

Roasting Process Optimization of Walnut Kernels for the Preparation of Walnut Cream Using Response Surface Methodology

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Abstract

Roasting has considerable effects on the quality of cream made of nuts. In this study, the roasting conditions of walnut kernels were optimized based on the stability parameters of the produced cream. Temperatures of 100-150°C for 10-30 minutes were used to roast walnut kernels. The amount of oil separation, peroxide, acidity and Thiobarbituric acid values of the cream, as well as color parameters were determined after three months of storage at 25°C. The results showed that the oil separation increased with temperature and time of roasting (from 4.16% at 100°C/10min to 7.85% at 150°C/30min). Peroxide, acidity and thiobarbituric acid values were significantly affected by temperature and time of roasting. In addition, it was shown that as the temperature increased, the redness and yellowness increased, but the lightness of the samples decreased. Finally, the temperature of 116°C for 12 minutes was chosen as the optimized roasting conditions for producing walnut cream.

Keywords: Nut spread, Oil separation, Optimization, Roasting, Walnut cream.

Introduction

Creams are semi-solid foods rich in fat that easily flow. These include water-in-oil emulsions with different formulations containing fat, water, emulsifier, stabilizer, salt, antioxidants and other compounds (Mousazadeh *et al.*, 2013). Nut cream that is produced conventionally is a viscous mixture of fine particulate matter of nut suspended in oil (nut paste), a sweetener such as sugar, corn syrup or honey, salt, and a stabilizing agent like a fat with a high melting point in order to prevent the separation of oil from the solid particles. Nut paste is produced by roasting and milling of nut kernels (Wong, 2000). Tomlins and Rukuni (2007) investigated

the effect of roasting and storage time on sensory attributes of peanut butter. They concluded that the brown color roasted taste and the burnt taste might be attributed to increased roasting time. They roasted peanuts at 160°C for 40 to 55 minutes before milling. The roasting condition for the production of nut creams depends on the type of nut and the method of roasting (Shakerardekani *et al.*, 2013). Different temperature and times of roasting have been used for producing creams from various nuts by many researchers. For example, the temperature of 175°C for 30 to 60 minutes was used for roasting peanuts (Chiou *et al.*, 1991), 20 minutes at

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135°C for macadamia (Birch *et al.*, 2009), 10 to 45 minutes at 104-162°C for hazelnuts (Ozdemir *et al.*, 2001), 30 to 120 minutes at 140-189°C for cashew nut kernels (Wanlapa and Jindal 2006). Raw nuts contain lipoxygenase, which increases the oxidation of damaged kernels. This enzyme is usually destroyed on roasting, but roasting may stimulate oxidation by non-enzymatic catalysts. High content of unsaturated fatty acids in nut creams render them to oxidation (Shakerardekani *et al.* 2013). Buranasompob *et al.*, (2007) showed that heating walnuts at 60°C for ten minutes could reduce the lipoxygenase activity by 81% in comparison with unheated kernels. Other researchers studied the effect of different roasting times and temperatures on the inactivation of lipoxygenase in different nuts and kernels and showed an 80 to 100% reduction in its activity (Chu and Resurreccion, 2004; Henderson *et al.*, 1991; Kermasha and Merche, 1987; McCurdy *et al.*, 1983). The literature review showed that the roasting conditions could significantly reduce the oil separation and oxidation process of nut creams. Thus, in this study, we aimed to extend the shelf life of walnut cream by slowing its oxidation reactions and reducing oil separation. To achieve this, we optimized the temperature and time of roasting of walnut kernels used for producing walnut cream.

Materials and Methods

Materials

Walnuts were harvested from the walnut collection orchard of Agricultural and Natural Resources Research and Education Center of Semnan province, Shahrood, Iran. The husks were manually removed, and the walnuts were dried in the field by natural air to 3% moisture. Then, they were shelled and the kernels were stored at -18°C before cream production. Sugar powder and salt were purchased from Sugar Co. of Shahrood and Iran mineral salts Co., respectively.

Methods

Cream production

Walnut kernels were roasted at 100, 125 and 150°C for 10, 20 and 30 minutes by a laboratory oven (TakAzma Co., Iran). The roasted kernels were cooled down to room temperature. Then, they were placed in a home food maker, where 20% of sugar powder and 0.2% of salt were added. An easily flow paste was made by milling and mixing them for about three minutes at the highest speed of the home food maker (Shakerardakani *et al.*, 2009). The produced paste was packed in polyethylene jars and stored at 25°C and 38% RH for three months. After the storage period, the percent of oil separation, peroxide value (PV), acidity, thiobarbituric acid value (TBA) and color (a*, b* and L*) were measured.

Analysis

Oil separation percentage was determined according to the method described by Shakerardekani *et al.*, (2009). The separated and concentrated oil on top of the jars was weighted and the oil off was calculated according to the following equation:

$$\text{oil separation \%} = \frac{W_{oil}}{W_{total}} \times 100$$

W_{oil} and W_{total} are the weight of the separated oil and the total weight of the sample, respectively. Peroxide value and acidity were determined according to the standard methods proposed by the Institute of Standards and Industrial Research of Iran (ISIRI) (1992). Peroxide value was measured by titration with 0.1 N sodium hyposulfite, based on the ability of peroxides to liberate iodine from potassium iodide. TBA value was determined based on the method described by Pokorny and Dieffenbacher (1989). TBA measurement is based on the reaction of one molecule of malonaldehyde and two molecules of TBA to form a red

complex, which can be determined by spectrophotometry at 532 nm. The oil extraction of the cream was carried out according to Ziaolhagh (2013) by n-hexane solvent. The sample and solvent were mixed with a ratio of 1:3 and kept in a dry and dark place for 48 hours at room temperature. Then, the miscella was filtered and evaporated in a rotary evaporator. The remaining oil was used for measurements.

For color measurements, approximately ten grams of each sample were placed in a Petri dish and allowed to stand to remove air bubbles. A ColorPage-HR7X Slim Genius scanner was used to obtain digital images. Samples were scanned at 300 dpi resolution. Image J version 1.46 software was used for image processing. The obtained images were converted from RGB color space to LAB and then the values of a^* , b^* and L^* were measured (Abràmoff *et al.*, 2005; Lino *et al.*, 2008).

Experimental design and statistical analysis

Response Surface Methodology (RSM) was used to explore the effects of temperature and time of roasting on stability and color characteristics of walnut creams. Thirteen treatments were conducted based on the central composite design (CCD). The center point was repeated six times to calculate repeatability of the method. Design Expert 8.0.7.1 software was used for analysis. The following second order polynomial regression model was applied to express the dependent variables as a function of the independent variables.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{112} X_1^2 X_2 + \beta_{122} X_1 X_2^2$$

The low and high limits of temperature and time were chosen according to the results of preliminary experiments. Temperatures higher than 150°C and 30 minutes caused burned flavor and dark color in the walnut kernels. The kernels failed to convert to a creamy texture at temperatures lower than 100°C and times lower than ten minutes.

Results

Model fitting

The experiments were carried out according to the CCD under the defined conditions. The results were fitted to second order polynomial models by multiple regression analysis for response variables including oil separation percent, PV, acidity, TBA and color attributes (a^* , b^* and L^*). The coefficients of the regression equations describing the effect of roasting conditions on cream attributes are given in Tables 1 and 2. The adequacy of the models was determined using sum of squares, p-value, R^2 , adjusted R^2 and lack-of fit analysis. The R^2 values for these variables were 0.87, 0.90, 0.86, 0.75, 0.99, 0.81, and 0.95, respectively. Since R^2 values were higher than 0.80 (except for a^*), the obtained regression models explained the variations well. The lack of fit is a measure of the fitness of a model. Non-significant lack of fit is good. In our study, the lack of fit did not result in a significant F-value in case of all responses. This indicates that these models are sufficiently accurate for predicting those responses. In our study, the coefficient of variations (CV) were less than 10 for all responses, except for TBA (Table 1), which shows better precision and reliability of the experiments carried out.

Separation of oil

Results of the analysis of variance (Table 1) showed that the effects of the roasting temperature and time on the oil separation percent were significant. The highest oil separation was observed in creams produced from walnuts roasted at 150°C for 30 minutes (7.85%). The Model F-value of 9.59 implied the model was significant. The analysis of variance showed that the linear and quadratic effects of temperature (A) were significant. The lack of fit F-value of 3.70 implied that the lack of fit was not significant relative to the pure

error and it showed that the model could fit well the effect of roasting conditions on oil separation.

Table 1. ANOVA table for the experimental variables as a linear (A and B), quadratic (A² and B²) and interaction (AB) terms of response variables (stability parameters) and coefficients for the prediction models

Source	Df	Oil separation (%)			PV(meq/kg)			Acidity(%)			TBA(mg/kg)		
		Coefficient	SS	p-value	Coefficient	SS	p-value	Coefficient	SS	p-value	Coefficient	SS	p-value
Model	5	26.76	14.46	0.0049	20.83	1.67	0.0020	-8.38	1.40	0.0056	-0.86	0.012	0.0402
A	1	-0.401	9.86	0.0007	-0.236	0.099	0.0904	0.135	0.80	0.0014	0.2	0.00312	0.1362
B	1	-0.043	0.89	0.1291	-0.197	0.016	0.4519	0.121	0.21	0.0335	-0.02	0.000047	0.7758
AB	1	0.0012	0.39	0.2904	0.00116	0.33	0.0085	0.00059	0.088	0.1343	0.000096	0.002328	0.0752
A ²	1	0.0017	3.16	0.0143	0.00087	0.82	0.0008	-0.00043	0.20	0.0360	-0.000085	0.007739	0.0066
B ²	1	-0.0019	0.099	0.5855	0.00145	0.058	0.1762	-0.00072	0.014	0.5156	0.000198	0.001084	0.1971
Residual	7		2.11			0.18			0.21			0.003737	
Lack of Fit	3		1.55	0.1194		0.12	0.1892		0.046	0.7845		0.000752	0.8015
Pure Error	4		0.56			0.061			0.17			0.002985	
Total	12		16.58			1.85			1.62			0.015	
R ²		0.8726			0.9032			0.8677			0.7568		
Adj-R ²		0.7816			0.8340			0.7732			0.5831		
CV		10.54			3.33			7.83			11.51		

A=roasting temperature, B=roasting time, SS=sum of squares, Number of replications:3

Table 2. ANOVA table for the experimental variables as a linear (A and B), quadratic (A² and B²) and interaction (AB) terms of response variables (color parameters) and coefficients for the prediction models

Source	Df	a*			b*			L*				
		Coefficient	SS	p-value	Coefficient	SS	p-value	Coefficient	SS	p-value		
Model	7	-3.33	76.18	< 0.0001	6	23.608	8.70	0.0445	5	-70.65	654.20	0.0001
A	1	0.192	12.56	< 0.0001	1	0.1545	2.96	0.0229	1	2.085	380.82	< 0.0001
B	1	1.58	2.16	0.0006	1	-1.939	1.57	0.0692	1	2.137	96.18	0.0020
AB	1	-0.035	14.46	< 0.0001	1	0.018	0.00019	0.9814	1	-0.0185	85.65	0.0028
A ²	1	0.00123	8.04	< 0.0001	1	-0.00114	1.41	0.0808	1	-0.00813	71.40	0.0045
B ²	1	0.016	0.26	0.0429	1	0.049	1.15	0.1071	1	-0.00559	0.86	0.6639
A ² B	1	0.000198	2.04	0.0006								
AB ²	1	-0.000157	0.20	0.0618	1	-0.000449	1.68	0.0621				
Residual	5		0.18		6	1.93			7		29.44	
Lack of Fit	1		0.033	0.3913	2	1.07	0.1968		3		22.15	0.1050
Pure Error	4		0.14		4	0.85			4		7.29	
Total	12		76.36		12	10.62			12		683.64	
R ²		0.9977			0.8187				0.9569			
CV		3.45			2.02				3.76			

A=roasting temperature, B=roasting time, SS=sum of squares, Number of replications:3

Fig. 1A shows that the oil separation in the samples was increased as the roasting temperature

increased from 100 to 150°C, with the highest at 150°C.

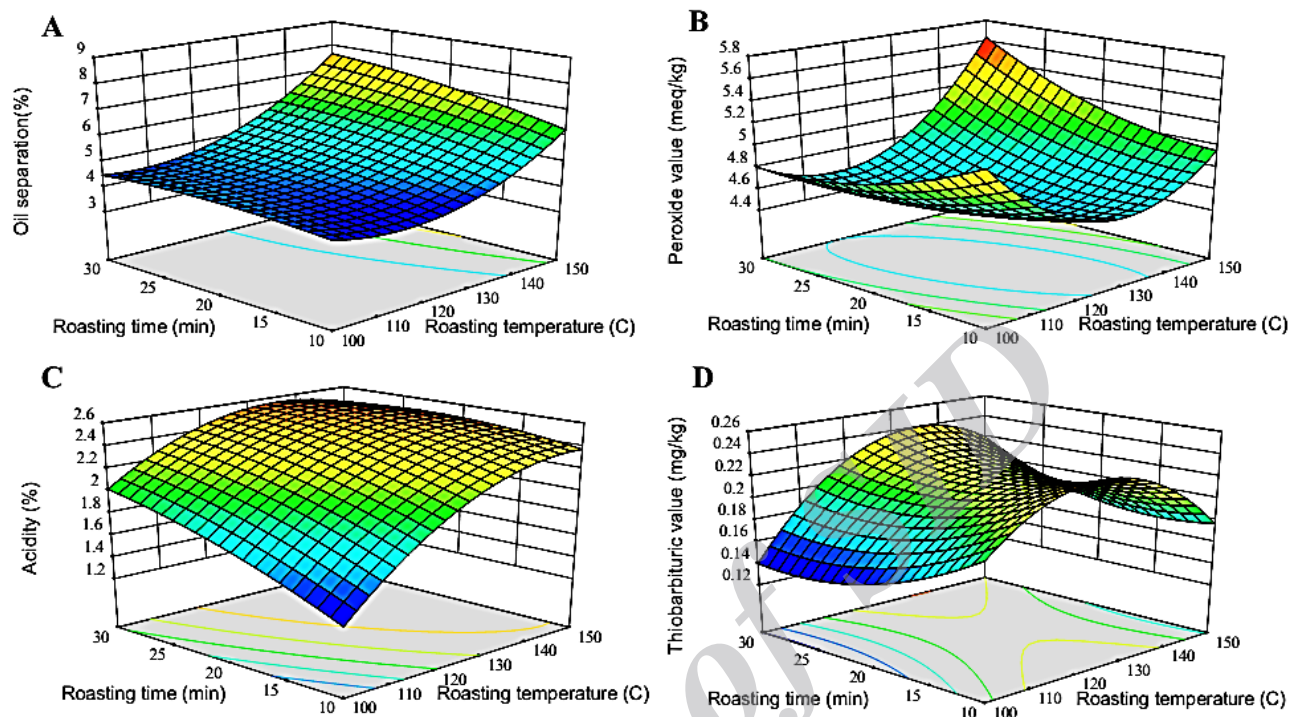


Fig. 1. Response surface of (A) oil separation (%), (B) Peroxide value (meq/kg), (C) acidity (%) and (D) Thiobarbituric acid value (mg/kg) of walnut creams as a function of roasting temperature and time.

Peroxide value

As shown in Table 1, the F value of the model is 13.06, which shows that the model is significant. The analysis of variance showed that the quadratic effect of temperature (A) and interaction effect of roasting temperature and time (AB) at 99% on the peroxide value were significant. The lack of fit of 2.6 implied that it was not significant. Therefore, the model could appropriately fit the effect of temperature and time on peroxide value.

As Fig. 1B shows, the temperature of 150°C for 30 minutes and the temperature of 100°C for 10 minutes caused the highest PV for the samples. The lowest peroxide value was related to 125°C and 20 minutes. These results showed that the peroxide value decreased as the temperature of roasting increased to 125°C, but at temperatures higher than 125°C, PV began to increase to reach its highest value at 150°C.

Acidity

As shown in Table 1, the F value of the model is 9.18, which shows that the model is significant. There is only a 0.56 % chance that this F value could occur due to noise. The analysis of variance showed that the linear effect of temperature at 99% and the quadratic effect of temperature and linear effect of time at 95% on the acidity were significant. The lack of fit of 0.36 implied that it was not significant, and the model could appropriately fit the effect of temperature and time on acidity. The acidity of the samples increased with the roasting temperature and time. The lowest acidity was observed for cream produced from walnuts roasted at 100°C for 10 minutes, but the highest acidity was determined for those roasted at 125°C for 30 minutes (Fig. 1C).

Thiobarbituric acid (TBA) value

The model F value for this attribute was 4.36, meaning that the model was significant. The analysis of variance (Table 1) showed that only the squared effect of temperature on TBA was significant. The lack of fit of 0.34 showed that it was not significant. TBA value is the amount of malonaldehyde in 1000 grams of oil. Its measurement is an auxiliary method for other methods such as peroxide value and acidity. This value does not determine the initial stages of oxidation (Samad Louei *et al.*, 2007). It can be seen from Fig. 1 that the TBA value has increased as the temperature increased. It can be concluded that secondary oxidation compounds were produced by increasing the temperature. The highest malonaldehyde values were recorded for samples roasted at 125°C for 20 minutes. Samples roasted at 100°C for 20 minutes showed the lowest value for TBA. As it can be seen from Fig.1D, the TBA value of

samples were decreased as the roasting temperature increased from 125 to 150°C. This result is probably due to the destruction of malonaldehydes at higher temperatures.

Color

The quadratic models with F values of 305.21, 4.52 and 31.11 were obtained for a^* , b^* and L^* values of roasted walnuts, respectively. The analysis of variance showed that the linear, quadratic and interaction effects of temperature and time on a^* and the effect of all factors except for quadratic effect of temperature on L^* were significant. Table 2 shows that only the effect of roasting temperature on the b^* was significant. In our study, it was shown that a^* value (redness) and b^* value (yellowness) increased and the L^* value (lightness) decreased with temperature (Fig. 2).

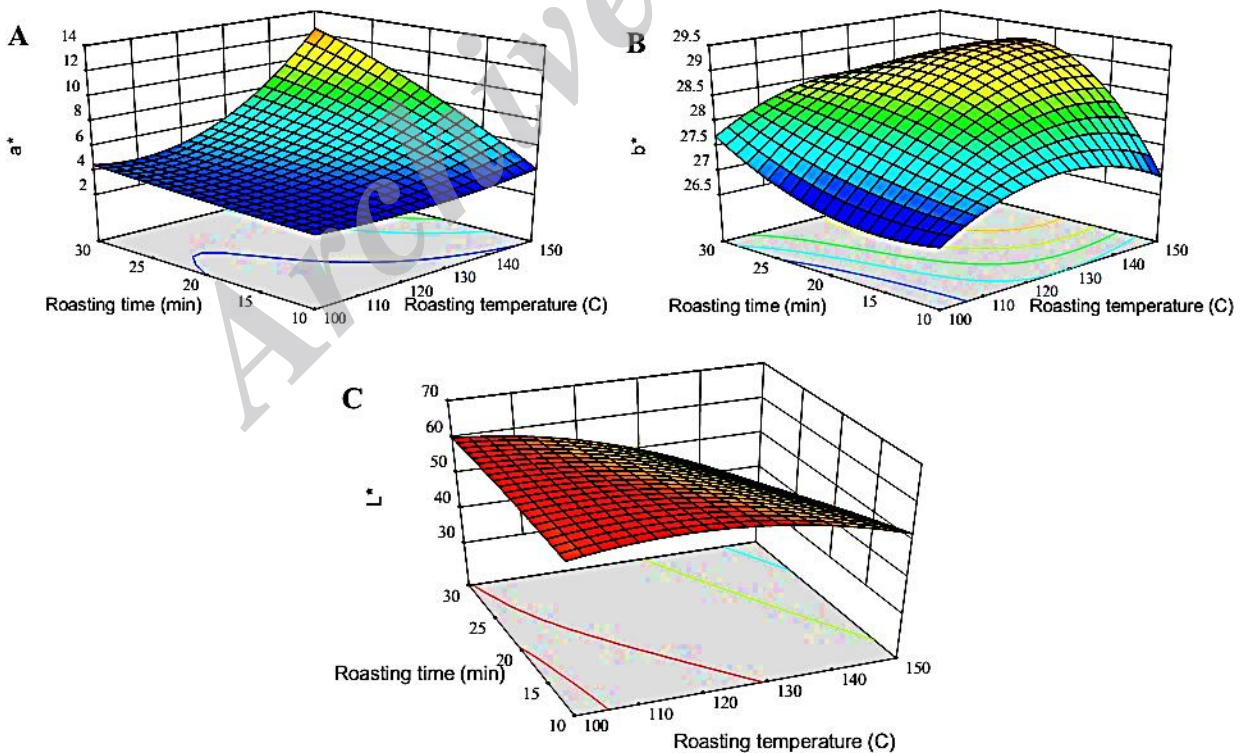


Fig. 2. Response surface of (A) a^* , (B) b^* and (C) L^* of walnut cream as a function of roasting temperature and time.

Optimization and validation

Optimum values for independent variables were obtained using Design Expert software. The desired properties of produced cream, the oil separation, acidity, PV, TBA and redness (a^*) should be minimum, while whiteness (L^*) and yellowness (b^*) should be maximum. According to the RSM analysis, temperature of 116°C and time of 12 minutes were considered as the optimized conditions for roasting of walnut kernels with the desirability of 0.73. The criteria and desirability function response surface for this optimization are shown in Fig. 3. The desirability is reduced as the roasting temperature and time deviate from 116°C

and 12 minutes, respectively. To validate the optimization process, we produced the walnut cream using the optimized temperature and time of roasting at three replications. After three months of storage at room temperature, we measured the previously mentioned properties. The results were compared with the predicted values using SPSS 19 by one sample t-test. The t-test results showed that there were no significant difference between the measured and predicted values. Thus, it was shown that our optimization of roasting conditions could be used for the production of walnut cream.

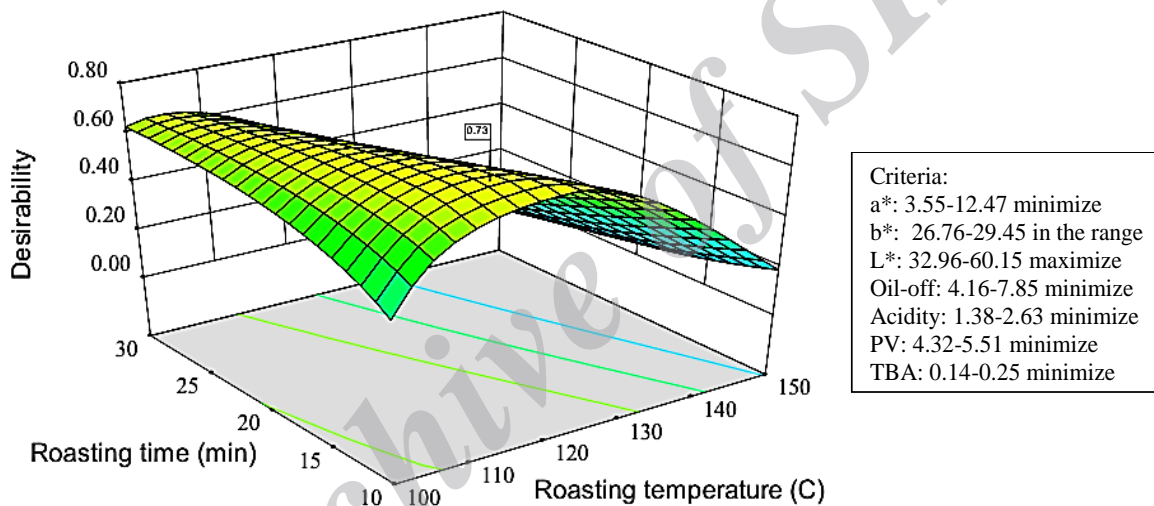


Fig 3. The desirability function response surface for process optimization along with the used criteria

Discussion

The different specific gravities of solid particles and oil in cream samples caused the oil to separate from the solids portion (Muego-Gnanasekharan and Resurreccion 1992). Gu *et al.*, (2009) showed that heating the emulsion gels containing soy and sun flower oils at 95°C for ten minutes entraps more oil in the gel network. The oil absorption capacity involves bonding of oil to nonpolar amino acids in the side chains of proteins. Ciftci *et al.*, (2008) and Ereifej *et al.*, (2005) showed that the colloidal stability of sesame paste decreased as the

temperature increased, which is likely due to the lower viscosity of oils at higher temperatures. Higher temperatures affect the microstructure of nut pastes, which causes the increased separation of oil (Aryana, 2000). In this study, the amount of separated oil from the walnut cream roasted at higher temperatures was more than those roasted at lower temperatures during storage. This observation was thought to be due to the effect of higher temperatures on the microstructure of the cream and the viscosity of walnut oil.

The oxidative effects of roasting is due to the destruction of natural antioxidants, breakdown of fatty acids and cell physical changes (Nikzadeh and Sedaghat, 2008). The free fatty acids increased with roasting temperature of hazelnut up to 120°C and then decreased. This may be related to the hydrolytic inactivation of enzymes (Ozdemir *et al.*, 2001). Luh *et al.*, (1981) showed that roasting of pistachios at 150°C for 30 minutes decreased fatty acids. Heating of walnut kernels at 60°C for ten minutes decreased enzyme activity by 81% compared to unheated samples (Buranasompob *et al.*, 2007).

Kashani and Valadon (1983) indicated the significant effect of roasting on peroxide value of pistachios. After roasting, the PV increased due to destructive reactions in its oil. Ozdemir *et al.* (2001) demonstrated that PV of hazelnuts decreased as the roasting temperature increased, but at 158°C or more, it increased significantly. They concluded that although roasting decreased the lipase activity and facilitated peroxidase activity, esterases are heat stable and may be active even after roasting. Generally, this enzyme destroys during roasting, but after that non-enzymatic catalysts could initiate oxidation. The high content of unsaturated fatty acids in nut creams make them susceptible to oxidation. Metaloproteins along with Fe and Cu salts are the main catalysts of nut cream oxidation (Shakerardekani *et al.*, 2013). Buranasompob *et al.*, (2001) found that the peroxide value of unheated walnut kernels were significantly higher than that of heated walnuts. Branch *et al.* (1987) reported that the peroxide value and acid number of oils extracted from heated peanuts were higher than those of unheated peanuts.

Roasting could affect the browning of walnuts due to the increasment in reducing sugar content. The higher reducing sugar concentration causes the color to change to brown, and L* value to decrease (Wall and Gentry, 2007). According to a study by Yeh *et al.*, (2002), the

L* value of peanut spreads decreased significantly as the temperature increased, while a* and b* increased with temperature. Kahyaoglu and Kaya (2006) indicated that temperature and time of roasting significantly affected the color values of sesame. They showed that during roasting there was an initial lightening due to low moisture content, denaturation of proteins and the concentration of oil particles in protein matrix. As the roasting temperature increased, the L* decreased by time. They also showed that b* and a* were increased with increasing roasting time and temperature. Birch *et al.*, (2009) reported that roasting macadamia for longer times caused the lightness (L*) to decrease, but a* and b* were increased as the the roasting time increased from 0 to 30 minutes. For roasted pistachios, the L* decreased by roasting time and temperature. As the roasting time and temperature increased, the a* and b* values of roasted pistachios increased and decreased, respectively (Kahyaoglu, 2008).

Conclusions

This study confirmed that the studied cream characteristics were significantly influenced by the time and temperature of roasting. The RSM analysis showed that the oil separation increased at higher temperatures and longer times of roasting. At lower temperature and shorter times, the oxidation of cream was higher. The optimized conditions of roasting walnut kernels to produce oxidative stable walnut cream with the lowest oil separation were determined as 116°C for 12 minutes. Thus, it is important to use the most appropriate temperature and time for roasting nut kernels before formulation of any nut cream, as they have considerable effects on the characteristics and shelf life the cream.

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بهینه‌سازی فرآیند برشته کردن مغز گردو جهت تولید کرم گردو با استفاده از روش سطح پاسخ

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چکیده

فرآیند برشته کردن مغزهای آجیلی بر کیفیت کرم تولیدی از آن‌ها تأثیر زیادی دارد. در این بررسی، شرایط برشته کردن مغز گردو با در نظر گرفتن عوامل مؤثر بر پایداری کرم تولید شده از آن بهینه‌سازی شده است. در این پژوهش برای برشته کردن مغزهای گردو از دماهای بین ۱۰۰ تا ۱۵۰ درجه سانتی‌گراد و برای مدت زمان بین ۱۰ تا ۳۰ دقیقه استفاده شد. مقدار روغن جدا شده از کرم، عدد پراکسید، عدد اسیدی و عدد تیوباربتوریک اسید و همچنین پارامترهای رنگ نمونه‌های تولید شده، پس از سه ماه نگهداری در دمای ۲۵ درجه سانتی‌گراد تعیین شدند. نتایج نشان داد که میزان جدا شدن روغن از کرم با افزایش دمای برشته کردن افزایش می‌یابد (از ۴/۱۶ درصد در ۱۰۰ درجه سانتی‌گراد به مدت ۱۰ دقیقه تا ۷/۸۵ درصد در ۱۵۰ درجه سانتی‌گراد به مدت ۳۰ دقیقه). عددهای پراکسید، اسیدی و تیوباربتوریک اسید نمونه‌ها به شدت تحت تأثیر دما و زمان برشته کردن قرار گرفتند. همچنین، با افزایش دما، بر میزان قرمزی و زردی رنگ کرم‌ها افزوده، و از میزان روشنی رنگ نمونه‌ها کاسته شد. در نهایت، نشان داده شد که دمای ۱۱۶ درجه سانتی‌گراد به مدت ۱۰ دقیقه بهترین شرایط برشته کردن برای تولید کرم گردو می‌باشند.

واژه‌های کلیدی: برشته کردن، بهینه سازی، پخشینه گردو، جدا شدن روغن، کرم گردو.