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ORIGINAL ARTICLE

Ultrasound-assisted extraction of bioactive compounds from ciriguela (*Spondias purpurea* L.) peel: Optimization and comparison with conventional extraction and microwave

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Abstract The ciriguela (*Spondias purpurea* L.) residue resulting from its pulp and juice processing stands out due to the high content of bioactive compounds. This study aimed to optimize the ultrasound-assisted extraction (UAE) process of phenolic compounds from ciriguela peel. The response surface method was used to investigate the effects of process-independent variables (ultrasonic amplitude, UA): 20%, 60% and 100%, and ultrasonic exposure time (T): 5, 10 and 15 min on the dependent variables (content of total phenolic compounds (TPC), DPPH – 1,1-diphenyl-2-picrylhydrazyl free radical scavenging (IC_{50}) and ferric reducing-antioxidant power (FRAP) of ciriguela peel extract. The UA and time influenced TPC, IC_{50} and antioxidant activity by FRAP. However, the antioxidant activity of DPPH had no significant influence on the variables used. Ideal conditions were set at UA = 100% (200 W) and T = 15 min. The extract of phenolic compounds from the ciriguela peel obtained by optimized ultrasound was compared with other extraction techniques (conventional and microwave-assisted). UAE showed better results concerning the extraction yield of phenolic compounds and high antioxidant activity (35.15 mg GAE/g, IC 50 = 0. 19 mg/mL), compared to conventional extraction (30.10 mg GA/g and IC 50 = 1.68 mg/mL) and microwave-assisted (23.31 mg GA/g and IC 50 = 4.29 mg/mL). These results demonstrate

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the efficacy and better usefulness of the ciriguela residue in obtaining the extracts of bioactive compounds using the ultrasound-assisted extraction technique.

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1. Introduction

Brazil is a country with favorable geographical and climatic characteristics for fruit production. In the semiarid region, the Caatinga biome has an immense biodiversity of fruits, particularly those of the genus *Spondias*, mainly ciriguela (*Spondias purpurea* L.), which originated in Mexico and Central America. This fruit is rich in secondary metabolites, particularly phenolic compounds and is of biological interest (Maldonado-Astudillo et al., 2014).

In recent decades, there has been an increase in the international consumption of tropical fruits. Considering this increase, the processing of fruits into products such as juices and beverages showed high growth, mainly due to the possibility of producing new sources of functional and healthy ingredients (Jeddou et al., 2017). However, this high growth in the pulp and fruit juice industries has generated a large volume of waste, which can be exploited for the production of highly valued substances (Ben-Othman et al., 2020; Saleem and Saeed, 2020). Fruit residues are rich in bioactive compounds. However, these substances degrade easily during food extraction, processing and storage (Renard, 2018).

The extraction of the compounds of interest present in fruit residues can generate products with high added value (Kringel et al., 2020). This extraction can be carried out by conventional methods, using maceration and/or agitation with solvents and by Soxhlet extraction, which is based on the capture of the compounds of interest from a solute or matrix, with or without heat. However, it requires long times to reach the maximum concentration of the compounds of interest and a high solvent demand and thermal degradation due to the long processing time (Caldas et al., 2018). Considering these disadvantages, more sustainable methods are sought, in which solvent consumption and the extraction time are minimized and the extraction yield is increased (Pintać et al., 2018).

The recent interest in operating an environmentally sustainable way as well as safety and economic aspects led to the best choice of applying new "green" extraction techniques (Noroozi et al., 2021). The innovative or nonconventional technologies that have been used include the application of pulsed electric fields, ultrasound-assisted extraction (UAE), microwave-assisted extraction, high pressure and solvent acceleration (Putnik et al., 2018). UAE stands out and uses mechanical waves that present frequencies between 20 and 100 kHz (Dadan et al., 2018). UAE has several advantages: shorter processing time, better penetration, low solvent consumption, higher yield, good reproducibility, improves the extraction rate and quality of the extract, allows the possibility of using more economical and safer alternative solvents for the environment and health (Chemat et al., 2017; Marić et al., 2018). The UAE process allows complete extraction of target compounds in short time mainly via production of hydrodynamic cavitation phenomenon. Different stages of hydrodynamic cavitation phenomenon were: nuclei formation, expansion phase, maximum radius, collapse phase, explosion and release of energy (Noroozi et al., 2021).

The main contribution of this manuscript was the consideration of the possibility of the valorization of ciriguela peel generated during ciriguela processing or beverage production. The growing global interest in emerging extraction technologies, which aim to minimize environmental impacts, justifies the use of ciriguela residue as a source of antioxidants, which have a high content of phenolic compounds. Since the use of RSM has not been reported yet for modeling the UAE of phenolic compounds from ciriguela peel, the aim of this study was to evaluate and optimize the extraction process of phenolic compounds from using UAE from ciriguela peel, maximizing the yield of phenolic compounds and antioxidant activity of ciriguela peel extracts, in addition to comparing conventional and microwave-assisted extraction processes.

2. Materials and methods

2.1. Materials

Ciriguela residues (*Spondias purpurea* L.) were supplied, as a byproduct, by the fruit pulp industry, located in João Pessoa, Paraíba, Brazil (07° 09′ S 36° 49′ W).

2.2. Chemicals

Ethanol (Vetec, Rio de Janeiro, Brazil) and distilled water were used for extraction. Folin-Ciocalteu phenol reagent, gallic acid ($C_7H_6O_5$), 2,4,6-tripyridyl-s-triazine (TPTZ) and 2,2-diphenyl-1-picrylhydrazyl radical (DPPH$^{\bullet}$) were obtained from Sigma Aldrich (St. Louis, MO, USA).

2.3. Obtaining the ciriguela residue flour (FRC)

The ciriguela peels were separated from the seeds manually and subjected to drying at 60 °C for 24 h (Caldas et al., 2018) in an oven with circulation and air renewal (Marconi; model MA035) until the moisture reached at or below 10%. Next, they were crushed in a 631/2 multiuse mill (Tecnal) and sieved using a sieve mesh #40 (425 μm). The FRC was packed in 140-μm low-density polyethylene bags, wrapped with laminated paper and frozen at −18 °C for further analysis.

2.4. Extraction processes of phenolic compounds

2.4.1. Preliminary analysis

In order to optimize the extraction conditions, initially preliminary extractions were carried out using UAE. The extractions were performed at specified conditions, ultrasound power (120 W) and extraction time (10 min). To evaluate the extracts by ultrasound, water (100%) and ethanol (20%, 50% and

80%) were used as solvents, acidified with 0.1% HCl. Ethanol is in compliance with good manufacturing practice and it is considered as a GRAS (generally-recognized-as-safe) solvent (Santos et al., 2010). The parameters used were: 10 g of FRC mixed with 40 mL of the solvent, was placed in a 100-mL beaker and submitted to an ultrasonic probe (Ultronique, Ecosonics) that has an ultrasonic frequency of 20 kHz. The extract was filtered using Whatman N° 2 filter paper. The extracts were stored in amber bottle which were kept in a freezer (−20 °C) until analysis.

2.4.2. Ultrasound-assisted extraction (UAE)

Ultrasound experiments were carried out using 10 g of FRC placed in an extraction unit with 40 mL of solvent (selected in the pre-tests). The sample container was covered with aluminum-foil paper to prevent oxidative change from light. In the UAE procedure, the sonicator (QR1000 Ultronique, Ecosonics - Brazil) used in this study has a constant frequency of 20 kHz, that has a maximum power of 1000 Watts (W) and a horn microtip with diameter of 25.4 mm. UAE process variables including ultrasonic intensity and time were investigated as outlined in Table 3. However, the temperature was controlled using a water bath around the extraction flask. The obtained extracts were filtered and kept in the dark at −20 °C for further analysis.

The UAE optimization of FRC phenolic compounds was developed using Statistica 7.0 software (StatSoft, Tulsa, USA). The central experimental design, comprised 8 factorial points (levels ± 1) and 5 central points (level 0). The independent variables were ultrasonic amplitude (UA) (20, 60 and 100%) and time (t) of exposure to ultrasound (5, 10 and 15 min), and the dependent variables (response) were the total phenolic compounds (TPC), ferric reducing-antioxidant power (FRAP) and 1,1-diphenyl-2-picryl hydrazyl (DPPH) free radical scavenging activity expressed as the IC_{50}.

Five replicates of the central point of the experimental design were used to estimate the value of the pure error and sum of squares. Because the various responses were the result of the interactions of the independent variables, the data for all the responses were adjusted to the second-order polynomial regression equation, Eq. (1).

$$y = \beta_0 + \sum_{j=1}^{k} \beta_j x_j + \sum \sum_{i<j} \beta_{ij} x_i x_j + \sum_{j=1}^{k} \beta_{jj} x_j^2 + \in \quad (1)$$

Where Y represents the predicted response, β_0 is the constant regression coefficient, βi, βjj and βij are the linear, square and interaction coefficients, respectively, Xi and Xj are the independent variables, and ε is noise or error (Azarpazhooh and Ramaswamy, 2012). The quality of the adjusted polynomial models was expressed by the regression coefficient (R^2), adequate precision (AP) and variation coefficient (CV).

The experimental data were adjusted to the proposed model, and analysis of variance (ANOVA), the lack of fit test (F test), determination of the regression coefficients and obtaining the response surfaces were performed using Statistica 7.0 software (StatSoft, Tulsa, USA) at the 5% significance level.

2.4.3. Microwave-assisted extraction (MAE)

For microwave extraction, 0.33 g of FRC was mixed with 20 mL of ethanol-water (80%), and then the mixture was subjected to a CEM Discover microwave (Discover System model 908005) using a power of 800 W, a temperature of 120 °C and an extraction time of 15 min (Best result of preliminary analysis). After exposure to electromagnetic waves, the supernatant obtained was filtered and stored in an amber container at −20 °C until further analysis.

2.4.4. Conventional extraction by maceration (MCE)

Three replicates (10.0 g) of FRC were extracted in 40 mL of 80% ethanol-water (v/v), by using a dynamic maceration, brought to room temperature (25 °C) and kept under stirring for 1 h. After extraction, suspended solids were removed by filtration through qualitative filter paper and the extracts obtained were stored at −20 °C until further analysis.

2.5. Physico-chemical analysis of FRC

2.5.1. Soluble solids and titratable acidity

The according to the methodology described by AOAC (2006). Soluble solids was expressed as °Brix and acidity were expressed as g of citric acid/100 g of FRC.

2.5.2. pH

The pH was measured directly measurements using a pH meter with a glass electrode (AAKER).

2.5.3. Moisture

The moisture content was determined using an infrared moisture balance (MARTE-IDSO, São Paulo, Brazil) and heating at 105 °C for 30 min. The results are expressed as (%) (AOAC, 2006).

2.5.4. Water activity

Water activity was measured using a water activity analyzer (DECAGON, AQUA LAB − 4TE) at 25 °C.

2.5.5. Color

The color was evaluated in a colorimeter (Minolta CR 400; Konica Minolta, Sensing Inc.), using the color standards of the CIELab ("Commission Internationale de L'Eclairage"). The instrumental color was determined on the surface of the ciriguela residue flour. The colorimeter was previously calibrated with a white standard before each analysis using a xenon lamp, illuminant C (Y = 92.78; x = 0.3139; y = 0.3200), an observation angle of 10° and a measuring area of 8 mm in diameter.

2.6. Analysis of bioactive compounds and antioxidant activity

2.6.1. Total phenolic compounds (TPCs)

The TPC content was determined spectrophotometrically, where the absorbance was quantified at 725 nm using Folin–Ciocalteu reagent and ethanol as the solvent, according to the methodology described by Wettasinghe and Shahidi (1999). Briefly, the reaction was conducted in test tubes. An amount of 0.5 mL of each sample was incubated with 8.0 mL of distilled water and 0.5 mL of Folin-Ciocalteau reagent. After 3 min 1.0 mL of sodium carbonate solution was added and left to react for 60 min in the dark. The TPCs

was calculated using a standard curve prepared from the aqueous solutions of gallic acid (0.1–1 mg/mL) and results were expressed in mg of gallic acid equivalent (GAE)/g of FRC.

2.6.2. Antioxidant activity by DPPH

The determination of the free-radical scavenging capacity was evaluated with the stable radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) as described by Brand-Wiliams et al. (1995) and modified by Sánchez-Moreno et al. (1998). The extract was diluted at three different concentrations of total phenolics. Briefly, 0.1 mL of appropriately diluted FRC extracts samples was added to 3.9 mL of DPPH (0.03 mg/ml) in methanol. The decrease in absorbance at 517 nm was monitored until the reaction reached a plateau (30 min) of reaction, using a spectrophotometer (Shimadzu UV-1650PC). The results were expressed as the IC_{50} (concentration of the extract in μg/mL that can react with 50% of the radicals present in the DPPH solution).

2.6.3. Ferric reducing antioxidant power (FRAP)

The FRAP test was performed according to the methodology reported by por Thaipong et al. (2006). The absorbance was measured at 593 nm, using a spectrophotometer (Jenway 6705 UV/Vis). FRAP reagent was freshly prepared by mixing a 10 mM 2,4,6-tris (1-pyridyl)-5-triazine (TPTZ) solution in 40 mM HCl with a 20 mM, $FeCl_3$ solution and 0.3 M acetate buffer (pH 3.6) in a proportion 1:1:10 (v/v/v). A calibration curve was prepared with aqueous solution of $FeSO_4$ (2.5, 5.0, 7.5 and 10 mg/L). In test tube 90 μL of the filtered and duly diluted extract with 270 μL of distilled water and 2.7 mL of FRAP reagent. Then, the reaction mixture was incubated at 37 °C for 30 min. Antioxidant activity by FRAP were expressed as μmol of ferrous equivalent per g of extract (μmol Fe^{2+}/g).

2.7. Statistical analysis

All the experiments were performed in triplicate, and the results were expressed as the mean values ± standard deviation. Statistical analysis of the model was performed using Statistica 7.0 software.

3. Results and discussion

3.1. Physico-chemical properties

The data on the soluble solid content, pH, titratable acidity, moisture and colorimetric parameters of the FRC are presented in Table 1.

The FRC presented 4.63 °Brix of soluble solids (Table 1), indicating the presence of water-soluble compounds and substances, such as sugars, acids, vitamin C and some pectins (Maldonado-Astudillo et al., 2014). The pH (3.79) characterized FRC as an acid product. The FRC acidity obtained as 1.57 g/100 g of citric acid (Table 1) was similar to that reported in the study by Neris et al. (2017), which characterized ciriguela peel and obtained 1.66 g/100 g of citric acid. The water activity found in the FRC (0.178) was considered a low value (the value varies from 0 to 1). The presence of moisture in plant matter indicates the possibility of microbial growth during storage (Shardul et al., 2013), and FRC showed low moisture (5.83%). The colorimetric parameters (L a* b*) remained on the positive side, indicating a shade of red to yellow, and the value of L was 61.86, indicating that the FRC has clear luminosity.

Table 1 Physico-chemical characterization of FRC.

Parameters	Mean values ± SD
Soluble solids (°Brix)	4.66 ± 0.15
pH	3.79 ± 0.15
Acidity (g/100 g citric acid)	1.57 ± 0.06
aw	0.178 ± 0.04
Moisture (%)	5.83 ± 0.01
L*	61.86 ± 1.05
a*	16.26 ± 0.34
b*	31.52 ± 0.17

Means ± standard deviation (n = 3).

3.2. Preliminary experiments – Solvent selection

Selection of the best solvent for extraction by UAE was based on the values of phenolic compounds of the extracts. Table 2 shows the data on phenolic compounds from the extracts obtained using water and ethanol (20, 50 and 80%) as solvent.

Ethanol has been found to possess the highest affinity for phenolics and hence it is the first choice for the extraction of phenolic compounds from fruit and vegetable waste (Ramić et al., 2015). Table 2 shows that the extract obtained with 80% ethanol had a higher content of phenolic compounds. This was expected because the solubility of polyphenols increases with increasing concentrations of ethanol (He et al., 2016). Noroozi et al. (2021) studying continuous ultrasound-assisted extraction of *Cucurbita pepo* seeds, revealed that with increasing ethanol concentration, the phenolic compounds recovery increased and reached a maximum value at the ethanol concentration of around 80%, and then slightly decreased. This is because the polarity of the solvent decreases, and the similarity in the solvent to the polyphenols polarity increases, causing an increase in the solubility of polyphenols. Increase in ethanol concentration increases the yield of phenolic compound until a maximum ethanol concentration and then it

Table 2 Extraction of phenolic compounds from FRC using different solvents.

	TPC (mg GAE/g of FRC)
Water	23.04^{c} ± 1.50
Etanol 20%	43.70^{b} ± 0.37
Etanol 50%	45.93^{b} ± 2.29
Etanol 80%	56.38^{a} ± 0.98

Means ± standard deviation (n = 3).
Means in each column followed by diferente superscript letters were significantly diferente (p > 0.05), by Tukey's test.

has negative effect on the yield. The ethanol concentration near 100% i.e. highly pure ethanol solvent causes dehydration of the tissue of plant along with denaturation of the protein leading to decreased yield at such high concentration (Kumar et al., 2021). Caldas et al. (2018) observed that the highest phenolic content was found for medium values of ethanol concentration (60%), within the studied range (8–92%), which may be related to the different polarities of phenolics present in grape skin. Savic and Savic Gajic (2020) analyzed the extraction of polyphenols from wheatgrass (*Triticum aestivum* L.) obtained maximum yield of phenolic compounds using 56% ethanol concentration.

3.3. Experimental design of FRC by UAE

The contents of the experimental variables used in this study and the results obtained for the quality parameters of the experimental design are shown in Table 3. TPC ranged from 14.56 to 35.15 mg GAE/g of FRC, antioxidant activity by IC_{50} from 0.19 to 3.51 mg/mL and antioxidant activity by FRAP from 7.624,40 to 17.373,73 μmol Fe^{2+}/g.

ANOVA for the quadratic model of the response surface is shown in Table 4. Not significant variables were omitted, and the other coefficients were used in the final predictive equations. The data showed a good fit with Eq. (1), which was statistically acceptable at $p < 0.05$.

The adequacy of the model was also evaluated by the residuals, which represent the difference between the observed and predicted values of the response (Maran et al., 2017). The residuals are thought of as the elements of variation unexplained by the regression model (Savic Gajic et al., 2019). The obtained residuals are plotted against the expected values in the normal probability plot (Fig. 1 a, b and c). The obtained plots of the model after excluding nonstatistically significant terms indicate that the residuals are normally distributed. The slight deviation of points from the straight line in the reduced model indicates a better prediction of the regression model.

3.4. Effect of process variables on TPC

The TPC of the FRC extracts and ANOVA of the results obtained are presented in Tables 3 and 4, respectively. Data analysis showed that the TPC was significantly affected by UA and t ($p < 0.05$). Additionally, it was possible to determine the significant regression coefficients for the TPC, as shown in Eq. (2).

$$TRC = 25.26 + 4.56X_1 + 4.39X_2 + 2.29X_1^2 + 0.63X_2^2 \quad (2)$$

The variables that significantly influenced the model for TPC were as follows: UA (X_1 and X_1^2) and t (X_2^2), as shown in Table 4. The lack of fit was significant for the TPC model, however, when the pure error value is low, that is, the reproducibility is very good, resulting in a false result of lack of fit, since the Fcalculated value is high due to the denominator being very low. Therefore, it does not explain the lack of fit. The model has statistical and predictive significance, and there is no lack of fit. To visualize the influence of variables on the TPC, the response surface graph (Fig. 2) was constructed.

As observed in tests 4, 2 and 12 (Table 3), the TPC increased slowly with the increase in UA and reached a peak at 100% UA, both obtained using 15 min of extraction time and resulting in 22.53, 30.01 and 35.15 mg GAE/g of FRC, respectively. Significant increases in TPC were observed as the UA range was increased during the extraction of phenolic compounds from the pomegranate peel (Sharayei et al., 2019). Opposite results have been obtained in some studies in which UA was not a significant factor in the extraction of phenolic compounds (Espada-Bellido et al., 2017; Saifullah et al., 2020).

The increase in UA causes an additional effect of cavitation and temperature increase, causing the explosion of bubbles, resulting in material swelling, solvent uptake, and pore

Table 3 Face-centered composite design 3^2 of UAE using two variables and the resulting quality response parameters of the FRC extract.

Exp. n0	UA (%)	Time (min)	TPC (mg GAE/g of FRC)	IC 50 (mg/mL)	FRAP (μmol Fe^{2+}/g)
1	60 (0)	10 (0)	29.40 ± 0.68	1.46 ± 0.13	15.403,95 ± 23.82
2	60 (0)	15 (+1)	30.01 ± 0.98	1.03 ± 0.08	15.653,02 ± 63.52
3	60 (0)	10 (0)	28.96 ± 0.30	1.40 ± 0.14	14.290,19 ± 45.91
4	20 (−1)	15 (+1)	22.53 ± 0.18	2.17 ± 0.23	11.456,33 ± 67.92
5	20 (−1)	10 (0)	20.42 ± 0.48	2.52 ± 0.09	10.857,03 ± 30.40
6	100(+1)	10 (0)	28.03 ± 0.25	2.64 ± 0.04	14.657,94 ± 85.25
7	20 (−1)	5 (−1)	14.56 ± 0.54	3.51 ± 0.04	7.624,40 ± 38.18
8	100 (+1)	5 (−1)	21,70 ± 0,10	2.99 ± 0.06	12.148,17 ± 90.78
9	60 (0)	10 (0)	29.73 ± 0.20	1.96 ± 0.07	15.749,00 ± 57.54
10	60 (0)	5 (−1)	25.06 ± 0.60	2.97 ± 0.07	9.573,12 ± 34.99
11	60 (0)	10 (0)	29.05 ± 0.30	1.95 ± 0.16	16.462,94 ± 74.69
12	100 (+1)	15 (+1)	35.15 ± 0.43	0.19 ± 0.02	17.373,73 ± 29.19
13	60 (0)	10 (0)	29.43 ± 1.22	1.48 ± 0.17	15.770,72 ± 97.94

UA: Ultrasound Amplitude.
TPC: Total Phenolic Compound.
FRAP: Ferric Reducing Antioxidant Power.
DPPH: scavenging activity of DPPH.
IC50: The concentration of extract required to scavenge 50% of 2, 2-diphenyl-1-picryl-hydrazyl free radical.
*Analytical results are the means ± SD (n = 3).

Table 4 ANOVA for the response surface quadratic model.

Source	df	TPC (mg GAE/g of FRC)	IC 50 (mg/mL de fenol)	FRAP (μmol Fe^{2+}/g)
Regression	4	984.98*	24.61*	2929070.19*
Residual	34	73.12	5.87	229854.43
Lack of fit	4	63.21*	4.55*	157999.76*
Pure Error	30	9.90 ^{ns}	1.31 ^{ns}	71854.67 ^{ns}
Cor Total	38	1058.10	30.48	3158924.62
R-Squared		0.93	0.81	0.93

ns: Not significant (p > 0,05).
*Significant at (p < 0,05).

enlargement in the materials (Bimakr et al., 2019; Gam et al., 2020; Gogoi et al., 2019). Cavitation effect works by imploding cavitation bubbles and thermal effect works by swelling and loosening the cell structure, which increase solute solubility and diffusivity (Poodi et al., 2018). The increase of ultrasonic power accelerates the destruction of cell walls, thereby promoting the extraction of the TPC into the extraction solvent (Gam et al., 2020). In addition, the penetration of the solvent into the solid matrix is improved, facilitating the mass transfer rate of phenolic compounds from the food matrix to the solvent (Gogoi et al., 2019).

The extraction time showed a positive linear effect on the extraction of phenolic compounds from FRC and led to a gradual increase in the TPC. Similar results were obtained in the study of extraction of antioxidants from plum seeds (Savic and Savic Gajic, 2021). The contact time allows for a higher mass transfer rate, resulting in better extraction efficiency. The operating time is very important during extraction because it helps to reduce electricity consumption as the operating time decreases (Chakraborty et al., 2020). The same effect was obtained in other studies (Fernandes et al., 2020; Saifullah et al., 2020).

As the extraction time increased, a higher TPC was obtained, according to tests 7 (14.56 mg GAE/g of FRC), 5 (20.42 mg GAE/g of FRC) and 4 (22.53 mg GAE/g of FRC), using 20% of ultrasonic amplitude and extraction time of 5, 10 and 15 min, respectively (Table 3). Gogoi et al. (2019) observed that the increase in extraction time from 8.00 to 14.00 min increased the yield of phenolic compounds, which subsequently decreased with a further increase in treatment time. Opposite results were obtained in some studies in which time was not a significant factor in the ultrasound-assisted extraction of phenolic compounds (Jovanovic et al., 2017; Li et al., 2016).

3.5. Effect of process variables on the antioxidant activity by DPPH (IC_{50})

IC_{50} represents the concentration of the extract necessary to inhibit 50% of DPPH free radicals. The following equation describes the IC_{50} predicted by the model (Eq. (3)) according to the coded and significant variables ($p \leq 0.05$).

$$IC = 2.14 - 0.39X_1 + 1.01X_2 - 0.29X_1^2 \quad (3)$$

The variables that significantly influenced the model for IC_{50} were UA (X_1 *and* X_1^2), and t (X_2). The not significant value of the lack of fit showed that the model was considered predictive for IC50 ($R^2 = 0.81$) (Table 4). The not statistically significant terms could be excluded from the second order polynomial equation in order to improve the prediction ability of the proposed model. The regression coefficients indicate that the linear effects and quadratic effect UA have a negative impact on the response.

UA caused a negative signal effect in IC_{50}, and the increase in IC_{50} was related to the increase in UA (Fig. 3). Similar results were obtained by Sharayei et al. (2019), in which the IC_{50} increase was observed when a UA up to 60% was used; additionally, when the UA was further increased, the IC_{50} decreased. Li et al. (2016) reported significant increases in the antioxidant activity (DPPH) as the ultrasound time was increased. In a study of the extraction of phenolic compounds from pomegranate peel, an increase in UA up to 60% was observed to increase the antioxidant activity (Sharayei et al., 2019).

3.6. Effect of the process variables on the antioxidant activity by FRAP

Through factorial experimental design, it was possible to determine the significant regression coefficients for antioxidant activity by FRAP, as shown in Eq. (4).

$$FRAP = 12883.45 + 2357.01X_1 + 2484.01X_2 + 820.78X_1^2 + 926.32X_2^2 \quad (4)$$

However, only the linear terms UA (X_1 and X_1^2) and t (X_2 and X_2^2) ($p \leq 0.05$) showed positive signal effects in the extraction. The not significant value of the lack of fit showed that the model was considered predictive for antioxidant activity by FRAP ($R^2 = 0.93$) (Table 4). The lack of fit was significant for antioxidant activity by FRAP model, however, when the pure error value is low, that is, the reproducibility is very good, resulting in a false result of lack of fit, since the Fcalculated value is high due to the denominator being very low. Therefore, it does not explain the lack of fit. The model has statistical and predictive significance, and there is no lack of fit.

The increase in UA likely promoted more efficient extraction of phenolic compounds, resulting in the increased antioxidant activity by FRAP (Fig. 4). This effect may be due to the cavitation process and vibration mechanics produced by the pressure of ultrasonic radiation, which can accelerate the pen-

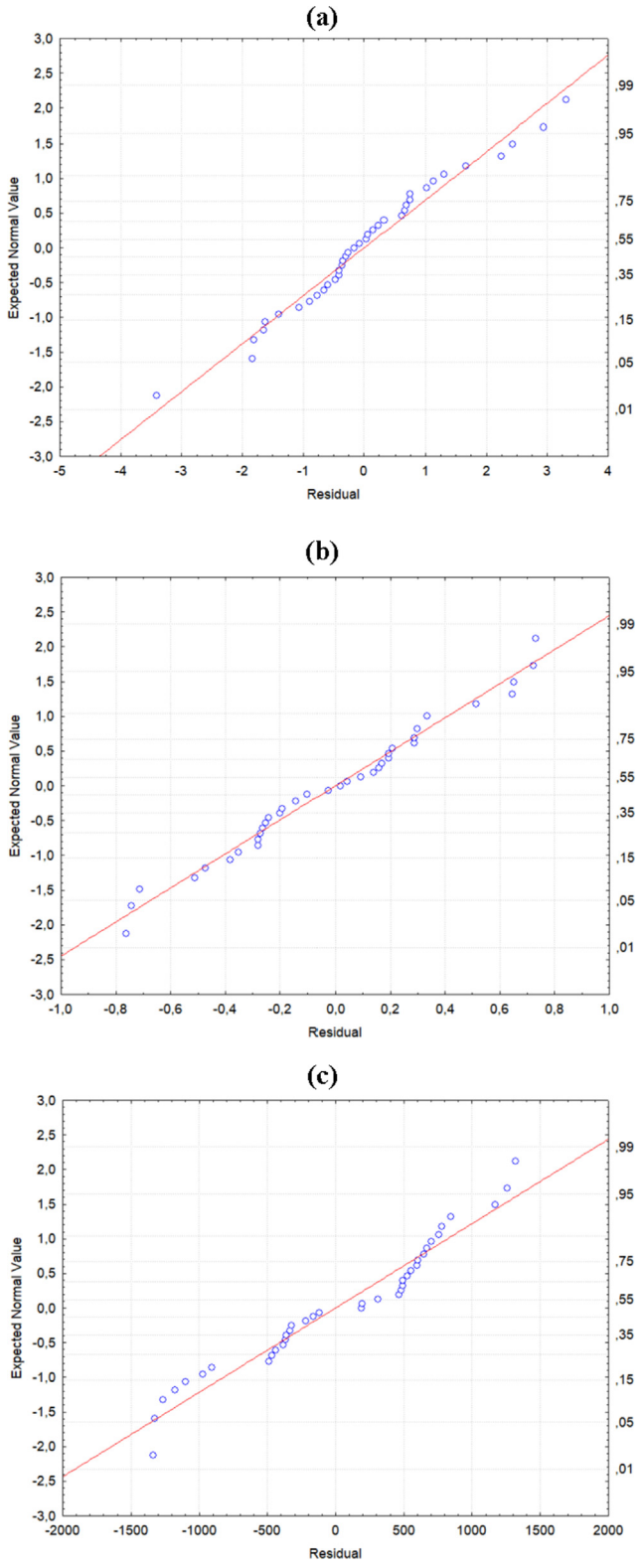

Fig. 1 Normal probability plot of studentized residuals for the reduced polynomial model: (a) TPC; (b) IC50; (c) antioxidant activity by FRAP.

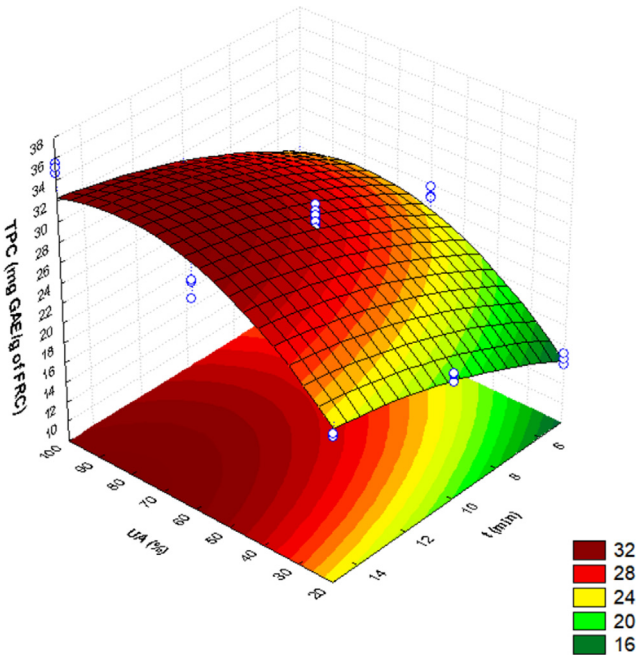

Fig. 2 Effect of the interaction of process variables on the extraction of TPC from FRC.

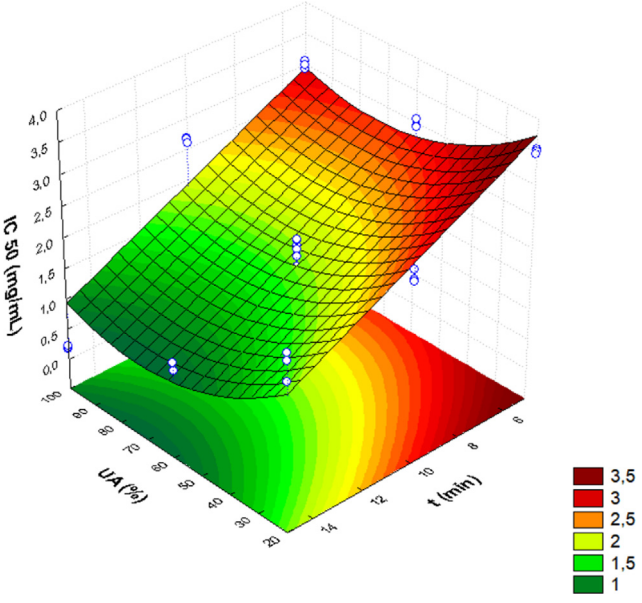

Fig. 3 Effect of the process variables on the IC_{50} of extracts of FRC phenolic compounds.

etration of solvents and improve the extraction efficiency and antioxidant activity of the obtained extracts (Wang et al., 2016).

Tests 7, 10 and 8, all using an extraction time of 5 min, showed significant increases in the antioxidant activity by FRAP as the UA was increased (7.624,40 (20% UA),

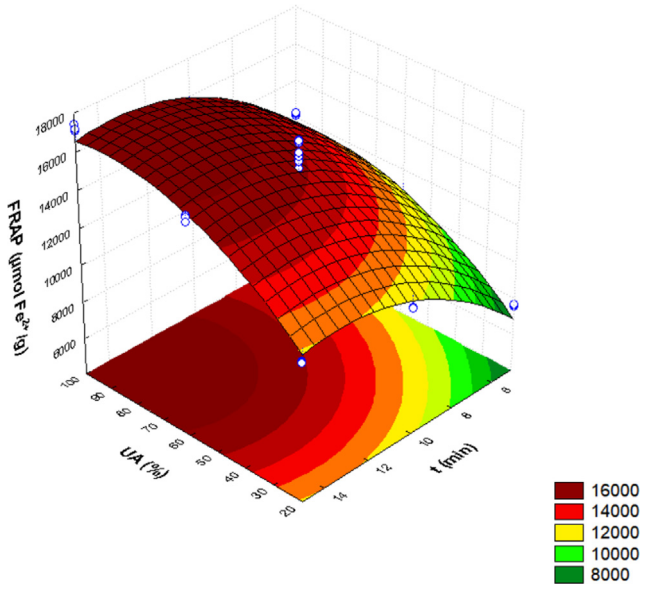

Fig. 4 Effect of the influence of process variables on the antioxidant activity by FRAP using FRC extracts.

9.573,12 (60% UA) and 12.148,17 µmol Fe^{2+}/g (100% UA), respectively (Table 3). The same trend was obtained by several authors using UAE (Chen et al., 2018; Li et al., 2016; Sharayei et al., 2019). UAE contribute to more efficient extraction of the compounds, improving the cavitation phenomena, mechanical agitation, and process efficiency and facilitating the transport of bioactive compounds, in addition to contributing to the reduction of extraction time (Chakraborty et al., 2020).

Higher FRAP values were obtained when longer extraction times were used (Fig. 4). Li et al. (2016) also reported significant increases in antioxidant activity by FRAP as the ultrasound time was increased.

3.7. Comparison of different extraction methods in the recovery of phenolic compounds from FRC

The optimization of the extraction of FRC phenolic compounds resulted in a higher content of phenolic compounds and high antioxidant activity using the following optimal extraction conditions—UA (100%) and t (15 min). Comparing the total phenolic compounds obtained by different methods, it could be noted that the ultrasound-assisted extraction was significantly more efficient (Table 5).

The extract obtained in the UAE showed greater phenolic recovery, indicating that the application of ultrasonic waves is a promising alternative, aiming to increase the extraction yield of phenolic compounds (Table 5). This can be seen in other studies (Caldas et al., 2018; Fernandes et al., 2020; Martínez-Ramos et al., 2020; Rezende et al. 2017) who investigated the recovery of phenolic compounds from grape residue, jabuticaba peel, mango peel and acerola residue, respectively, using different extraction methods. Compared to the UAE with maceration and soxhlet extraction, the UAE gives higher yields of desired compounds for shorter extraction times and at lower temperatures (Savic Gajic et al., 2019). The higher extraction efficiency using the UAE was due to the effect of cavitation (Vinatoru et al. 2017), facilitating the penetration of the solvent through the healthy cells is better, which reflects the increase in the mass transfer (Savic and Savic Gajic, 2020).

The higher rate extraction for UAE is attributed to the cavitation process, which causes the rupture of cellular structures and greater penetration of the solvent into the internal structure of the particles, increasing the intraparticle diffusivity (Chemat et al., 2017). During sonication, ultrasonic waves create shock waves within the cell wall and liquid jets are formed as a result of cavitation of the liquid media due to compression and rarefaction cycle of ultrasonic waves (Gogoi et al., 2019). Cavitation is a process that results in swelling of the material, increased temperature, solvent absorption, softening of plant surfaces and enlarged pores in materials, factors that are favorable to mass transport (Chakraborty et al., 2020; Gam et al. 2020).

The conventional techniques require the use of organic solvent for the extraction of bioactive compounds from plant material. In addition to evaporation and recycling of the solvent after using these techniques. These facts cause the increase of solvent consumption, energy consumption and generation of hazardous solvent residues after its evaporation from the sample (Savic Gajic et al., 2021). Meregalli et al. (2020) compared conventional extraction and UAE in the extraction of bioactive compounds obtained from red araçá and observed an increase of 23.45% in the levels of phenolic compounds and a 25.00% reduction in the time of extraction using UAE compared with conventional extraction by maceration. Additionally, ultrasonic extraction produced a higher yield of propolis phenolic compounds than extraction by maceration and microwave (Oroian et al., 2020). Bimakr et al. (2017) investigated the effects of ultrasound-assisted extraction on the extractive value (EV) of bioactive phenolics from Malva sylvestris leaves and its comparison with agitated bed extraction (ABE) technique. In comparison the free radical scavenging activity (FRSA) and TPC analyses results revealed that UAE afforded extracts with relatively higher FRSA and TPC values compared with ABE in much shorter time (48.77 min). Opposite results were obtained in some studies evaluated the impact

Table 5 Extraction of phenolic compounds from FRC using different extraction methods.

Methods of extraction	TPC (mg GAE/g of FRC)	IC 50 (mg/mL)	FRAP (µmol Fe^{2+}/g)
Ultrasound	35.15^{a} ± 0.43	0.19^{a} ± 0.02	$17{,}373.73^{a}$ ± 29.19
Microwave	23.31^{c} ± 0.17	4.29^{c} ± 0.03	$15{,}784.15^{a}$ ± 80.16
Conventional	30.10^{b} ± 0.07	1.68^{b} ± 0.11	$16{,}040.50^{a}$ ± 25.13

Means ± standard deviation (n = 3).
Means in each column followed by different superscript letters were significantly different ($p > 0.05$), by Tukey's test.

of different extraction methods of phenolic compounds (Da Rocha and Noreña, 2020; Rocchetti et al., 2019).

4. Conclusions

Ciriguela peel (*Spondias purpurea* L.) is considered a sustainable source of natural antioxidants and phenolic compounds. The optimum condition of the UAE of phenolic compounds from the FRC extract was obtained using an ultrasonic amplitude (UA) of 100% and a time (t) of 15 min. The extraction process affected the content of bioactive compounds and antioxidant activity. In this work, the impacts of different extraction technologies (including conventional and nonconventional) that is, mechanical agitation, ultrasound and microwave—were evaluated in terms of recovering phenolic compounds from ciriguela peel. Additionally, UAE was more efficient in recovering the compounds of interest.

Declaration of Competing Interest

The authors 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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