## COLOUR CHANGE AND WEIGHT LOSS DURING THE ROASTING PROCESS FOR PRODUCTION OF CAROB POWDER

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#### Abstract

The effect of different roasting times (0, 5, 10, 15, 20, 30, 45, 60, 75 and 90 min) and temperatures (135, 150 and 165 °C) on the weight loss and apparent colour change of carob powder was investigated. The roasting time and temperature significantly (P<0.05) affected the colour parameters L and b and weight loss of the samples. However, the colour parameter a was only affected by the roasting time. The total colour difference increased by about 60 % when the temperature was increased from 135 to 165°C. Furthermore, the significant changes in weight loss (WL) and colour parameters were simulated using three-dimensional models. The changes in L were well described by the paraboloid model, whereas the Gaussian model was more suitable to describe the weight loss and total colour difference of the carob powder.

Keywords: Ceratonia siliqua L., carob powder, roasting, weight loss, colour

## KEÇİBOYNUZU TOZU ÜRETİMİNDE KAVURMA PROSESİ SÜRESİNCE RENK DEĞİŞİMİ VE AĞIRLIK KAYBI

#### Öz

Farklı kavurma süreleri (0, 5, 10, 15, 20, 30, 45, 60, 75 ve 90 dakika) ve sıcaklıklarının (135, 150 ve 165°C) keçiboynuzu tozunun görünür renk değişimi ve ağırlık kaybı üzerine etkisi araştırılmıştır. Kavurma süresi ve sıcaklığı örneklerin renk parametrelerinden olan L ve b değerleri ile ağırlık kaybın önemli derecede (P<0.05) etkilemiştir. Ancak, renk parametrelerinden a değeri sadece kavurma süresinden etkilenmiştir. Sıcaklığın 135'den 165°C'ye artması ile toplam renk değişiminde %60'lık bir artış görülmüştür. Ayrıca ağırlık kaybı ve renk parametrelerindeki önemli değişikliklerin üç boyutlu modellere uygunluğu test edilmiştir. Keçiboynuzu tozunun ağırlık kaybı ve toplam renk değişiminin tanımlanmasında Gaussian modeli en uygun model olarak belirlenirken, paraboloid model ise renk parametrelerindeki değişim için en uygun model olarak belirlenmiştir.

Anahtar kelimeler: Ceratonia siliqua L., keçiboynuzu tozu, kavurma, ağırlık kaybı, renk

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#### INTRODUCTION

Carob is a fruit of the perennial leguminous tree Ceratonia siliqua L., which is native to the Mediterranean basin and Southwest Asia (1). The dry fruit mass of carob consists of 90 % pod and 10% seeds (2, 3). The seeds are used to produce locust bean gum; mainly used in the food industry (4). The pod of carob is rich in sugars (40-60 %, mostly sucrose), D-pinitol, minerals, dietary fibre and polyphenols (1, 5-10). Pods are utilized as a processed carob syrup (pekmez in Turkish), carob powder for human consumption (1, 11, 12) as well as animal feed. The carob powder produced by roasting and milling of the carob kibbles is mainly used as a substitute or extender of cacao in baking and chocolate manufacturing (13-15). In Turkey, carob powder is also consumed as a hot drink like Turkish coffee.

During the production of carob powder, kibbled carob is subjected to high roasting temperatures for variable times depending on the type of roaster used and the desired characteristics of final product, i.e. lightly, medium or highly roasted carob powder. In conventional roasting, a temperature of 120-180 °C is used for 10 to 60 minutes (16). Moisture loss and important chemical reactions, including sugar caramelisation and/or the Maillard reaction takes place during the roasting. These changes significantly affect the overall product quality and palatability (14). The food industry can therefore control some important parameters i.e. physical (weight loss and density), chemical (Maillard reaction) and organoleptic (colour, texture, aroma and flavour) to obtain the desired quality of their products (17-22).

Monitoring physical properties of foods is relatively easy and rapid as compared to the chemical properties while roasting. The physical changes, mostly using a kinetic approach with regression analysis during the roasting of certain foods, such as coffee (17, 21, 23), cocoa (24) and nuts (25-29), have been studied by different researchers to optimise the process. Three-dimensional (3D) modelling has also been studied to understand the colour changes during roasting of hazelnuts (30), sesame seeds (18) and pistachio nuts (31).

Per the best of our literature knowledge, there are no reports that present the information regarding the effects of roasting conditions on physical parameters, such as colour and weight loss of carob powder. The current study was aimed to investigate the effects of temperature and time of the roasting process on weight loss and colour parameters (L, a, b and total colour difference TCD) of the carob powder. Furthermore, the compatibility of different mathematical models for expressing the observed changes during roasting was also studied to predict colour changes and weight loss of carob powder.

### **MATERIALS AND METHODS**

#### Roasting

The carob sample was obtained from a local producer in Antalya, Turkey. Kibbled carob samples (~8 x 10 mm size) were weighed in glass plates and placed in an oven (Model UM 500, Memmert, Schwabachi, Germany, equipped with fan) at various temperatures i.e. 135, 150 and 165∞C and time intervals i.e. 0, 5, 10, 15, 20, 30, 45, 60, 75 and 90 minutes to roast them. After cooling to room temperature, the roasted samples were grinded in a mill (Model MKM 6000, Bosch, Leipzig, Germany) and filtered (60 mesh, VIBRO, Retsch, Haan, Germany) to obtain fine powders which were stored in polyethylene bags at 4°C for further analysis.

#### Analyses

Moisture determination of the samples was carried out according to Nicoli et al. (2006) (32). Weight loss (g/100 g) was calculated from the weight of carob samples before ( $W_0$ ) and after roasting (W) using equation 1:

$$WL = 100(W_0 - W) / W_0 \tag{1}$$

Colour analyses of the carob powder samples were carried out using a colour meter (model Chroma Meter CR-400, Konica-Minolta Sensing Inc., Osaka, Japan). Colour parameters were expressed as L (darkness/whiteness), a (greenness/ redness) and b (blueness/yellowness) on the Hunter scale. The total colour difference (TCD) was calculated using equation 2:

TCD = 
$$\sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2}$$
 (2)

where  $L_0$ ,  $a_0$  and  $b_0$  are the colour values of the unroasted carob powder (32, 33). The instrument was standardised against a white tile (L = 97.02, a = 0.08, and b = 1.75) before each measurement. Each colour value was obtained as the mean of three measurements.

#### Statistical analysis

The experiment was set up as a randomised plot with a factorial design in the roasting temperature and the roasting time (3x10) in triplicate. The data were then subjected to variance analysis, and appropriate mean separation was conducted using Duncan's multiple-range test in the SAS System for Windows V7 software (SAS, Institute Inc., Cary, NC, USA).

#### Modelling of the weight loss and colour change

The significant changes in the weight loss and colour parameters were plotted as a function of roasting time and temperature. The following plane, paraboloid, Gaussian and Lorentzian non-linear equations (Eq. 3–6) were employed for 3D modelling using the Sigma Plot (9.0) data processing program:

$$y = y_0 + aT + bt \tag{3}$$

$$y = y_0 + aT + bt + cT^2 + dt^2$$
(4)

$$y = ae^{\left(-\frac{1}{2}\left(\left((T - T_0)/b\right)^2 + \left((t - t_0)/c\right)^2\right)\right)}$$
(5)

$$y = \frac{a}{((1 + ((T - T_0) / b)^2)(1 + ((t - t_0) / c)^2)))}$$
(6)

where y is the dependent variable (weight loss and colour parameters);  $y_0$ ,  $T_0$  and  $t_0$  are intercepts; *a*, *b*, *c* and *d* are regression parameters; and *T* and *t* are the independent variables of temperature (°C) and time (min).

#### **RESULTS AND DISCUSSION**

# Weight loss and colour parameter changes of carob powder

The average moisture content of unroasted kibbled carob powder samples was 8.03 g/100 g that reduced to 1.92, 1.37 and 0.90 g/100 g after 90 min of roasting at 135, 150 and 165°C, respectively. These moisture values were found to be lower than those reported by Yousif and Alghzawi (14) but similar with our previous data (16). Either a difference in the raw material characteristics and roasting conditions or different ambient conditions could cause differences in the moisture contents of the samples.

As expected, both loss of moisture and heatinduced reaction products, such as CO2 and organic components (sugar and protein), during roasting (17, 18, 23) caused a decrease in the weight, and thus the WL value of the samples increased. Figure 1 shows the behaviour of weight loss of the samples at temperatures of 135, 150 and 165 °C. The changes in WL values during the roasting process at different temperatures and times were found to be significant (P<0.01; Table 1). Higher WL values of the carob powder were found at higher roasting temperatures and longer roasting times (Fig. 1). At tested roasting temperatures, the observed change in the WL values of the carob samples was almost linear at roasting times from 0 to 20 min followed by partly non-linear deceleration behaviour of samples roasted at 135°C and 150°C. The WL mean values of the carob powders at different roasting temperatures and times are given in Table 2 and Table 3, respectively. The WL values of the samples increased by 36.32 % when the roasting temperature was increased from 135 to 165°C while the WL mean value was found as 18.25 % after 90 min of roasting. The current findings related to the WL behaviour of carob powder were like few previous studies on coffee beans (17, 23) where a quasi-linear behaviour was followed by a noticeable deceleration.

Table 1. Variance analyses of roasting variables on quality parameters of the carob powder

Source of		WL	WL L		а		b		TCD		
Variation	DF	MS	F	MS	F	MS	F	MS	F	MS	F
RT	2	111.71	1050.55*	504.07	387.51*	0.84	2.41 <sup>№</sup>	72.20	57.67*	572.82	262.67*
RP	9	384.98	3620.36*	502.50	386.30*	5.76	16.49*	108.88	86.97*	565.97	259.53*
RT x RP	18	14.22	133.72*	68.45	52.62*	1.16	3.33*	22.09	17.64*	82.05	37.63*
Error	60	0.11		1.30		0.35		1.25		2.18	

\*: significance at P<0.01 NS: not significant; RT: roasting temperature; RP: roasting period

Roasting Temperature (°C)	WL (%)	L	а	b	TCD
135	6.52° ± 0.87	$56.86^{a} \pm 0.66$	11.01 ± 0.21	$20.04^{a} \pm 0.28$	5.93° ± 0.68
150	7.48 <sup>♭</sup> ± 1.02	54.72 <sup>₅</sup> ± 1.10	10.77 ± 0.17	18.32⁵ ± 0.57	8.34⁵ ± 1.17
165	$10.24^{a} \pm 1.58$	$48.94^{\circ} \pm 2.25$	11.09 ± 0.19	16.95° ± 1.13	14.41ª ± 2.42

Table 2. Effects of roasting temperatures on quality parameters of the carob powder

Values are mean ± standard error, N=30

Values in a column followed by different superscript letters are significantly (P < 0.05) different (Duncan's multiple-range test)

Roasting Periods (min)	WL (%)	L	а	b	TCD
0	$0.00^{i} \pm 0.00$	62.08 <sup>ª</sup> ± 0.30	8.88 <sup>d</sup> ± 0.07	20.78ª ± 0.10	$0.00^{h} \pm 0.00$
5	$1.56^{\circ} \pm 0.10$	60.04 <sup>b</sup> ± 0.37	10.96 <sup>bc</sup> ± 0.34	21.16ª ± 0.41	$3.38^{9} \pm 0.36$
10	2.76 <sup>h</sup> ± 0.15	59.02 <sup>bc</sup> ± 0.28	10.59° ± 0.23	20.95ª ± 0.48	3.82 <sup>g</sup> ± 0.32
15	4.05 <sup>9</sup> ± 0.17	58.07 <sup>cd</sup> ± 0.34	10.91 <sup>bc</sup> ± 0.28	20.86 <sup>ª</sup> ± 0.49	4.79 <sup>fg</sup> ± 0.32
20	5.25 <sup>t</sup> ± 0.18	57.15⁴ ± 0.30	11.32 <sup>ab</sup> ± 0.14	$20.90^{a} \pm 0.39$	5.66 <sup>ef</sup> ± 0.33
30	7.11° ± 0.35	55.95° ± 0.34	11.24 <sup>ab</sup> ± 0.25	20.33ª ± 0.55	6.81° ± 0.55
45	11.11 <sup>d</sup> ± 0.69	51.88 <sup>t</sup> ± 1.67	$11.68^{a} \pm 0.28$	18.28 <sup>♭</sup> ± 0.76	11.02 <sup>d</sup> ± 1.79
60	13.66° ± 0.97	47.22 <sup>g</sup> ± 2.77	11.64ª ± 0.16	15.82° ± 1.22	16.01° ± 3.00
75	17.02 <sup>₅</sup> ± 1.50	42.60 <sup>h</sup> ± 3.27	11.18 <sup>abc</sup> ± 0.28	12.92⁴ ± 1.58	21.25 <sup>₅</sup> ± 3.55
90	18.25ª ± 1.69	41.07 <sup>i</sup> ± 3.22	$11.17^{\text{abc}} \pm 0.31$	12.37⁴ ± 1.70	$22.89^{a} \pm 3.56$

Table 3. Effects of roasting time on quality parameters of the carob powder

Values are mean ± standard error, N=9

Values in a column followed by different superscript letters are significantly (P < 0.05) different (Duncan's multiple-range test)



Fig. 1 Changes in the weight loss (WL) value of the carob powder obtained by the roasting processes at different times and temperatures

Figures 2–4 illustrate the colour changes in the carob powder during the roasting process at 135, 150 and 165°C. The roasting temperature did not cause a noticeable effect on the colour parameter values of the samples during the initial roasting period while a different behaviour was observed in the subsequent roasting period. The higher the roasting temperature caused a greater change in colour. The colour parameters *L* and *b* were significantly (*P*<0.01) decreased by the roasting process. When the temperature was increased from 135 to 165°C, the *L* and *b* values decreased by 13.93 % and 15.42 %, respectively. A similar behaviour in these colour parameters was also

reported by Kahyaoglu (31) while roasting pistachio nuts. However, an increment in the L values was reported in hazelnut (28), sesame (18), coffee (32) and peanut (34) during the initial period of the roasting process. Kahyaoglu and Kaya (18) reported that low moisture content, denaturation of protein and a high concentration of oil particles embedded in the protein matrix could cause an increase in the L value during the initial period of roasting. Considering these explanations, the decrease in the L values in the present study can be related to the low oil content of the carob pod (12) rather than a low moisture content or denaturation of protein.



Fig. 2 Changes in the L value of carob powder obtained by the roasting processes at different times and temperatures



Fig. 3 Changes in the b value of carob powder obtained by the roasting processes at different times and temperatures



Fig. 4 Changes in the total color difference (*TCD*) value of carob powder obtained by the roasting processes at different times and temperatures

The colour *a* value did not significantly change by an increase in roasting temperature but roasting time was positively correlated with the colour a value (Tables 2 and 3). In fact, the colour *a* value of the samples increased by around 20 % during the 90 min of roasting time. The *TCD* of the samples significantly increased at higher temperatures and longer roasting time (Table 2). There was 60% increase when the temperature was increased from 135 to 165°C. That increase in the *TCD* was almost fivefold between 30 and 75 min roasting time at 165°C, which implies that the chemical reactions that result in the colour change of the carob powder particularly accelerate in this time interval. The light red-brownish colour of the unroasted carob changes to dark brown after roasting due to Maillard reactions and caramelization of the compounds in carob powder (16).

# Modelling of the weight loss and colour parameters during roasting

In the present study, non-linear regression analysis was employed by using 3D equations (Eq. 3–6) to model the weight loss and changes in colour parameters as a function of the roasting temperature and time. The details of the 3D non-linear equations employed (plane, paraboloid, Gaussian and Lorentzian) were reported by Şimşek (30).

The coefficient of determinations  $(R^2)$  and standard error of estimations (SEE) of the tested non-linear equations are given in Table 4. The  $R^2$ values, which describe the predictive models for the dependent variables were 70-98 %. The maximum  $R^2$  and minimum SEE values were used for the degree of fitting of the experimental data to the prescribed models. The changes in the WL values of carob powder were plotted in the Gaussian model (Eq. 5;  $R^2 = 0.976$ ). The partial regression parameters were predicted by fitting the experimental data of the WL parameters in the Gaussian model (Table 5). The predicted WL function of the carob powder is plotted in Figure 5. It is clear from the plot that the WL parameters are behaving in a sigmoidal way for roasting time and in a curvilinear way for temperature.

eristic	Plane	Paraboloid	Gaussian	Lorentzian				
R²	0.922	0.933	0.976*	0.944				
SEE	1.947	1.876	1.133	1.720				
R <sup>e</sup>	0.801	0.811*	0.771	0.772				
SEE	4.076	4.128	4.542	4.539				
R²	0.695	0.717*	0.679	0.684				
SEE	2.393	2.395	2.554	0.532				
R²	0.789	0.799	0.973*	0.966				
SEE	4.480	4.552	1.678	1.874				
	eristic R <sup>e</sup> SEE R <sup>e</sup> SEE R <sup>e</sup> SEE R <sup>e</sup> SEE R <sup>e</sup> SEE	eristic Plane $R^{e}$ 0.922   SEE 1.947 $R^{e}$ 0.801   SEE 4.076 $R^{e}$ 0.695   SEE 2.393 $R^{e}$ 0.789   SEE 4.480	eristic Plane Paraboloid $R^{e}$ 0.922 0.933   SEE 1.947 1.876 $R^{e}$ 0.801 0.811*   SEE 4.076 4.128 $R^{e}$ 0.695 0.717*   SEE 2.393 2.395 $R^{e}$ 0.789 0.799   SEE 4.480 4.552	eristicPlaneParaboloidGaussian $R^e$ 0.9220.9330.976*SEE1.9471.8761.133 $R^e$ 0.8010.811*0.771SEE4.0764.1284.542 $R^e$ 0.6950.717*0.679SEE2.3932.3952.554 $R^e$ 0.7890.7990.973*SEE4.4804.5521.678				

Table 4. The values of regression coeffient (R<sup>2</sup>) and standard error of estimation (SEE) for 3D models.

\* The coefficient ( $R^2$ ) was preferred for mathematical modeling of the quality parameters.

Quality Characteristics	3D	PRC	E	SE	Р	
WL	Gaussian	T <sub>0</sub>	1087.4	19492	0.9560	( <i>R</i> <sup>2</sup> = 0.976)
		$t_0$	87.4	3.836	<0.0001	(SEE=1.133)
		a	88490.0	15647152	0.9955	
		b	227.1	2364	0.9243	
		С	40.1	2.5	<0.0001	
L	Paraboloid	Уо	-78.98	159.09	0.624	( <i>R</i> <sup>2</sup> = 0.811)
		а	2.16	2.13	0.322	(SEE=4.128)
		b	-0.222	0.10	0.033	
		С	-0.01	0.01	0.267	
		d	-0.002	0.001	0.865	
0	Paraboloid	Уо	53.87	92.29	0.565	( <i>R</i> <sup>2</sup> = 0.717)
		а	-0.33	1.24	0.791	( <i>SE</i> E=2.395)
		b	-0.03	0.06	0.588	
		С	0.0008	0.0041	0.855	
		d	-0.0009	0.0006	0.177	
TCD	Gaussian	T <sub>0</sub>	825.8	7118.7	0.9086	( <i>R</i> <sup>2</sup> = 0.973)
		t <sub>o</sub>	93.5	5.5	<0.0001	( <i>SEE</i> =1.678)
		а	6816706	907261779	0.9941	
		b	134.1	709.5	0.8516	
		С	40.5	3.2	<0.0001	

Table 5. Mathematical models of the quality parameters of the roasted carob powder

PRC, Partial regression coefficientE, Estimated value of PRC



Fig. 5 3-D plot of the weight loss (WL) of the carob powder

The changes in *L* and *b* values were obtained from the paraboloid model (Eq. 3) by using the highest  $R^2$  and the lowest *SEE* values (Table 4). The paraboloid model that describes the changes in the *L* values and the predicted *L* function of the carob powder is shown in Figure 6. Although the highest value of  $R^2$  was used, the paraboloid model (Fig. 7) still did not significantly correspond to the changes in the *b* value (Table 5). Şimşek (30) reported that the changes in the *L* and *b* values of hazelnuts during the roasting process agreed with the paraboloid model.



Fig. 6 3-D plot of the L value of the carob powder

The experimental data of *TCD* did fit ( $R^2 = 0.973$ ) in the Gaussian model. The predicted values of the partial regression coefficient of the Gaussian model and the surface function of the *TCD* values are shown in Table 5 and Figure 8, respectively. Considering the fitting quality of the current experimental data to the Gaussian model, the *TCD*, which comprises all colour coordinates, can be used for roasting of carob in terms of colour quality.



Fig. 7 3-D plot of the b value of the carob powder

### CONCLUSION

This work presents the weight loss and the change in colour parameters (L, a, b and TCD) of carob powder during different roasting conditions. The results showed that roasting temperature affected the weight loss and all the colour parameters except colour *a* value. The roasting time was a key factor in determining the overall quality of the product due to its significant (P < 0.01) effects on all controlled parameters. It was also observed that the heat-induced reactions, which lead to significant changes in the weight loss and colour parameters of the carob powders, accelerates during 30-75 min of roasting time at all tested temperatures. Therefore, the optimum roasting degree can be standardized between 30 and 75 min of roasting time. The 3D non-linear equations were also used successfully to predict the potential changes during roasting. The changes in the L values of the carob powder were well described by the paraboloid model while the Gaussian model was found to be the best for describing the changes in the WL and TCD values of the carob powder.

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Fig. 8 3-D plot of the TCD value of the carob powder

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