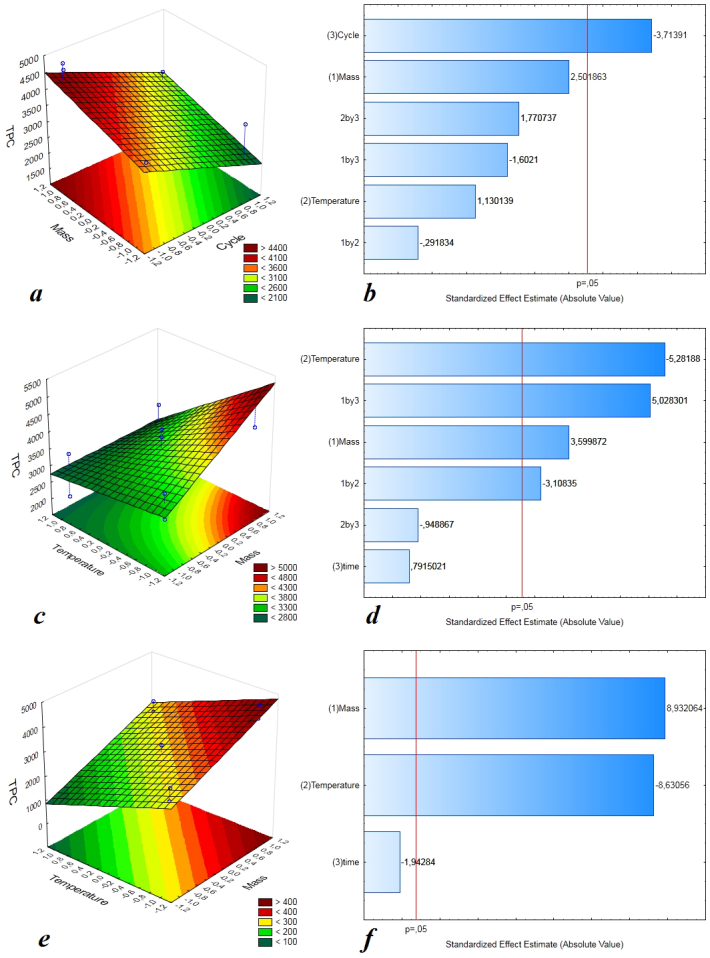

Figure 2. Response surface for the effects and Pareto graph on the extraction of the Total Phenolic Compounds (TPC) content by Percolation solid-liquid, Conventional Solid-Liquid (CSL), and Ultrasonic-assisted Solid-Liquid (USL) extractions in the freeze-drying sample with linear effects (L) and quadratics (Q). Response surface for the effects of mass and cycle (a), Pareto graph (b), on the extract LYO for PSL; response surface for the effects of temperature and mass (c), Pareto graph (d), on the extract LYO for CSL; response surface for the effects of temperature and mass (e), Pareto graph (f), on the extract LYO for USL.

The TPC of the CSL extract increased significantly with decreasing temperature (run from 7 to 6), reflecting the characteristics of these compounds being thermosensitive. Thus, the increase in temperature results in degradation (Joana Gil-Chávez et al., 2013). The increase in mass caused a decrease in TPC due to possible solvent saturation (Cacace & Mazza, 2003). The response surface and Pareto graphs are shown in Figure 2cd; the variables were evaluated statistically (Table SM2 contained in supplementary material). The R^2 of the CSL represented a good adjustment in the analysis of variance and the Fisher test showed that the variables had a significant influence on the TPC response. Through the CCD, it was possible to measure the effect of different planning conditions and the interaction between them (Figure SM2b contained in

supplementary material). The model that describes the behavior of the TPC for CSL (mg GAE·100 g^{-1}) is represented by Equation 5:

$$TPC = 3498.202 + 432.044 \cdot m - 603.913 \cdot T - 373.054 \cdot m \cdot T + 603.480 \cdot m \cdot t \quad (5)$$

where: m = mass (g), T = temperature (°C), and t = time (h).

The model that describes the USL process and represented by Equation 6 was built from the R^2 of the significant variables: mass and temperature, and according to ANOVA and Fisher's test (Table SM2 contained in supplementary material) the equation is significant and predictive, therefore allows the prediction of TPC values for USL extracts from the LYO sample. The response surface to better visualize the effects is shown in Figure 2e. It is noted that the mass has more influence than temperature and that the best extraction condition was 9 g/30 °C. The fit of the model can be checked in Figure SM2c contained in supplementary material.

$$TPC = 2828.087 + 836.747 \cdot m - 808.503 \cdot T \quad (6)$$

Where: m denotes mass (g) and T is the temperature (°C).

The determination of the chemical profile for the LYO was performed in the extracts of the 2-PSL, 6-CSL and 2-USL for LYO.

3.2.3.2 Total flavonoid content

The highest amounts of TF for LYO sample are in the tests with shorter extraction cycle and time. The characteristics of the particle, smaller size and greater porosity, provide a greater contact area between solute and solvent, and consequently increases the mass transfer in the process. Other authors have also reported the influence of particle size and porosity in the extraction process (Ameer et al., 2017; Migliato et al., 2009; Seraglio et al., 2018). Considering the extraction time, PSL is effective. TF readings on LYO sample extracts are shown in Table 4.

3.2.3.3 Total anthocyanins content

The smallest mass was positive and effective for the TA extraction process in the sample pre-treated by LYO (Table 4). The maximum extraction of TA in the LYO sample was in run 3 by CSL. However, this amount is similar to that found in the extracts by PSL. In this sense, the simplest method (PSL) proved to be more efficient for extraction of TA from jambolans.

When extracting anthocyanin from the fruit of jambolan with a solution containing ethanol and hydrochloric acid, Faria et al. (2011) reported lower results than this research, proving the importance and good performance of the pre-treated material and the use of water as a solvent to obtain "green" extracts of jambolan rich in bioactive compounds.

3.2.3.4 Antioxidant activity by DPPH and FRAP

The Table 4 shows the values of the antioxidant activity in the extracts from the LYO samples obtained by PSL, CSL, and USL. In general, the extracts from the LYO sample showed greater antioxidant activity than the ACO samples, and this increase is correlated with the higher levels of TPC (Benherlal & Arumughan, 2007), resulting from the particle size and porosity.

The antioxidant activity by DPPH is inversely proportional to the IC50 value. That is, the lower the IC50 value, the greater its antioxidant capacity by reducing DPPH by 50%. The pulp and peel of the jambolan are rich in flavonoids, mainly anthocyanins, carotenoids, vitamin C, and sugars (Faria et al., 2011; Rufino et al., 2010; Tavares et al., 2016). From these results, it can be inferred that, for LYO (the lower mass condition) and the PSL processes were efficient in extracting compounds with high stability and antioxidant power.

3.3 Chemical profile of the best extracts

Studies carried out on fruits from Ecuador classify fruits according to the TPC in levels: low (less than 100 mg GAE·100 g^{-1}); medium (between 100 and 500 mg GAE·100 g^{-1}); and high (above 500 mg GAE·100 g^{-1}) (Vasco et al., 2008). Based on this concept, jambolan pulp and peel samples are considered to be high TPC products (Table 3 and Table 4).

CG-MS was responsible for the identification of volatile compounds and HPLC for the quantification of phenolics and anthocyanins.

3.3.1 Gas chromatography coupled to mass spectrometry

The volatile compounds were identified from the comparison of the mass spectrum of the substance with the database for GC-MS. The identified components are shown in Table 5.

Table 5. Compounds identified by Gas Chromatography coupled to Mass Spectrometry (GC-MS) in jambolan extracts.

CHEMICAL COMPOUNDS	RT (min)	% AREA					
		PSL[1]	CSL[1]	USL[1]	PSL[2]	CSL[2]	USL[2]
Benzoic acid, methyl ester	11.06	1.19	2.6	3.04	2.92	2.95	4.25
Butyrolactone	5.25		1.07	5.45		1.19	1.54
Bicyclo (2,2,1) heptane, 2-chloro-2,3,3-trimethyl	22.50			40.3			
1,2-Benzenedicarboxylic acid bis (2-methylpropyl)	40.85			12.0			
Canfeno	22.50		34.6				
Cyclohexane, 1-methyl-3- (1-methylethenyl) -, (+/-)	22.50	49.9			46.2	21.8	34.6
Diethyl phthalate	31.20	1.33				0.84	
Ethanone, 2,2-dimethoxy-1,2-diphenyl	41.38			7.83			
Glycerol 1,2-diacetate	21.60					0.66	
Heptane, 2,2,3,5-tetramethyl-	7.29				1.7		
Hexadecanoic acid, methyl ester	42.77	6.29	2.0	2.47	3.8	1.92	3.91
Phthalic acid, 5-methylhex-2-yl butyl ester	40.85	4.7					
Phthalic acid, trans-hex-3-enyl isobutyl ester	40.85				2.23		
Phthalic acid, decyl isobutyl ester	40.85					4.31	
Phthalic acid, 2-ethoxyethyl ethyl ester	31.20						1.12
Pentanic acid, isobutyl ester of ddd-trimethyl d-carboxyisopropyl isobutyl ester	31.33						1.39
2,2,7,7-Tetramethyloctane	7.30						1.11

[1]Air circulation oven. [2]Freeze-drying. PSL = Percolated Solid-Liquid, CSL = Conventional Solid-Liquid, USL = Ultrasonic-assisted Solid-Liquid. RT - RetentionTime (min), and Area % - relative area detected by Gas Chromatography coupled to Mass Spectrometry (GC-MS).

The compound cyclohexane, 1-methyl-3- (1-methylethenyl), also known as carvestrene, is an isomer of limonene and is commonly used in cosmetics in the creation of perfumes. In addition, D-limonene is used in the food industry to mask the bitter taste of alkaloids. In agriculture, it is used as an insecticide and a disinfectant in handkerchiefs. Due to its high antimicrobial power; it presents an alternative to acetone to dissolve polystyrene (Zheng et al., 2005; Zhou et al., 2013).

In ACO by CSL, camphene, a bicyclic monoterpene, was found in large quantities. According to Simões & Schenkel (2002), terpenoids are common in essential oils such as monoterpenes (90% of volatile oils) and sesquiterpenes; this compound is found in seeds, flowers, leaves, roots, and plant wood, also in mosses and algae. Oxygenated monoterpenes are targets of interest to the fine chemicals, agrochemicals, and fragrance industries.

The presence of the compounds hexadecenoic acid, methyl ester (fatty acid, palmitic acid) and benzoic acid stand out. Benzoic acid is an aromatic compound, classified as carboxilic acid; usually applied as a food preservative and is also an important precursor to the synthesis of other organic substances, such as salicylic acid and 2-acetylsalicylic acid (aspirin). The volatile compounds present in the aqueous extracts of jambolans showed the value of their biological activity and can be used in several therapeutic applications.

3.3.2 High-Performance Liquid Chromatography (HPLC)

The high antioxidant capacity of jambolan is attributed to the presence of phenolic compounds and anthocyanins, about 74 individual phenolic compounds have been detected in the edible parts of the jambolan, mainly in the peel (Tavares et al., 2016).

Table 6 shows the content of compounds quantified by HPLC. The compounds found in highest concentrations were the flavonoids: (+) - catechin, tyrosol, and epicatechin, compounds with high antioxidant potential, both due to the ability to bind to metal ions and to capture free radicals; prominence in the prevention and treatment of neurodegenerative diseases (Revilla & Ryan, 2000; Reynertson et al., 2008). The catechin content ranged from 7.29 to 32.06 mg·100 g^{-1}, with similar results in CSL-ACO and PSL- LYO. Epicatechin had little variation between extracts, showing the influence of sample granulometry and porosity.

Table 6. Chemical composition profile of the jambolan extracts determined by High Performance Liquid Chromatography on a Diode Array Detector (HPLC-DAD).

COMPOUNDS	CONCENTRATION (mg/100 g of extract)					
	PSL[1]	CSL[1]	USL[1]	PSL[2]	CSL[2]	USL[2]
PHENOLICS						
Trans-caftaric acid	0.74	0.90	0.74	0.80	0.66	0.81
Tyrosol	14.19	17.12	9.61	16.05	10.22	11.78
(+) – Catechin	20.62	32.06	7.29	15.01	8.56	15.56
Caffeic acid	0.12	0.21	0.12	0.06	0.10	0.13
(+) – Epicatechin	8.65	8.66	7.43	5.97	6.39	6.85
P-coumaric acid	1.50	1.57	1.51	1.43	1.35	1.47
Ferulic acid	0.18	0.22	0.17	0.15	0.14	0.16
Miricetine	0.95	1.67	0.98	1.21	0.98	1.20
Trans-resveratrol	0.89	0.86	0.86	n.q.	n.q.	n.q.
Quercetin	1.30	1.35	1.30	1.32	1.21	1.35
Kaempfenol	1.01	1.02	0.99	1.00	0.92	1.03
ANTHOCYANINS						
Cyanidin - 3,5 - diglucoside	16.32	31.91	20.03	27.95	20.27	27.10
Delphinidin - 3 - O - glucoside	6.26	8.27	6.96	7.82	8.51	10.71
Malvidin - 3, 5 - diglucoside	2.39	3.97	1.93	4.21	2.42	3.04
Cyanidin - 3 – O - glucoside	10.39	19.02	10.17	20.54	12.44	17.25
Peonidin - 3 - O - glucoside	0.11	0.13	0.11	0.23	0.08	0.11
Malvidin – 3 – O - glucoside	0.34	0.59	0.24	0.66	0.40	0.54

[1]Air circulation oven. [2]Freeze-drying. PSL = Percolated Solid-Liquid, CSL = Conventional Solid-Liquid, USL = Ultrasonic-assisted Solid-Liquid. n.q. = not quantifie.

The extracts showed tyrosol values between 9.61 and 17.12 mg·100 g^{-1}. Wine samples showed tyrosol concentrations ranging from 9.04 to 46.86 mg·100 g^{-1}, that may be influenced by factors such as temperature,

nitrogen availability, and sugar content of grapes, which affect yeast activity during fermentation (Sartor et al., 2017).

Trans-resveratrol, a stilbene abundant in grapes, was identified in extracts and could be quantified in the ACO sample, and in LYO, there was interference at the peak, making quantification impossible. When identifying this compounds in wines, Sartor et al. (2017) found concentrations of trans-resveratrol between 0.50 and 4.94 mg·100 g^{-1}. This compound is widely studied because it plays an important role in health due to its antioxidant potential and cardioprotective effect (Shrikanta et al., 2015).

The main anthocyanin in the jambolan extracts was cyanidin-3,5-diglucoside, followed by cyanidin-3-O-glucoside. The identifications were confirmed by comparing retention times with those of authentic standards. The results are consistent with reports from previous studies (Brito et al., 2007). This study demonstrated that the insertion of an appropriate treatment to the material before the extraction process can be an alternative to improve the yield and the quality of the extracts (Baliga et al., 2011).

4 Conclusions

The combination of processes, drying, and extraction, prove the efficiency in obtaining bioactive compounds from the shell and pulp of a jambolan. In addition to verifying the efficiency of a solvent-free residual process, the chemical characterization provided important data, showing the potential use of aqueous extracts of jambolan rich in bioactive compounds for in vivo evaluation of the antioxidant potential, and treatment of degenerative diseases. The volatile compound cyclohexane, 1-methyl-3-(1-methylethenyl) was found by GC in the extracts of jambolan peel and pulp, with higher amounts in the LYO samples. Among the components quantified by HPLC in the obtained extracts were the anthocyanins: cyanidin-3,5-diglucoside; cyanidin-3-O-glucoside; and the phenolics: catechin, tyrosol and epicatechin.

In general, the increase in temperature during the process negatively could affect the result of the extraction of bioactive compounds and, consequently, reduce their antioxidant potential.

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Supplementary Material

Supplementary material accompanies this paper.

Table SM1 Analysis of variance for total compounds phenolics in extraction methods for drying in Air Circulation Cven.

Table SM2 Analysis of variance for total compounds phenolics in extraction methods for drying in Freeze-Drying sample

Fig. SM1 Response surface, and Pareto graph for total phenolic compounds by percolated solid-liquid extractions in the drying in air circulation oven

Fig. SM2 Graph of observed values *versus* predicted values for the significative ANOVA in the drying in air circulation oven and freeze-drying methods

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