Optimization of ultrasound-assisted osmotic treatment of Aleo vera gel impregnated with grape pomace phenolic compounds using response surface methodology

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Abstract: This study was focused on investigating the use of pulsed ultrasound-assisted osmotic dehydration (UAOD) for enhancing the infusion with phenolic compounds extracted from grape pomace (GPx) into Aloe vera gel (AVG). The response surface methodology (RSM) was used and a dehydrated product with high antioxidant content was produced. AVG were initially immersed in a sucrose solution of 50° Brix with three levels of GPx (10, 20, 30 v v^{-1}) and subjected to pulsed ultrasound (US) treatment with a probe of 2 cm diameter, two levels of ultrasound amplitude (UA) (50% and 100%), and three immersion time (IT) (30, 135, 210 min). The pulse on and off times were set at 15 min and 1 min, respectively. A multiple regression analysis was carried out to develop second-order polynomial models with high values of determination coefficient (R^2>0.81). The optimal conditions were obtained using the Design-Expert software. The best condition obtained for the UAOD optimization was that UA = 59%, 20% grape pomace was extracted in 50% sucrose, IT = 173 min (moisture loss 49.9%, predicted 51.1%), solid gain was 4.3 % (predicted 5.0%), total phenolic content was 30.5 mg g^{-1} (predicted 31.8 mg g^{-1}), and DPPH radical scavenging activity was 15.3% (predicted 15.0%). UAOD appears to be a promising process for antioxidant incorporation and preparing added value product.

Keywords: ultrasound, osmotic dehydration, grape pomace, response surface methodology

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1 Introduction

The development and consumption of functional food, or foods that promote health is a great challenge for food industry. New sources of nutraceuticals and other natural and nutritional materials should be identified and foods supplemented with bioactive constituents should be developed. Grape pomace (GP) is a by-product of beverage manufacture which generates a large amount of residue for about 20%–25% weight of the crushed grape. Grape pomace is usually discarded, causing disposal problems. GP is mainly consisted of peels (skins), seeds and stems (Minjares-Fuentes et al., 2014), containing phenolic

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compounds, such as anthocyanins (e.g., malvidin and peonidin), flavan-3-ols (e.g., catechin and proanthocyanidins), flavonols (e.g., quercetin, myricetin), stilbenes and phenolic acids (Makris et al., 2007). Anthocyanins have possessed several health benefits, and a bright attractive hue and water solubility therefore they can be replacing for synthetic colors (Nayak et al., 2006). Extraction of anthocyanin from GP and turning them into useful products have a positive environmental effect. Infusing with bioactive compounds from GP into Aloe vera which has a short life, can make it versatile for use in different products. The direct result of bioactive compounds infused to solid foods was that led to produce a wide range of new products appearing on the market. Some physiological active compounds such as calcium, iron or vitamins are mixed with liquid foods such as milk, yogurts, or restructured foods e.g. breakfast cereals, cakes, bread etc. during formulation. However, these techniques are not appropriate for some functional solids foods which maintaining their cellular structures are important (Fito et al., 2001). It is reported that osmotic pretreatment to solid foods can be improved the nutritional, sensorial and functional properties without changing its integrity. Osmotic dehydration is a feasible method for infusing with bioactive compounds such as minerals, phenolic compounds, probiotics and vitamins into solid matrix without altering their natural matrix (Adsare et al., 2016). Therefore, applying osmotic dehydration pre-treatment has two benefits in food industry, which is removal of water and impregnating the food pieces (solid food matrix) with solutes from the osmotic solution (Bellary and Rastogi, 2016).

Many researchers investigated the infusion of active compounds into food matrix without affecting its tissue. The list of these compounds are minerals (Ortíz et al., 2003), phenolic compounds (Rózek et al., 2007; Rózek et al., 2010), curcuminoids (Bellary and Rastogi, 2016), probiotics (Puente et al., 2009), and vitamins (Hironaka et al., 2011). It is important to notice that the rate of mass transfer during osmotic treatment is generally low, therefore, to improve mass transfer phenomena in may fruit during or before osmotic dehydration process, a number of technique has been proposed, such as subjecting materials to pulsed-vacuum (Corrêa et al., 2016), and ultrasound assisted osmotic dehydrating (Fernandes and Rodrigues, 2008).

Ultrasound is a method which attracts enormous interests of food scientist. It is with the frequency ranging from 20 kHz to 100 MHz, enhancing the change of physical and chemical properties of food products (Zheng and Sun, 2006). During power ultrasounds, compression and expansion of the material are usually referred as sponge effect which has a double effect on moisture removal and solid gain (Knorr et al., 2004). This phenomenon results in creating microscopic channels in fruits tissue, therefore, expanded and escaped gas trapped in the pores are eased. So the empty pores are filled with the osmotic solution. This mechanism may explain the increase in mass diffusion when ultrasonic treatment is used (Simal et al., 1998).

Aloe vera (*Aloe barbadensis* Miller) (AV) is a member of the *Liliaceae* family with its parenchymatous tissue made up of polysaccharides (~60% of the dry matter) and a major fraction of water (Garcia-Segovia et al. 2010). In view of its highly therapeutic and functional properties, and beneficial for human health, application of it in the formulation of food products is increasing (Miranda et al. 2009). However, Aloe vera nature is highly perishable. Therefore, drying is one of preservation method to prolong its shelf life.

As consumers are very concerned about nutrient availability on food structure, the combination of convective drying with different pretreatments has been proposed for fruits and vegetables by many authors (Fito and Chiralt, 2003; Perez and Schmalko, 2009). Only a few studies have dealt with drying Aloe vera, such as osmotic dehydration of the watermelon pulp (Falade et al., 2007) and spray-drying of the watermelon juice (Quek et al., 2007), but none has dealt with the infusing with GPx into their texture and enriching it with nutraceuticals. Therefore, the objectives of the present work were investigating the effect of ultrasound assisted osmotic dehydration and superiority over OD.

2 Materials and Methods

2.1 Chemicals

Materials used in this research included Folin–Ciocalteu (FC) reagent, Gallic acid 2, 2-diphenyl-1-picrylhydrazyl (DPPH), and all chemicals and solvents were obtained from Sigma–Aldrich (St. Louis, MO) and Merck (Darmstadt, Germany).

2.2 Raw materials

The fresh whole leaves of AV were obtained by the local market. The leaves were cut for extracting their acibar (yellow color liquid), and then they were hanging vertically for 1 h for draining. After separating the leaf tissue, the Aloe vera gel (AVG) was homogenized and mixed with 2% agar and frozen. The red-grape (GP) (*Vitis vinifera* L. cv.) pomace was obtained from a grape beverage industry in Iran. The pressed pomace (65.87% ± 0.76% moisture content) was immediately transferred to a room freezer at −20°C and stored there to avoid enzymatic degradation of the polyphenols until further use. A phenolic extract was prepared from the pomace in accordance with modification procedure (Drosou et al., 2015). GP (100 g) was placed into an extraction thimble with 1000 mL water in the extraction flask. The water was refluxed for 5-6 h, till the completion of 10 extraction circles. Anthocyanin concentration and total soluble solids in the extract were found to be 145.67 mg L^{-1} and 4-5 ^{o} Brix, respectively. The extract was stored in a cold room at 4–5°C and used within 7 days.

2.3 Agar preparation

The agar gel was prepared by dispensing 2% agar in distilled water (W/V) in a beaker and heated to 95˚C in a microwave oven until it was completely dissolved. Then the gel was mixed with AVG in volume ratio of 1:4. Then the beaker was placed in an ice bath to cool the gel quickly to a temperature of 45˚C and poured into cylindrical Plexiglas containers with height and diameter of 2 cm×2 cm, and refrigerated at 4˚C for 12 h.

2.4 Ultrasound assisted osmotic dehydration (UAOD)

AVG cylinders (0.05±0.01 kg) were immersed in an aqueous solution of sucrose and subjected to ultrasonic waves for 30, 135, 210 min in an ultrasonic bath. UAOD experiments were conducted at temperature of 50°C± 2°C, and the equipment was protected from exposure to light in order to minimize the light effect. AVG cylinders (0.05±0.01 kg) were immersed in about 250 mL osmotic solution of 50 °Brix with different grape pomace extract (10%, 20%, 30%) subjected to the intermittent acoustic treatment with a pulse of on time 15 min and off time 1 min. The experiments were done in 250 mL beakers. The ultrasound frequency was set at 25 kHz, and the power output of ultrasonic bath was set at 400W. Two levels of output amplitude (200, 400W) were used. The immersion treatment time varied between 30min and 210 min. In all experiments, the weight ratio of osmotic solution and gel was over 10:1 to avoid significant modifications to the concentration of the osmotic medium. The experiments were carried out at 50°C. The maximum temperature rise was less than 2°C, which was negligible for the process. All experiments were repeated three times. After the osmotic treatment, samples were removed from the solution, drained, placed on absorbent paper to remove the excess of osmotic solution, and weighed.

2.5 Determination of moisture loss and solid gain

The moisture loss (ML) and solid gain (SG) of AVG samples after osmotic treatments were calculated by using the following equations according to Hawkes and Flink (1978):

$$ML = 100\frac{M_0 x_0 - M_t x_0}{M_0} \quad (1)$$

$$SG = 100\frac{M_1 s_1 - M_0 x s_0}{M_0} \quad (2)$$

Where, M_0 and M_t are the sample mass at time 0 and time *t* (kg); x_0 and x_t are the moisture fractions at time 0 and at time *t* (kg kg^{-1} wet basis); s_0 and s_t are the solids fraction at time 0 and time *t* (kg kg^{-1} wet basis). These equations are based on the assumption that no solids leaked into the solution.

2.6 Extraction of phenolic compounds from UAOD AVG

In order to determine the extent of phenolic infused in to the AVG after osmotic dehydration, an extraction procedure was used. Five grams of osmotic-treated AVG was grounded and extracted with 50 mL deionized water (solvent) for 5 h at room temperature. Then, it was vacuum filtered using Buchner funnel with Whatman no. 1 filter paper (155 mm).

2.7 Determination of total phenolic content (TPC)

Folin–Ciocalteau method was used to measure the total phenolic content (TPC) of UAOD treated samples (Singleton et al., 1999). TPC was presented as Gallic acid equivalents in 1 mg per 1 kg. For the test, 100 µL the sample solutions (100 mg in 10 mL methanol), 6 mL double-distilled water and 500 µL Folin-Ciocalteau reagent were mixed. Subsequently, after waiting for 8.8 min at room temperature, 1.5 mL sodium carbonate (20% w v^{-1}) were added to the solution. The extracts were mixed and allowed to stand for 30 min at room temperature before measuring the absorbance at 765 nm. A mixture of water and reagents was used as a blank. The calibration curve formula for Gallic acid equivalents (concentration range 0.04–0.40 mg mL^{-1}) in methanol was achieved.

2.8 Determination of antioxidant capacity

The 2, 2-diphenyl-1-picrylhydrazyl (DPPH) free radical-scavenging activity of the UOD pre-treated AVG (prediluted to 90 mg L^{-1} concentration) was measured by DPPH assay as described by Ramadan et al. (2003) with a slight modification. Aliquots of each extract (100 µL) were added to 3 mL ethanolic DPPH solutions (0.1 mMol). Discolorations were measured at 517 nm using an UV-1601 spectrophotometer (Shimadzo, Kyoto, Japan) after remaining for 30 min in the dark. The radical-scavenging activity was calculated as a percentage of DPPH discoloration according to Equation 3.

$$\text{DPPH\%} = (A_{\text{cont}} - A_{\text{Sample}}) \times 100 / A_{\text{cont}} \qquad (3)$$

Where, A_{cont} was defined as absorbance of the control; A_{sample} was defined as absorbance of the extracts. Graphs of scavenging efficacy percentage against extract concentration (50-200 µL) in the solution were drawn.

2.9 Experimental design and statistical analysis

Optimization of the processing conditions for Aleo vera gel was carried out using response surface methodology. A Face Centered Experimental Design (FCED) response surface methodology and a Design-Expert Software Version 11.0.7.1 (Minneapolis, USA) was used to analyze the independent effect [ultrasound amplitude, UA (A), grape pomace extraction, GPx (B), Immersion time, IT (C)], with 3 levels for each variable on the moisture loss (ML, %), solids gain (SG, %), total phenolic content (TPC, mg g^{-1}) and 1,1-diphenyl-2-picryl hydrazyl free radical scavenging (DPPH, %). Each variable was coded at three levels of -1, 0 and 1 (Table 1). Six replicates at the center of the design were used to estimate the pure error and sum of squares. Since the various responses were the result of various interactions of independent variables, the following second order polynomial regression equation was fitted to the experimental data of all responses (Equation 4).

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i=1}^{j-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j + \varepsilon \qquad (4)$$

where, Y is the predicted response, β_0 is a constant, ε is noise or error, X_i and X_j are the independent variables, β_i, β_{jj} and β_{ij} are linear, squared, and interaction coefficients, in the order given. Regression coefficient (R^2), adequate precision (AP) and variation coefficient (CV) expresses the quality of the fitted polynomial models.

2.10 Optimization

Utilization of the desirability function proposed by Derringer and Suich (1980) made obtainment of simultaneous optimization possible. For a process optimization, a number of response variables are either maximized or minimized. In order to optimize several response processes, employing the desirability function is very much frequent. For discovery of the most suitable view of the surface response, multiple trial and error attempts may be in order (Myers and Montgomery, 2001). In the current study, for maximization of moisture loss, solids gain, total phenolic content, and DPPH of Aole vera gel, a desirability function was created over time.

3 Results and Discussion

3.1 Model description

Ultrasound amplitude (UA), grape pomace extraction (GPx) and immersion time (IT) represented the UAOD experimental conditions. The design for combined effects, incorporative of 20 experiments, was in accordance with Table 1.

Table 1 Independent variables and their coded and uncoded values for optimization

Coded value	Ultrasound amplitude (UA) (%)	Grape pomace extract (GPx) (%)	Immersion time (IT) (min)
-1	0	10	30
0	50	20	135
1	100	30	210

Table 2 CCD in actual forms of process variables and experimental data of response variables for UAOD of Aloe vera gel

	Extraction conditions			Analytical results			
Exp.no	UA(%)	GPx(w v^{-1})	IT(min)	ML(%)	SG(%)	TPC(mg g^{-1})	DPPH(%)
1	0	10	30	29.06	2.11	15.76	9.54
2	0	30	30	30.82	2.39	16.86	8.57
3	100	30	30	34.94	2.57	19.97	12.89
4	100	10	30	36.00	2.67	20.78	13.87
5	50	20	30	37.33	2.87	20.99	12.67
6	100	20	135	37.93	3.25	32.02	13.74
7	0	20	135	40.94	3.28	14.67	13.78
8	50	20	135	51.06	4.41	31.67	13.87
9	50	20	135	52.82	4.50	30.80	14.24
10	50	20	135	54.94	4.54	32.67	14.87
11	50	20	135	55.11	4.68	31.77	14.65
12	50	30	135	45.12	4.72	32.23	15.34
13	50	10	135	45.50	5.34	25.74	15.87
14	50	20	135	45.94	4.26	25.87	16.23
15	50	20	135	46.16	3.30	29.56	16.98
16	100	30	240	46.56	3.37	25.67	12.87
17	50	20	240	48.58	3.54	28.97	13.77
18	100	10	240	51.52	3.62	25.57	12.45
19	0	30	240	52.17	3.78	16.87	10.23
20	0	10	240	52.67	3.99	14.66	10.99

Table 2 gives information for data on solid gain, water loss, total phenolic content and DPPH evaluated under the different UAOD experimental conditions (Equation 4). A polynomial response surface model of the second-order, was fitted to each response variable (Y). For determining effects of process variables with significance on each response, a variance analysis procedure was utilized. Table 3 is a summarization of terms of significance and their coefficients in the final model. A major feature of a model taken into consideration, is suitability validation, making sure that lack of fit is not of significance (P>0.05). In other words, the model is of significance and its prediction of the response variables is satisfactory. Acceptable actual and fitted values correlation can also confirm this. The relative importance of the influence of individual variables and their interactions were achieved through employment of the P-values. The R^2 of water loss, solid gain, total phenolic content and DPPH were 0.82, 0.90, 0.87 and 0.87 respectively. The large R^2 and the values of adjusted R^2 demonstrated the suitability of the models for the design of space navigation.

Table 3 Regression coefficients of predicted polynomial models for the investigated responses from AVG

Source	Df	Sum of Square			
		ML(%)	SG(%)	TPC(mg g^{-1})	DPPH(%)
Model	9	1008.48□□□	13.35□□□	704.87□□□	79.83□□□
A-UA	1	0.17^{ns}	0.00□	204.03□□□	16.15□□
B-GPx	1	2.64^{ns}	0.08□	7.85^{ns}	0.80^{ns}
C-IT	1	694.60***	3.24□□□	30.21□	0.77□
AB	1	6.62^{ns}	0.02^{ns}	2.02^{ns}	0.17^{ns}
AC	1	39.74^{ns}	0.28^{ns}	16.76^{ns}	2.59^{ns}
BC	1	4.77^{ns}	0.05^{ns}	0.51^{ns}	0.32^{ns}
A^2	1	90.71**	2.59□□	96.48□□	7.94□
B^2	1	0.05^{ns}	1.73□□	0.41^{ns}	0.06^{ns}
C^2	1	13.55^{ns}	2.93□□□	50.33□	13.79□□
Residual	10	222.44	1.53	102.54	12.18
Lack of fit	5	137.72^{ns}	0.28^{ns}	72.51^{ns}	4.87^{ns}
Pure error	5	84.72	1.25	30.03	7.31
Cor Total	19	1230.92	14.88	807.42	92.00
Variance analysis of quadratic model					
R^2		0.82	0.90	0.87	0.87
Adjusted R^2		0.66	0.80	0.76	0.75
Predicted R^2		0.37	0.73	0.27	0.47
Adequate			10.44	7.52	8.93
Precision		7.74			
CV		10.54	10.69	12.99	8.25

Note: ns means no significant effect at level of P<0.05; □ means P<0.05; □□ means P<0.01; □□□ means P<0.001.

3.2 Effect of process variables on ML

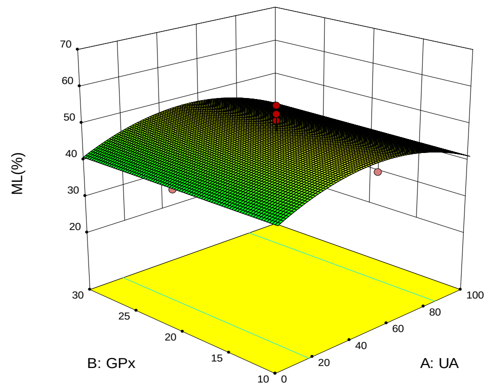
Table 3 indicated that the linear coefficients, quadratic and interaction effect of UA, GPx and UT were of significance. The determination coefficient (R^2) of the predicted models in this response was 0.37 and F-value for lack of fit of the model was not of significance (*P*>0.05). These values would be of suitable fitness to the mathematic model. Based on the sum of squares, the importance of the independent variables on polyphenol content could be arranged in the following order: linear term of IT, quadratic term of UA, followed by linear term of UA. Associated regression equation for the responses based on actual values is given as Equation 5:

$$ML = 30.42 + 0.28\ UA + 0.08\ IT - 2.79E003UA^2 \quad (5)$$

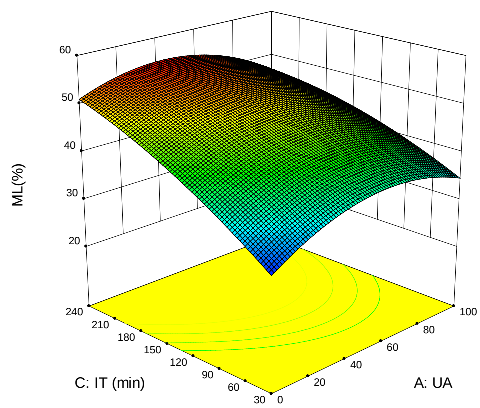
Figure 1 represents three-dimensional (3D) plots for moisture loss as a function of UAOD conditions. Such data was obtained by keeping one variable at the midpoint of the testing ranges and varying the other two within the experimental range.

As shown in Figure 1(a), increase of UA until 50% resulted in enhancement of the ML. This effect is probably due to the cavitation phenomena, concentration gradient, and ML increasing. Microchannel formation as a result of the acoustic cavitation phenomenon is linked to the indirect effect of ultrasound. Cavitation takes place in the water inside or outside the product cells, when ultrasound waves travel through the sample, resulting in cell and tissue disruption and the consequent formation of cavities and microchannel. Popular belief holds that the presence of these micro channels is the primary effect of the ultrasound technology in mass transfer phenomena enhancement in food processing (Miano et al., 2016).

After this time, the ML started to decrease. Thus, an excessive UA is not efficient to increase ML. Additionally, increasing GPx results in increased moisture loss. This followed the results of other studies focus on ML. Oladejo and Ma (2016) reported that the acoustic cavitation produced through increasing ultrasound intensity and GPx had a sponge like effect, that is, compressive and expansion stress appears alternately on the surface of the sweet potato. This led to breakdown of the semi-permeable membrane of the sweet potato, thus microscopic channels formed inside the sweet potato sample through which water came out. Based upon Figure 1(b), the response surfaces showed that the ML increased with the elevation of the IT. As the immersion time increased, the semi-permeable membrane of the AVG weakened, therefore, allowed more loss of water. In addition, this might be a result of the improvement of mass transfer and solubility at higher immersion time. The moisture loss is directly proportional to the ultrasonic probe treatment time. Pretreatments had been proven to enhance water loss during the OD process. Similar findings were reported in the study effect of ultrasound-assisted osmotic dehydration pretreatment on the sweet potato (Oladejo and Ma, 2016).



(a) UA and GPx



(b) UA and IT

Figure 1 Response surfaces of the total moisture loss of AVG

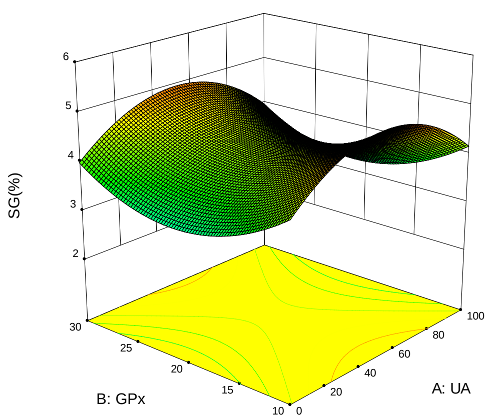
3.3 Effect of process variables on SG

As demonstrated in Table 3, the quadratic and linear terms of UA, GPx and IT were of significance at *P*<0.005 and *P*<0.05. Associated regression equation for the responses based on actual values on SG is given as Equation 6:

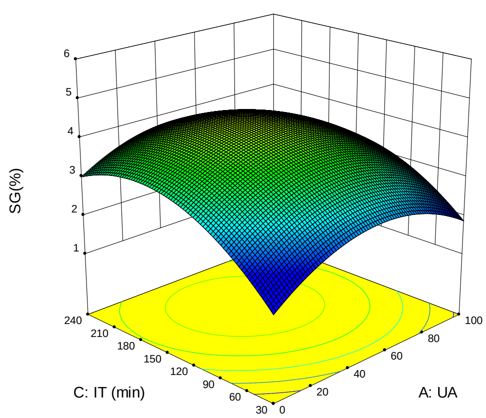
$$SG = 4.21 + 0.04\ UA - 0.33\ GPx + 0.0\ 3IT - 3.88\ UA^2 + 7.94\times10^{-3}\ GPx^2 - 9.3610^{-5}\ IT^2 \quad (6)$$

Figure 2 provides three-dimensional (3D) plots for solids gain as a function of UAOD conditions.

As represented in Figure 2(a), with an increase of GPx and UA, solid gain would increase. The difference in the osmotic driving force between the osmotic solution and the AVG resulted in lysis (breakdown) of cell wall, allowing movement of the solutes into the membrane of sweet potato. The creation of microscopic channels within the membrane of AVG by the ultrasonic effect also resulted in solute uptake (Hamedi et al., 2018). Similarly, the counter-current flow of the osmotic solution employed in the current study may have led to impregnation of solutes into the AVG membranes.



(a) UA and GPx



(b) UA and IT

Figure 2. Response surfaces of the solids gain of AVG

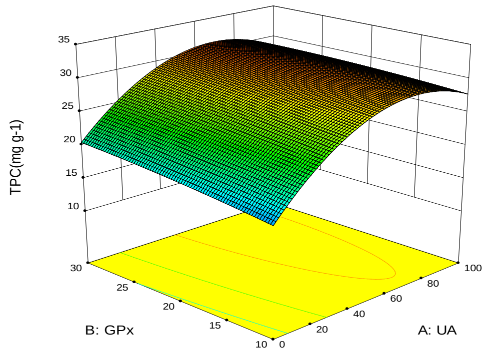
3.4 Effect of process variables on TPC

The values of the regression coefficients provide a general sense as how far the responses are affected by the control variables, quantitatively. The results in Table 3 demonstrated that the quadratic and linear terms of UA and IT were of significance ($P \leq 0.05$). The interaction terms showed no obvious effect of significance ($P > 0.05$) on the TPC. The regression model was highly significant with satisfactory coefficient of determination (R^2 = 0.839). Moreover, the CV was within the acceptable range (11.95) and the lack-of-fit tests did not lead to significance of F value (0.1), indicating that the model was adequately precise for predicting the TPC. According to the sum of squares, the importance of the independent variables on TPC may be arranged in the following order: ultrasound amplitude (A), quadratic term of ultrasound amplitude (UA), quadratic term of immersion time (IT^2), and the immersion time(IT). The values of the coefficients for TPC based on actual values were used for determining the final predictive model by neglecting the non-significant cross-terms given as Equation7:

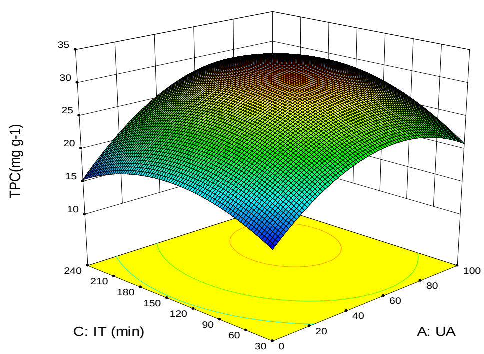
$$TPC = 9.76 + 0.33\ UA + 0.12\ IT - 2.43\times10^{-3}\ UA^2 - 4.01\times10^{-4}\ IT^2 \quad (7)$$

In order to visualizing the influence of variables on TPC, three-dimensional surface plots (Figure 3) were constructed in accordance with Equation 7.

As Figure 3 presents, total phenolic content increases with increasing GPx and ultrasound amplitude. Higher ultrasound amplitude resulted in solid gain enhancing and higher mass transfer, thus, total phenolic contents in higher power ultrasound grew. The increase of ultrasound amplitude (80%) and water loss resulted in total phenolic content decreasing. Applied ultrasound may speed up the formation of free radicals and increase the polymerization level of phenolic compounds (Stojanovic and Silva, 2007). Higher power ultrasound, introducing the cavitation phenomenon, might also rupture the surface of agar gels, resulting in more leakage and loss of phenolic compounds than gaining. Different GPx concentration of the osmotic solution generated different trends in gaining phenolic by migration into the sample matrix. With the increase of the GPx concentration, Osmotic pressure increased and mass transfer enhanced. Based upon Figure 3(b), the TPC was increased to a small degree by elevating GPx. This may be attributed to infusion with phenolic compounds into the fruit matrix. Rózek et al. (2010) investigated the infusion with grape phenolic into fruits and vegetables and reported that total phenolic content and antiradical activity of plant foods increased, such as apple, banana, and potato.



(a) UA and GPx



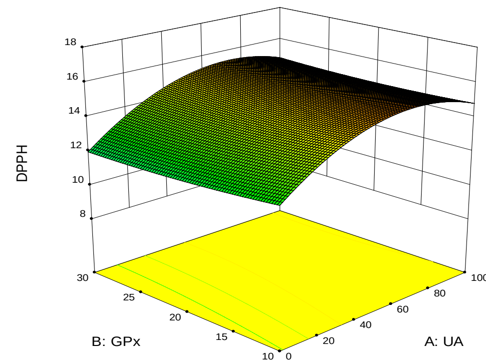
(b) UA and IT

Figure 3 Response surfaces of the TPC of AVG

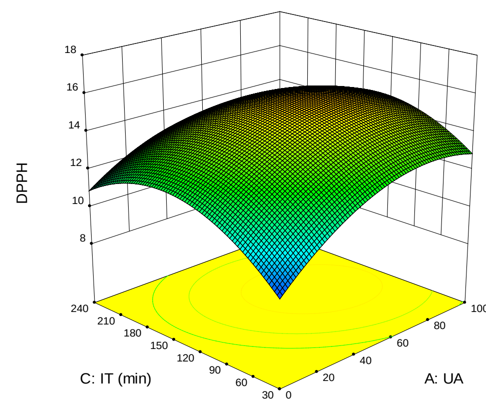
3.5 Effect of process variables on DPPH radical-scavenging capacity

The results in Table 3 demonstrated that the quadratic and linear terms of UA and IT were of significance ($P \leq 0.05$). The interaction term coefficients were not an effect of significance on DPPH ($P > 0.05$). The regression model was highly significant with satisfactory coefficient of determination ($R^2 = 0.825$). Furthermore, the variation coefficient was within the acceptable range (7.75). The lack-of-fit tests did not lead to a significance on F value (0.77), indicating that the model was adequately precise for predicting the DPPH. According to the sum of squares, the importance of the independent variables on the DPPH value could be ranked in the order of the linear terms of ultrasound amplitude (UA), quadratic terms of immersion time (IT^2), and quadratic terms of ultrasound amplitude (UA^2) followed by the linear term of immersion time(IT). The values of the coefficients for DPPH based on actual values were employed for determining the final predictive model by neglecting the non-significant cross-terms given as Equation 8:

$$DPPH=8.40+0.09\,UA+0.05\,IT-6.58\times10^{-4}\,UA^2-1.98\times10^{-4}\,IT^2 \quad (8)$$



(a) UA and GPx



(b) UA and IT

Figure 4 Response surfaces of the DPPH of AVG

Figure 4(a) demonstrates the interactive effect of GPx and UA on DPPH activity of pre-treated samples. DPPH was increased up to 80% readily with increasing UA, and followed by a slight decrease, while slightly elevating GPx resulted in increase of DPPH. DPPH radical scavenging activity assay is capable of evaluating the electron donation potency of different antioxidative compound easily (Rezaiea et al., 2015). The DPPH free radical is commonly used for evaluation of antioxidant activities of compounds. The results showed that DPPH antioxidant activity had a linear relationship with the total phenolic content. Wang et al. (2011) reported the same observation on extraction of phenolic compounds from pomegranate. Phenolic compounds have a strong antioxidant capacity and show different behaviors in response to osmotic solution concentration, ultrasound, oxygen, temperature and other factors. While GPx increased in the osmotic solution and higher power ultrasound applied, the changes in the antioxidant activity of the osmo-treated samples demonstrated changes similar to that of the total phenolic compounds. The higher water loss and soluble solid gain usually correlate with a higher leakage of low molecular or water-soluble compounds of sample extract, including chemical composition building up the antioxidant properties (Mieszczakowska-Frąc et al., 2016). The reaction of antioxidants presenting in the agar gel extracts with DPPH, reveals quite a high activity, signaling that the osmotic treatment is a successful way for phenolic impregnation into a solid matrix, particularly in the case of agar gel.

3.6 Optimization and model validation

The desirability function method described in the materials and methods section was employed to analyze the process parameters concerning the dependent variables (moisture loss, solids gain, total phenolic compounds, DPPH) optimization.

Table 4 Predicted and experimental values of the response variables at optimum conditions for UAOD and conventional methods

Characteristics	Predicted values	Experimental values
ML (%)	51.125[a]	49.87[a] ±1.072
SG (%)	4.305[a]	4.98[a] ± 0.06
TPC (mg g^{-1})	30.529	31.78[a] ± 0.91
DPPH (%)	15.264[a]	14.98[a] ±1.12

Note: Predicted values mean obtained using response surface quadratic model; Experimental values mean average value ± standard deviation of triplicate determinations from experiments; a means no significant difference (P< 0.05) at same column.

The desired levels for each of the operational conditions (UA, GPx, and IT) were selected within the range defined by Hamedi, et al. (2018), while the dependent variables were defined as maximum. Each of the dependent variables was analyzed separately. The optimal conditions were obtained by Design-Expert software. The final result of this optimization suggested that a UAOD containing 59% ultrasound amplitude, 20% grape pomace extract, and 173 min immersion time could be a good mixture of these three factors to attain the best antioxidant capacity. These new UAOD conditions were submitted to the same experimental procedures applied as those from the commencement of this study. There was no significant difference between the estimated and observed values (P < 0.05), indicating a good fit between the models and the experimental data (Table 4).

4 Conclusion

The optimum process parameters for the ultrasound assisted osmotic dehydration (UAOD) of AVG in sucrose solutions with grape pomace extract were obtained by utilizing RSM. The developed models demonstrated good correlation with the experimental data at 95% confidence level. The optimum osmotic dehydration conditions were 50% sucrose (w v^{-1}) at 50°C, 59% UA, 20% grape pomace extract and 173 min immersion time, achieving maximum ML, SG, TPC and DPPH. The results showed that retention of the antioxidants such as phenolic, was greatly enhanced in the dehydrated Aole vera gel using UAOD pretreatment. These results showed that this process can be employed by the industry without high energy requirements and costs while having a high characteristic valued by consumers.

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