PAPER

OPTIMIZATION OF MICROWAVE AND AIR DRYING CONDITIONS OF QUINCE (*CYDONIA OBLONGA*, MILLER) USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Effects of slice thickness of quince (*Cydonia oblonga* Miller) , microwave incident power and air drying temperature on antioxidant activity and total phenolic content of quince were investigated during drying in microwave and air drying. Optimum conditions were found to be: i) for microwave drying, 285 W and 4.14 mm thick (maximum antioxidant activity) and 285 W and 6.85 mm thick (maximum total phenolic content), and ii) for air drying, 75 °C and 1.2 mm thick (both maximum antioxidant activity and total phenolic content). Drying conditions were optimized by using the response surface methodology. 13 experiments were carried out considering incident microwave powers from 285 to 795 W, air temperature from 46 to 74 °C and slice thickness from 1.2 to 6.8 mm.

- Keywords: Microwave, air drying, antioxidant activity, total phenolic content, response surface methodology -

1. INTRODUCTION

Edible fruits are sources of nutrients such as carbohydrates, vitamins, and minerals as well as non-nutrient compounds such as polyphenols. Nowadays, it is commonly admitted that there is a positive relationship between a diet rich in vegetables and fruits and a reduced incidence of degenerative diseases such as cancer and cardiovascular events (GIBNEY *et al.*, 2009). Health beneficial properties of quince fruit (*Cydonia oblonga* Miller) have known from ancient times. Quince is the only species in the genus Cydonia, which falls into Pomoideae subfamily of the Rosaceae along with apple and pear (PACIFICO *et al.*, 2012). Quince is used extensively in Europe as a dwarfing rootstock for pear. Total world production of fresh quince was 540.337 tons in 2010 and about 25% of this was produced in Turkey (TSI, 2012).

Drying process is one of the most important preserving operations that causes time and energy consumption in the food industry. That is why new methods are aimed to decrease drying time and energy consumption. New methods combined different systems such as using microwave drying together with traditional drying methods to reduce drying time (SECMELER, 2003). Over the past two decades, there has been an increasing attraction in microwave drying to reduce drying time and increase the removal of water from agricultural products. Microwave drying has several advantages such as short drying time, higher drying rate, better quality of the dried products and decrease energy consumption (SANGA *et al.*, 2000).

Response surface methodology (RSM) is one of the most commonly used optimization technique in food science. This method is preferred because of the simplicity and high efficiency. RSM covers a group of techniques used to study the relationship between one or more measured responses and input variables. (ARTEGE *et al.*, 1994). It has been successfully applied to optimize food processing operations by many researchers (FRANK, 2001; LEE *et al.*, 2006; LUCIANE *et al.*, 2001; MIRHOSSEINI *et al.*, 2008; PIETRASIK and LI-CHAN, 2002).

2. MATERIAL AND METHODS

2.1. Material

Fresh quinces were obtained from a local market in Konya. These samples were transferred to laboratory in cool bags and they stored in refrigeration temperature (4 °C) until the assay, Initial moisture content of fruits was detected as 80% in average. Prior to drying, round shaped samples (2 cm in diameter) were obtained from fruit slices. Thickness varied from 1.2 to 6.8 mm according to experimental design.

2.2. Drying

Quinces were dried until the moisture content decreased to 40% of the initial moisture content, since burning was observed on the quince slices in microwave drying below this moisture content level. Microwave drying experiments were performed using a domestic microwave oven (ARCELIK ARMD 580, Turkey). The dimensions of the microwave cavity were 345 mm x 340 mm x 225 mm. Three power levels were selected as high (720 W), medium (540 W) and low (360 W) for drying experiments. One dish containing 1 slice of sample to make effective drying was placed on the centre of a turntable fitted inside the microwave oven. Quinces were placed uniformly as a thin layer onto the stainless steel trays (0.3 m x 0.2 m) and dried using air oven (Nüve FN055 Ankara, Turkey, 55 L volume) at three different temperatures (50, 60 and 70 °C). Quince slices were placed uniformly as a thin layer onto the stainless steel trays (0.3 m x 0.2 m) and dried under direct sunlight in April in Konya, Turkey (BALLADIN & HEADLEY, 1999).

2.3 Extraction

The phenolic compounds were isolated from samples using a modified version of the method described by SHAHIDI *et al.* (2001). One gram of sample was extracted 3 times using 10 ml of 70% (v/v) aqueous methanol (Merck, Germany) at room temperature by a homogenizer (Ika Ultra Turrax Tube Disperser) for 1 min. The slurry was centrifuged at 4000 rpm for 15 min. Supernatants were collected and combined in a rotary flask and then evaporated at 45 °C under vacuum by a Rota vapor R-3000 rotary evaporator (Laborato 4001, Heidolph). The extracted phenolics were dissolved in 25 ml methanol and then filtered using filter paper. Methanolic solutions of phenolic were stored -25 °C until analysis.

2.4 Determination of total phenolic content and antioxidant activity

Total phenolic compounds were prepared by using Folin-Ciocalteu Reagent (YOO *et al.*, 2004). The free radical scavenging activity of the extract was determined using 1, 1-diphenyl-2-picrylhydrazyl (DPPH) in order to determine antioxidant activity (LEE *et al.*, 1998). Total phenolic contents were calculated by a standard calibration curve prepared using gallic acid. Total phenolic content results were given as gallic acid equivalents in milligrams per 1000 g fruit. Antioxidant activity results were expressed as percentage activity (%).

2.5 Experimental Design

Response Surface Methodology (RSM) was used to optimize drying conditions, based on better preserve the antioxidant activity and total phenolic content of quince fruit. Box–Behnken design was selected for RSM analysis. Box–Behnken designs require only three levels, coded as −1, 0 and +1. The effects of the two independent processing parameters: slice thickness (X_1, mm), incident microwave power (X_2, Watt) in microwave operation and additionally slice thickness (X_1, mm), processing temperature level (X_2, °C) in air drying on two dependent variables (antioxidant activity and total phenolic content) were investigated using RSM. The total number of experiments in this study was 13 based on two levels and a two factor experimental design, with five replicates at the centre of the design for estimation of a pure error sum of squares. Minitab 16 (Minitab Inc. State College, PA) was used for the experimental design, data analysis and regression modeling. The independent variables were; X_1 (2–4 mm), X_2 (360-720 W) in microwave process and X_1 (2 – 4 mm), (X_2 50 - 70 °C) in air drying. Experimental data from the Box–Behnken design was fitted into a second-order polynomial model.

$$Y= b_o + b_1X_1 + b_2X_2 + b_1^2 X_1^2 + b_2^2 X_2^2 + b_1b_2X_1X_2 \quad (1)$$

Where Y is the predicted response, X_1 and X_2 are independent variables, b_0 is a constant; b_1, b_2, b_1^2, b_2^2, b_1b_2 are linear, quadratic and interaction coefficients, respectively.

3. RESULTS AND DISCUSSION

3.1 Microwave drying

Independent variables [thickness of slice (X_1) and microwave power level (X_2)] observed and predicted values of antioxidant activity and total phenolic content for microwave drying were given in Table 1. During drying in microwave oven antioxidant activity decreased in the studied variables. The influences of interaction between thickness-thickness were determined as statistically important ($p \leq 0.05$) whereas the other parameters were found insignificant ($p > 0.05$) (Table 2a). Statistical analysis indicated that the fitted model (Eq.1) to experimental results displayed high performance to predict the antioxidant activity of quince samples within the studied ranges of variables. Regression coefficient (R^2) and adjusted regression coefficient (R^2_{adj}) were calculated as 0.994 and 0.990, respectively (Table 2a). 3D surface plots for the significant terms were shown in Figs. 1 and 2 (MINITAB 16). Surface plot given in Fig. 1a indicated that antioxidant activity was not affected by thickness and power level. Thickness-thickness square parameter was the only parameter which affected on antioxidant activity ($p \leq 0.05$) (Table 2a). Antioxidant activity of quince increased up to 4 mm of slice thickness and then decreased (Fig. 1a).

The interaction between thickness-thickness, thickness-power, power-power, power and thickness alone showed significant effects ($p \leq 0.05$) (Table 2b) on total phenolic content of quince dried in microwave oven. Total phenolic content of quince dried in microwave oven was fitted in Eq.2. Re-

Table 1. Experimental Design of Microwave Drying and Corresponded Responses (Antioxidant Activity and Total Phenolic Content)

Run	X_1 Thickness (mm)	X_2 Power (W)	Antioxidant Activity (%) Observed	Antioxidant Activity (%) Predicted	Total Phenolic Content (mg/1000g) Observed	Total Phenolic Content (mg/1000g) Predicted
1	4	285	83	82	1661	1635
2	2	720	66	65	796	791
3	1.2	540	68	68	810	791
4	6	360	77	77	1499	1503
5	4	540	70	73	882	898
6	2	360	75	76	1125	1157
7	4	540	75	73	879	898
9	4	795	63	66	691	718
10	4	540	73	73	895	898
11	6.8	540	67	68	859	880
12	4	540	77	73	916	898
13	6	720	68	65	605	572

gression coefficient (R^2) and adjusted regression coefficient (R^2_{adj}) were calculated as 0.880 and 0.795, respectively (Table 2b). The linear coefficient for thickness was over seventy times greater than that of power level. There was interaction of significance between thickness and power level.

$$Y_1 = 80.13 + 5.58\,X_1 - 0.66\,X_1^{2} \quad (1)$$

$$Y_2 = 2088.05 + 290.44\,X_1 - 4.87\,X_2 + -7.86\,X_1^{2} - 0.39\,X_1X_2 \quad (2)$$

Antioxidant activity and total phenolic content values were found higher in microwave drying than that of air drying. Drying time decreased 60-120 times in microwave drying. BERTELI & MARSAIOLI (2005) have studied the influence of drying methods on moisture content of product. Very short heating–cooling cycles take place in microwave drying. The use of microwave for drying has become common because it enhances the product quality and processing speed (DIAZ *et al.*, 2003). Fresh fruits are well known for their antioxidant activity which is usually attributed to the polyphenol content (WANG *et al.*, 1996). Total phenolic content of 2020 mg GAE/kg sample and 78% inhibition for antioxidant activity were reported in fresh quince samples before the drying experiment (HAMAZU *et al.*, 2005). KARADENIZ *et al.* (2005) reported that antioxidant activity of some quince varieties in Turkey found between 51-68%.

Table 2 A. Analysis of variance of antioxidant activity of quince in microwave drying
B. Analysis of variance of total phenolic content of quince during microwave drying

A

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	318.926	318.926	63.7851	10.34	0.004
Linear	2	268.848	48.119	24.0594	3.9	0.073
Thickness	1	0.239	26.655	26.6549	4.32	0.076
Power	1	268.608	6.093	6.0932	4.05	0.353
Square	2	49.997	49.997	24.9984	4.05	0.068
Thickness*Thickness	1	49.906	48.507	48.5071	7.86	0.026
Power*Power	1	0.091	0.091	0.091	0.01	0.907
Interaction	1	0.081	0.081	0.0812	0.01	0.907
Thickness*Power	1	0.081	0.081	0.0812	0.01	0.912
Residual Error	7	43.174	43.174	6.1677		
Lack-of-Fit	3	17.322	17.322	5.774	0.89	0.518
Pure Error	4	1328	1328	332		
Total	12	362.099				

B

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	1081629	1081629	216326	263.08	0
Linear	2	849631	300620	150310	182.79	0
Thickness	1	7932	72176	72716	87.77	0
Power	1	841699	122155	122155	148.55	0
Square	2	452240	152240	76120	92.57	0
Thickness*Thickness	1	17411	6877	6877	8.36	0.023
Power*Power	1	134829	134829	134829	163.97	0
Interaction	1	79758	79758	79758	97	0
Thickness*Power	1	79758	79758	79758	97	0
Residual Error	7	5756	5756	822		
Lack-of-Fit	3	4428	4428	1476	4.45	0.092
Pure Error	4	1328	1328	332		
Total	12	1087385				

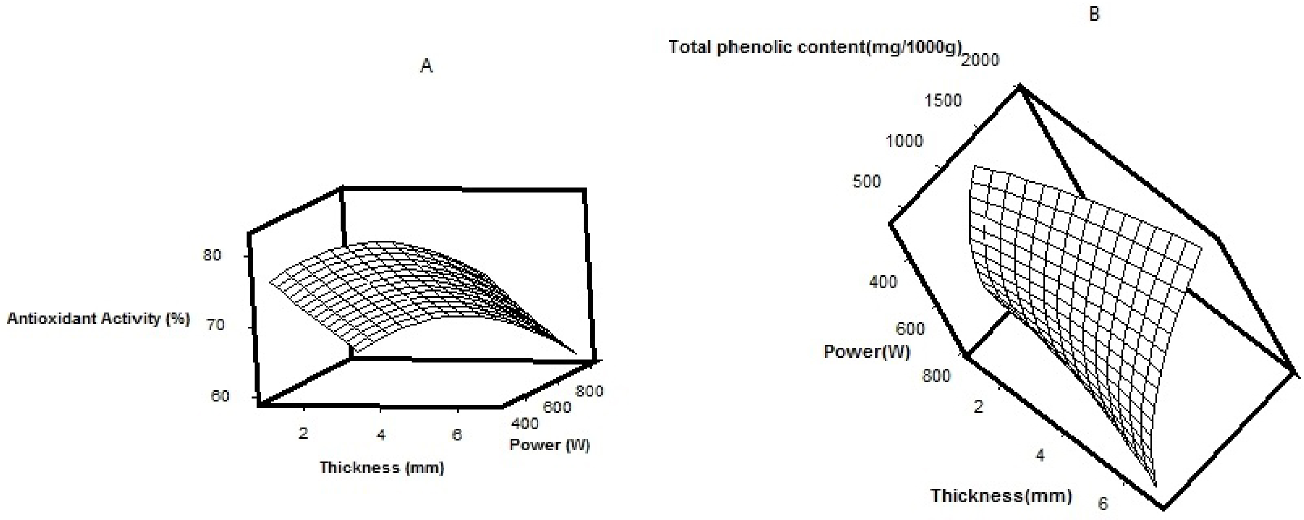

Fig. 1 - Effect of thickness and power on antioxidant activity during microwave drying (A) and on total phenolic content during microwave drying (B).

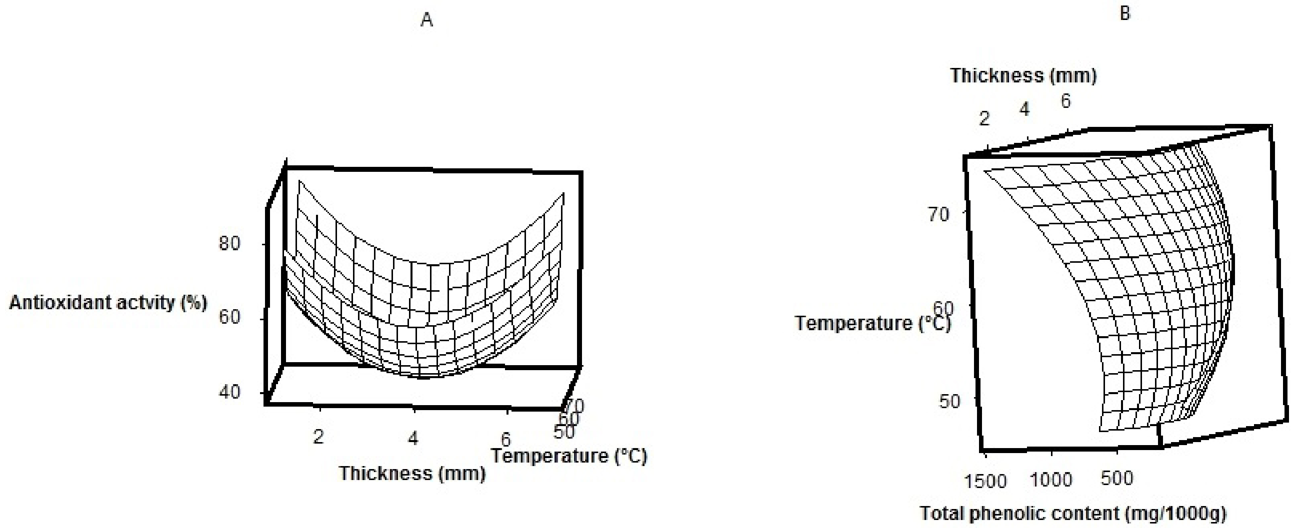

Fig. 2 - Effects of thickness and temperature on antioxidant activity oven drying (A) and on total phenolic content oven drying (B).

Table 3. Experimental Design of Oven Drying and Corresponded Responses (Antioxidant Activity and Total Phenolic Content)

Run	X_1 Thickness (mm)	X_2 Temperature (°C)	Antioxidant Activity (%) Observed	Antioxidant Activity (%) Predicted	Total Phenolic Content (mg/1000g) Observed	Total Phenolic Content (mg/1000g) Predicted
1	2	50	63	59	484	469
2	6	70	59	63	457	472
3	4	74	67	65	696	720
4	4	60	38	40	229	264
5	4	60	39	40	280	264
6	2	70	65	64	952	903
7	1.2	60	58	61	685	730
8	6	50	58	58	491	540
9	4	60	40	40	258	264
10	6.8	60	63	60	520	475
11	4	60	42	40	289	264
12	4	46	55	58	485	460
13	4	60	40	40	266	264

3.2 Air drying

Independent variables [thickness of slice (X_1) and temperature of air (X_2)], observed and predicted values of antioxidant activity and total phenolic content for air drying were given in Table 3. Antioxidant activity of quince samples decreased after air drying. Thickness of samples and process temperature had considerable effect on antioxidant activity. Additionally, the influences of interaction between thickness-thickness and temperature-temperature were statistically significant (p≤0.05) whereas the other parameters were insignificant (p>0.05) (Table 4a). Effect of thickness and temperature on the antioxidant activity in air drying was given in Fig. 2a. Increase in thickness from 2 to 4 mm resulted in lower antioxidant activity at different drying temperatures. The fitted model to antioxidant activity results of quince samples after air drying was given in Eq. 3. Model parameters for Eq. 3 (R^2 and R^2_{adj}) were calculated as 0.953 and 0.920, respectively (Table 4a).

The interaction between thickness-thickness, temperature-temperature, thickness-temperature and temperature of process showed significant effects (p≤0.05) on total phenolic content after air drying (Table 4b). Changes in total phenolic content was given in Fig. 2b and increase in thickness and power level caused firstly decrease and then increase in total phe-

Table 4.A Analysis of variance of antioxidant activity of quince in oven drying
B Analysis of variance of total phenolic content of quince in oven drying

A

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	1420.7	1420.7	284.13	28.7	0
Linear	2	50.57	750.9	375.45	37.9	0
Thickness	1	1.7	141.46	141.46	14.3	0
Temperature	1	48.87	721.43	721.43	72.9	0
Square	2	1369.7	1369.7	684.87	69.2	0
Thickness*Thickness	1	571.44	749.98	749.99	75.8	0
Temperature*Temperature	1	798.3	798.3	798.3	80.6	0
Interaction	1	0.36	0.36	0.357	0.04	0.86
Thickness*Temperature	1	0.36	0.36	0.357	0.04	0.86
Residual Error	7	69.3	69.3	9.9		
Lack-of-Fit	3	58.38	58.38	19.46	7.13	0.04
Pure Error	4	10.92	10.92	2.729		
Total	12	1490				

B

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	533725	533725	106745	59.7	0
Linear	2	132129	125964	62982	35.3	0
Thickness	1	649.25	14	14	0.01	0.93
Temperature	1	67203	118914	118914	66.6	0
Square	2	338638	338638	169319	94.8	0
Thickness*Thickness	1	154271	198402	198402	111	0
Temperature*Temperature	1	184367	184367	184367	103	0
Interaction	1	62959	62959	62959	35.2	0
Thickness*Temperature	1	62959	62959	62959	35.2	0
Residual Error	7	12508	12508	1787		
Lack-of-Fit	3	10381	10381	3460	6.51	0.05
Pure Error	4	2128	2128	532		
Total	12	546233				

nolic content similar to the antioxidant activity. Total phenolic content of quince by air drying was fitted in Eq. 4. Model parameters for Eq 4 (R^2 and R^2_{adj}) were calculated as 0.977 and 0.960, respectively (Table 4b). The high values of regression coefficient indicate a high degree of correlation between the experimental and fitted values.

$$Y_3 = 449.60 - 20.10\,X_1 - 12.54\,X_2 + 2.59\,X_1^2 + 0.10\,X_2^2 \quad (3)$$

$$Y_4 = 4925.38 - 6.43\,X_1 - 161.10\,X_2 + 42.22\,X_1^2 + 1.63\,X_2^2 - 6.27\,X_1X_2 \quad (4)$$

3.3 Sun drying

Sun drying was chosen as traditional drying method and this process maintained about 24 h. The use of microwave oven seems to be more advantageous considering the time factor. In sun drying, total phenolic content and antioxidant activity were determined as 1544 mg GAE/ 1000 g and 74%, respectively. These values were close to the results observed after microwave drying and higher than that of air drying.

In order to determine the optimal residual activity, response optimizer tool in MINITAB 16 (Minitab Inc. State College, PA) was used. The optimum conditions were found as 4.14 mm thickness and 285 W power level for maximum antioxidant activity, 6.85 mm thickness and 285 W power level for maximum total phenolic content in microwave drying. When air drying was analyzed, 1.2 mm thickness and 75 °C temperature were selected for maximum antioxidant activity and total phenolic content.

4. CONCLUSIONS

The effect of power level and slice thickness on the antioxidant activity and total phenolic content were investigated for quince samples after drying. RSM was used to optimize the factors in order to obtain maximum level of antioxidant activity and total phenolic content of quince samples. All independent variables including thickness of slice (mm), power level (W) and processing temperature (°C) had significant effects on the response values. Furthermore square and interaction parameters showed significant effects ($p \leq 0.05$). A desirable quadratic mathematical model was built by using Box–Behnken design. The antioxidant activity and total phenolic content decreased after drying. Nevertheless optimum drying conditions were obtained for both microwave and air drying. Additionally, the use of microwave provides time saving compared to the other drying methods.

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Paper Received December 11, 2013 Accepted May 8, 2014