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Optimization of ultrasonic-assisted extraction of antioxidants from lemon peel waste using response surface methodology and multi-response desirability function

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The aim of this study was to model ultrasound-assissted extraction (UAE) of antioxidants from lemon peels using an experimental design coupled with multi-response desirability function. A Box-Behnken Design (BBD) was employed to investigate the effects of sonication time (2, 6, 10 min), ratio of acetone in extraction solvent (0, 50, 100%) and volume of solvent (10, 15, 20 mL) on antioxidant capacity of the peel. 1,1-Diphenyl-2-picryl-hidrazil ($DPPH^{\bullet}$), 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) ($ABTS^{+}$), *N*,*N*-dimethyl-*p*-phenylene diamine dihydrocholide ($DMPD^{+}$) radical scavenging activity, β-caroten bleaching and reducing power antioxidant assays were responses in the optimization process. It was found that the optimum UAE parameters were extraction time of 6 min, 80% acetone, volume of 12 mL as extraction solvent. Under these optimum UAE conditions, maximum antioxidant capacity of lemon peel was obtained. By this rapid, easy and efficient process, extraction of valuable antioxidants in by-products such as lemon peels can be conducted.

Keywords: Lemon peel, antioxidant activity, optimization, ultrasound-assisted extraction, Box-Behnken Design.

Introduction

Molecular oxygen produced by plants during photosynthesis is critical to all living things. Oxygen consumption during cell growth gives rise to the formation of a number of reactive oxygen species (ROS)[1]. Oxygen which is known to play a vital role in many biological processes can lead to formation of free radicals such as superoxide, hydrogen peroxide, peroxynitrite, lipid and hydroxyl radicals derivatives by interacting with specific molecules of body during natural processing. Low concentrations of ROS show an effect that regulates intracellular signaling and homeostasis, while high concentrations play a major role in protein, lipid and DNA damage[2].

Antioxidants are compounds that delay, prevent or eliminate oxidative damage of any molecule[3]. A great number of plants including vitamins, flavanoids and carotenoids especially show a huge antioxidant activity. Citrus plants belonging to the family Rutaceae and their fruits are a source of numerous benefits for human health such as antioxidants, anticarcinogens, anti-inflammatory, antidiabetic and anti-lubricant effects[4,6] and they are known for their high levels of flavanone and vitamin in comparison to other fruits and vegetables. Similarly, lemon fruits are rich in nutrients compounds such as ascorbic acid, pectin, fibers, essential oils, different organic acids and phenolic compounds[7].

In the fruit juice and fruit product industry, a large amount of citrus peel occurs as waste for every year[8]. Generally, butylated hydoxyanisole (BHA), butylated hydroxytoluene (BHT) and propyl gallate (PR) which are synthetic antioxidants are used to increase the storage stability of foodstuffs in industrial processes. However, due to the possible toxicity of chemicals used as antioxidants, there has been growing interest in natural antioxidants[9]. Hence, recently, researchers have focused on inexpensive, quick, and not harmful to the environment extraction techniques along with medicinal plants to extract natural antioxidants. Various extraction methods such as maceration, Soxhlet, pressurized liquid, supercritical fluid, microwave-assisted and ultrasound-assisted extraction can be used in extraction of natural antioxidants from various plants[10]. Soxhlet extraction and macera-

tion, which are classical extraction methods, are very prolonged processes that require large amounts of toxic organic solvents, which are able to lead thermal degradation of target molecules. Use of green extraction techniques such as ultrasound-assissted extraction (UAE) has been known as faster, facile, environmentally friendly way of extraction of antioxidants from natural materials along with minimizing energy costs[11,14]. This extraction method is generally affected by several variables, such as type and volume of extraction solvent, solvent to solid ratio, extraction time and temperature. Hence, optimization of these parameters is very crucial to provide high amount of antioxidant compounds. The parameters affecting the response in multivariate experiments and their interactions can be determined by use of statistical experimental design[13,15]. Response Surface Methodolgy (RSM) allows statistical evaluation of the data and determination of the most suitable conditions for the experiment, so experimental design provides advantages in terms of the efficiency of the procedures and shortens time spent for optimization process.

In addition, if various depented variables are wanted to be optimized simultaneously, Derringer's desirability function is a useful and practical method in the optimization of a process having multiple responses. A detailed description of Derringer's desirability functions has been discussed in these sources[16,17].

In this work, optimum conditions for UAE of antioxidants from waste lemon peels were investigated by statistical experimental design containing three factors sonication time, ratio and volume of extraction solvent. Afterwards, the multicriteria decision making approach, Derringer's desirability function based on five different antioxidant capacity tests, was used to obtain high efficiency. The objectives of the study contribute to valorisation of the waste products and decrease chemical solvent consumption and time spent especially for optimization process.

Experimental

Citrus material preparation:

Lemons were bought from local market in Istanbul, Turkey during the June-July 2018. The lemons were washed and then peeled. The peels were chopped into small pieces and dried in an oven at 40°C for 4–5 days. The dried materials were ground into powder with grinder[18]. The powder was stored at +4°C until use.

Optimization of UAE:

Extraction process was carried out based on a Box-Behnken Design (BBD) using three levels of each factor (high, center point and low levels). Table 1 has shown factors levels used in BBD. 1000 mg of the powdered peels of lemon extracted three different levels were examined by selecting sonication time (2, 6, 10 min), ratio of extraction solvent in acetone (0, 50, 100%) and amount of extraction solvent (10, 15, 20 mL) with ultrasonic bath (Bandelin Electronic, 320 W 35 kHz). Table 2 displays the BBD matrix of the experiment of 15 trials. After extraction process, the homogenates were centrifuged at 10000 rpm for 15 min. Then, the supernatants were evaporated for about 12–15 h until dryness. Dried extracts were diluted in suitable solvent prior to antioxidant capacity analysis.

Table 1. Factors levels used in BBD

Factor	Notation	Factor Levels		
		Low (–1)	Center point (0)	High (+1)
Sonication time (min)	A	2	6	10
Ratio of acetone in extraction solvent (%)	B	0	50	100
Volume of extraction solvent (mL)	C	10	15	20

$DPPH^{\bullet}$ radical-scavenging assay:

Brand-Williams method was used for the free radical scavenging activity of the lemon peel extracts. This method is based on the decreasing the intensity of the purple color of DPPH when treated with antioxidants. 20 mg/L radical solution in ethanol was prepared, then 0.75 mL of samples (1000 μg/mL) were taken and 1.5 mL of DPPH was added of this sample solution. After the mixture was shaken and kept in dark for 30 min. The absorbance was recorded at 517 nm using UV-spectrophotometer[19].

$ABTS^{+}$ radical-scavenging assay:

This cation radical scavenging activity of the lemon peel extracts were measured with Arnao's method. In this method, 4 mM ABTS and 2.6 mM potassium persulfate solution were mixed equal volumes to prepare the $ABTS^{+}$ radical solution. The solution was incubated at room temperature in dark for

12 h. Then, the solution was diluted with approximately 60 mL methanol to obtain an absorbance of about 1.1 units at 734 nm. 150 μL of lemon peel extracts (1000 μg/mL) and 2850 μL of the $ABTS^{\bullet+}$ solution were mixed and kept in the dark for 2 h. The absorbance was measured at 734 nm. Trolox solutions (100–500 μM) were used as standards and results are calculated as μM Trolox equivalents (TE)/μg extract[20].

$DMPD^{\bullet+}$ scavenging assay:

Firstly, 100 mM DMPD+ radical solution was prepared for this method developed by Fogliano *et al.*[21]. 1 mL of this solution was added to 50 mL of 0.1 *M* acetate buffer (pH 5.3) in a test tube. Then, 0.2 mL of 0.05 *M* iron(III) chloride was added to this mixure. The extracts were transferred to test tubes and 0.5 mL of distilled water was added. After 10 min, the absorbance was measured spectrophotometrically at 505 nm.

β-Carotene bleaching test:

After trans-β-carotene (10 mg) was dissolved in 10 mL of chloroform, 0.2 mL of this solution was added to 20 mg of linoleic acid and 200 mg of Tween-40 mixture. After chlorofom blown away, 50 mL of distilled water was supplemented to the mixture and vigorously shaken to obtain emulsion. Afterwards, 0.2 mL of extract solution (1000 μg/mL) was taken and 5 mL of emulsion was added to this extracts. The tubes were incubated in a water bath at 50°C. After 60 and 120 min of incubation, absorbance was measured at 470 nm. Butylated hydoxyanisole (1 mg/mL) was used as positive control. As a negative control solution, 5 mL of the above emulsion along with 0.2 mL of distilled water was mixed and used[22].

Reducing power assay:

The reducing power of the lemon peel extract was determined by the Oyaizu method. 1000 μg/mL extract was mixed with 2.5 mL of 0.2 *M* phosphate buffer, pH 6.6 and 2.5 mL of 1% (w/v) potassium ferricyanide. After the mixture was incubated at 50°C for 30 min, 2.5 mL of 10% (w/v) trichloroacetic acid was added to it and centrifuged at 10000 rpm for 5 min. Then 2.5 mL of supernatant portion was stirred with 2.5 mL of distilled H_2O and 0.5 mL of 0.1% (w/v) iron(III) chloride and finally the absorbance was spectrophotometrically measured at 700 nm[23].

Results and discussion

Experimental design for extraction process:

Box-Behnken Design (BBD) (Table 2) for the following

Table 2. Design matrix and responses of BBD

Run	Factors			Responses				
	A	B	C	Y_1	Y_2	Y_3	Y_4	Y_5
1	2	100	15	49.26	499.0	0.96	0.607	27.98
2	10	100	15	82.20	479.0	0.97	0.506	33.36
3	2	0	15	81.20	278.4	0.90	0.558	90.90
4	10	0	15	91.10	367.8	1.05	0.607	92.02
5	2	50	10	93.50	384.3	0.99	0.641	88.16
6	10	50	10	93.50	396.7	0.99	0.661	87.94
7	2	50	20	92.40	423.1	0.99	0.658	86.18
8	10	50	20	90.70	545.5	1.01	0.637	85.97
9	6	100	10	77.00	527.8	0.96	0.561	-0.47
10	6	0	10	91.00	343.1	1.01	0.571	87.44
11	6	100	20	79.20	516.7	0.96	0.424	-5.96
12	6	0	20	90.80	263.1	1.03	0.625	86.72
13	6	50	15	92.40	422.5	0.98	0.685	85.31
14	6	50	15	92.80	382.5	0.98	0.672	84.15
15	6	50	15	93.40	413.7	0.98	0.054	84.35

A: Sonication time (min); B: ratio of acetone in extraction solvent (%); C: volume of extraction solvent (mL);

Y_1: $DPPH^{\bullet}$ radical scavening activity (%); Y_2: $ABTS^{+}$ radical scavening activity (%); Y_3: β-caroten bleaching test; Y_4: reducing power; Y_5: $DMPD^{+}$ radical scavening activity (%).

variables sonication time (A), ratio of acetone in extraction solvent (B), and volume of extraction solvent (C) according to the mentioned above conditions (15 experiments) were undertaken. The effects of these factors on DPPH$^{\bullet}$ radical scavenging activity (Y_1), ABTS^{+} radical scavenging activity (Y_2), β-caroten bleaching test (Y_3), reducing power (Y_4) and DMPD^{+} radical scavening activity (Y_5) were determined to reveal the major factors for the UAE process. Analysis of variance (ANOVA) was calculated to determine the most important effects and interactions. The results of analysis ANOVA and the regression model were summarized in Table 3. A p-value less than 0.05 in the ANOVA table means that there is statistical significance of a factor at a 95% confidence level. The F-test was also used to predict the statisti-

Table 3. Analysis of variance (ANOVA) for BBD

Source	Sum of squares	DF≠	Mean square	F-Ratio	p-Value	Estimated effect	Regression coefficient
Y_1							
A	1177	1	1177	119.99	0.0001*	24.26	12.13
B	552	1	552	56.25	0.0007*	16.61	8.30
C	10	1	10	1.08	0.3466	2.3	1.15
AA	483	1	483	49.24	0.0009*	–22.88	–11.44
AB	133	1	133	13.53	0.0143*	–11.52	–5.76
AC	41	1	41	4.18	0.0965	–6.4	–3.2
BB	111	1	111	11.34	0.0200*	–10.98	–5.49
BC	1	1	1	0.15	0.7174	–1.2	–0.6
CC	31	1	31	3.12	0.1377	–5.76	–2.88
Total error	49	5	9.81				
Total	2536	14	R^2 = 0.9806	Adj. R^2 = 0.9458			
Y_2							
A	1177	1	1177	119.99	0.0001*	24.26	12.13
B	552	1	552	56.25	0.0007*	16.61	8.30
C	10	1	10	1.08	0.3466	2.3	1.15
AA	483	1	483	49.24	0.0009*	–22.88	–11.44
AB	133	1	133	13.53	0.0143*	–11.52	–5.76
AC	41	1	41	4.18	0.0965	–6.4	–3.2
BB	111	1	111	11.34	0.0200*	–10.98	–5.49
BC	1	1	1	0.15	0.7174	–1.2	–0.6
CC	31	1	31	3.12	0.1377	–5.76	–2.88
Total error	49	5	9.81				
Total	2536	14	R^2 = 0.9806	Adj. R^2 = 0.9458			
Y_3							
A	0.00125	1	0.00125	17.61	0.0085*	0.025	0.0125
B	0.00495	1	0.00495	69.74	0.0004*	0.050	0.025
C	0.00023	1	0.00023	3.26	0.1310	0.010	0.005
AA	0.00011	1	0.00011	1.55	0.2683	0.010	0.005
AB	0.00078	1	0.00078	11.04	0.0209*	0.028	0.014
AC	0.0	1	0.0	0.13	0.7363	0.003	0.0015
BB	0.00005	1	0.00005	0.72	0.4363	0.007	0.0035
BC	0.00011	1	0.00011	1.55	0.2679	0.010	0.005
CC	0.00005	1	0.00005	0.72	0.4363	0.007	0.0035
Total error	0.00035	5	0.000070				
Total	0.00787	14	R^2 = 0.9549	Adj. R^2 = 0.8738			

Table-3 (contd.)

Y_4							
A	0.00035	1	0.00035	0.41	0.5506	–0.013	–0.0066
B	0.00864	1	0.00864	10.07	0.0247*	0.065	0.0325
C	0.00101	1	0.00101	1.18	0.3270	–0.022	–0.011
AA	0.00002	1	0.00002	0.03	0.8803	–0.004	–0.002
AB	0.00562	1	0.00562	6.55	0.0507	0.075	0.0375
AC	0.00042	1	0.00042	0.49	0.5153	–0.0205	–0.01025
BB	0.04181	1	0.04181	48.71	0.0009*	–0.212	–0.106
BC	0.00912	1	0.00912	10.62	0.0225*	0.096	0.048
CC	0.00262	1	0.00262	3.06	0.1407	–0.053	–0.0265
Total error	0.00429	5	0.00086				
Total	0.07274	14	R^2 = 0.9410	Adj. R^2 = 0.8348			
Y_5							
A	4.59	1	4.59	0.06	0.8090	1.515	0.757
B	10933	1	10933	154.55	0.0001*	73.93	36.96
C	2.71	1	2.71	0.04	0.8525	–1.16	–0.58
AA	368.50	1	368.50	5.21	0.0713	19.98	9.99
AB	4.56	1	4.56	0.06	0.8097	–2.13	–1.06
AC	0.0	1	0.0	0.00	1.0000	0.0	0.0
BB	4151	1	4151	58.68	0.0006*	–67.06	–33.5
BC	0.13	1	0.13	0.00	0.9677	–0.36	–0.18
CC	210	1	210	2.96	0.1458	–15.07	–7.535
Total error	353.7	5	70.7				
Total	16164.4	14	R^2 = 0.9781	Adj. R^2 = 0.9387			

≠DF; degree of freedom; *Significant.

cal significance of all terms in the model equation within a 95% confidence interval. Determination coefficients (R^2) and adjusted determination coefficients (Adj. R^2) were close to 1, which indicate a high correlation between the experimental and predicted values. Likewise, R^2 must be greater than 0.80 in order to demonstrate an accurate model[24].

As shown in the Pareto charts (Fig. 1), A and B factors had a significant effect in the UAE process for the responses of Y_1, Y_2 and Y_3, while C factor and some interactions between the factors in the selected levels were negligible. Also since A and B factors have a positive effect on the responses, a higher response can be achieved by increasing their amounts. For the investigation of significant effects in the UAE process for the responses of Y_4 and Y_5, as can be seen in Fig. 1d and 1e, only B factor was found as statistically significant at the studied range. AA and BB, quadratic contributions of the factors also effect the UAE process significantly, except Y_3.

Multi-response optimization:

A Derringer's desirability function was employed to simultaneously optimize five responses including DPPH$^{\bullet}$ radical scavenging activity (Y_1), $ABTS^+$ radical scavenging activity (Y_2), β-caroten bleaching test (Y_3), reducing power (Y_4) and $DMPD^+$ radical scavening activity (Y_5). If several responses have to be optimized simultaneously and to determine the most favorable extraction parameters, this method is very efficient and powerful. Response surface graphs were used to envisage the profiles of estimated desirability's values. The graphs are provided on Fig. 2, illustrating the overall desirability (from 0 to 1) for the maximization of responses, by the altering of factors in UAE process. The optimum UAE parameters found by means of this chemometric tool also conformed with the observed effects the statistical analysis of each UAE parameters previously described above. After desirability 3D graphs were plotted as a function of the factors examined, namely A, B and C, a maximum desirability =

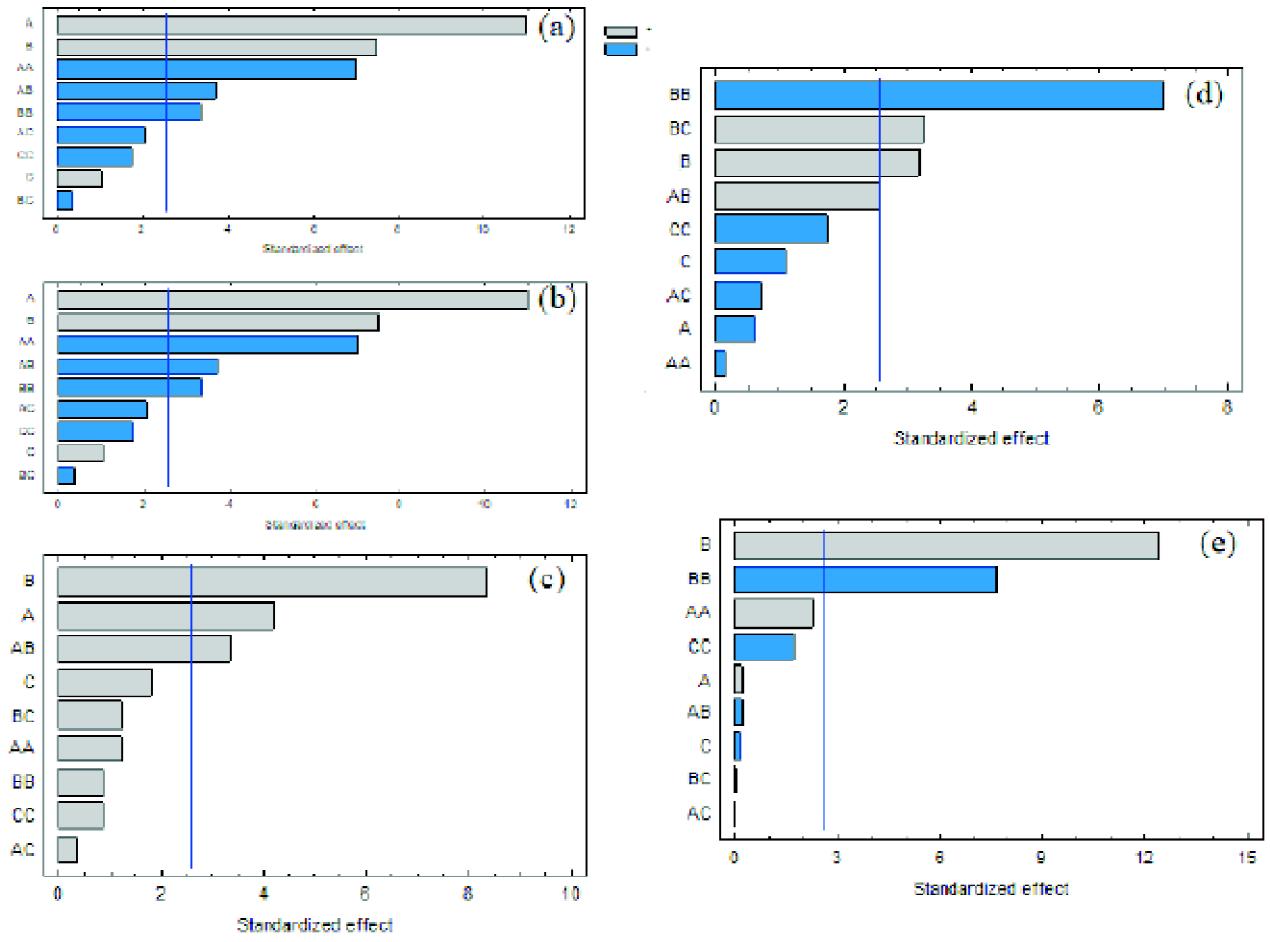

Fig. 1. Pareto charts obtained from BBD for: (a) Y_1: DPPH$^{\bullet}$ radical scavening activity (%); (b) Y_2: ABTS^{+} radical scavening activity (%); (c) Y_3: β-caroten bleaching test; (d) Y_4: reducing power; (e) Y_5: DMPD^{+} radical scavening activity (%).

1.0 was achieved for the UAE parameters: Sonication time (A) = 6 min, ratio of acetone in extraction solvent (B) = 80% and volume of extraction solvent (C) = 12 mL.

Comparison of the developed UAE method and other UAE techniques:

Citrus fruit have been intrigued for many years because

Table 4. Comparison of the developed UAE method with the other techniques used for by-products of citrus fruits

By-product	UAE conditions			
	Min	Extraction solvent in water (%)	L/S* (mL/g)	Reference
Citrus peel (*Citrus reticulate*)	23–25	–	40	25
Sour orange peel	10	100 acidified water	20	26
Yuzu (*Citrus junos* Sieb ex Tanaka) fruit	119.67	65.55 ethanol	37.168	27
Citrus limon residues	15.05	63.93 ethanol	40	28
Fortunella margarita Swingle polysaccharides	87	100 water	32	29
Mandarin (*Citrus reticulate* Blanco cv. Sainampueng) peel	40	80 acetone	20	30
Lemon peel	6	80 acetone	12	This study

*L/S: Liquid/Solid.

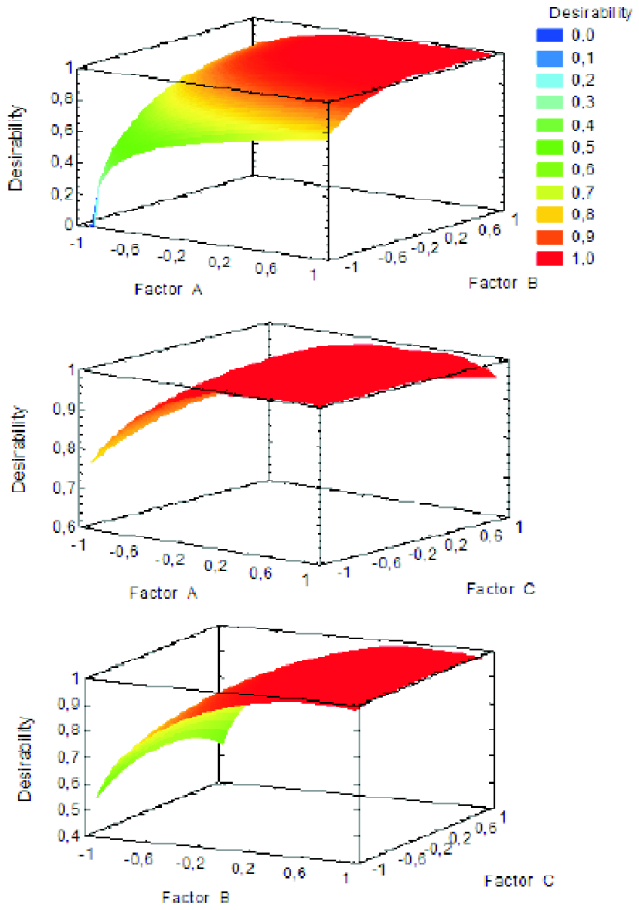

Fig. 2. Response surface graphs for the overall desirability function (the other factor was fixed at its optimum value).

of antioxidant properties. Especially in the fruit juice industry, large amounts of waste are produced and these wastes are important in terms of natural antioxidant sources. When the results obtained are compared with the other studies (Table 4), it is clear that the optimization process of this study is advantageous in sonication time and solvent consumption.

Conclusions

The optimum UAE conditions developed in this work regarding extraction of antioxidants in lemon peel were determined using multi-response desirability function. UAE is a suitable, practical and rapid method for the extraction of antioxidants from by-products and can be employed for the replacement of the conventional extraction methods which requires more consumption of solvent and time in the industrial scale extraction of the antioxidants. The results implied that antioxidant activity of lemon peel was effected most significantly sonication time and extraction solvent content in the investigated levels. Also, some interactions were observed between the studied factors, which were understood by the parabolic view of 3D graphs. The optimum UAE conditions included the use of 12 mL 80% acetone in water for 6 min. These easily achievable extracts can be used in pharmaceutics and cosmetics to develop nutraceutical or food supplements.

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