Journal of Applied Science and Agriculture, 8(6) November 2013, Pages: 937-945

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Evaluation of Response Surface Methodology Efficiency for Optimization of Subcritical Water Extraction of phenolic compounds from Barberry Fruits

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ARTICLE INFO

Article history:
Received 19 October 2013
Received in revised form 16 November 2013
Accepted 19 November 2013
Available online 11 January 2014

Key words:
Antioxidant activities Barberry Extraction yieldTotal phenolic contentsVitamin C

ABSTRACT

Seedless barberry fruit (*Berberis vulgaris*) is a good source of anthocyanins, phenolic compounds and vitamin C. It is important that the methods be used for the extraction of valuable compounds can be achieved, most functional materials with minimal impurities and damage. Since two decades, subcritical water extraction (SWE) is well known as green technology for separation and extraction of plant materials. SWE can be applied for extraction of bioactive compounds. In this study, the SWE of total phenolic contents of seedless barberry fruit was optimized using response surface methodology. Response surface methodology is a series of statistical techniques that can be used to design experiment, modeling and evaluation the effect of variables on the final obtained results and optimization of process conditions. In this study a quadratic model was applied to predict the behavior of samples. Central composite design was employed using 13 treatment and 5 repeat in central points. The independent variables factors were temperature (110-170ºC) and time (10-50 minutes) for evaluation of total phenolic contents as well as antioxidant activities, vitamin C and extraction yield of extracted barberry. Effects of extraction temperature and time were found to be significant on all responses. Optimal SWE conditions were identified as 157.50 °C temperature and 29.64 minutes for total phenolic contents (568.75 mgGAE/100 mL), antioxidant activities (91.15%), vitamin C (294.274 mg/L) and extraction yield (92%). Experimental values for response variables at these optimal conditions match well with the predicted values. Barberry extracts obtained by SWE showed more than 91% DPPH radical scavenging activities. High correlation was seen between total phenolic contents and antioxidant activities in the SWE method. Results of this research showed response surface methodology is a useful tools for optimization of variables factors of barberry bioactive compounds extraction using SWE in order to use of this optimize condition in industry.

To Cite This Article: Akram Sharifi, Seyed Ali Mortazavi, Abdolmajid Maskooki, Mehrdad Niakousari, Amir Hossein Elhamirad., Evaluation of Response Surface Methodology Efficiency for Optimization of Subcritical Water Extraction of phenolic compounds from Barberry Fruits. ***J. Appl. Sci. & Agric.,*** *8(6): 937-945, 2013*

INTRODUCTION

Barberry shrubs are grown in many countries essentially for medical and ornamental purposes. In Iran, however, the seedless variety of this shrubs are grown as commercial plant to obtain dried fruit and consumed as a food additive. It has been well known that the seedless barberry fruit is a good source of anthocyanins, phenolic compounds, citric acid and vitamin C. Therefore, it is important that the methods be used for the extraction of valuable compounds can be achieved, most functional materials with minimal impurities and damage (Herreroa *et al*., 2010).

Subcritical water extraction (SWE) , is an extraction technique using water as the solvent, but with modified physical properties; it is considered a recent alternative for the isolation of antioxidant constituents from plant materials. With SWE systems, temperatures between 100 and 374 ºC (the critical point of water is at 374 ºC and 22 MPa) are generally employed and the pressure must be high enough to keep the water in the liquid state (Ramos *et al*., 2002). Under subcritical conditions, the intermolecular hydrogen bonds of water break down and the dielectric constant of water decreases (Singh *et al*., 2011).

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Today, the attitude towards green processes has increased, SWE method is fast, inexpensive, recycling capabilities and environmentally friendly. Compared with toxic organic solvents that used in conventional extraction methods, water is used in this technique.

Some studies have focused on the environmental aspects of the SWE method (Siriwong *et al*., 2009; Okuda *et al*.,2009). Other studies carried out on the ability to selectively extract different classes of compounds by SWE. (Zaibunnisa *et al*.2009).

SWE may therefore be a good candidate for the extraction of antioxidant compounds from plant materials (Hassas-Roudsari *et al*.,2009). SWE has been used to extract the most active antioxidant compounds from rosemary (Ibanez *et al*., 2003); phenolic compounds from flax shive (Kim & Mazza, 2006) and anthocyanins from red grape skin (Ju and Howard, 2005) ; Many investigations were carried out and reported that this novel technique has also been used to extract flavonoids, phenolic acids and anthocyanins from red grape skins (Luque-Rodríguez *et al*., 2007); nutraceuticals such as glycyrrhetic acid, glycyrrhizin and liquiritin from licorice roots (Baek *et al*., 2008); catechins and epicatechin from tea leaves and grape seeds (Pineiro *et al*.,2004); and flavones, anilines and phenols from orange peel (Lamm and Yang, 2003) and Caffeine from tea waste (Shalmashi *et al*., 2010).

In this study, a subcritical water extraction (SWE) was employed for the extraction of anthocyanins and total phenolic contents from barberry fruit (*berberris vulgaris*) and SWE process was optimized by using response surface methodology.

Methodology:

Sample preparation:

Seedless barberries of *Berberis vulgaris* variety were provided for the experiment from Birjand barberry gardens in southern Khorasan province in Iran. The fruits were separated from the stems and stored. All reagents and chemicals used in the study were manufactured by Merk in Germany.

Subcritical water extraction:

Subcritical water extraction was carried out using a SWE device were designed, installed and operated at the laboratory of Research Institute of Food Science and Technology (RIFST), Iran. This system consists of a distilled water tank, a pump (Comet type: MTP AX 2/70 m) establishing a pressure of up to 170±5 bar, an extraction cell having capacity of 140 ml (thick wall to withstand the pressure), an heating coil, a pressure gauge and a temperature control system. Barberries were first milled using a Black & Decker grinder (Model no. JBG60, USA). The powdered sample was then loaded into the cell. Extraction was carried out at temperature range 110-170ºC for 10-50 minutes. The impurities of liquid (extract) was separated using filter paper (Whatman paper No. 4) under vacuum condition. The filtered extracts were cooled and stored at 4±1ºC in a dark Polypropylene bags until used in the analysis.

Determination of antioxidant activity:

The radical scavenging activity of the extract for DPPH (2,2-diphenyl-1-picrylhydrazyl) was monitored according to the method explained by Galvez *et al* (2007). The samples were prepared by re-diluted at a ratio of 10:40 (extract : ethanol 80%) was determined. Aliquots of each extracts (0.5 ml) were added to 3.5 ml of ethanolic solutions of DPPH (0.004%). Discoloration of obtained solutions were measured at 517 nm after incubation for 30 min at room temperature in the dark using spectrophotometric method. The Inhibition ability of free radical DPPH in one percent was evaluated based on equation 1:

$$\text{DPPH \%} = (A_{blank} - A_{sample} / A_{blank}) \times 100 \quad (1)$$

where A_{blank} was defined as absorbance of the control whereas A_{sample} was defined as absorbance of the sample (the extracts) (Galvez *et al*.,2007).

Determination of total phenolic content:

Total phenolic contents were determined using the Folin–Ciocalteau method. 0.5 ml of extracts were mixed with 2.5 ml of Folin–Ciocalteau reagent (pre-diluted at a ratio of 1:10) and allowed to stand at room temperature for 3 min. 2 ml of sodium bicarbonate (75 g/L) was added to the mixture. After standing for 30 min at room temperature, absorbance values were measured at 765 nm using Uv-Vis spectrophotometer. Results were expressed as mg gallic acid equivalents (GAE)/g sample (Shahidi and Naczk, 2004).

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The measurement of vitamin C:

For vitamin C measurements, 50 g of frozen fruits were stabilized in *meta*-phosphoric acid (5%), homogenized, filtered and the extract injected into an HPLC-system (KNAUER-Germany). Identification of ascorbic acid in the samples was made by comparison of the spectra and retention times with those obtained for the standard solutions. (Melendez *et al*.,2004).

determination of extraction yield:

Extraction yield was determined by calculating the amount of solids of extracted according to AOAC standard method (AOAC. 2000).

Statistical analysis and optimization using RSM:

Response surface methodology (RSM) with central composite design was applied to identify optimum levels of two variables of the temperature (110-170ºC) and time (10-50 minutes) regarding of four responses; total phenolic contents, antioxidant activity, vitamin C and extraction yield in the barberry fruit extracts. The coded and uncoded independent variables used in the RSM design are listed in Table 1. Analysis of the experimental design data and calculation of predicted responses were carried out using design expert software (Design expert 7.1.5). A second-order polynomial equation was used to express the responses as a function of the independent variables as indicated in equation 2:

$$Y = b0 + b_1 A + b_2 B + b_{11} A^2 + b_{22} B^2 + b_{12} AB \quad (2)$$

Where Y represents the measured response variables includes vitamin C (mg/L), total phenolic contents (mg GAE/100 mL), antioxidant activities (%) and extraction yield (%) and b0 is regression coefficients. These coefficients represent the linear, quadratic and interaction effects of the variables.

Table 1: coded levels of independent variables used in the RSM design.

Independent variables	coded levels		
	+1	0	-1
Temperature (A ºC)	110	140	170
Time (B minutes)	10	30	50

RESULTS AND DISCUSSION

Modeling of the extraction process:

The statistical analysis indicated that the proposed model (Equation 2) was adequate according to the significance of the F-test ($p \leq 0.05$), not significant of lack of fit, the values of R^2 and adjusted R^2 and the coefficient of variation. With respect to the analysis of variance tables, it can be found that all the parameters of the fitted model are quite significant ($p \leq 0.05$). In order to investigate the influence of parameters on the analysis tables of variance, statements with no significant F- test were removed from the model and other statements with significant differences were retained in the model (Noshad *et al*.,2011).

Total phenolic contents:

Figure 1.a shows the effects of temperature and extraction time on the total phenolic contents in barberry extract. The results indicated that the increasing temperature from 110-170^{o}C enhanced the total phenolic contents by almost two fold in the samples; however extraction time has no significant effect on total phenolic contents .This is evident from the numerical coefficient for temperature and time in the model, too (Figure 1.b). Analysis of variance showed that only the quadratic statements of temperature (A^2) and linear statements (A) had significant influence on the amount of total phenolic contents (Table. 2). Nevertheless, statements related to the effect of linear (B) and time quadratic statements (B^2) as well as the statements of temperature and time interaction (AB) had no significant influence on the amount of extraction. Thus, these coefficients were excluded from the model. The adjusted equation of this response is given in Equation 3:

$$Y = + 436.70 + 126.34 A + 93.57 A^2 \quad (3)$$

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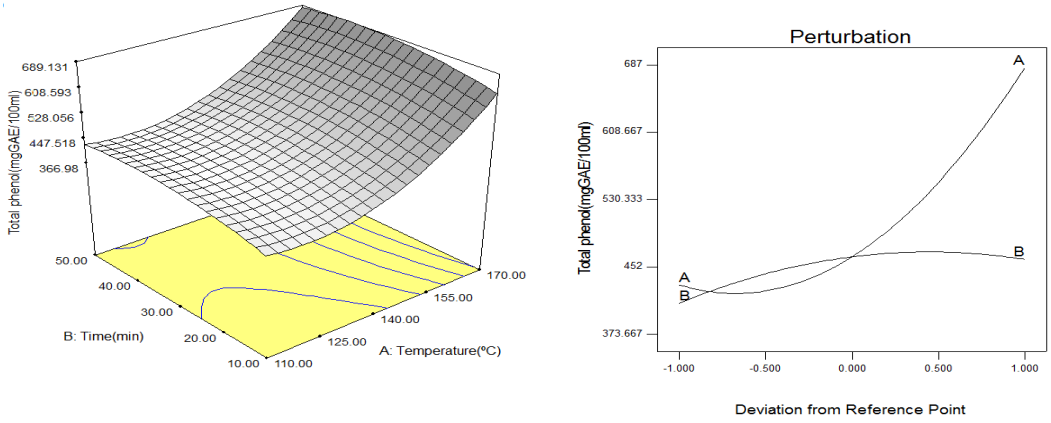
The coefficient of determination (R^2) of the predicted models in this response was 0.9526 and p-value for lack of fit was 0.4717. These values would give a relative good fit to the mathematic model in equation (table. 2).



b **a**

Fig. 1: (a) Response surface contour plot showing total phenolic contents in barberry extract with respect to different temperature levels (°C) and treatment time (min)

(b) Plot showing the significant of two independent parameters of temperature and time on efficiency of extraction of total phenolic contents in barberry extract

Table 2: ANOVA for the quadratic model of total phenolic contents (mg GAE/100 mL)

	Sum of square	DF	Mean square	F-value	Prob > F
Model	1.240×10^5	5	24801.70	28.12	0.0002*
A	95769.93	1	95769.93	108.60	0.0001
B	3888.76	1	3888.76	4.41	0.0739
A^2	24183.78	1	24183.78	27.42	0.0012
B^2	2241.07	1	2241.07	2.54	0.1549
AB	0.2900	1	0.2900	3.327×10^4	0.9860
Residual	6173.22	7	881.89	-	-
Lack of Fit	2677.59	3	892.53	1.02	0.4717ns
R-Squared	0.9526	-	-	-	-
Adj R-Squared	0.9187	-	-	-	-

The positive effect of increased temperature on the extraction of phenolic compounds using subcritical water have been reported by many researchers (Shi *et al*.,2003; Rodriguez-Meizoso *et al*., 2006; Singh and Saldana, 2011).Water under subcritical conditions acts as relative polar and even non polar solvent can dissolve polar compounds more than non-polar compounds. Higher temperatures lead to decrease in water polarity, hence enhances the water capability to dissolve nonpolar compounds such as polyphenols. Furthermore, increasing the temperature from 25 to 200°C, decreases the water dielectric constant from 75 to 35 which is close to dielectric constant of methanol and Venice Ethanol (Singh and Saldana, 2011; Cacace and Mazza,2006). Ibanez *et al*. (2003) reported that at 25°C rosmanol content as the main compound of extracted juice from rosemary (over 50%), but increasing the temperature to 200°C leads to an increase in compounds with low polarity such as carnosic acid and carnosol. It is also known that increasing temperature reduces the adhesion and viscosity of water, which in turn could lead to increased diffusion rate and mass transfer during extraction process (Ramos *et al*., 2002). Another reason for higher extraction efficiency of total phenolic contents at higher temperature could be due to the hydrolytic degradation of the polysaccharide network and lignin of cell wall at high temperature (Rangsriwong *et al*., 2009).

Antioxidant activities:

The data in table of analysis of variance (Table .3) indicate that the linear statements of temperature (A) and (B) the extraction time have the most positive effects and quadratic statements time (B^2) have a negative impact on the amount of the extracted antioxidant. However, the interactive terms of temperature-time (AB) had no significant influence on the fitted model. The antioxidant activity of the extracts increased as temperature increased from 110 to 170 (Figure 2.a). However, antioxidant activity in the extract increased when extraction

time increasing from 10 to about 40 minutes, and then remains unchanged (Figure 2.a). The best equation of the fitted response is given in Equation 4:

$$Y = + 87.47 + 4.81\ A + 2.94\ B + 2.73A^2 - 3.06\ B^2 \quad (4)$$

The coefficient of determination (R^2) of the predicted models in this response is 0.9592 and the p-value for lack of fit is 0.4216. These values would give a fairly good fit to the model (Table. 3).

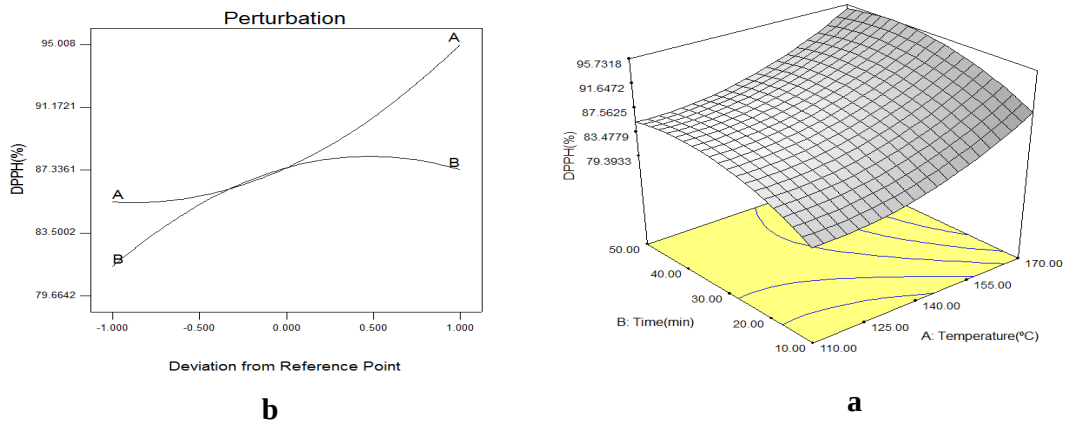


Fig. 2 (a) Response surface contour plot showing variation in antioxident activity in barberry extract with respect to different temperature levels (°C) and extraction duration (min)

(b) Plot showing the significant of two independent parameters of temperature and time on antioxident activity of the extract

Table 3: ANOVA for the quadratic model of antioxidant activity (DPPH%)

	Sum of square	DF	Mean square	F-value	Prob > F
Model	244.22	5	44.84	32.94	0.0001*
A	138.63	1	138.63	101.82	0.0001
B	51.78	1	51.78	38.03	0.0005
A^2	20.60	1	20.60	19.01	0.0060
B^2	25.89	1	25.89	15.13	0.0033
AB	6.59×10^{-3}	1	6.59×10^{-3}	4.84×10^{-3}	0.9465
Residual	9.53	7	1.36	-	-
Lack of Fit	4.48	3	1.49	1.18	0.4216ns
R-Squared	0.9592	-	-	-	-
Adj R-Squared	0.9301	-	-	-	-

Temperature had more positive effect on antioxidant activity than extraction time. The results obtained in this study were in a good agreement with findings that reported by Rodriguez-Meizoso *et al*. (2010) on extraction process on a variety of microalgae, Klorofita (Haematococcus pluviales microalga). Most of researchers attributed this observation to reduction of dielectric constant that can be increase the solubility of organic compounds such as polyphenols, reduction of the solubility of inorganic compounds and the extraction of specific components with high antioxidant power (Rodriguez-Meizoso *et al*., 2010). As it can be seen in Figure 3.b, antioxidant activity were reduced with an increase of time to more than 45 minutes. This phenomenon can be attributed to the destruction of antioxidant compounds in longer extraction period under SWE conditions (Gan and Latiff ,2011).

The high correlation between extraction yield of phenolic compounds and antioxidant activity (r=0.88) will confirm that (Figure. 3).

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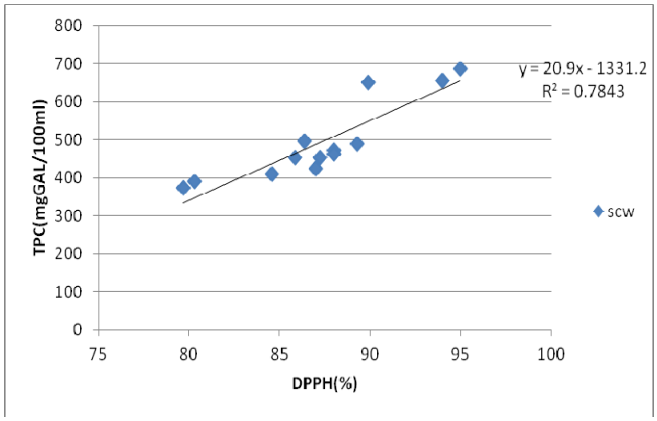

Fig. 3: Correlation between antioxidant activities and total phenolic content in barberry extract

Vitamin C:

Figure 4.a illustrates the effects of operating temperature and extraction time on vitamin C in barberry extract. The results indicate increasing the operating temperature and extraction time, significantly reduced the efficiency of vitamin C and temperature had the most negative effects on the amount of vitamin C (Figure 4.b).

The table of variance shows, linear terms of temperature (A) and time (B) and the quadratic statement of temperature (A^2) have the negative effect on the efficiency of vitamin C content in the extract (Table.4).

The lowest content of vitamin C were obtained at temperature of 170°C and 50 minutes extraction times. However, in the SWE process where the materials usually encountered extremely high temperature, destruction or change in vitamin C structure and hence their reduction were not unexpected (Rodriguez-Meizoso *ET AL*., 2006).

The equation of the fitted response is given in Equation 5:

$$Y = + 428.39 – 186.89A – 39.41\ B\ -90.55A^2 \quad (5)$$

Coefficient of determination (R^2) of predicted model (Equation 5) is 0.9906 and the p-value for the lack of fit is 0.0044. These R^2 and P indicate that the model can suitably fit the experimental data (Table. 4) .

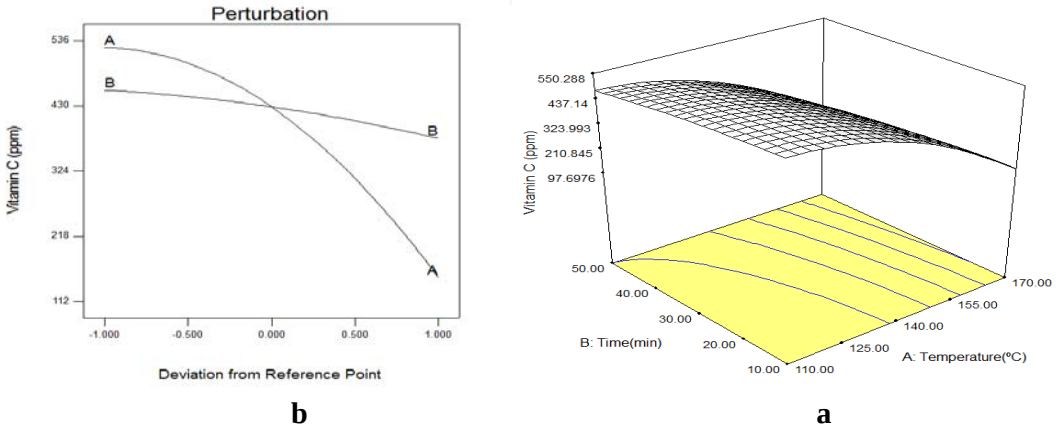

Fig. 4: (a) Response surface contour plot showing variation in vitamin C in barberry extract with respect to different temperature levels (°C) and extraction duration (min)

(b) Plot showing the significant of two independent parameters of temperature and time on vitamin C of the extract

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Table 4: ANOVA for the quadratic model of vitamin C (mg/L)

	Sum of square	DF	Mean square	F-value	Prob > F
Model	2.48×10^{-5}	5	49711.01	147.60	0.0001*
A	2.096×10^{-5}	1	2.096×10^{-5}	622.23	0.0001
B	9316.52	1	9316.52	27.66	0.0012
A^2	22644.31	1	22644.31	67.23	0.0001
B^2	404.52	1	404.52	1.20	0.3094
AB	12.25	1	12.25	0.036	0.8542
Residual	2357.42	7	336.80	-	-
Lack of Fit	2242.42	3	747.47	25.96	0.0044ns
R-Squared	0.9906	-	-	-	-
Adj R-Squared	0.9839	-	-	-	-

Extraction yield:

Figure 5.a shows increasing the operating temperature and extraction time, significantly increased the extraction yield and temperature had the most positive effects on the extraction yield (Figure 5.b). Temperature rise can increase the vapor pressure of extractable compounds, therefore tendency of these compounds to extraction increases (Reverchon and De Marco, 2006).

Analysis of variance showed that linear terms of temperature (A) and time (B) and the quadratic statements of temperature (A^2) and temperature and time interaction (AB) had significant influence on the extraction yield (Table. 5), quadratic statements of time (B^2), had no significant influence on the extraction yield, thus, this coefficient was excluded from the model.

The best equation of the fitted response is given in Equation 6 and the coefficient of determination (R^2) of the predicted models in this response is 0.932.

$$Y= + 0.90+0.024A +0.016B +0.012\ A^2+0.014AB \quad (6)$$

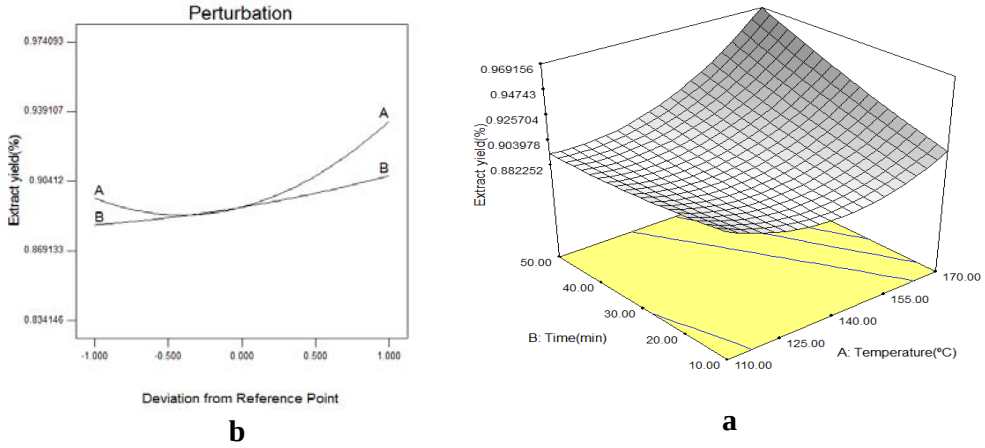

Fig. 5: (a) Response surface contour plot showing variation in extraction yield in barberry extract with respect to different temperature levels (°C) and extraction duration (min)

(b) Plot showing the significant of two independent parameters of temperature and time on extraction yield of the extract

Table. 5: ANOVA for the quadratic model of extraction yield (%)

	Sum of square	DF	Mean square	F-value	Prob > F
Model	6.19×10^{-3}	5	1.24×10^{-3}	19.25	0.0006*
A	2.55×10^{-3}	1	2.55×10^{-3}	55.30	0.0001
B	1.46×10^{-3}	1	1.46×10^{-3}	22.69	0.0021
A^2	3.72×10^{-4}	1	3.72×10^{-4}	5.78	0.0472
B^2	3.98×10^{-5}	1	3.98×10^{-5}	0.62	0.4574
AB	8.02×10^{-4}	1	8.02×10^{-4}	12.47	0.0096
Residual	4.50×10^{-4}	7	6.44×10^{-5}	-	-
Lack of Fit	4.50×10^{-4}	3	1.47×10^{-4}	58.57	0.0009ns
R-Squared	0.932	-	-	-	-
Adj R-Squared	0.883	-	-	-	-

Optimization of the extraction process:

The optimum SWE conditions to obtain high amount of phenolic and antioxidant activities, vitamin C and extraction yield of extracted barberry using RSM technique are presented in the Table. 6. In this study, the optimum condition in the extraction of the barberry would be achieved if the values of the total phenolic contents, vitamin C, antioxidant activities and extraction yield reached maximum. For this purpose, initially, were adjusted optimization purposes, response levels and independent variables. The best responses were obtained using desirability function method.

Table 6: The optimum SWE conditions

Temperature(ºC)	Time(min)	DPPH(%)	Total phenol(mgGAE/100ml)	Vitamin C(mg/L)	Extraction yield(%)	Desirability
157.50	29.64	91.15	568.75	294.274	0.920	0.544

Conclusion:

The aim of this study was to optimize the extraction process of phenolic compounds and anthocyanins in seedless barberry through response surface methodology. the quadratic polynomial model was used in order to predict the behavior of the sample. Optimal SWE conditions were identified as 157.50 °C temperature and 29.64 minutes time for maximum total phenolic contents (568.75 mgGAE/100 mL), vitamin C (294.274 mg/L) and extraction yield (92%). Barberry fruits extracts obtained by SWE showed more than 91% DPPH radical scavenging activities. Experimental values for response variables at these optimal conditions match well with the predicted values. The results showed that the response surface methodology is an appropriate method for optimizing the extracted variables of barberry bioactive material in order to utilize these optimum conditions in industry may be obtained in the SWE process of barberry, respectively.

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