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An insight into chayote (*Sechium edule*) peels valorization: phytochemical characterization and bioactive potential †

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Abstract: A Box-Behnken Design was applied to investigate the influence of ethanol %, time, temperature, and ultrasonic power on the Ultrasound-Assisted Extraction (UAE) of phenolic compounds, carotenoids, and antioxidant capacity from chayote peel. The recovery of total phenolics (406 mg GAE/100 g dw) and antioxidant compounds (FRAP value of 82.83 mg AAE/100 g dw and ABTS value of 319 mg AAE/100 g dw) were maximized using 37% ethanol, 55 °C and 224 W, for 30 min. The extraction of carotenoids (17.14 mg/100 g dw) was maximized using 75% ethanol, 30 °C and 200 W, for 61 min.

Keywords: *Sechium edule*; chayote peel; ultrasound-assisted extraction (UAE); Box-Behnken Design (BDD); phenolics and carotenoids profile

1. Introduction

Chayote, scientifically known as *Sechium edule* (Jacq.) Swartz, is an underutilized vegetable in many regions, including Portugal. It is a member of the Cucurbitaceae family, which includes cucumbers, pumpkins, and squashes. Chayote is known for its interesting nutritional and phytochemical composition, and it has been associated with various potential health benefits [1]. Literature reports that chayote peels extracted with 100% water have a Total Phenolic Content (TPC) of 746.46 ± 58.73 mg GAE/ 100 g dry weight (dw), mostly represented by the phenolic acids (e.g., caffeic acid, phenylacetic acid, 4-hydroxybenzoic acid, protocatechuic aldehyde and dihydroxybenzoic acid isomer IV) and flavonoids (Hispidulin, apigenin 7-*O*-apiosyl-glucoside, apigenin, chrysoeriol 7-*O*-apiosyl-glucoside and neohesperidin) [2]. Chayote peels are also rich in carotenoids (1.7 mg/ 100 g dw), with a β-carotene content of 0.36 mg/ 100 g dw [3]. Hence, the application of green-extraction techniques to recover these valuable bioactive compounds and further investigation on the phytochemical profile of chayote peels is required to promote its sustainable use in food, cosmetic and pharmaceutical sectors.

This study aimed to determine the optimal ultrasound-assisted extraction (UAE) conditions for recovering carotenoids, phenolic compounds, and antioxidant capacity from chayote peel. A response surface methodology using the Box-Behnken Design (BDD) was employed to investigate the impact of ethanol percentage, extraction time, temperature, and ultrasonic power on the recovery of these valuable compounds.

2. Material and Methods

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2.1. Sampling

Samples of chayote green variety at maturity stage were supplied by a local farm located at Cinfães, Douro (Portugal), and were collected in October 2021 from 10 plants (random sampling) to obtain a representative set of fruits. Then, peels were separated from pulp, dried for 18 h at 52 °C in processed food (Excalibur 9 Tray Dehydrator, Model 4926 T, USA), grounded (Moulinex A320), sieved through 0.75 mm stainless steel sieve, thoroughly mixed and stored at 8 °C under light-free conditions until extractions.

2.2. BBD Optimization and Validation

The optimization of UAE of phenolics and carotenoids from chayote peels was done by Box-Behnken Design (BBD). Four independent variables were considered: X1 - 25-75% of ethanol; X2 - 30-80 minutes; X3 - 35-55°C; and X4 - 60-80% ultrasonic power amplitude. Total phenolic content, TPC (Y1); Ferric reduction antioxidant power, FRAP (Y3); 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid radical scavenging activity, ABTS (Y2) and total carotenoid content, TC (Y4), were taken as the dependent variables (Table 1). Desirability indices were constructed to obtain the optimum experimental conditions to maximize the bioactivities of chayote peel.

2.3. Ultrasound Assisted Extraction (UAE)

For UAE procedures, 1 g of freeze-dried powder of chayote peels were mixed with 30 mL extraction solvent in a 3.5 cm inner diameter cylindrical flask. After that, the flask was covered with aluminum foil and placed in the ultrasonic bath (Bandelin SONOREXTM Digital 10 P Ultrasonic baths DK 102 P, Bandelin Electronic GmbH, Berlin, Germany). The extraction was then carried at conditions defined by the BBD (Table 1). All extractions were performed in triplicate, using a solid to sample ratio of 1:30 g/mL, with occasional stirring. The obtained extracts were filtered, centrifuged (5000 rpm for 15 min at 4 °C), lyophilized for 48 h and stored at 4 °C until further use.

2.4. Characterization of Chayote Peel Extracts

The total phenolic content (TPC), total carotenoid content (TC), and antioxidant activity evaluated by FRAP and ABTS assays were performed as previously described [4]. TPC results were expressed as milligrams of gallic acid equivalents (GAE) per 100 grams of dry weight (dw); TC results as mg/ 100 g dw; and FRAP and ABTS results as mg of ascorbic acid equivalents (AAE)/ 100 g dw.

2.5. HPLC-PDA Polyphenol Composition Profile

The phenolic profile of the optimal extract was characterized by HPLC with a photodiode array detector and a C18 column as previously described [4]. The extract was analyzed three times, and the results were expressed as mg/ 100 g dw.

2.6. HPLC Vitamin A, Vitamin E, Carotenoids, and Chlorophylls Composition Profile

The vitamin A, vitamin E, carotenoids, and chlorophylls composition profile of the optimal UAE-chayote peel extract was characterized by HPLC with a photodiode array detector and a C18 column as previously described [4]. The extract was analyzed three times, and the results were expressed as mg/ 100 g dw.

2.7. Statistical Analysis

Results were expressed as means ± standard deviation. Design-Expert software version 7.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used for establishing the experimental design of the optimization process. IBM SPSS Statistics 22.0 software (SPSS Inc., Chicago, IL, USA) was employed to analyze data HPLC analyses. Tukey´s multiple range test, at a significance level of $p < 0.05$, was used for the comparisons of the mean values.

Table 1. Experimental conditions and results of total phenolics content (Y1), antioxidant activity (Y2, Y3) and total carotenoids (Y4) obtained by UAE of chayote peels.

Independent variables					Dependent variables							
Run	X1 EtOH (%)	X2 Time (min)	X3 T (ºC)	X4 Power (%)	Y1 - TPC (mg GAE/100 g dw)		Y2 - FRAP (mg AAE/100 g dw)		Y3 - ABTS (mg AAE/100 g dw)		Y4 - TC (mg/100 g dw)	
					Exp.[a]	Pred.[b]	Exp.[a]	Pred.[b]	Exp.[a]	Pred.[b]	Exp.[a]	Pred.[b]
1	25	30	55	60	275.85	305.02	87.39	77.76	289.25	293.69	4.89	5.06
2	50	55	45	70	237.87	250.78	49.77	54.03	244.73	290.57	16.99	16.50
3	50	55	45	90	386.60	357.69	81.65	74.69	364.20	328.99	10.47	10.03
4	50	55	45	70	237.87	250.78	49.77	54.03	244.73	290.57	16.99	16.50
5	50	55	45	70	276.06	282.58	62.21	54.03	293.53	290.57	15.43	16.50
6	25	30	35	60	302.22	257.17	80.36	76.05	341.74	302.18	5.44	6.87
7	75	80	35	80	255.19	257.17	52.13	65.05	214.57	257.00	16.65	17.87
8	100	55	45	70	69.29	86.64	10.06	8.18	129.39	87.17	17.94	17.12
9	75	80	55	80	229.89	259.18	53.79	59.21	225.38	260.98	16.93	16.23
10	75	30	55	60	237.50	239.11	55.70	58.36	264.96	254.38	4.44	4.97
11	50	55	45	70	215.49	250.78	41.19	54.03	274.40	290.57	16.20	16.50
12	25	80	35	80	321.33	329.36	76.42	74.87	325.51	332.13	4.72	4.92
13	50	55	25	70	411.13	361.82	89.90	85.69	356.41	344.36	15.35	12.78
14	75	80	35	60	283.85	277.67	71.60	61.72	252.84	239.01	14.91	16.44
15	50	105	45	70	332.41	293.79	72.33	63.72	327.35	271.57	15.10	13.86
16	75	30	35	60	225.49	228.69	34.32	38.84	186.26	217.80	15.47	15.90
17	50	55	45	70	268.68	250.78	60.62	54.03	343.51	290.57	15.69	16.50
18	75	30	55	80	384.89	331.94	76.04	68.02	234.64	239.90	5.53	6.05
19	25	30	35	80	261.55	296.52	65.01	66.29	310.33	314.40	7.13	6.52
20	25	30	55	89	390.92	406.74	69.02	80.01	299.39	309.27	8.28	7.49
21	75	80	55	60	195.71	191.89	41.84	43.86	205.84	239.63	10.01	12.02
22	25	80	55	60	290.80	267.21	40.70	43.28	237.74	239.63	8.34	6.86
23	25	80	35	60	256.88	340.98	67.62	78.95	251.48	284.09	1.27	2.15
24	50	5	45	70	310.33	308.16	65.42	69.63	246.73	268.59	9.29	8.40
25	50	55	65	70	377.75	386.27	81.75	81.56	361.73	339.86	8.89	9.33
26	75	30	35	80	200.50	233.73	37.96	36.48	205.84	199.97	11.99	14.21
27	25	80	55	80	315.16	343.39	52.44	51.22	284.71	291.05	11.44	12.41
28	0	55	45	70	282.90	224.75	39.93	37.41	193.31	201.62	1.58	2.27
29	50	55	45	70	268.68	250.78	60.62	54.03	343.51	290.57	16.69	16.50
30	50	55	45	50	288.36	276.47	66.55	69.11	294.12	295.42	7.86	6.17

[a] Experimented values are expressed as average of triplicate determinations from different experiments. [b] Predicted valued based on BBD evaluation.

3. Results and Discussion

3.1. Analysis of BBD

Table 1 shows the experimental extraction conditions and the experimental and predicted values of TPC, FRAP, ABTS and TC of chayote peel extracts. For all the responses, there was a close agreement between the experimental values and the theoretical values predicted by BBD. TPC varied between 69.29 and 411.13 mg GAE/ 100 g dw, FRAP from 10.06 to 89.90 mg AAE/100 g dw, ABTS from 129.39 to 364.20 mg AAE/ 100 g dw, and TC from 1.27 to 17.94 mg/ 100 g dw. The lowest values of Y1-Y3 responses were recorded at 45°C, 100% ethanol, 55 min and 70% (200 W) of ultrasound power (run 8), while the lowest value of TC (Y4 response) was achieved at 80°C, 25% ethanol, 35 min and 60% (170 W) of ultrasound power (run 23).

3.2. Validation of the BBD model

The optimal UAE conditions to maximize the phenolic, antioxidant and carotenoid, composition of chayote peel were predicted by BBD. For this purpose, individual desirability's of the three responses were combined into a single number and then searched the greatest overall desirability. With a desirability of 89.9% (Figure 1a), the optimum conditions predicted by the BBD model to maximize the combined Y1, Y2 and Y3 responses were 37% ethanol, 55 °C and 224 W, for 30 min. The experimental values agreed within a 95% confidence interval with the predicted values, $p = 0.104$. Regarding Y4 response, the carotenoids extraction from chayote peel was maximized using 75% ethanol, 30 °C and 200 W, for 61 min (desirability of 100%, Figure 1b). The experimental value agreed within a 95 % confidence interval with the predicted value, $p = 0.203$. Thereby, the adequacy of the models in predicting the optimum UAE conditions of phenolics, carotenoids and antioxidant compounds from chayote peels was confirmed.

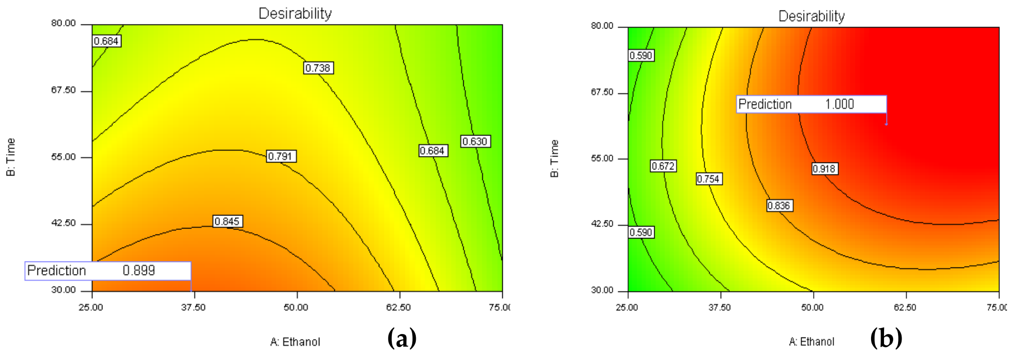

Figure 1. Desirability index for combined responses Y1 (TPC), Y2 (FRAP) and Y3 (ABTS) **(a)** and for Y4 (TC) response **(b)**.

The TPC values obtained in this work (406 mg GAE/ 100 g dw) are in the same range than those reported by [2], ~ 500 mg GAE/ 100 g dw, when a UAE probe device (20 kHz, 375 W) was used and extractions were performed with 25% ethanol, 140 rpm, 30 min, at 25 °C and a sample/solvent ratio of 100 mg/mL. Nevertheless, these authors showed that extraction with 100% water stood out as the most potent in obtaining phenolic compounds (746.46 ± 58.73 mg GAE/ 100 g dw). The TC values obtained in this work are significantly higher than values reported by [3], 1.7 mg/ 100 g dw, when powdered samples were extracted in acetone and transferred to petroleum ether phase for carotenoids estimation.

3.3. Phenolic Composition Profile of Optimal Extract

HPLC-DAD was employed to evaluate the phenolic composition profile of chayote peel extracts; Table 2 summarizes the identified phenolic compounds by the chromatographic analysis, which could contribute to the antioxidant activity observed in the optimal extract. The phenolic composition determined by HPLC-DAD revealed the presence of compounds belonging to different families, with 4-hydroxyphenilacetic acid (33.32 ± 1.67 mg/100 g dw), gallic acid (15.09 ± 0.75 mg/100 g dw), protocatechuic acid (14.99 ± 0.75 mg/100 g dw), ferulic acid (14.9 ± 0.75 mg/100 g dw) and *p*-coumaric acid (11.2 ± 0.56 mg/100 g dw) being the major contributors to the demonstrated antioxidant properties of the produced UAE-chayote peel extract. These phenolic compounds have been previously identified in chayote peel extracts [2]; however, different amounts have been quantified depending on the variety, as well as from the extraction conditions employed. Similar as reported by [2], the phenolic acid caffeic acid and the flavonone naringin were also quantified in the UAE- chayote peel extract. The phenolic acids accounted for 71% of the total compounds quantified, followed by 21% of flavonols (mostly represented by myricetin).

Table 2. Content (mg/100 g dw) of the identified phenolic compounds in the optimal UAE-chayote peel extract. Results were expressed as mean ± standard deviation (n = 3).

Compounds	UAE (mg/ 100 g dw)
Gallic acid	15.09 ± 0.75
Protocatechuic acid	14.99 ± 0.75
4-hydroxyphenilacetic acid	33.32 ± 1.67
4-hydroxybenzoic acid	ND
4-hydroxybenzaldehyde	1.03 ± 0.05
Chlorogenic acid	4.91 ± 0.25
Vanillic acid	4.28 ± 0.24
Caffeic acid	0.91 ± 0.05
Syringic acid	0.35 ± 0.18
p-coumaric acid	11.2 ± 0.56
Ferulic acid	14.9 ± 0.75
Sinapic acid	2.21± 0.11
Cinnamic acid	6.47 ± 0.32
∑ Phenolic acids	**118.93 ± 5.95**
(+)-Catechin	1.80 ± 0.09
(-)Epicatechin	4.50 ± 0.23
∑ Flavanols	6.30 ± 0.31
Naringin	5.98 ± 0.30
Naringenin	1.06 ± 0.05
Pinocenbrin	2.13 ± 0.11
∑ Flavanones	**9.16 ± 0.46**
Rutin	1.03 ± 0.05
Quercetin-3-*O*-glucopyranoside	2.05 ± 0.10
Quercetin-3-*O*-galactoside	ND
Myricetin	20.96 ± 1.05
Kaempferol-3-*O*-glucoside	4.28 ± 0.21
Kaempferol-3-*O*-rutinoside	1.34 ± 0.06
Quercetin	2.13 ± 0.11
Tiliroside	0.91 ± 0.04
Kaempferol	2.29 ± 0.11
∑ Flavonols	**34.92 ± 1.75**
∑ Stilbenes (Resveratrol)	**1.56 ± 0.08**
Phloridzin	0.56 ± 0.02
Phloretin	0.34 ± 0.02
∑ Others	**0.90 ± 0.05**
∑ All phenolic compounds	**167.03 ± 8.35**

ND: not detected.

3.4. Vitamin A, Vitamin E, Carotenoid, and Chlorophyll Composition Profile of Optimal Extract

The HPLC analysis of the UAE-chayote peel extract showed that tocopherol esters were the main class of carotenoids (1047.20 ± 121.41 µg/ g dw extract), followed by retinol esters (245.89 ± 34.85 µg/ g dw extract) and α-tocopherol (219.13 ± 12.80 µg/ g dw extract). The content of α-tocopherol was higher than that reported for spinach, 75-88 µg/g DW, suggesting that chayote peel might have the potential to supply nutritionally relevant vitamin E in the diet.

4. Conclusions

This study successfully applied a BBD as a practical approach to optimize the UAE conditions of phenolics and carotenoids from chayote peels, which can be further safely applied to food or cosmetic industries creating an added value to this residue.

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