Optimizing extraction conditions of crude fiber, phenolic compounds, flavonoids and antioxidant activity of date seed powder

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Abstract The objective of this study was to optimize the extraction conditions of crude fiber, phenolic compounds, flavonoids, and antioxidant activity from date seeds powder, using Response Surface Methodology. A central composite design with four independent variables; concentration of ethanol ($X_1 = 25, 50$ and 75% v/v), solvent: sample ratio ($X_2 = 40:1, 50:1$ and $60:1$ v/w), temperature ($X_3 = 45, 55$ and $65\text{ }^\circ\text{C}$), and extraction time ($X_4 = 1, 2$ and 3 h) and a three level face centered cube design were used. A total of twenty nine experimental runs with five replicates at the central point were used to study the response variables using two extraction cycles. Maximum phenolic compound content ($71.6\text{ mg GAE}/100\text{ g}$) was extracted using 50% ethanol solution with $40:1$ solvent: sample ratio for 1 h at $55\text{ }^\circ\text{C}$. While the maximum antioxidant activity ($55.02\text{ }\mu\text{mol Fe(II)}/\text{g}$) was obtained using similar ethanol concentration and solvent: sample ratio except at lower temperature ($45\text{ }^\circ\text{C}$) for 2 h . On other hand, the maximum flavonoids content ($455.77\text{ mg CEQ}/100\text{ g}$) was reached by using 50% concentration, $50:1$ solvent: sample ratio at $65\text{ }^\circ\text{C}$ for 3 h . In contrast, the content of fiber was not affected by the different extraction conditions. Results indicate that using combination of extracted conditions, have a great potential for extracting all depending compounds except crude fiber.

Keyword Response surface methodology · Date seeds powder · Crude fiber · Phenolic compounds · Flavonoids · Antioxidant activity

Introduction

The date palm trees (*Phoenix dactylifera* L.) have gained more attention among Arab countries over years, particularly the Gulf countries. According to FAO (2014), dates are the most produced dried fruit in 2014, with 7.5 million tons (including table dates and dates used for industrial purposes). The United Arab Emirates (UAE) is the fourth leading producer of dates, producing 900 thousand tons annually. This represents 13% of the world's date production. Based on that, date industries yield a significant amount of unutilized date seeds (pits) from different processing operations (molasses and paste), which can reach up to 8–15% of the total weight in completely ripe date fruit depending on variety and quality grade (Al-Farsi et al. 2007). Lately, date pits have been used for animal feeding and for energy recovery as fuel (Joardder et al. 2012).

Date fruits contain a high level of soluble sugars (44–88%), fat (0.20–0.50%), protein (2.30–5.60%), and crude fiber (6.40–11.50%) (Al-Farsi et al. 2007; Al-Shahib and Marshal 2003; El-Sohaimey and Hafez 2010). Additionally, dates are considered a good source of polyphenols (3.0–8.36 g/100 g), such as simple phenolic acids and flavonoids (Al-Farsi et al. 2007). Comparatively, with date flesh compositional analysis, seeds contain 72.59–83.10% carbohydrates, 10.19–12.67% fat, 5.17–5.67% protein, 1.12–1.18% ash and nearly 9.40% crude fiber (Besbes et al. 2004; Nehdi et al. 2010). Therefore, date seeds as a by-product are a good source of crude fiber, which makes it

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suitable raw material for the preparation of fiber-based foods and crude supplements.

In addition to the crude fiber, phenolic compounds are bioactive substances that are widely distributed in plants, as well as they are important constituents of the human diet. Date seeds contain high levels of antioxidants 580–929 μm Trolox equivalents/g) and phenolic (3102–4430 $\mu\text{g}/100\text{ g}$) as reported by Al-Farsi et al. (2007). Whereas, polyphenols and flavonoids took considerable interest for their antioxidant properties as they act as free radical scavengers and inhibitors of low-density lipoprotein (LDL), of cholesterol oxidation and of DNA breakage (Barbosa-Pereira et al. 2013; Galleano et al. 2010; Haminiuk et al. 2012; Rehecho et al. 2011).

Since dates have high nutritive benefits as well as being a valuable raw material for several food industries (i.e. jam, syrup, cookies, etc.), numerous ways of date utilization were improved. With increasing awareness of the physiological benefits of crude fiber and phenolic compounds as antioxidants, there is an urgent need to enrich food with natural antioxidants and provide the public with natural and cheap resources of these compounds. Therefore, extraction of active ingredients is essential when it has therapeutic value for the human health, particularly if the extraction was done from neglected by-products.

The aim of this work is to maximize the utilization of date seeds by-product, by preparing extracts as well as choosing optimal extraction conditions of phenolic compounds, flavonoids and crude fiber under conditions compatible with food requirements.

Materials and methods

Date seeds powder

Ten kilograms of raw seeds of mixed date varieties (mainly Khalas, and Barhi) were obtained from Al Foah Dates Co., Al Ain, UAE. Date seeds were washed with water to get rid of any adhering date flesh, then air-dried. Collected seeds were grounded in a heavy-duty hammer mill (W4SS, Buffalo, NY, USA), sieved to 300–500 μm (Laboratory Test Sieve, Endecotts LTD., London, England). Date seed powder was packed in plastic bags and stored at room temperature in a dark place until used for extraction.

Chemicals

Absolute ethanol was acquired from Panreac (Barcelona, Spain). Other chemicals were of analytical grade and obtained from Sigma–Aldrich–Fluka Co. Ltd. (St. Louis, Mo, USA) unless otherwise be specified.

Experimental design

Optimization of extraction conditions of phenolic compound, flavonoids and crude fiber from date seeds powder were carried out using a central composite design (CCD) and response surface methodology (RSM), according to Myers and Montgomery (2001, 2002). A four factors and a three level face centered cube design consisting of twenty-nine experimental runs were conducted with five replicates at the central point (Jing et al. 2012). The orders of all experiments were performed randomly to minimize error, due to extraneous factors. The four independent variables are X_1 , concentration of solvent; X_2 , solvent: sample ratio; X_3 , extraction temperature and X_4 , extraction time at three levels (−1, 0, 1).

To determine the influence of selected parameters on the response variables, data were fitted to a second-order polynomial equation according to Bruns et al. (2006) and Sarkis et al. (2014) using the following equation:

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=1}^3 \beta_{ij} X_i X_j$$

where Y is the predicted response, β_0 , β_i , β_{ii} and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms respectively, and X_i , and X_j are the independent variables.

To determine the influence of selected parameters on the response variables, the complete set of experimentally observed response values for the extraction yield design is given in Table 1.

Extraction procedure

A portion of 10 g of date seed powder (300–500 μm) was weighed, transferred into conical flasks and mixed with ethanol solution. To monitor the temperature during extraction procedure, suspension samples were incubated in a calibrated incubator with shaking (SS15 Shel Lab Floor Shaking Incubator, Shedon Manufacturing, Inc., USA) for limited time according to the extraction time. Subsequent to the extraction, centrifugation was done using Sigma 2–16 Benchtop Laboratory Centrifuge at 3000 rpm/10 min (SciQuip Ltd, UK), followed by a filtration through Whatman filter paper No. 4. Two cycles of extraction, centrifugation and filtration were used to maximize the extraction yield as shown in Fig. 1.

The solid residues remained after filtration were dried at 60 °C in a conventional oven to get the Fiber Concentration Extract (FCE), which was used as the main source of crude fiber. While the supernatant portion was evaporated under vacuum at 40 °C using LABOROTA 4000 Eco rotary evaporator (Heidolph's Corporate, Germany) to

Table 1 Significant regression coefficient of the predicted quadratic polynomial models for the investigated responses and model parameters ($P < 0.05$)

Constant	Responses ^b									
	FCE					PC				
	Crude fiber		L^*		a^*		b^*		Phenolic compounds	
	Coef	P value	Coef	P value	Coef	P value	Coef	P value	Coef	P value
β_0	18.17	0.000	46.61	0.000	12.81	0.000	20.65	0.000	17.85	0.000
<i>Linear</i>										
β_1	-0.93	-	1.54	0.000	-0.00	-	0.62	0.000	4.02	0.001
β_2	0.06	-	0.60	-	-0.04	-	0.44	0.046	13.13	0.000
β_3	0.11	-	-0.43	-	-0.64	0.000	-0.53	0.001	2.51	0.015
β_4	0.16	-	-0.01	-	-0.47	0.000	-0.40	0.029	16.26	0.000
<i>Quadratic</i>										
β_{11}	-0.11	-	-2.68	0.000	0.84	0.000	-0.85	0.001	-20.61	0.000
β_{22}	0.06	-	0.64	-	-0.32	0.012	-0.06	-	12.43	0.000
β_{33}	-0.63	-	-0.74	-	-0.41	0.000	-0.21	-	23.92	0.000
β_{44}	-1.31	-	0.26	-	-0.44	0.000	-0.05	-	18.96	0.000
<i>Interaction</i>										
β_{12}	0.45	-	-1.68	0.008	0.05	-	-0.54	0.039	-14.92	0.000
β_{13}	-1.66	0.005	0.48	-	0.23	0.001	0.12	-	-3.923	0.001
β_{14}	-1.10	-	-0.55	-	0.31	0.005	0.35	-	-14.63	0.000
β_{23}	-0.16	-	2.47	-	-0.60	0.013	1.28	0.027	41.37	0.000
β_{24}	-0.01	-	1.25	-	-0.43	0.022	0.28	-	36.23	0.000
β_{34}	0.78	-	-0.11	-	-1.05	0.000	-0.50	-	25.87	0.000
R^{2a}	70.77	-	90.35	-	96.68	-	91.20	-	98.43	-
CV	1.15	-	0.77	-	0.13	-	0.33	-	2.30	-
Whereas 1 is solvent concentration, 2 is Solvent/sample ratio, 3 temperature and 4 extraction time										
CV is the coefficient of variation										
^a R^2 for multiple determination										
^b The maximum predictable response was obtained based on a total of 29 experiments required for determining 12 regression coefficients										

Fig. 1 Flow diagram of preparation of phenolic concentrates (PC) and fiber concentration extract (FCE) from date seed powder

prepare the Phenolic Concentrate (PC). Crude PC extracts were kept in dark glass bottles inside the freezer till analyzed for phenolic compounds, flavonoids and antioxidant activity.

Determination of FCE yield

FCE yield is referred to the solid residues remained after the extraction and expressed as a percentage compared with the initial dry date seed powder.

FCE yield was carried out in triplicates and calculated using the following equation:

$$Y\% = \left(\frac{C}{Z} \right) * 100$$

where C is the weight (g) of solid residue remained after centrifugation for 2 cycles; Z is the weight (g) of dry date seed powder, and Y % is the yield (g/100 g).

Analysis of the response variables in FCE

Crude fiber

Crude fiber content was determined in the FCE, the remained residue powder after extraction, according to AOAC (2000). All samples were analyzed in triplicates.

Color

Color of FCE remained from date seed powder after extraction was measured using Hunter Lab Color Flex EZ Spectrophotometers. L^* , a^* , and b^* values were directly read

in a glass cuvette with spectrophotometer, calibrated with black and white tiles. In this coordinate system, the L -value is a measure of lightness, ranging from 0 (black) to 100 (white), the a -value ranges from -100 (greenness) to $+100$ (redness) and the b -value ranges from -100 (blueness) to $+100$ (yellowness). All samples were analyzed in triplicates.

Analysis of the response variables in PC

In order to evaluate the potential of extract conditions, total phenolic compounds, flavonoids and antioxidant activity were determined in PC liquid portion as follow:

Total phenolic compounds

Total phenolic compounds were determined in the PC extracts using Folin–Ciocalteu reagents (Singleton et al. 1999). The PC extract or Gallic acid (40 μ l) standard was mixed with 1.8 ml of Folin–Ciocalteu reagent (prediluted 10-fold with distilled water) and vortexed, then allowed to stand at room temperature for 5 min. Followed by adding 1.2 ml of sodium bicarbonate (7.5%) to the mixture and then vortexed. After standing for 60 min at room temperature with exclusion of light, absorbance was measured at 765 nm using UV-2450 UV–vis Spectrophotometer (Shimadzu, Kyoto, Japan). The total phenolic compounds were calculated from the calibration standard curve of Gallic acid in the range of 10–100 mg/ml. All samples were analyzed in triplicates, and the results were expressed as milligram Gallic acid equivalents (GAE) per 100 g sample in a dry basis (Sarkis et al. 2014; Shui and Leong 2006).

Total flavonoid content

Total flavonoids content were determined in PC extracts according to the colorimetric assay of Kim et al. (2003). Four milliliter of distilled water was added to 1 ml of PC. Followed by adding 5% of sodium nitrite solution (0.3 ml), and then adding 10% aluminum chloride solution (0.3 ml) and vortexed. Test tubes were incubated at ambient temperature for 5 min, then 2 ml of 1 M sodium hydroxide was added to the mixture. Immediately, the volume of the reaction mixture was made to 10 ml with distilled water. The mixture was thoroughly stirred by a vortex and the absorbance of the pink color developed was determined at 510 nm using a UV-2450 UV-vis Spectrophotometer (Shimadzu, Kyoto, Japan). A calibration curve of catechin (in the range of 50–500 mg/ml) was used and all samples were analyzed in triplicates. The results were expressed as milligrams catechin equivalents (CEQ) per 100 g dry sample.

Antioxidant activity of ethanoic extracts

The FRAP assay was carried out to measure the antioxidant activities of PC extracts, according to the procedure described by Benzie and Strain (1996, 1999) and Saikia et al. (2016). Briefly, the FRAP reagent was prepared from sodium acetate buffer (300 mM, pH 3.6), 10 mM TPTZ solution (40 mM HCl as solvent) and 20 mM iron (III) chloride solution in a volume ratio of 10:1:1, respectively. The FRAP reagent was prepared fresh on a daily basis and warmed to 37 °C in a water bath before use. One hundred microliters of the diluted sample were added to a 3 ml of the FRAP reagent. The absorbance of the mixture was measured at 593 nm using a UV-2450 UV-vis Spectrophotometer (Shimadzu, Kyoto, Japan) after 4 min. The standard curve of FeSO₄ solution in the range of 10–100 µmol/ml was used for calibration, and the results were expressed as µmol Fe(II) per gram dry material. All samples were analyzed in triplicates.

Statistical analysis

Response surface methodology was performed using Minitab Software (Minitab version 16.0, Minitab Inc., State College PA, USA), to investigate the obtained results, to build and to evaluate models, and plot the three-dimensional response surface curves. Since there were four independent variables in this study, and the visualization was possible only for three variables, the fourth factor was kept constant at middle level.

Results were given as mean ± SD of three replicates. Data was analyzed using one way analysis of variance (ANOVA) by SPSS (version 19, SPSS Inc., Chicago IL,

USA) and Tukey's multiple comparison tests were used to make comparisons between treatment means. The significance was accepted at *P* value of ≤0.05.

Results and discussion

Response surface analysis

Relevant independent variables of X_1 , X_2 , X_3 and X_4 used in this study were selected depending on previous investigations. Ethanol was used because it is a preferred solvent concerning safety and health issues (Herrero et al. 2005), and it is the second most important solvent subsequent water in polarity. In addition, the most of the flavonoids and phenolic compounds are hydrosoluble antioxidants which extracted using medium polar and polar solvents as ethanol. Different hydro-ethanol mixtures (X_1) were used in this study, whereas, the extraction of polyphenols depends on the polarity of solvents. Therefore, the combination of alcohol and water is more efficient in the extraction of polar and ionic compounds (Markom et al. 2007). Different solvent/sample ratios (X_2) were used to increase the surface area exposed to the solvent for the highest extraction. Besides, three levels of temperatures (X_3) were used. The minimum temperature was 45 °C as a warm condition to increase penetration and diffusion, while the maximum temperature did not exceed 65 °C (less than the boiling point of ethanol 78.5 °C) for the stability of the analyte and avoid the degradation of compounds and to reduce losing of ethanol due to high temperature. Regarding extraction time (X_4), 1, 2 and 3 h were used to increase the effectiveness of extraction. In addition to these variables, extraction was done in two cycles to increase the efficiency of the extraction procedure.

Table 1 shows the coefficients of the full quadratic polynomial equation for the investigated responses and model parameters ($P < 0.05$). Data showed that the regression model at $P < 0.05$ of all dependent variables was highly significant. In addition, it had high R^2 for color values (L^* , a^* and b^*), phenolic compounds and antioxidant activity ranged between 90.33 and 98.43. In contrast, it had low R^2 for crude fiber and flavonoids (70.77, 74.94 respectively). Data showed that the coefficient of variation (CV) was low that gave better reproducibility, therefore, the used model was suitable and explained the variability of all tested extraction factors.

Data in Table 1 shows that all the independent variables had positive linear effects, significant quadratic effects, and significant interactions among factors on phenolic compounds, flavonoids and antioxidant activity. The time (X_4) had significant effects, and it had the largest positive linear regression on phenolic compounds and flavonoids, which

indicate that with increasing extraction time the level of these compounds increases in the extract. While the concentration of ethanol (X_2) had significant and positive linear effects on color parameters. On another hand, temperature (X_3) had significant effects on phenolic compounds, and it had the same effects when combined with other independent variables as solvent concentration ethanol or solvent/sample ratio. This is agreed with Espada-Bellido et al. (2017) who mentioned that solvent composition, temperature and extraction cycle had significant effects on extraction of total phenolic compounds from mulberry. Obviously, none of the four independent variables or their interaction had effects on crude fiber content except for the interaction between the concentration of ethanol and temperature, which showed a significant effect at $P < 0.05$.

Data shows that the interaction among the four independent variables had significant effects on phenolic compounds. While, the interaction between X_1 and X_2 (ethanol concentration and solvent: sample ratio), X_2 and X_4 (solvent: sample ratio and time), and X_3 and X_4 (temperature and time) shows significant effects on both flavonoids and antioxidant activity.

Regarding color values, lightness (L -values) of the FCE was affected significantly only by the concentration of ethanol and its interaction with solvent: sample ratio. While temperature and time which were the most independent variables had individual significant effects on redness (a -values). Contradictory, all independent variables had individual significant effects on yellowness (b -values).

In case of analysis of variance of the regression parameters of the predicted response surface quadratic models in Table 2, data shows that linear, quadratic and interactions had highly significant effects at $P < 0.05$ in the response of all dependent parameters (color values, phenolic compounds, flavonoids and antioxidant activity) except crude fiber. The interaction regression of phenolic compounds and flavonoids was higher than the linear regression. In contrast, the linear regression of color values (L^* , a^* and b^*) were higher than the interaction regression. The interaction regression of a^* values (24.56) was higher than L^* (3.34) and b^* (5.44) values. Therefore, analysis of the model parameters shows that the most influential parameters were phenolic compounds, and flavonoids under these experimental tested conditions. In contrast, the crude fiber was the only parameter that did not affect by any of these extraction conditions.

FCE Yield % and crude fiber

Crude fiber (CF) refers to one type of dietary fiber that remained after dissolving all soluble fiber and some of the insoluble fiber in food. It is mainly composed of cellulose,

Table 2 Analysis of variance of the regression parameters of the predicted response surface quadratic models

Regression	DF ^a	SS	R ²	F value
<i>Crude fiber</i>				
Linear	4	12.15	3.03	2.29
Quadratic	4	10.14	2.53	1.91
Interaction	6	22.51	3.75	2.83
Total model	14	44.81	9.33	2.34
L^*				
Linear	4	26.91	6.72	11.32*
Quadratic	4	50.50	12.62	21.24*
Interaction	6	11.91	1.98	3.34*
Total model	14	89.34	21.34	11.97*
a^*				
Linear	4	3.37	0.89	48.87*
Quadratic	4	2.91	0.72	39.78*
Interaction	6	2.69	0.44	24.56*
Total model	14	8.98	2.16	37.74
b^*				
Linear	4	9.36	2.34	21.02*
Quadratic	4	4.46	1.11	10.01*
Interaction	6	3.63	0.60	5.44*
Total model	14	17.47	4.06	12.16*
<i>Phenolic compounds</i>				
Linear	4	1338.05	334.51	63.03*
Quadratic	4	3216.76	804.19	151.52*
Interaction	6	2927.05	487.84	91.92*
Total model	14	7481.86	1626.54	102.16*
<i>Flavonoids</i>				
Linear	4	45,898	11,474	3.76*
Quadratic	4	68,849	17,212	5.63*
Interaction	6	98,882	16,480	5.4*
Total model	14	42,766	3054	4.93*
<i>Antioxidant activity</i>				
Linear	4	916.53	229.13	12.10*
Quadratic	4	1075.82	268.95	14.20*
Interaction	6	915.92	152.65	8.06*
Total model	14	2908.27	650.74	11.45*

^aDegrees of freedom

* Significant

cutins, suberins, and lignin. Food has a significant level of dietary fiber, has significant therapeutic implications for many diseases, such as diabetes, hyperlipidemia, and obesity and preventive effect against hypertension, prostate cancers, coronary heart disease, high cholesterol, colorectal, weight-loss and intestinal disorders (Slavin 2005; Tariq et al. 2000). Therefore, the amount of crude fiber was measured in the remaining DFP to detect the effectiveness of using the FCE as a fiber supplement.

Data in Table 3 shows the effect of interacted extraction conditions on the yield percentage of FCE and its content of crude fiber. The yield of FCE and crude fiber content differs among a range between 25.40–74.13 and 14.62–20.16 g/100 g respectively. The interacted extraction conditions caused maximum yield of FCE (74.13%) were 25% E/W, 50:1 at 45 °C for 2 h provided at the same time low crude fiber content (16.57%), which represent 22.35% only of the FCE. Whereas, the minimum yield of FCE (25.40%) obtained using 25% E/W, 50:1 at 55 °C for 3 h gave the maximum crude fiber content (20.16%) that represents 79.37% of the FCE. Despite these changes, Table 2 shows no significant effects of independent variables on crude fiber content in FCE extracts. Similar results were reported by Al-Farsi and Lee (2008); that no significant differences were observed between dietary fiber yields produced by four different purifying solvents (water butanol, water butanone, acetone butanol and acetone butanone) from date seeds.

Figure 2a shows the response of surface of the crude fiber as a function of the interaction between independent variables. As it can be seen, there is a linear reduction in crude fiber content in FCE with increasing the solvent concentration, temperature and time. In addition to a slight increment in crude fiber with increasing the solvent: sample ratio. The trend shows that the maximum achieved crude fiber (20.16%) was achieved using 25% ethanol concentration, 55 °C, 3 h and 50:1 solvent: sample ratio. This maximum percentage of the presence crude fiber in FCE is still very low, therefore, all tested extraction conditions are not suitable and need to be changed to prepare a fraction from date seeds rich in crude fiber.

Color of FCE

The aim of measuring the color of FCE is to ensure that it has no significant effects on the appearance and color of food products when FCE incorporates in product formulation as a source of fiber. Table 4 shows the analysis of variance of color values (L^* , a^* , and b^*) at $P < 0.05$ for the control sample (dates seed powder) compared with all extracted FCE.

Results show that all interacted extraction conditions increased the lightness (L -value) of FCE significantly compared to the control sample. Although, the increment did not reach to 50% of the lightening scale (the maximum value was 47.92% using 50% E/W). In addition, there was a significant increment in yellowness (b^*) between control sample and all extracted FCE, except those extracted using 50% E/W, 40:1 ratio for 2 h at 65 °C or 50% E/W, 60:1 ratio for 3 h at 55 °C (random run no. 10 and 8 respectively). In contrast, there were no significant effects of most of the extraction conditions on the intensity of redness

values (a^*) of all FCE extracts compared to the control sample.

Increasing the intensity of yellowness and reducing the redness of the control sample is might be due to losing some of red pigments in the liquid PC extracts.

Effect of extraction conditions on phenolic compounds

Results in Table 3 shows the effect of all independent variables on the extraction of phenolic compounds from date seed powder at $P < 0.05$. Data shows that phenolic compounds in PC varied from 7.63 to 71.72 mg GAE/100 g among various extraction conditions.

Figure 2b and Table 3 depict that the majority of phenolic compounds (above 40 mg GAE/100 g) were obtained using 50% ethanol concentration in interaction with other independent variables. These results are corresponding with Al Farsi and Lee (2008) findings, that the extraction capacity and selectivity of 50% acetone significantly showed the highest extraction capacity for gallic, p-hydroxybenzoic, caffeic, and p-coumaric and ferulic, compared to water. With increasing the concentration to 75% E/W, the phenolic compounds were going down, ranging between 11.91 and 28.46 mg GAE/100 g, this is due to a reduction in both solvent polarity, and phenolic compounds solubility. These results are matching with findings of Kchaou et al. (2013) and Wijekoon et al. (2011) that the recovery of phenolic compounds is influenced by the polarity of the solvent used in extraction.

Additionally, time shows linear increasing effects on phenolic compounds with increasing the time from 1 to 3 h. Contradictory, temperature and solvent: sample ratio illustrated gradual reduction of phenolic compounds. This finding is agreed with Larrauri et al. (1997) who mentioned that using extraction temperature above 60 °C significantly reduced total phenolic content compared with extractions at 40 and 50 °C.

Effect of extraction conditions on flavonoids content

The relationship between interacted extraction conditions and total flavonoids content in PC extracts are given in Table 3. Data shows that flavonoid content ranged between 36.30 and 455.77 mg CEQ/100 g. The maximum amount of flavonoids, 455.77 mg CEQ/100 g, was detected using 50% ethanol, 50:1 ratio at 65 °C for 3 h (random order no. 21) and with reducing temperature to 45 °C, under same conditions, the amount of flavonoids content decreased to half (216 mg CEQ/g, random order no. 22). This agreed with Shi et al. (2003) who mentioned that heating weakens the phenol–protein and phenol–polysaccharide interaction in plant tissue that increased the

Table 3 Experimental data and the observed response values (Mean $\pm$ SD) of different combinations of the four independent variables (X_1 , X_2 , X_3 and X_4) for the FCE yield %, crude fiber, phenolic compounds, flavonoids and antioxidants in PC using hydroalcohol solutions from date seed powder

Run order ^a	Standard order ^b	Coded and actual level of variables				FCE yield %	Crude fiber ^c	Phenolic compounds (mgGAE/100 g)	Flavonoids (mg CEQ/100 g)	Antioxidants (μ mol Fe(II)/g)
		Cocn. X_1	Ratio X_2	Temp X_3	Time X_4					
4	1	50(0)	50:1(0)	55(0)	2(0)	46.76 $\pm$ 0.22 ^c	18.88 $\pm$ 0.16 ^{cde}	17.50 $\pm$ 0.13 ^{ab}	192.54 $\pm$ 0.56 ^d	10.66 $\pm$ 0.42 ^b
14	2	75(1)	50:1(0)	45(-1)	2(0)	29.44 $\pm$ 0.18 ^a	18.47 $\pm$ 0.16 ^{cde}	28.46 $\pm$ 0.31 ^c	155.21 $\pm$ 0.76 ^c	28.03 $\pm$ 1.00 ^e
27	3	50(0)	50:1(0)	55(0)	2(0)	45.49 $\pm$ 0.19 ^c	18.13 $\pm$ 0.09 ^{cde}	16.59 $\pm$ 0.27 ^a	188.62 $\pm$ 2.06 ^d	11.00 $\pm$ 0.18 ^b
6	4	75(1)	60:1(1)	55(0)	2(0)	71.09 $\pm$ 1.05 ^f	17.72 $\pm$ 0.13 ^{abcd}	11.91 $\pm$ 0.61 ^a	86.22 $\pm$ 1.83 ^b	13.59 $\pm$ 0.24 ^{bc}
11	5	50(0)	50:1(0)	65(1)	1(-1)	48.21 $\pm$ 0.75 ^c	16.25 $\pm$ 0.09 ^{bc}	24.84 $\pm$ 0.23 ^b	134.40 $\pm$ 0.86 ^c	19.47 $\pm$ 0.08 ^c
23	6	50(0)	50:1(0)	55(0)	2(0)	45.49 $\pm$ 0.38 ^c	18.02 $\pm$ 0.03 ^{cde}	18.63 $\pm$ 0.43 ^b	83.70 $\pm$ 0.61 ^b	11.19 $\pm$ 0.08 ^b
17	7	25(-1)	50:1(0)	55(0)	3(1)	25.40 $\pm$ 0.07 ^a	20.16 $\pm$ 0.02 ^{efg}	43.81 $\pm$ 0.64 ^d	303.04 $\pm$ 0.05 ^g	37.81 $\pm$ 0.16 ^g
9	8	75(1)	50:1(0)	65(1)	2(0)	37.94 $\pm$ 0.69 ^b	14.94 $\pm$ 0.35 ^a	23.78 $\pm$ 0.30 ^b	135.44 $\pm$ 0.54 ^c	21.99 $\pm$ 0.29 ^d
3	9	50(0)	60:1(1)	45(-1)	2(0)	35.36 $\pm$ 0.29 ^b	17.72 $\pm$ 0.18 ^{abcd}	23.46 $\pm$ 0.16 ^b	188.38 $\pm$ 0.96 ^d	21.60 $\pm$ 0.17 ^d
19	10	25(-1)	50:1(0)	45(-1)	2(0)	74.13 $\pm$ 0.39 ^f	16.57 $\pm$ 0.06 ^{bc}	10.70 $\pm$ 0.17 ^a	51.85 $\pm$ 0.34 ^a	10.57 $\pm$ 0.16 ^b
25	11	25(-1)	50:1(0)	65(1)	2(0)	54.95 $\pm$ 0.04 ^d	19.28 $\pm$ 0.09 ^{def}	19.85 $\pm$ 0.30 ^b	36.67 $\pm$ 0.28 ^a	11.19 $\pm$ 0.13 ^b
2	12	50(0)	60:1(1)	55(0)	1(-1)	68.72 $\pm$ 0.24 ^e	18.41 $\pm$ 0.11 ^{cde}	7.63 $\pm$ 0.45 ^a	77.56 $\pm$ 0.39 ^b	11.1 $\pm$ 0.10 ^b
15	13	25(-1)	60:1(1)	55(0)	2(0)	46.41 $\pm$ 0.11 ^c	17.89 $\pm$ 0.07 ^{bcd}	34.82 $\pm$ 0.37 ^c	201.29 $\pm$ 0.67 ^e	34.09 $\pm$ 0.99 ^g
24	14	50(0)	40:1(-1)	55(0)	3(1)	52.30 $\pm$ 0.11 ^d	15.46 $\pm$ 0.37 ^{ab}	18.40 $\pm$ 0.51 ^b	73.75 $\pm$ 0.41 ^a	7.23 $\pm$ 0.07 ^a
29	15	50(0)	50:1(0)	55(0)	2(0)	45.11 $\pm$ 0.17 ^c	17.99 $\pm$ 0.72 ^{abcd}	19.01 $\pm$ 0.23 ^b	85.99 $\pm$ 0.52 ^b	11.16 $\pm$ 0.19 ^b
13	16	50(0)	50:1(0)	45(-1)	1(-1)	35.51 $\pm$ 0.27 ^b	16.74 $\pm$ 0.33 ^{bc}	67.82 $\pm$ 0.59 ^g	323.71 $\pm$ 0.26 ^h	30.95 $\pm$ 0.07 ^f
18	17	75(1)	50:1(0)	55(0)	3(1)	26.56 $\pm$ 0.28 ^a	14.87 $\pm$ 0.18 ^a	21.86 $\pm$ 0.25 ^b	122.14 $\pm$ 1.06 ^c	31.75 $\pm$ 0.37 ^f
22	18	50(0)	50:1(0)	45(-1)	3(1)	52.50 $\pm$ 0.46 ^d	14.62 $\pm$ 0.19 ^a	44.89 $\pm$ 0.22 ^d	216.90 $\pm$ 0.36 ^e	24.69 $\pm$ 0.14 ^e
7	19	75(1)	50:1(0)	55(0)	1(-1)	51.30 $\pm$ 0.30 ^d	15.55 $\pm$ 0.29 ^{ab}	17.86 $\pm$ 0.59 ^{ab}	77.77 $\pm$ 0.62 ^b	15.29 $\pm$ 0.23 ^{bc}
28	20	50(0)	50:1(0)	55(0)	2(0)	45.00 $\pm$ 0.11 ^c	17.77 $\pm$ 0.40 ^{abcd}	17.29 $\pm$ 0.35 ^{ab}	187.24 $\pm$ 0.12 ^d	10.60 $\pm$ 0.24 ^b
5	21	75(1)	40:1(-1)	55(0)	2(0)	63.20 $\pm$ 0.14 ^e	18.26 $\pm$ 0.25 ^{cde}	13.22 $\pm$ 0.30 ^a	204.77 $\pm$ 0.31 ^e	10.30 $\pm$ 0.05 ^b
1	22	25(-1)	40:1(-1)	55(0)	2(0)	46.40 $\pm$ 0.36 ^c	17.89 $\pm$ 0.06 ^{bcd}	14.88 $\pm$ 0.33 ^a	141.57 $\pm$ 0.59 ^c	15.89 $\pm$ 0.22 ^{bc}
21	23	50(0)	50:1(0)	65(1)	3(1)	47.28 $\pm$ 0.22 ^c	17.12 $\pm$ 0.16 ^{bcd}	52.00 $\pm$ 0.32 ^e	455.77 $\pm$ 0.71 ^k	25.61 $\pm$ 0.10 ^e
20	24	50(0)	40:1(-1)	55(0)	1(-1)	46.40 $\pm$ 0.42 ^c	17.71 $\pm$ 0.20 ^{abcd}	71.72 $\pm$ 0.37 ^g	413.00 $\pm$ 0.45 ^j	35.63 $\pm$ 0.15 ^g
16	25	50(0)	40:1(-1)	45(-1)	2(0)	49.45 $\pm$ 0.40 ^c	16.22 $\pm$ 0.05 ^{bc}	68.73 $\pm$ 0.28 ^f	331.13 $\pm$ 0.55 ^h	55.02 $\pm$ 0.03 ^h
12	26	50(0)	60:1(1)	65(1)	2(0)	41.94 $\pm$ 0.05 ^c	16.75 $\pm$ 0.34 ^{bc}	15.72 $\pm$ 0.44 ^a	88.71 $\pm$ 0.35 ^b	10.64 $\pm$ 0.20 ^b
26	27	25(-1)	50:1(0)	55(0)	1(-1)	41.90 $\pm$ 0.10 ^c	15.69 $\pm$ 0.13 ^{ab}	11.89 $\pm$ 0.56 ^a	36.30 $\pm$ 0.15 ^a	7.93 $\pm$ 0.35 ^a
10	28	50(0)	40:1(-1)	65(1)	2(0)	44.91 $\pm$ 0.27 ^c	15.45 $\pm$ 0.27 ^{ab}	71.60 $\pm$ 0.44 ^g	244.56 $\pm$ 0.06 ^f	28.87 $\pm$ 0.53 ^e
8	29	50(0)	60:1(1)	55(0)	3(1)	44.17 $\pm$ 0.38 ^e	18.97 $\pm$ 0.41 ^{cde}	47.42 $\pm$ 0.24 ^d	181.05 $\pm$ 0.53 ^d	27.61 $\pm$ 0.60 ^e

Values are expressed as mean $\pm$ SD (n = 3)(X_1) Solvent concentration, (X_2) Solvent/sample ratio, (X_3) Temperature and (X_4) Extraction time^aNo randomized order^bRandomized order^c% crude fiber in DFP Values are expressed as mean $\pm$ SD (n = 3)

Fig. 2 Response surface plot of (a) phenolic compounds, (b) flavonoids and (c) antioxidant activity among different independent variables (solvent concentration, solvent: sample ratio, temperature and extraction time)

Fig. 2 continued

Table 4 Influence of extraction conditions on L^* , a^* and b^* values of FCE

Sample random no.	Solvent Concn%	L^*	a^*	b^*
Control ^a	–	39.61 ± 0.3422 ^a	15.14 ± 0.2196 ^c	16.35 ± 0.6137 ^a
17	25%	42.82 ± 0.4474 ^{bc}	12.37 ± 0.5430 ^{abcd}	18.31 ± 0.4539 ^{bc}
19		42.56 ± 0.0461 ^{bc}	14.14 ± 0.0888 ^{de}	19.62 ± 0.1446 ^{cdef}
25		41.98 ± 0.3550 ^b	12.38 ± 0.4471 ^{abcd}	18.47 ± 0.4277 ^{bc}
15		43.31 ± 0.3704 ^{cd}	13.31 ± 0.5772 ^{bcde}	20.06 ± 0.6698 ^{defg}
1		43.38 ± 0.1501 ^{cd}	13.40 ± 0.5320 ^{bcde}	19.63 ± 0.4253 ^{cdef}
26		45.13 ± 0.3775 ^{efgh}	13.38 ± 0.5033 ^{bcde}	20.62 ± 0.4517 ^{defg}
12	50%	46.42 ± 0.3487 ^{ijk}	11.61 ± 0.6863 ^{ab}	19.61 ± 0.4821 ^{cdef}
24		43.38 ± 0.4178 ^{ed}	12.17 ± 0.7446 ^{abcd}	19.33 ± 0.5529 ^{cde}
23		47.05 ± 0.4483 ^{jk}	12.88 ± 0.7729 ^{abcd}	21.03 ± 0.5774 ^{fgh}
16		45.15 ± 0.1625 ^{efgh}	13.59 ± 0.6814 ^{cde}	20.59 ± 0.0907 ^{defg}
4		46.23 ± 0.1569 ^{hijk}	12.59 ± 0.2550 ^{abcd}	20.26 ± 0.0984 ^{defg}
11		44.57 ± 0.4427 ^{ef}	12.82 ± 0.7507 ^{abcd}	20.59 ± 0.5047 ^{defg}
21		44.67 ± 0.5802 ^{ef}	10.99 ± 0.8792 ^a	19.22 ± 0.5802 ^{cd}
20		43.29 ± 0.3704 ^{cd}	13.33 ± 0.5772 ^{bcde}	20.09 ± 0.6698 ^{defg}
22		47.92 ± 0.2081 ^k	13.20 ± 0.2959 ^{bcde}	21.18 ± 0.0700 ^{gh}
3		43.34 ± 0.3911 ^{cd}	13.29 ± 0.5874 ^{bcde}	20.06 ± 0.6698 ^{defg}
2		45.67 ± 0.4712 ^{fghij}	12.78 ± 0.8470 ^{abcd}	21.15 ± 0.5862 ^{gh}
13		46.46 ± 0.4944 ^{ijk}	12.03 ± 0.8672 ^{abc}	20.81 ± 0.5557 ^{efgh}
10		45.93 ± 0.0808 ^{ghijk}	12.41 ± 0.0400 ^{abcd}	16.60 ± 0.1266 ^a
8		45.62 ± 0.3350 ^{fghij}	12.44 ± 0.5000 ^{abcd}	17.28 ± 0.5934 ^{ab}
9	75%	44.78 ± 0.3412 ^{efg}	12.84 ± 0.5167 ^{abcd}	19.81 ± 0.4776 ^{cdefg}
6		43.30 ± 0.3694 ^{cd}	13.35 ± 0.5692 ^{bcde}	20.26 ± 0.6698 ^{defg}
18		45.16 ± 0.3003 ^{efgh}	13.06 ± 0.5755 ^{bcd}	20.32 ± 0.5236 ^{defg}
7		46.67 ± 0.1385 ^{jk}	13.45 ± 0.0832 ^{bcde}	20.47 ± 0.1126 ^{defg}
5		45.98 ± 0.4823 ^{hijk}	13.20 ± 0.8951 ^{bcde}	20.52 ± 0.6439 ^{defg}
14		44.05 ± 0.1436 ^{de}	13.66 ± 0.1553 ^{cde}	20.53 ± 0.1014 ^{defg}

Values are expressed as mean ± SD (n = 3)

Values within the same row, followed by the same letter, are not statistically different ($P < 0.05$)^aDate seed powder (used as a control sample)

migration of polyphenol compounds (as flavonoids) into the solvent. Moreover, data shows that all the detected quantities above 323.71 mg CEQ/100 g were achieved using 50% E/W concentration at 40:1 or 50:1 solvent: sample ratio.

According to Table 1, extraction time had the highest positive linear regression and significant effects on the extraction of flavonoids from date seed powder. These results were supported with data finding in Table 3, using 25% solvent concentration and 55 °C temperature, the flavonoids contents jumped from 36.3 to 141.57 and 201.29 mg CEQ/g when extraction time increased from 1 to 2 h respectively.

Figure 2b and c illustrated that the effects of interacted independent variables on phenolic compounds and flavonoids show similar behavior. This is due to the heterogeneous structures of phenolic compounds that contain a large number of components ranging from simple

molecules to highly polymerized molecules and considering flavonoids as a type of phenolic compounds.

Effect of extraction conditions on antioxidant activity

Data in Table 3 shows that antioxidant activity of PC extracts ranged between (7.23–55.02) μmol FE (II)/g. The maximum antioxidant activity (55.02 μmol FE (II)/g) was achieved using 50% concentration with heating at 45 °C for 2 h at 40:1 ratio (random run no. 16). Using the same extraction conditions with increasing temperature to 65 °C (random run no. 10), the amount of antioxidant activity was reduced to half (28.87 μmol FE (II)/g). While increasing both temperature and time to 55 °C and 3 h respectively (random run no. 24), the amount of antioxidant activity was reduced to eighth (7.23 μmol FE (II)/g).

Data shows that the antioxidant activity of all extracts was lower than its corresponding phenolic compounds content, except for random run numbers 6, 2, 18 and 1. This needs further structure study to know more about types of compound with their antioxidant activities extracted under these conditions.

The response surface plot in Fig. 2d and Table 1 shows that there is a linear increase and it has a significant effect on antioxidant activity with increasing the concentration of ethanol, solvent: sample ratio and time. As the upper design points of these variables produced a greater response on antioxidant activity compared to lower design points. This may be due to the effect of these variables on types of compounds extracted and types of free fractions produced.

Conclusion

In conclusion, response surface methodology can be used to maximize the extraction conditions of phytochemicals from date seed powder to produce functional foods and to improve the value of the dates' industry. Whereas, ethanol concentration was found to be the most effective independent variable in extracting phenolic compounds from date seed powder. While both ethanol concentration and time were found to be the most effective independent variables in extracting flavonoids. In addition, all independent variables except temperature had significant effects on antioxidant activity. In contrast, the content of fiber extracted is not modified by all extraction conditions used in this study.

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