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ORIGINAL PAPER

Solid–liquid extraction of bioactive compounds from *Spondias mombin* L. by-products: optimization and identification of phenolic profile

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Abstract

The reuse of vegetable by-products has become one main global challenge. In this study, a central composite rotate design was used to obtain the optimal conditions (extraction time, ethanol concentration and extraction temperature) for the hydro-ethanolic extraction of bioactive compounds from *Spondias mombin* L. residue. The optimized extract (35 min, 70 °C and ethanol concentration of 55%) was analyzed for yellow flavonoids, tannins, ferric reducing antioxidant power (FRAP) as well as HPLC profile of phenolic compounds. This extract had 1666.18 ± 127.56 mg GAE 100 g^{-1} dm of total phenolic compounds, 38.03 ± 0.49 µg mL^{-1} of DPPH scavenging activity (IC_{50}) and extraction yield of 17.44 ± 0.17%. The optimized extract showed strong FRAP antioxidant activity and high content of tannins and phenolic acids. Results indicated that 2,5-dihydroxybenzoic, salicylic, 4-hydroxybenzoic acid and ellagic acids were the main components in the optimized extract. High amounts of flavonoids were also noted, highlighting rutin, catechin and myricetin hydrates. The present results show that extracts from *Spondias mombin* L. agro-industrial waste would be helpful to design functional food products.

Keywords Yellow mombin · Fruit by-product · Phenolic profile · Antioxidant activity · Optimization extraction · Flavonoids

Introduction

The production and consumption of tropical fruits are increasing worldwide due to its nutritional values and sensorial characteristics. The Brazilian production of tropical fruits reached 602.65 thousand tonnes in 2018, corresponding to 2.52% of the entire world's production (FAO 2018). Native fruits from the genus *Spondias* spp. such *Spondias mombin* L., also known as yellow mombin, are well accepted by consumers mostly due to their pleasant and exotic flavor. This fruit also contains a high level of ascorbic acid, carotenoids, and phenolic compounds, which contribute to its antioxidant activity (Contreras-Calderón et al. 2011). Because of these characteristics, yellow mombin is widely used in the North and Northeast of Brazil for the production of juice, popsicles, ice cream, yogurt, jam, and frozen pulp (Tiburski et al. 2011; Silva et al. 2012c).

The agro-industrial activity generates large amounts of by-products that are often discarded, causing environmental pollution and economic losses. It is estimated that 30–40% of residues (peel, stalks, seeds or seed and residual pulp) are generated during fruit processing (Nascimento Filho and Franco 2015). The reuse of vegetable waste has become one of the most challenging issue in food technology due to their potential as sources of bioactive compounds that may be used for the development of new and valuable food products. The sustainable use of agro-industrial waste can result in the production of value-added products, thus contributing to reduce environmental impacts and to boost economic growth (Sepúlveda et al. 2018).

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Fruit and vegetable by-products are rich in various bioactive substances such as carotenoids, phenolic compounds, anthocyanins, flavonols, catechins, flavones, and flavanones, which have proven antioxidant activity in biological systems (Varzakas et al. 2016). For the extraction of these antioxidant compounds, several techniques are employed, such as conventional techniques, including maceration, Soxhlet, heat reflux, agitation, boiling, leaching and distillation, and more advanced techniques, such as microwave assisted extraction, extraction assisted by ultrasound, supercritical extraction and extraction of pressurized liquid (Huang et al. 2013; Krishnan and Rajan 2017; Favareto et al. 2019; Monroy et al. 2020).

Maceration is a commonly used procedure for extracting antioxidant compounds from plant materials because it is a fast, cheap, and easy to perform method (Sarraf et al. 2021). The maceration with increased temperature can promote quick extraction, improving extraction efficiency, and being suitable for thermolabile compounds (Jovanović et al. 2017; Palsikowski et al. 2020). Thus, the maceration technique can be considered adequate to maximize the solid–liquid extraction process, combining the use of temperature, short extraction time, and "green solvents" (Albuquerque et al. 2018).

Besides, studies that approach the extraction of bioactive compounds partly use some toxic organic solvent, such as acetone, chloroform, hexane, and methanol, to perform the low-pressure extractions (LPE) (such Soxhlet, maceration, ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE)) or supercritical fluid extraction (SFE) (Guindani et al. 2016; Krishnan and Rajan 2017; Santos Felix et al. 2018; Favareto et al. 2019; Fernández-Barbero et al. 2019; Paludo et al. 2019). According to Monroy et al. (2020), water and ethanol have been widely used as solvents in extraction processes, due to their low cost, low toxicity, in addition to being "green solvents" with the possibility of direct use in food and pharmaceutical products.

Palsikowski et al. (2020) reported that ethanol was considered the best solvent choice compared to hexane and ethyl acetate for the ultrasound-assisted extraction (UAE) of compounds with antioxidant activity from the leaves of *B. forficata*. Studies that used ethanol as extraction solvent found the highest yield of the extraction of bioactive compounds from the leaves of *Duguetia furfuracea* and *pitanga* (Favareto et al 2019; Garmus et al. 2019).

Several studies have been carried out on the extraction of antioxidant compounds from tropical fruit residues processed in Brazil such as guava, jabuticaba, grape (Casagrande et al. 2019; Fernández-Barbero et al. 2019; Lima et al. 2019; Paludo et al. 2019), and fruits from *Spondias* spp. In fact, many researchers have shown higher antioxidant activity in the leaves, pulp and peel of yellow mombin, umbu (*Spondias tuberosa* L.), and jocote (*Spondias purpurea* L.) (Contreras-Calderón et al. 2011; Omena et al. 2012; Tiburski et al. 2011; Silva et al. 2012a). Besides, *Spondias mombin* leaves are rich in phytochemicals related to wound healing and anti-inflammatory properties, being a valuable resource to the pharmaceutical industry (Silva et al. 2012a; Cabral et al. 2016). The use of these material can be useful for the prevention of free radical damage, which helps to treat herpes and diseases associated with inflammation (Tiburski et al. 2011). Therefore, by-products from the processing of exotic tropical fruits may have applicability potential and can be used as food additives such as antimicrobials, antioxidants, coloring, flavoring and thickening agents (Ayala-Zavala and González-Aguilar 2010).

Few studies have investigated the antioxidant capacity of by-products from *Spondias* spp. Fruits. The determination of optimum conditions for the extraction of phenolic compounds from these bioresources have also been target of little investigation although the optimization of the extraction process is essential for obtaining extracts rich in polyphenolic substances (Santos Felix et al. 2018). Considering the limited information available in the literature, a more detailed study of the mechanisms responsible for the bioactive effects of yellow mombin bagasse extract is required. Thus, the development of a practical extraction method such as maceration combined with the use of non-toxic solvent as ethanol, considered as "green solvent", may be a viable alternative to extract antioxidant compounds from agro-industrial waste which can be possibly applied in the food, medicinal or pharmaceutical industry.

The extraction efficiency of antioxidant compounds from plant materials is mostly affected by extraction time, temperature, and concentration of extraction solvent. The role of each parameter in the extraction process is variable and depends on the food matrix (Xu et al. 2017). Thus, is fundamental to determine the optimal conditions for extraction in order to reduce production costs and improve product yield. The response surface methodology (RSM) is a widely used statistical tool to optimize procedures for the extraction of compounds of interest in plants and fruits since several factors can have an impact on the extraction efficiency such as concentration and type of solvent, temperature, time, etc. The RSM is a statistical method that consists of evaluating the effects of multiple factors, as well as their interactions in one or more response variables by a mathematical model, it is advantageous in terms of the reduced number of experimental tests when it is necessary to optimize a variable of answer (Casagrande et al. 2019).

Therefore, this study aimed to use the RSM to optimize the conditions for the solid–liquid extraction of the antioxidant compounds present in the yellow mombin bagasse (temperature, time, and solvent concentration) on the total phenolic compounds, antioxidant activity and extraction yield, and validate the optimization condition characterizing

the optimized extract in terms of phenolic profile and antioxidant properties.

Material and methods

Solvents and reagents

The chemicals 2,2′-diphenyl-2-picrylhydrazyl hydrate (DPPH), hydroxy-2,5,7,8-tetramethylchromane-2-carboxyl acid (Trolox) and 2,4,6-tris(2-pyridyl)-s-triazine (TPTZ), methanol, acetonitrile and acetic acid were purchased from Sigma-Aldrich (São Paulo, Brazil). The standards for phenolic compounds (3,4-dihydroxybenzoic acid, 4 hydroxybenzoic acid, caffeic acid, ellagic acid, ferulic acid, gallic acid, 2,5-dihydroxybenzoic acid, salicylic acid, syringic acid, trans-cinnamic acid, p-coumaric acid, vanillic acid, myricetin, quercetin, naringenin, catechin, hesperetin and rutin) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Folin–ciocalteau was obtained from Êxodo Científica (São Paulo, Brazil) and ethanol from Neon (São Paulo, Brazil). All chemicals used in the experiments were of analytical grade.

Plant material and sample characterization

Approximately 4 kg of *Spondias mombin* L. residue (peel, seed and residual pulp) were provided by a local fruit processing unit located in Paraiba, Brazil. Whole yellow mombin waste was oven dried at 55 °C for 24 h (Crizel et al. 2015). After dehydration, the sample was grounded using a knife mill (SL-31, Solab, Piracicaba, SP, Brazil) and stored at – 80 °C until use. Fresh and dried yellow mombin bagasse were submitted to chemical composition analysis. Moisture and ash content were analyzed by the gravimetric method according to AOAC (2005). Total lipid content was achieved using the Soxhlet procedure, and protein percentage was measured by Kjeldahl's method according to AOAC (2005). Carbohydrate content was calculated by difference (total carbohydrates = 100 − Σ(Moisture + Ash + Lipids + Proteins). The total and reducing sugars were also determined based on the anthrone (Yemm and Willis 1954) and 3,5-dinitrosalicylic acid (DNS) methods (Miller 1959), respectively. The non-reducing sugars were calculated as the difference between total and reducing sugars. Physicochemical evaluations of acidity, pH and water activity were also performed according AOAC (2005). Acidity was determined by titration with 0.1 N NaOH. pH values were measured using a digital pH meter (iON, PHS-3E-BI, Brazil). Water activity (Aw) was quantified using an Aqualab device (4TE Decagon Devices, Inc. São Paulo, Brazil).

Preparation of *Spondias mombin* L. by-product extract

Prior to the extractions, preliminary tests were carried out (data not shown) to define the solid/liquid ratio and homogenization time of the mixture. For the extraction, 3.0 g of the dried residue was homogenized for 5 min in 30 mL of the ethanol solutions using the concentrations pre-established in the experimental design (Table 1). The mixture was then incubated in a water bath (SL-154/10-4, Solab, Piracicaba, Brazil) following the extraction time and temperature described in the experimental design matrix (Table 1). After the end of the extraction time, the crude extract was centrifuged at 8.960×*g* for 20 min at 10 °C (Hettich LAB TECHNOLOGY, Tuttlingen, Germany), filtered on qualitative filter paper (80 g m^{-2}) to remove solid particles, and the ethanolic fraction was removed under vacuum (180 mbar) with a rotary evaporator at 45 °C. The volume was completed to 100 mL with distilled water and the obtained extracts were stored in dark flasks at − 80 °C until the analyses.

Experimental design

The extraction conditions were optimized for obtaining the highest antioxidant potential from *Spondias mombin* L. residue extract, by using a response surface methodology (RSM) with central composite rotational design (CCRD) (Table 1). Three independent variables were tested: extraction time (x_1), extraction temperature (x_2) and ethanol concentration (x_3). The total phenolic compounds (Y_1), DPPH scavenging activity (Y_2) and extraction yield (Y_3) were selected as the responses. The range of three independent variables presented in Table 1 was based on preliminary experiments.

The operational conditions of the experimental procedure were performed in duplicate according to the 2^3 central composite rotational design (CCRD) with 8 factorial points, 6 star corner points and 3 central points (Table 1), totaling 34 experimental runs. The experimental results were fitted in a second-order polynomial equation (Eq. 1), where β represents the constant regression coefficients of the model, while

Table 1 Independent variables and levels of CCRD planning for extraction of compounds with antioxidant potential from the yellow mombin residue

Factors[a]	Levels				
	$-\alpha$[b]	− 1	0	+1	$+\alpha$
Extraction time (min.) − x_1	10	30	60	90	110
Extraction temperature (°C) − x_2	20	30	45	60	70
Ethanol concentration (% v/v) − x_3	13	30	55	80	97

[a]Independent variables

[b]α = 1.68

Y and x are the dependent and independent variable, respectively. Additional confirmation experiments were conducted to verify the validity of the statistical experimental design.

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_1^2 + \beta_5 x_2^2 + \beta_6 x_3^2 + \beta_7 x_1 x_2 + \beta_8 x_1 x_3 + \beta_9 x_2 x_3. \quad (1)$$

After determining the optimal extraction conditions for phenolic compounds from *Spondias mombin* L. residue, the optimized extract was submitted to total phenolic compounds, DPPH scavenging activity, extraction yield, yellow flavonoid, total and condensed tannins, ferric reducing antioxidant power (FRAP) and phenolic profile analyses.

Determination of total phenolic compounds (TPC) and extraction yield

TPC were determined using the Folin Ciocalteau method with modifications (Waterhouse 2002). Extracts aliquots (50 µL) were diluted in 1.950 µL of distilled water and mixed with 150 µL Folin Ciocalteau solution (2 M). After incubation for 5 min, 350 µL of 20% Na_2CO_3 solution was added. The mixture was then shaken and incubated for 30 min at 40 °C in a water bath. The absorbance was measured at 765 nm using an UV–Vis spectrophotometer (5100, Labnova, Santo André, Brazil). The results were calculated using a standard curve of gallic acid and expressed as mg gallic acid equivalents (GAE) per 100 g sample. The extraction yield was determined by gravimetry according to Prado et al. (2010).

DPPH scavenging activity (2,2-diphenyl-2-picrylhydrazyl)

The DPPH test of the extracts was performed according to Rufino et al. (2007). Aliquots of 0.1 mL of previously diluted extracts were mixed to 3.9 mL of methanolic DPPH solution (0.06 mM) and kept in the dark at 25 °C for 30 min. Absorbance was measured at 515 nm using methanol as a blank. The inhibition curves were prepared and IC_{50} values were calculated using the graph by plotting inhibition percentage against extract concentration. Results were expressed as IC_{50}, which corresponds to the effective concentration of sample required to scavenge DPPH radical by 50%.

Yellow flavonoid content

The yellow flavonoids content was determined according to Francis (1982). The absorbance was recorded at 374 nm and the yellow flavonoid content was calculated using the Eq. 2. Results were expressed as mg yellow flavonoids per 100 g of the sample.

$$Yellow\ flavonoid\ content\left(\mathrm{mg\,100\,g^{-1}}\right) = \frac{dilution\ factor \times A_{374}}{76.2} \times 100. \quad (2)$$

Total and condensed tannins content

Measurement of total tannins were performed using Folin–Ciocalteau method (Makkar et al. 1993). Results were expressed as mg of tannic acid equivalent per 100 g of sample. Condensed tannins (CT) percentage was determined using butanol-HCL test (Porter et al. 1985). Absorbance was recorded at 550 nm and the percentage of condensed tannins was calculated according to Eq. 3 (Barman et al. 2017):

$$CT(\%) = \frac{A_{550} \times 78.26 \times dilution\ factor}{\%\ dry\ matter}. \quad (3)$$

Ferric reducing antioxidant power (FRAP)

The FRAP test was evaluated as described by Benzie and Strain (1999). Absorbance was read at 593 nm and the reducing power was expressed as mg Trolox Equivalent per 100 g sample and calculated based on calibration curve prepared with different concentrations of Trolox solution (2.0–27.0 µmol L^{-1}).

Phenolic compounds profile

The profile of phenolic compounds was analyzed in quadruplicate using high performance liquid chromatography equipped with LC-20 AT safety module (Shimadzu Corporation, Japan). The separation was performed on a C18 column (SUPELCOSIL™ LC-PAH, 250 mm × 4.6 mm ID, particle size 5 µm) (Sigma-Aldrich, St. Louis, MO, USA). Chromatographic separation was performed using a gradient elution of (A) water/acetic acid 2% (v/v) and (B) acetonitrile:methanol 2:1 (v/v) described below: 90% A in 0 min, 88% A in 3 min, 85% A in 6 min, 82% A in 10 min, 80% A in 12 min, 70% A in 15 min, 65% A in 20 min, 60% A in 25 min, 50% A in 30–40 min, 75% A in 42 min and 90% A in 44 min, as described by Prasad et al. (2009) with adaptations by Meireles (2017). The flow rate was maintained at 1.0 mL min^{-1} and the column temperature was maintained at 40 °C with an injection volume of 20 µL. The total execution time was 50 min. Peaks of all components were detected at 280 nm. Calibration was performed by injecting the standards three times at five different concentrations (0.05, 0.1, 0.25, 0.5 and 1.0 mg mL^{-1}). Individual phenolic compounds were identified by spectroscopic UV spectrum interpretation, retention time, and chromatographic comparison with authentic Sigma Aldrich standards. The quantification was

based on detection of peak areas using LabSolutions software version 5.42 SP4 Copyright (Shimadzu Corporation) versus pre-determined calibration curves.

Statistical analysis

The results of fresh and dried *Spondias mombin* L. residue were compared by Student's t test ($p < 0.05$). To optimize the process, the response function (Y) was used to perform the analysis of variance (ANOVA) for the regression. The desirability profile was used to find the optimal conditions of the process, using desirable condition (value 1) and undesirable condition (value 0). For phenolic compounds and extraction yield, the desirable conditions were those with the highest values for these parameters, for the DPPH scavenging activity, the desirable conditions were the lowest IC_{50} values. The Spearman's test was used to determine the correlations among data obtained in the CCRD. For the optimized extract, the Pearson's correlation test was performed for the variables phenolic compounds, DPPH scavenging activity, extraction yield, yellow flavonoids, total and condensed tannins, and Ferric reducing antioxidant power (FRAP). Pareto's chart, response surface graphs and desirability parameters were generated for the response functions (Y_1, Y_2 and Y_3) using Statistica software (v. 8.0 Statsoft®, Tulsa, USA).

Results and discussion

Characterization of *Spondias mombin* L. residues

The partial chemical composition of yellow mombin residue is described in Table 2. Fresh and dehydrated samples are composed mainly of carbohydrates and water. Compared with results on this work, higher moisture content was reported by Silva et al. (2012b) in fresh yellow mombin bagasse (83.3 g 100 g^{-1}). Yellow mombin residues presented lipid and protein content of 4.73 and 2.01 g 100 g^{-1} for fresh, and 8.74 and 3.44 g 100 g^{-1} for dehydrated samples, respectively. These results are considerable when compared to yellow mombin pulp, which presented values of 0.26% for lipids and 0.82% for proteins according to Mattieto et al. (2010), suggesting that the peel and endocarp may contain higher amounts of lipids and proteins.

After dehydration, the yellow mombin bagasse presented higher acidity and lower pH value than the fresh sample due to the concentration of organic acids (Table 2). The acidity of dried *Spondias mombin* L. residue were similar to those reported by Tiburski et al. (2011) for the pulp. In fact, according to these authors, fresh and dried yellow mombin waste can be considered of low and medium acidity, respectively, suggesting that the acidity increment observed in dried residues compared to fresh samples is a good indicator of conservation. It was also observed that, although the drying process was partial, the dehydrated residue had high water activity (Aw) (0.783), which can be associated with the short time spent in the drying process. Results of Aw were very similar to the water activity found by Silva et al. (2012b) for the fresh yellow mombin bagasse.

Table 2 Partial chemical composition, acidity, pH and water activity (Aw) of fresh and dehydrated yellow mombin residue

Parameters	*Spondias mombin* L. residue		
	Fresh	Dried	p-value
Moisture[a]	65.27 ± 3.66	16.76 ± 0.21	< 0.001
Ash[a]	1.13 ± 0.17	2.63 ± 0.11	< 0.001
Lipids[a]	4.73 ± 0.25	8.74 ± 0.73	< 0.001
Proteins[a]	2.01 ± 0.07	3.44 ± 0.07	< 0.001
Carbohydrates[a,b]	26.68 ± 3.74	69.24 ± 1.20	< 0.001
Total soluble sugars (TSS)	4.19 ± 0.34	8.80 ± 0.18	< 0.001
Reducing sugars (RS)	1.40 ± 0.05	3.15 ± 0.04	< 0.001
Non-reducing sugars (NRS)	2.79 ± 0.32	5.65 ± 0.21	< 0.001
Acidity (%)	0.71 ± 0.03	1.78 ± 0.05	< 0.001
pH	3.22 ± 0.03	3.10 ± 0.02	< 0.01
Aw	0.987 ± 0.003	0.783 ± 0.013	< 0.001

[a]Data expressed in g 100 g^{-1} of the sample

[b]Carbohydrates = 100 − Σ(Moisture + Ash + Lipid + Protein)

As expected, the carbohydrate content increased after dehydration from 26.68 to 69.24 g 100 g^{-1}. An increase in the content of total sugars (from 4.19 to 8.80 g 100 g^{-1}), reducing sugars (from 1.40 to 3.15 g 100 g^{-1}) and non-reducing sugars (2.79 for 5.65 g 100 g^{-1}) was also noted (Table 2). The total soluble sugars content for the fresh residue was close to the results reported by Amariz et al. (2018) in yellow mombin bagasse.

Optimization of the extraction of antioxidant compounds

Table 3 shows the mean values of response variables in all conditions proposed in the factorial design. Assay 7 (90 min, 60 °C and 30% of ethanol) and 12 (60 min, 70 °C and 55% of ethanol) showed the best responses for phenolic compounds (1013.88 and 970.72 mg GAE 100 g^{-1}), antioxidant activity (42.60 and 34.31 IC_{50} μg mL^{-1}) and extraction yield (17.08 and 16.93%), while assays 13 (60 min, 45 °C and 13% ethanol) and 14 (60 min, 45 °C and 97% ethanol) showed the lowest values for these variables (760.04 and 856.34 mg GAE 100 g^{-1} for phenolic compounds, 179.35 and 103.92 IC_{50} μg mL^{-1}, and 15.92 and 12.53% for extraction yield). Considering the extraction time factor, it is possible to

Table 3 Levels and results of the variables of the factorial planning 2^3+3 central point + star configuration (2 × 3) to obtain extract from yellow mombin residue

Assay	x_1	x_2	x_3	Y_1	Y_2	Y_3
1	− 1(30)	− 1(30)	− 1(30)	787.98	92.61	15.93
2	− 1(30)	− 1(30)	+ 1(80)	913.43	53.31	13.47
3	− 1(30)	+ 1(60)	− 1(30)	963.15	58.36	16.72
4	− 1(30)	+ 1(60)	+ 1(80)	971.18	53.61	14.97
5	+ 1(90)	− 1(30)	− 1(30)	989.10	77.96	16.06
6	+ 1(90)	− 1(30)	+ 1(80)	991.63	54.52	14.17
7	+ 1(90)	+ 1(60)	− 1(30)	1013.88	42.60	17.08
8	+ 1(90)	+ 1(60)	+ 1(80)	999.89	64.24	14.79
9	− α(10)	0(45)	0(55)	995.83	48.09	15.93
10	+ α(110)	0(45)	0(55)	974.80	42.42	16.55
11	0(60)	− α(20)	0(55)	964.78	58.49	15.67
12	0(60)	+ α(70)	0(55)	970.72	34.31	16.92
13	0(60)	0(45)	− α(13)	760.04	179.35	15.98
14	0(60)	0(45)	+ α(97)	856.34	103.92	12.53
15	0(60)	0(45)	0(55)	997.41	37.23	16.89
16	0(60)	0(45)	0(55)	964.47	33.00	16.91
17	0(60)	0(45)	0(55)	996.75	33.89	16.82

x_1 maceration time (min), x_2 maceration temperature (°C), x_3 concentration of ethanol (% v/v), Y_1 phenolic compounds (mg GAE 100 g^{-1} of dry material), Y_2 antioxidant activity (IC_{50} µg mL^{-1} extract), Y_3 extraction yield (%)

consider that the point of imminence for the conditions of higher extraction corresponds to assay 12.

According to the factorial design, it was possible to verify by means of the Pareto's chart (Fig. 1) that the ethanol concentration was the factor of greatest influence on the content of phenolic compounds, antioxidant activity (quadratic) and extraction yield (linear) of obtained extracts, being directly proportional to the antioxidant activity and inversely proportional to phenolic compounds and yield. As observed in the Pareto's chart, the ethanol concentration showed great influence on the response variables, and this variation directly reflected the antioxidant activity of the extract. The lowest antioxidant activity values were observed in the assays that used the highest (97%) and lowest (13%) concentrations of ethanol (assays 13 and 14), while those using the center point concentration (55%) showed the highest antioxidant activity values (Table 3).

The extraction time and temperature directly influenced the phenolic content and extraction yield in the linear model. Time × extraction temperature interactions were also significant ($p < 0.05$), with inversely proportional effects on phenolic compounds' content and extraction yield. The extraction temperature × ethanol concentration interaction resulted in reduction in the extraction process of phenolic compounds but promoted an increase in antioxidant activity. On the other hand, the extraction time × ethanol concentration interaction is directly proportional to the increase of the antioxidant activity of extracts. According to Albuquerque et al. (2018), time, temperature and solvent proportion are variables of interest for the extraction of nutritional or bioactive compounds from vegetable materials in maceration extraction systems.

The adjustments of mathematical models of the optimization process of response variables (Y_1, Y_2 and Y_3) were performed through ANOVA in the regression model. Only significant effects were considered, as shown in the Pareto's chart (Fig. 1), and the significance of models were analyzed by the F test. In the ANOVA performed for the content of phenolic compounds, antioxidant activity and extraction yield, R^2 values were equal to 0.6871, 0.896 and 0.9586, respectively. According to the F test, the calculated F values for phenolic compounds, antioxidant activity and extraction yield were 17.87, 66.51 and 173.12, respectively, being higher than tabulated F under the study conditions ($F_{tab} = 2.34$ for phenolic compounds and antioxidant activity and $F_{tab} = 2.25$ for extraction yield), suggesting that the models for these parameters are significant ($p < 0.05$) and predictive.

Therefore, Eqs. 4, 5 and 6 represent the proposed second-order polynomial model to experimental data, containing only statistically significant terms:

$$Y_1 = 995.09 + 21.60x_1 + 21.50x_2 + 26.29x_3 - 56.98x_3^2 - 27.02x_1x_2 - 25.06x_2x_3 \quad (4)$$

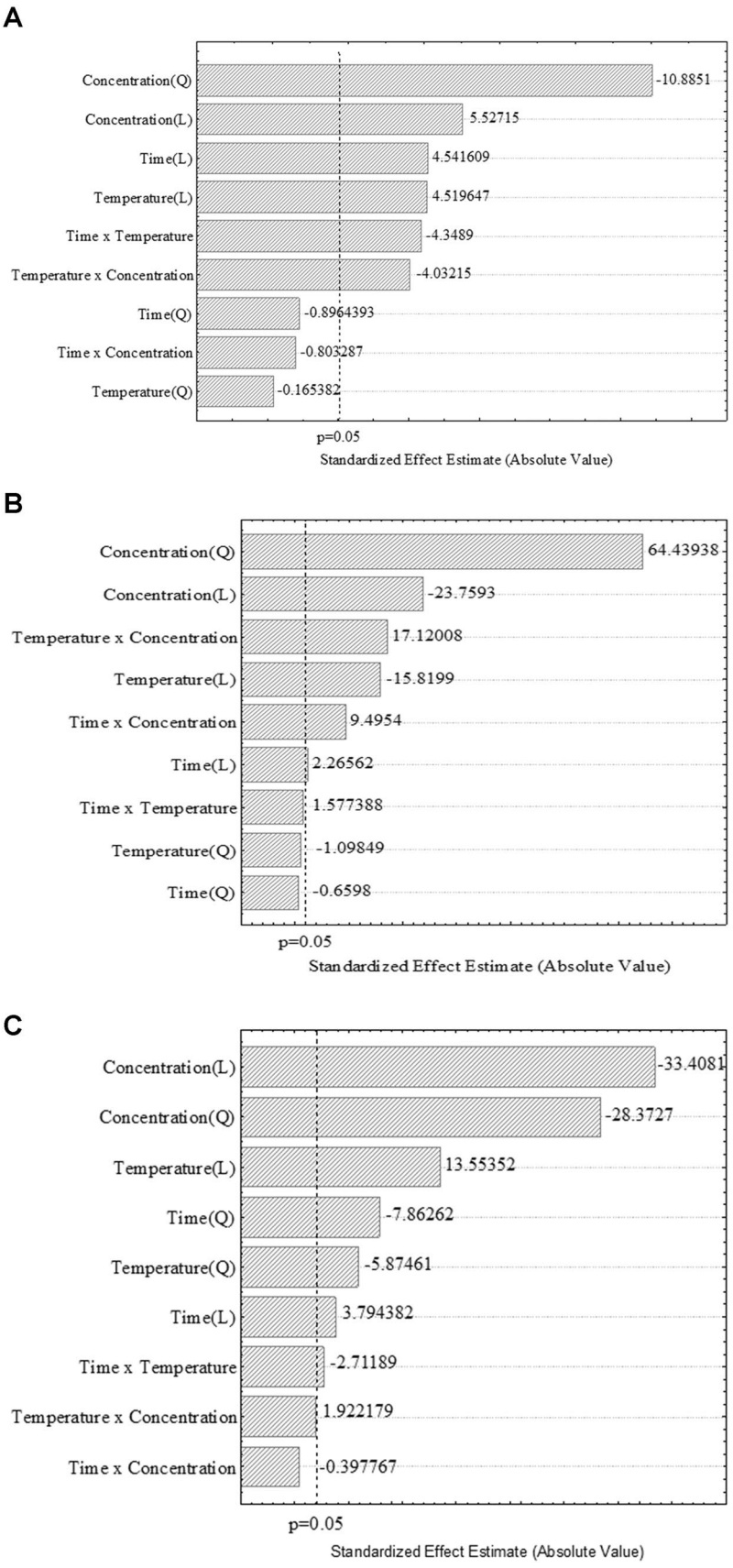

Fig. 1 Pareto chart of the response variables: phenolic compounds (**A**), antioxidant activity (**B**) and extraction yield (**C**)

$$Y_2 = 36.27 - 1.07x_1 - 7.49x_2 - 11.25x_3 + 33.59x_3^2 + 5.87x_1x_3 + 10.59x_2x_3 \quad (5)$$

$$Y_3 = 17.00 + 0.13x_1 - 0.29x_1^2 + 0.45x_2 - 0.22x_2^2 - 1.12x_3 - 1.04x_3^2 - 0.12x_1x_2. \quad (6)$$

Spearman's correlation (Table 4) showed that all significant correlations were negative, and the phenolic compounds (Y_1) showed a moderate negative correlation ($p < 0.001$ and $r_s = -0.483$) with IC_{50} (Y_2). A strong negative correlation ($p < 0.001$ and $r_s = -0.650$) was found between antioxidant activity and extraction yield (Y_3). These results suggest that phenolic compounds were the main substances responsible for the antioxidant capacity of the extracts.

From Eqs. 3–5, response surfaces were generated (Fig. 2). The three independent variables promoted a greater impact on the extraction of phenolic compounds (Fig. 2A–C), as observed through the analysis of the Pareto's chart (Fig. 1). Evaluating the three response surfaces, it was possible to observe the optimal regions (Y_1 close to 1050 mg 100 g^{-1} of the sample) when the x_1, x_2 and x_3 was respectively between 100 and 110 min of maceration, 60 and 70 °C and 50 and 70% ethanol. The values obtained in all tests were higher than those observed by Santos Felix et al. (2018), who evaluated the use of Doehlert matrix and mix designs to optimize the extraction of antioxidant compounds from yellow mombin bagasse. Omena et al. (2012) obtained higher contents of phenolic compounds when evaluating the antioxidant potential of umbu and jocote (2022 and 2547 mg GAE 100 g^{-1}).

For the antioxidant activity (Fig. 2D–F), as well as in phenolic compounds, the extraction time and temperature promoted small changes in IC_{50} values (Y_2). The higher antioxidant potential (IC_{50} close to 20 µg mL^{-1} of extract) was observed in extracts obtained at a temperature range of 55 to 75 °C and an ethanol concentration range of 40 to 65%. The results obtained for antioxidant activity were higher than those observed by Satpathy et al. (2011) in *Spondias pinnata* K. extracts and by Silva et al. (2012a) in leaves of *Spondias mombin* and *Spondias tuberosa*, showing that extracts obtained from yellow mombin residue displayed high antioxidant activity against the DPPH radical.

The extraction yield (Fig. 2G–I) showed an optimum region (yield close to 17%) when subjected to extraction between 40–80 min, with temperatures of 40 and 70 °C and ethanol concentration between 30 and 60%. The extraction yield values were higher than those reported by Omena et al. (2012) evaluating, respectively, the antioxidant extraction yield of umbu and jocote peel (11.3 and 5.9%) and seeds (5.6 and 1.4%). These results are an important parameter in determining the technical and economic viability of the processes (Palsikowski et al. 2020).

All independent variables caused variations in response variables. The extraction time was the factor that had lower impact in the studied variables. This can be attributed to the possible saturation of the extraction solution. The balance between the concentrations of solutes of the bagasse in the solution is obtained after a certain time, since the behavior over time of the extraction process is explained by Fick's diffusion law. Thus, after some time, an increase in extraction time causes only a small increase in compounds concentration. Alternatively, it can be attributed to the fact that relatively longer extraction times can promote increased phenolic oxidation (Chirinos et al. 2007; Silva et al. 2007).

Temperature significantly affected the extraction of phenolic compounds, and consequently antioxidant activity and extraction yield. This effect was more important at intermediate concentrations, between 40 and 70%. In other conditions it is possible that phenolic compounds may have been degraded with increasing temperature. At the highest temperatures (50 and 70 °C), the rupture of the cell wall of the yellow mombin bagasse may occur, as a result of weakened interactions and reduced affinity of the solid with the solute, as well as the increase in the solubility and diffusivity of phenolic compounds, improving mass transfer. It may also promote the reduction of viscosity and density of the medium, facilitating the penetration of the solvent in the sample and accelerating the extraction process (Krishnan and Rajan 2017; Lazar et al. 2016; Maran et al. 2017).

As phenolic compounds are composed of several classes of substances with antioxidant potential, each class can interact in a way with the solvent used in the extraction. The variation of solvent (ethanol) concentration in water contributed to the creation of a moderately polar medium, which promoted the potentiation of polyphenol extraction from *Spondias mombin* L. residues (Chirinos et al. 2007; Mokrani and Madani 2016). This result shows that due to the increase in the polarity of the extracting solvent, the extraction yield increases, indicating that the increase in the concentration of water in ethanol provides the increases the extraction yield. Possibly, phenolic compounds present

Table 4 Spearman's correlation coefficient (R) among the variables Y_1, Y_2 and Y_3

	Y_1	Y_2	Y_3
Y_1	–	− 0.483***	0.262^{ns}
Y_2	–	–	− 0.650***
Y_3	–	–	–

Y_1 phenolic compounds (mg GAE 100 g^{-1} of dry material), Y_2 antioxidant activity (IC_{50} µg mL^{-1} extract), Y_3 extraction yield (%)

Statistical significance (p-value) reported such as: ns for not significant, *** for $p < 0.001$

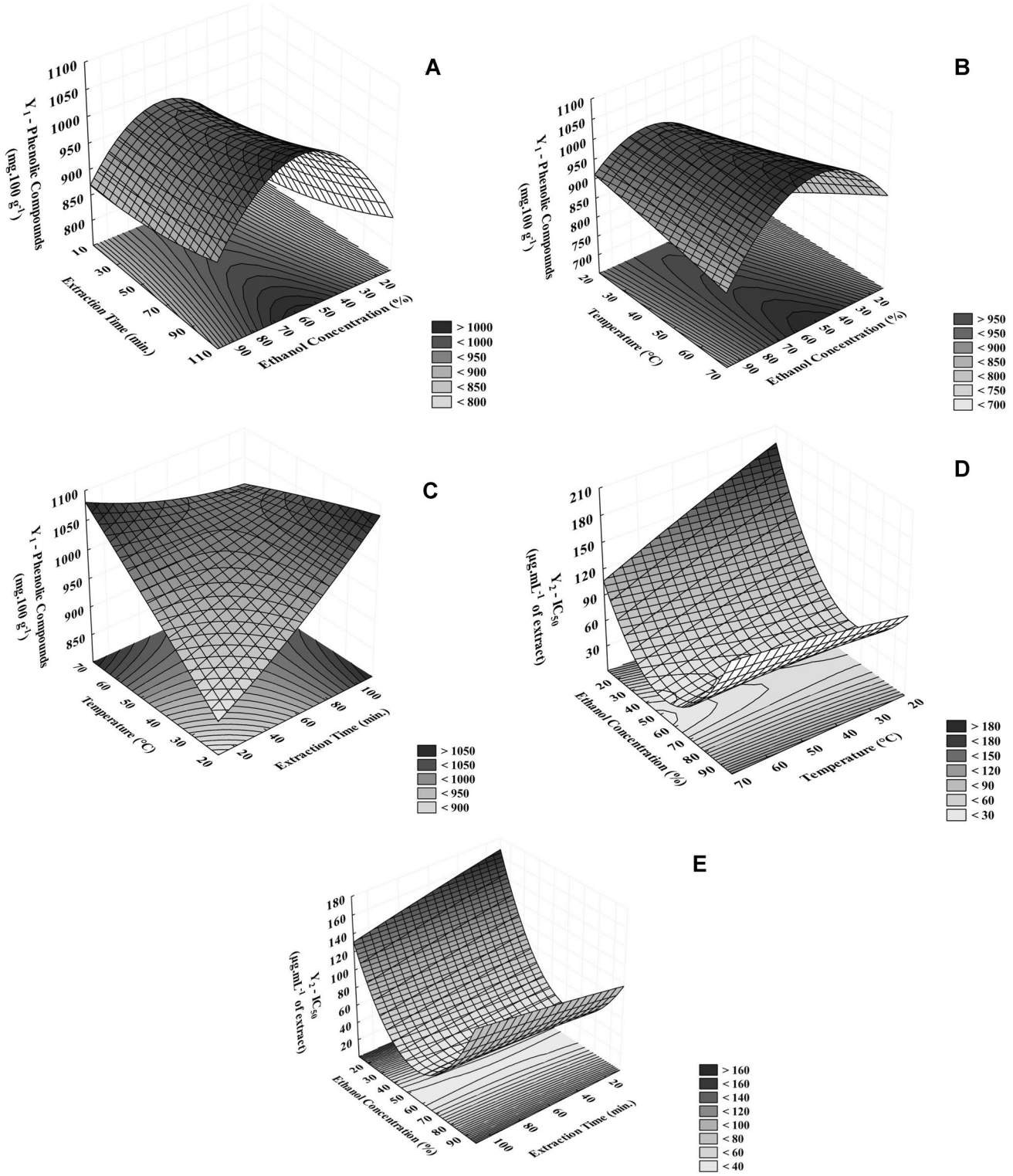

Fig. 2 Response surfaces generated for the response variables: phenolic compounds (**A**–**C**), antioxidant activity (**D**–**F**) and extraction yield (**G**–**I**)

in the yellow mombin bagasse possess stronger affinity for intermediate polarity solvents.

The use of solvents in conjunction with high temperatures may allow a greater solvation of the target compounds present in the matrix, promoting the increase in diffusion

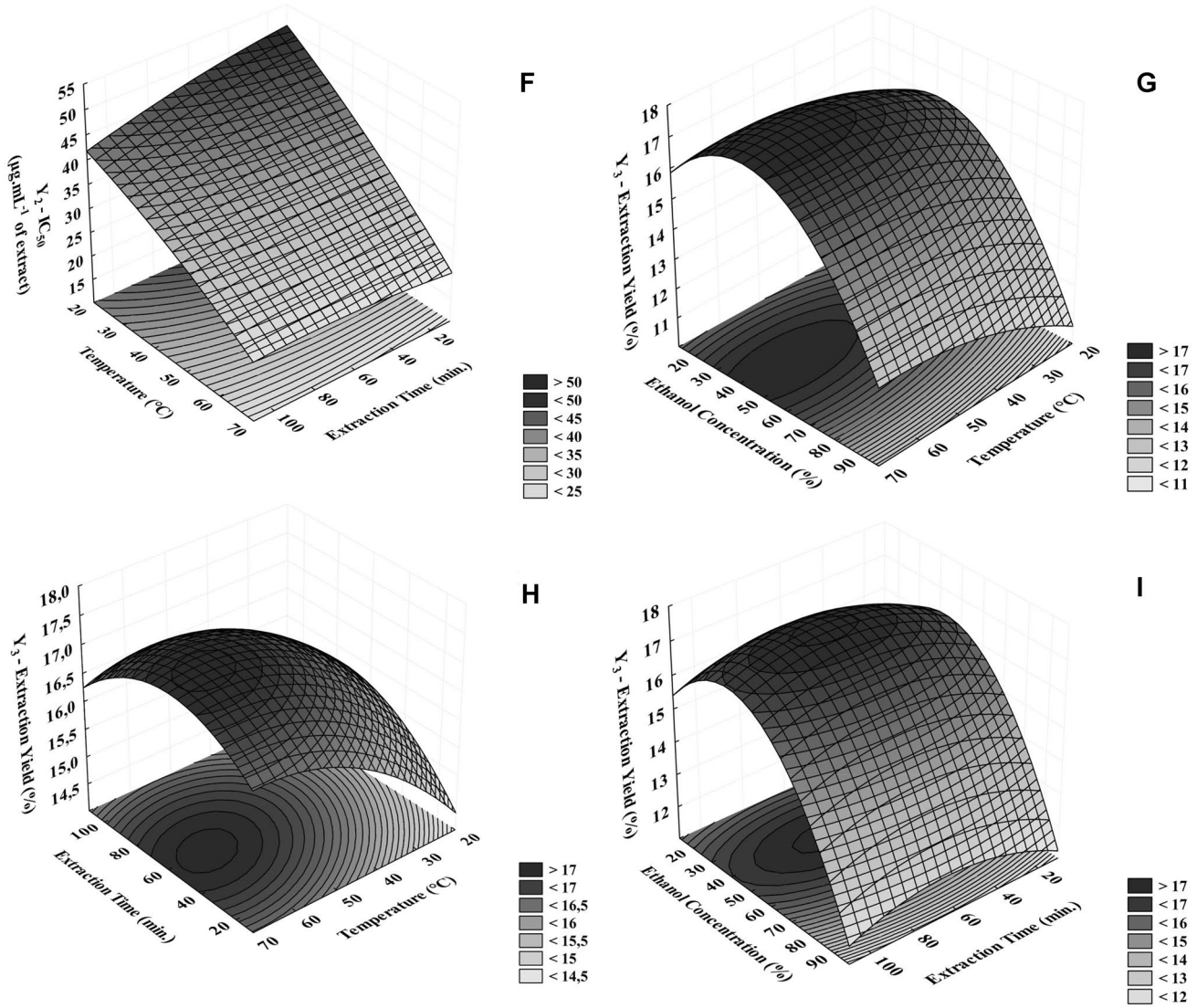

Fig. 2 (continued)

rates. Consequently, it may reduce the extraction time and solvent consumption (Mustafa and Turner 2011; Okiyama et al. 2018). Thus, the results of phenolic compounds, antioxidant activity and extraction yield may be related to the solvent polarity and solubility of polyphenols present in yellow mombin bagasse. It shows that ethanol concentration from 40 to 70% is indicated for the efficient extraction of polyphenolic compounds from *Spondias mombin* L. residues, resulting in high antioxidant potential of the extract.

In the desirability function (Fig. 3), only the extraction time showed different values (35 min) from those observed in experimental assays. The extraction temperature and ethanol concentration were 70 °C and 55%, respectively. The conditions estimated by the desirability function indicated optimum values for phenolic compounds, DPPH scavenging activity and extraction yield of 1.051.56 mg GAE 100 g^{-1}, 24.62 μg mL^{-1} extract and 17.42%, respectively. The optimization value was very close to 1 (0.9646). This is a satisfactory value since each independent variable can display different measurement scales, hence the optimization of multiple responses at the same time is a challenge.

Model fitting

According to the optimum conditions obtained from the desirability of antioxidant compounds extraction, Eqs. 3–5 indicated that the optimal condition for the extraction of antioxidant compounds from yellow mombin bagasse was 35 min of extraction at 70 °C using 55% ethanol in distilled water. The estimated content of phenolic compounds, antioxidant activity and extraction yield

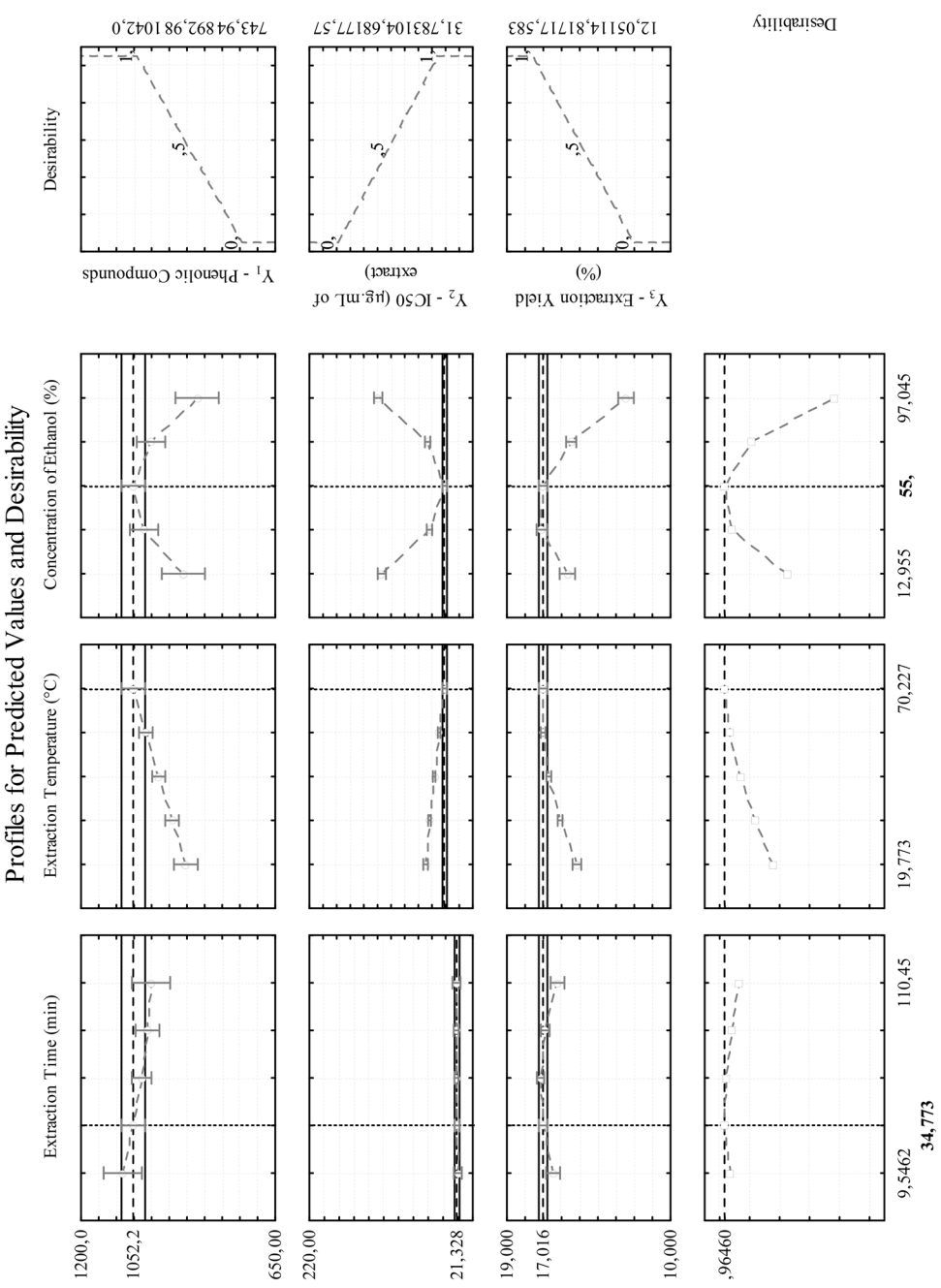

Fig. 3 Profile of the predicted values and desirability for the content of phenolic compounds, antioxidant activity and extraction yield of the extract of *Spondias mombin* L. residue

were 1051.56 mg GAE 100 g^{-1} of sample, 24.62 µg mL^{-1} of extract and 17.42%, respectively. For validation of the mathematical model, the results obtained from the extracts prepared under the desirability conditions were compared with estimated values. Observed values of phenolic compounds (1666.18 ± 127.56 mg GAE 100 g^{-1}) and antioxidant activity (38.03 ± 0.49 µg mL^{-1}) were consistent with predicted values calculated by the model, yet, significantly different ($p < 0.05$). For the extraction yield, however, average value of $17.44 \pm 0.17\%$ was observed. This obtained results is not significantly different from value predicted by the model ($p > 0.05$), enabling the validation of the proposed model. The results showed the difficulty of developing an optimal method to extract antioxidant compounds present in yellow mombin residue when working with three response variables at the same time.

Characterization of the optimized extract

The optimized extract showed high content of total tannins (835.12 ± 63.57 mg TAE 100 g^{-1} sample), with 41.78 ± 1.29% corresponding to condensed tannins. Tannins display several biological properties, such as antioxidant, antitumor, antimutagenic, antiviral, antibacterial and hemostatic activities (Okuda and Ito 2011). Condensed tannins or proanthocyanidins are composed of catechin, epicatechin and gallic acid esters, which may be associated with a wide range of potential human health benefits (De-Faria et al. 2012).

The optimized extract also presented 31.22 ± 0.81 mg of yellow flavonoids per 100 g of sample. Rufino et al. (2010) observed contents of yellow flavonoids equal to 7.1 ± 0.7 and 6.9 ± 1.7 mg 100 g^{-1} of fresh sample in yellow mombin and umbu fruits, respectively. These results show that dehydrated yellow mombin bagasse presents approximately 4.5 times more flavonoids than fresh fruits. This is an important finding since yellow flavonoids have beneficial health properties, acting as nutraceuticals and antioxidants (Tapas et al. 2008; Silva et al. 2012c).

Regarding the ferric reducing antioxidant power (FRAP), the extract obtained under optimum conditions presented FRAP value equal to 98.54 ± 4.37 µmol TE g^{-1} of the sample. The extract presented high FRAP values compared to results reported by Omena et al. (2012) for jocote and umbu peel (16.20 ± 1.05 and 4.98 ± 0.21 µmol TEAC g^{-1}) and seeds (13.73 ± 0.53 and 5.46 ± 0.08 µmol TE g^{-1}), respectively. Fernandes et al. (2016) reported lower FRAP values in 5 plant extracts (mustard, saffron, mint, ginger and fennel) compared to results observed in this experiment. According to the FRAP value, yellow mombin bagasse extract can be classified with good ferric reducing capacity (Fernandes et al. 2016).

For the optimized extract, no significant correlation of phenolic compounds, antioxidant activity (DPPH and FRAP) was observed with any of the other variables evaluated ($p > 0.01$ and $p > 0.05$, respectively). However, it is observed that phenolic compounds and condensed tannins were the most important phenol groups for the antioxidant activity (DPPH), and tannins (total and condensed) are the most important groups for the FRAP activity (Table 5). A strong significant Pearson's correlation of extraction yield and total tannins ($p < 0.001$ and $r = -0.995$) was observed. Yellow flavonoids and condensed tannins were also found to have strong significant correlation ($p < 0.05$ and $r = -0.974$). The negative extract yield and total tannins correlation may be due to ethanol concentration used in the extractor solution. It can also be attributed to the degree of polymerization of the extracted tannins since their solubility vary among different solvents (Khoddami et al. 2013). The negative effect of yellow flavonoids on condensed tannins can possibly be attributed to flavones and flavonols. These are the main chromophores responsible for yellow coloring, characteristic of flavonoids while condensed tannins are oligomers or polymers of flavan-3-ols units (such as catechin and epicatechin monomers) (Shadkami et al. 2009; Deveoglu and Karadag 2019). Thus, condensed tannins may have influenced the

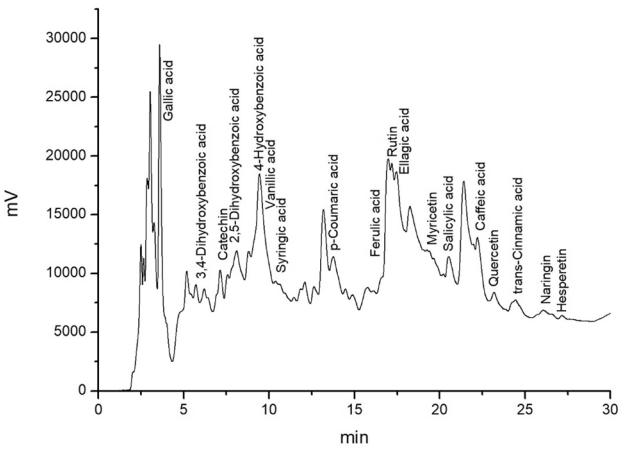

Fig. 4 HPLC phenolic profile of the optimized extract from *Spondias mombin* L. residue recorded at 280 nm

Table 5 Pearson's correlation coefficient (R) between different parameters of the optimized extract of *Spondias mombin* L.

	PC	AA	EY	YF	TT	CT	FRAP
PC	–	− 0.307^{ns}	0.109^{ns}	0.453^{ns}	− 0.039^{ns}	− 0.512^{ns}	−0.654^{ns}
AA	–	–	− 0.249^{ns}	− 0.185^{ns}	0.161^{ns}	0.401^{ns}	0.835^{ns}
EY	–	–	–	0.922^{ns}	− 0.995***	− 0.908^{ns}	−0.569^{ns}
YF	–	–	–	–	− 0.904^{ns}	− 0.974**	−0.647^{ns}
TT	–	–	–		–	0.869^{ns}	0.483^{ns}
CT	–	–	–	–	–	–	0.799^{ns}
FRAP	–	–	–	–	–	–	–

PC phenolic compounds, *AA* antioxidant activity, *EY* extraction yield, *YF* yellow flavonoid, *TT* total tannins, *CT* condensed tannins, *FRAP* ferric reducing antioxidant power

Statistical significance (p-value) reported such as: ns for not significant, ** for $p < 0.01$, *** for $p < 0.001$

Table 6 Phenolic profile of optimized extract from *Spondias mombin* L. residue

Phenolic compounds	mg 100 g^{-1} dm[a]
Phenolic acids	
3,4-Dihydroxybenzoic acid	20.97 ± 0.82
4 Hydroxybenzoic acid	79.99 ± 10.44
p-Coumaric acid	10.98 ± 3.46
Salicylic acid	113.49 ± 12.48
Syringic acid	10.23 ± 0.96
trans-Cinnamic acid	2.25 ± 0.50
2,5-Dihydroxybenzoic acid	259.84 ± 22.29
Vanillic acid	35.94 ± 21.34
Ferulic acid	27.29 ± 2.51
Ellagic acid	46.93 ± 6.99
Caffeic acid	36.94 ± 8.43
Gallic acid	7.99 ± 0.00
Total of phenolic acids	652.84 ± 72.37
Flavonoids	
Rutin	156.26 ± 23.38
Myricetin	33.95 ± 6.92
Quercetin	20.47 ± 6.55
Naringenin	1.22 ± 0.38
Catechin	66.90 ± 3.60
Hesperetin	2.25 ± 0.50
Total of flavonoids	281.03 ± 58.85
Contents of phenolic compounds	933.87 ± 66.49

[a]mg 100 g^{-1} of dry-matter

absorption of UV light during the analysis of yellow flavonoids increasing their value.

Evaluating the phenolic profile of the optimized extract, it was possible to identify 18 compounds. Some compounds have not been identified (Fig. 4). The extract showed high concentrations of phenolic acids (652.84 ± 72.37 mg 100 g^{-1} dm) and 12 compounds belonging to this class were found (Table 6). Phenolic acids are a group of compounds that exhibit high antioxidant activity, which is positively correlated to the number of hydroxyl groups attached to aromatic rings (Bogucka-Kocka et al. 2016). Among the acids found in the optimized extract, 2,5-dihydroxybenzoic acid (gentisic acid) (259.84 ± 22.29 mg 100 g^{-1} dm), salicylic (113.49 ± 12.48 mg 100 g^{-1} dm), 4-hydroxybenzoic acid (79.99 ± 10.44 mg 100 g^{-1} dm) and ellagic (46.93 ± 6.99 mg 100 g^{-1} dm) acids stand out, which are important compounds in the pharmaceutical area.

Moreover, hydroxycinnamic acids (p-coumaric, ferulic and caffeic) were found, which constitutes another important group of antioxidant compounds. These substances present high antioxidant potential, with caffeic acid being the highest in antioxidant potential and p-coumaric acid the lowest in potential among the three (Andreasen et al. 2001). Dutra et al. (2017) observed similar phenolic acids' profile to that of this study in jocote and umbu (fruit and pulp), and reported higher levels of 2,5-dihydroxybenzoic (gentisic) and 3,4-dihydroxybenzoic (protocatechuic) acids.

The extract obtained in the optimized conditions also had considerable amounts of flavonoids (281.03 ± 58.85 mg 100 g^{-1} dm), with 6 compounds of this class. Among these, rutin (156.26 ± 23.38 mg 100 g^{-1} dm), catechin (66.90 ± 3.60 mg 100 g^{-1} dm) and myricetin (33.95 ± 6.55 mg 100 g^{-1} dm) can be highlighted. Moreover, other flavonoids such as quercetin, hesperetin and naringenin were also found. The rutin and quercetin contents found in yellow mombin residue extract were similar to those reported by Dutra et al. (2017) for jocote and umbu (fruit and pulp).

Conclusions

The optimal conditions for the extraction of antioxidant compounds were extraction time of 35 min, 70 °C and 55% ethanol solution. Although the content of phenolic compounds and antioxidant activity are higher than values predicted by the experimental model, it could be concluded that the results obtained in the validation test were satisfactory, since they allowed obtaining extract from *Spondias mombin* L. pulp residue with high antioxidant power and a diversified phenolic profile, highlighting 2,5-dihydroxybenzoic and salicylic acids, hydroxycinnamic acids, in addition to considerable amounts of flavonoids such as rutin and catechin which are responsible for providing antioxidant potential to yellow mombin extract. Due to the variety of compounds with antioxidant potential found in the extract, the low environmental risk and reduced toxicity of ethanol, it could be concluded that the *Spondias mombin* L. by-product extract has the potential to be applied in the food and pharmaceutical industry.

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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