Application of response surface methodology for optimisation of Cornelian cherry - Capia pepper leather dried in a heat pump drying system

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Abstract

The heat pump drying system was optimised for cornelian cherry-capia pepper leather production development using response surface methodology. The central composite design was used to optimise the process parameters in terms of drying time, coefficient of the performance of heat pump, coefficient of the performance of the whole system, specific moisture extracted ratio, energy consumption, drying rate and colour values. The optimal condition of independent variables was obtained as a cornelian cherry pulp concentration of 47.419% and drying temperature of 33.574°C with composite desirability of 0.846. Moreover, hydroxymethylfurfural (HMF) and effective moisture diffusivity (D_{eff}) values of all runs were analysed. HMF was not determined in cornelian cherry-capia pepper leather. D_{eff} values of cornelian cherry-capia pepper leather were between 1.026×10^{-9} - 1.532×10^{-9} m^2s^{-1}. The drying behaviour of cornelian cherry-capia pepper leather with optimal conditions acquired with the central composite design was evaluated with seven thin-layer drying models. The statistical parameters based on R^2, root mean square of error and χ^2 values were determined between 0.8267 to 0.9845, 0.004087 to 0.035626 and 0.000853 to 0.066247, respectively. Page and Modified Page models were assumed to represent the heat pump drying behaviour of the cornelian cherry-capia pepper leather in thin layers compared to the other models.

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Introduction

Leather is traditionally produced by drying fruit/ vegetable pulp (Basumatary *et al*., 2020). The production steps of the leather show some differences according to geographical regions and demands of customers and producers. Sugar, starch or flour can also be used to produce leather. Production of leather usually involves the cooking of pulp and drying. Cooking pulp can inactivate the enzymes and decrease the level of the microbial population (Chen and Martynenko, 2018). The drying process prolongs shelf-life by inhibiting microbial growth. In addition, it can reduce the weight and volume of food so products can be transported and stored easily. Fruit leathers may have greater nutrient content, such as minerals, antioxidant capacity, fibre and energy, than fresh fruits due to the concentration of nutrients (Sharma *et al.,* 2016).

Cornelian cherry (*Cornus mas.* L.) fruits typically have a sour-sweet taste due to polyphenolic contents. (*i.e.*, gallic acid, rutin, resveratrol, quercitrin, chlorogenic acid and quercetin). These phenolic contents have high antioxidant capacity (Tontul and Topuz, 2017; Ünver, 2019). Fruits contain anthocyanins (*i.e.*, pelargonidin-3-glucoside, cyanidin-3-glucoside, cyanidin-3-rutinoside, delphinidin-3-galactoside, cyanidin-3-galactoside), also carotenoids (*i.e.*, lutein and alpha-carotene). It has been reported that fresh cornelian cherry fruit contains twice has several health benefits such as antimicrobial, anti-inflammatory, anticancer, antidiabetic and anti-atherosclerotic actions (De Biaggi *et al.,* 2018).

Capia pepper (*Capsicum annuum*) is consumed as fresh, cooked, powder, sauce, puree, paste, pickle, and spice (Kelebek *et al.,* 2020). It involves high amounts of flavonoids, capsaicinoids, quercetin and luteolin; also, it is known to be good source of carotenoids (*i.e.*, β-carotene, β-cryptoxanthin, alpha-carotene and lutein) (Kelebek *et al.,* 2020). The amount of ascorbic acid in 100 g of fresh capia pepper is stated to be approximately 130 mg. This value is 1.5-2 times more than the amount of ascorbic acid in an orange (Cerit, 2015). In addition to them, capia pepper is rich in vitamins (*i.e.*, A, C, E and folate). Capia pepper has been used for cell protection, prevention of degenerative diseases and reduction the risk of coronary heart diseases (Cerit, 2015).

Onion (*Allium cepa* L.) can be consumed fresh, as powder or dried. Onion has strong antioxidant activity due to its flavonoids, fructan and organosulfur compounds (Becerikli, 2017). It is effective in preventing cardiovascular diseases against pathogenic microorganisms. Besides, it has anti-inflammatory activity (Becerikli, 2017).

Garlic (*Allium sativum* L.) contains high amounts of allicin and polyphenols. It is a good source of organosulfur compounds. Traditionally it has been used against viral diseases (Rouf *et al.,* 2020).

Spices are defined as flowers, fruits, seeds and tropical plant roots with a sharp and intense flavour. It has been observed that spices have high antioxidant activity. Thanks to these effects, many studies have shown that they can prevent oxidative deterioration that causes poor taste and odour in foods (Embuscado, 2015).

Since sun drying is risky in terms of microbiological contamination, many producers use modern drying systems (*i.e.*, freeze drying, spray drying, hot air drying, microwave drying, and heat pump drying) (Tunçkal *et al*., 2016). On the other hand, heat pump dryer systems have many advantages, such as lower energy consumption, higher energy efficiency, better product quality, the ability to operate independently from outdoor weather conditions for food drying, a wide range of drying conditions, reducing operation cost compared to traditional hot air dryers (Tunçkal *et al.*, 2016).

Optimisation studies have gained importance in food engineering since 2000 to increase the efficiency of the food processes and the acceptability of the product by consumers. Optimisation of pasteurisation, extraction, extrusion and drying conditions are some of the applications where this procedure is used (Demir *et al.*, 2017; Süfer *et al.*, 2018). Response surface methodology (RSM) is a common statistical approach for optimisation that may also be used to the number of experiments minimised. For drying experiments due to obtain high-quality products optimisation of process parameters such as temperature, time, air velocity and thickness of material are important (Süfer and Palazoğlu, 2019).

Several types of research have been carried out so far and are only related to fruit-containing leathers. The objective of this study was to optimise the process conditions in terms of drying temperature and cornelian cherry pulp concentration by using RSM to achieve minimum specific energy consumption, maximum moisture diffusivity, maximum L^* and a^* values and also the minimum hydroxymethylfurfural (HMF) concentration during drying of cornelian cherry-capia pepper leather (without added sugar and salt) by a closed loop heat pump drying system for the first time.

Materials and Methods

Materials

Product

Cornelian cherries were bought from a district bazaar in Bursa. Capia pepper, garlic, onion and spices were purchased from a local market in Bursa. Apple pectin is obtained from Penguen Gıda, Bursa.

Drying system

In each experiment, prepared leather mixes weighing 225 g were dried with heat pump drying (HPD) that was manufactured in the Yalova University Air Conditioning and Refrigeration Technology Program Laboratory. The drying system consists of a heat pump, fan, channel system, 60x60x60 cm^3 drying chamber and 46x42x2 cm^3 stainless steel wire trays. In the experimental system, internal and external condensers connected to each other as serial were used for system performance. A digital thermostat that references the drying room outlet temperature (desired drying temperature) and the external condenser connected in series with the inner condenser are activated to keep the drying chamber at a constant temperature within the differential range of ±0.5°C (Tunçkal *et al*., 2016). While the drying system operates with the activation of the external condenser by assisting the thermostat, a part of the internal condenser load has been taken, thus increasing the performance. The evaporation capacity has also increased in proportion to the condensing capacity, and thus, more moisture is taken from the air leaving the drying room. The drying system works with an accuracy of ±0.8°C through digital thermostatic control.

Methods

Preparation of cornelian cherry-capia pepper leather samples

The parameters of the experimental design were the concentration of cornelian cherry pulp (%) and drying temperature (°C). In addition, minimum and maximum levels of parameters (Table 1) were selected according to laboratory trials. The experimental design was created using the Central Composite Design method.

Firstly, cornelian cherries and capia peppers were washed. Cornelian cherries were de-pitted by using a colander. The seeds of capia peppers were removed, and the peppers were steam blanched for 10 minutes. Then both of them were ground by a domestic blender. Garlic (2%) and onions (20%) were grated and pressed to obtain their juice. This juice (10 mL) and 1 g of each spice (black pepper, chilli pepper, mint, thyme, sumac and cumin) were mixed and kept for 4 hours in a closed glass jar. Subsequently, the juice was filtered to remove spices and added to the cornelian cherry - and capia pepper pulp and then blended. Apple pectin was dissolved in hot water (75°C) (0.05%), and 40 mL was added to this blend. Finally, the leather pulp was boiled at 98°C for 10 minutes. After cooling to room temperature, the leather pulp was spread uniformly (8x8 cm, 3 mm thickness) and dried by HPD.

Drying procedure

Before drying, the dryer was run for about 30 minutes to stabilise the drying conditions. Leather samples were dried at various temperatures (Table 1) and a constant drying air velocity (1.0 m/s). The air flow rate was measured in the drying trials using Testo 405-V1 and Testo 410-2. After the system was stabilised, 225 g of leather pulp was poured into a mould with a thickness of 3 mm, a width of 8 cm and a length of 8 cm.

The drying process was started after the samples were homogeneously placed in the drying tray. The load cell was used to measure the mass loss during the drying process, and the data were recorded to the computer at four-minute intervals with the help of the indicator. Relative humidity and temperature data were measured from the device, and enthalpy (h) values were determined from the psychometric diagram. In the experiments, the ambient temperature in which the drying system was placed was 24±1°C, and the humidity value was determined as 55±5% on average. The drying procedure continued until the samples' moisture content decreased about 0.090-0.116 g water/g dry matter. All experiments were carried out in three replications. While the refrigerant low- and high-pressure values were determined by the digital manifold, the circulating fluid temperatures in the system were measured using a 4-channel digital thermometer via pipes. The amount of energy consumed by the compressor and the whole system was measured using a digital meter and recorded at every top of the total.

Performance

Heat pump drying system analyses

The coefficient of performance (COP) of a heat pump drying system is defined in two ways: first, the specific moisture extracted ratio (SMER), known as water extracted from product per consumed energy; and second, COP, known as the heat delivered through the condenser to the drying air per the consumed energy at compressor (Jia *et al.,* 1990). The COP value of the heat pump (COP_{hp}) is determined as Equation (1).

$$COP_{hp} = \frac{\dot{Q}_{cd}}{\dot{W}_{comp}} \tag{1}$$

Here, $\dot{Q}_{cd}$ and $\dot{W}_{comp}$ indicated the amount of heat transferred by the internal condenser (kW) and power consumption of the compressor (kW), respectively.

The COP of the whole system (COP_{ws}) is calculated using Equation (2).

$$COP_{ws} = \frac{\dot{Q}_{cd}}{\dot{W}_{comp} + \dot{W}_{fan,i} + \dot{W}_{fan,e}} \tag{2}$$

Here, $\dot{W}_{fan,i}$ and $\dot{W}_{fan,e}$ are the power consumption of the fan (kW) for the inlet and outlet, respectively.

The amount of heat transferred by the internal condenser to the drying air is calculated using the following equation.

$$\dot{Q}_{cd} = \dot{m}_a(h_A - h_E) \tag{3}$$

Here, $\dot{m}_a$, h_A and h_E represent the mass flow rate of drying air (kg/s), specific enthalpy of drying air at the condenser outlet (kJ/kg) and specific enthalpy of drying air at the condenser inlet (kJ/kg), respectively.

SMER is defined as the total amount of energy consumed per the moisture absorbed from the product and calculated using the following equation (Jia *et al.,* 1990). The first hour of the drying time is taken into account in the SMER calculation.

$$SMER = \frac{\dot{m}_w}{\dot{W}_{comp} + \dot{W}_{fan,i} + \dot{W}_{fan,e}} \tag{4}$$

Here, SMER represents a specific moisture extraction ratio (kg/kWh) and represents the mass flow rate of water extracted from the product (kg/hr).

Mathematical modelling of drying curve

Seven different thin-layer drying models were used to select the best model for describing the drying curve of cornelian cherry-capia pepper leather samples. These models were given as following equations (Suna and Özkan-Karabacak, 2019):

Page; MR = exp(-k^{tn}) (5)

Modified Page; MR= exp [(-kt)^n] 6)

Logarithmic; MR = a exp(-kt) + c (7)

Lewis; MR = exp(-kt) (8)

Henderson & Pabis; MR = a exp(-kt) (9)

Two Term Exponential; MR = a exp(-k$_0$t) + b exp(-k$_1$t) (10)

Wang & Singh; MR=1+at+bt^2 (11)

Table 1. Central composite matrix of independent variables used in the response surface methodology and experimental design.

Values	Parameter	Levels	
		Low (-1)	High (1)
X_1	Cornelian cherry pulp concentration (%)	30	40
X_2	Drying temperature (°C)	40	50
Run no.	**Cornelian cherry pulp concentration (%)**	**Drying temperature (°C)**	
1	30	40	
2	40	40	
3	30	50	
4	40	50	
5	27.93	45	
6	42.07	45	
7	35	37.93	
8	35	52.07	
9	35	45	
10	35	45	
11	35	45	
12	35	45	
13	35	45	

The moisture ratio (MR) and drying rate (DR) of leather samples were calculated using by following Equation (12) and Equation (13), respectively.

$$MR = \frac{M - M_e}{M_i - M_e} \quad (12)$$

$$DR = \frac{M_{t+Dt} - M_t}{Dt} \quad (13)$$

In these equations, $M, M_i, M_e, M_t, M_{t+dt}$ are definite moisture content at a definite time (g water/g dry base), initial moisture content (g water/g dry base), equilibrium moisture content (g water/g dry base), moisture content at a specific t and $t+dt$ (min) times (g water/g dry base), respectively. The value of M_e is lower than that of M and M_i. For this reason, equilibrium moisture content (g water/g dry base) is considered to be zero.

The root mean square of error (RMSE) represents the deviation between experimental and predicted data. In the drying process of leather pulp, the best model was determined using the highest correlation coefficient (R^2) and the lowest RMSE and chi-square (χ^2). These parameters were calculated using by following Equation (14) and Equation (15):

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2\right]^{1/2} \quad (14)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2}{N-n} \quad (15)$$

$MR_{exp,i}$, $MR_{pre,i}$, N and n are the experimentally dimensionless moisture ratio for the test, the predicted dimensionless moisture ratio for the test, the number of observations and the number of constants in the model, respectively.

Calculation of effective moisture diffusivity

In determination, effective moisture diffusivity (D_{eff}) of leather samples was used by Fick's second law. The law is based on the fact that considering moisture change has been only by diffusion, changes in temperature and pressure are not negligible, and during the drying process, samples are not shrinkage (Süfer and Palazoğlu, 2019). The mathematical solution of Fics' law is given below:

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}\exp\left(-\frac{(2n-1)^2\pi^2 D_{eff} t}{4L^2}\right) \quad (16)$$

D_{eff} is effective moisture diffusivity (m^2/s), L is the half thickness of the slab in samples (m), and n is a positive number. Only the first term, Equation 16, is written logarithmic in practice (Equation 17).

$$MR = \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad (17)$$

The D_{eff} is calculated by the slope of ln MR graph drawn against the drying time (Equation 18).

$$D_{eff} = -\frac{slope 4L^2}{\pi^2} \quad (18)$$

Colour analysis

Colour parameters [L^*, a^*, b^*, Chroma (C^*) and hue angle ($h°$)] were measured using by CR-5 Konica Minolta Chroma Meter (Osaka, Japan). The sample was placed in the device's chamber, and the measurement was made in three replicates. L^*, a^* and b^* values describe lightness/darkness ($L^* = 0$ for black, $L^* = 100$ for white), greenness/redness ($a^* < 0$ for green, $a^* > 0$ for red), blueness/yellowness ($b^* < 0$ for blue, $b^* > 0$ for yellow). Also, C^* and hue angle $h°$ were estimated by these parameters and represent colour intensity and an angle between 0° and 360°, respectively.

Hydroxymethylfurfural analysis

Briefly, 1.00±0.01 g sample and 7 mL distilled water were mixed, and this material was shaken. This sample was centrifugated at 4500 g, 4°C for 10 min, and the supernatant was taken into a test tube. The residue was mixed with 2 mL distilled water and centrifugated at 4500 g, 4°C for 10 min. This process was repeated two times. Each supernatant was collected and mixed with 0.250 mL Carrez I (potassium ferrocyanide, 15%, w/v) and 0.250 ml Carrez II (zinc acetate 30%, w/v) solutions. Subsequently, this extract was centrifugated and completed 10 mL with distilled water. Each sample was filtered using a membrane filter (0.45 mm) then this filtrate was injected into high-performance liquid chromatography. C18 (25 cm × 4.6 mm, 5 µm) column was used as the immobile phase. Spectral measurement was done at 280 nm, flow rate was 1 mL/min, injection volume was 20 µL, and acetonitrile: distilled water (95:5 v/v) was used as the mobile phase. The temperature of the column was 32°C, and the program duration was 15 min (Rufián-Henares and Delgado-Andrade, 2009).

Experimental design

RSM optimises the production process by evaluating the relationship between variables and responses. Thus, this method has been used to decrease the number of analyses, reduce research costs, and determine the best formulation by optimisation (M'hir *et al.,* 2019). Two levels of Central Composite Design were used to optimise the responses. These levels were cornelian cherry pulp concentration (%) and drying temperature (°C). In this study; drying time (Y_1), total energy consumption (Y_2), COP_{hp} (Y_3), COP_{ws} (Y_4), SMER (Y_5), drying rate (Y_6), colour parameters (L^*, a^*, b^*, C^*, $h°$) (Y_7, Y_8, Y_9, Y_{10}, Y_{11}, respectively) and HMF (Y_{12}) were determined as responses. Two factors and their levels (1, -1) were determined for creating an experimental design. The mean of 1 was a high value, the mean of 0 was the optimum value, and the mean of -1 was a low value of parameters (Table 1). In this study, considering specified parameters and their levels, the experimental design involved 13 runs (Table 1). All computational values were examined used by Design Expert 9 software. The effect of the studied factors on the individual response was evaluated and described using a 5% test of significance.

$$Y = \beta_0 + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_i^2 + \sum_{i=1}^{2}\sum_{j=i+1}^{3}\beta_{ij} X_i X_j + \varepsilon \quad (19)$$

where β_0, β_i, β_{ii} and β_{ij} are intercept, linear, quadratic, and interaction regression terms, and ε is the error. X_i and X_j are the independent variables, and Y is the predicted response variable in RSM optimisation. A three-dimensional graphics were constructed to observe the effects of factors on each response visually; moreover, optimum levels for each factor were obtained.

Results and Discussion

Fitting the models

The analytical results of all the experiments designed based on the Central Composite Design model for drying leather samples are given in Table 1. Design Expert 9 Software program was used to constitute the polynomial equations and to perform the regression analysis, including the regression coefficient, lack of fit, R^2, adjusted R^2, P values and appropriate models in Table 2. The p-value was less than 0.05 showed the significance of the model terms in the circumstances the coefficients for the equations were considered significant. If the p-value was lower than 0.05, which indicated that the related model fits the design, the lack of fit values was considered non-significance. Furthermore, the R^2 value greater than 0.80 signified that the model was statistically significant. Depending on those criteria, the reduced cubic model was fitted for drying time (min) and drying rate (g/min) and the quadratic model was found as the best model for total energy consumption (kWh), COP_{hp}, COP_{ws}, SMER (kg/kWh), *a*, b*, C*, h°* values ($p<0.05$). Additionally, the reduced quadratic model was best fitted to the *L** value ($p<0.05$). The lack of fit values of all models was specified to be insignificant ($p>0.05$). High R^2 and adjusted R^2 values showed a good correlation between experimental and predicted values. The highest R^2 (0.9826) and adjusted R^2 (0.9652) values were found in the *b** value; the lowest values were determined as 0.7392 and 0.5902 in the *L** value, respectively.

Drying time

$$\text{Drying time}(Y_1) = 220.96 - 30.54\,X_1 - 68.07\,X_2 + 41.00\,X_1X_2 + 8.63\,X_1^2 - 16.63\,X_2^2 - 0.1792\,X_1^2X_2 + 10.32\,X_2^2X_1 \quad (20)$$

According to Equation 20, cornelian cherry pulp concentration and drying temperature had a negative effect on drying time. As the amount of cornelian cherry pulp used in the formulation increased, the drying time was shortened because the dry matter of the mixture to be dried was increased. In addition, the increase in drying temperature shortened the drying time. Similarly, Doymaz (2005) stated that when the air temperature increases drying rate also increases, and as a result of this interaction, drying time decreases. Drying time was positively influenced by interaction terms of cornelian cherry pulp concentration and drying temperature and quadratic term of cornelian cherry pulp concentration, while the drying temperature's quadratic term negatively influenced it. Interaction between the quadratic term of cornelian cherry pulp concentration and drying temperature ($X_1^2X_2$) had a negative effect on drying time, whereas it was positively influenced by the interaction between the quadratic term of drying temperature and cornelian cherry pulp concentration ($X_2^2X_1$). From Figure 1a it can be seen that when the drying temperature and cornelian cherry pulp concentration decreased, the drying time increased.

Coefficient of performance value of the heat pump

$$COP_{hp}\,(Y_2) = 2.66 + 0.2637\,X_1 + 0.0248\,X_2 + 0.0028\,X_1X_2 - 0.0499\,X_1^2 + 0.0087\,X_2^2 \quad (21)$$

Linear terms of cornelian cherry pulp concentration, drying temperature and their interaction term positively affected COP_{hp} value ($p<0.05$). Figure 1b demonstrated that lower cornelian cherry pulp concentration and lower drying temperature led to reduced COP_{hp}.

Coefficient of performance of the whole system

$$COP_{ws}\,(Y_3) = 2.37 + 0.2366\,X_1 + 0.0171\,X_2 + 0.0008\,X_1X_2 - 0.0428\,X_1^2 + 0.0053\,X_2^2 \quad (22)$$

From Equation 22, linear terms of cornelian cherry pulp concentration, drying temperature and their interaction term had a positive effect on the COP_{ws} parameter. However, the quadratic term of cornelian cherry pulp concentration had a negative effect on COP_{ws},. Similar to COP_{hp}, the decrease in both cornelian cherry

Table 2. Summary of regression analysis response.

Regression coefficient	Drying time (min)	Total energy cons. (kWh)	COP_{hp}	COP_{ws}	SMER (kg/kWh)	Drying rate (g/min)	*L**	*a**	*b**	*C**	*h°*
b0	+220.96	+3.19*	+2.66*	+2.37*	+0.0971*	+1.24	+24.51*	+10.71*	+1.34*	+10.52*	+16.36*
X_1 Cornelian cherry pulp conc. (%)	-30.54	-0.2746*	+0.2637*	+0.2366*	+0.0032	+0.1626	+1.83*	+4.52*	+2.95*	+4.77*	+1.24*
X_2 Drying temperature (°C)	-68.07	+0.0267	+0.0248	+0.0171	+0.0012	+1.20	+0.4456*	-3.85*	-3.76*	-5.26*	-4.41*
$X_1 X_2$	+41.00	-0.0042	+0.0028	+0.0008	-0.0008	+0.0083		-0.2025*	-0.0725*	-0.2075*	-0.1075*
X_1^2	+8.63	+0.0065	-0.0499	-0.0428	+0.0001		-0.7586*	-1.28*	-0.7961*	-1.31*	-0.3586*
x_2^2	-16.63	+0.0120	+0.0087	+0.0053	-0.0002	+0.7521*	+0.1063*	-0.9478*	-0.8339*	-1.26*	-0.9037*
$X_1^2X_2$	-0.1792					-0.0006					
$X_1X_2^2$	+10.32										
X_2^3						0.1389*					
Lack of Fit	0.2960	0.5908	0.9575	0.8160	0.0511	0.0600	0.1547	0.2814	0.5556	0.1514	0.8895
R^2	0.9448	0.8045	0.8350	0.8933	0.9266	0.9317	0.7392	0.9813	0.9826	0.9489	0.8531
Adj R^2	0.8675	0.6416	0.7172	0.8172	0.8742	0.8634	0.5902	0.9625	0.9652	0.8977	0.7481
P value	0.0070	0.0387	0.0115	0.0027	0.0008	0.0029	0.0325	0.0003	0.0002	0.0030	0.0079
PRESS value	2673.51	0.4645	0.0179	0.0145	0.0001	0.4254	23.82	1.58	0.7595	6.36	5.66
Model	Reduced cubic	Quadratic	Quadratic	Quadratic	Quadratic	Reduced cubic	Reduced Quadratic	Quadratic	Quadratic	Quadratic	Quadratic

COP_{hp}, coefficient of the performance of heat pump; COP_{ws}, coefficient of the performance of the whole system; SMER, specific moisture extracted ratio; *L*; a*; b*; C**, Chroma; *h°*, hue angle.

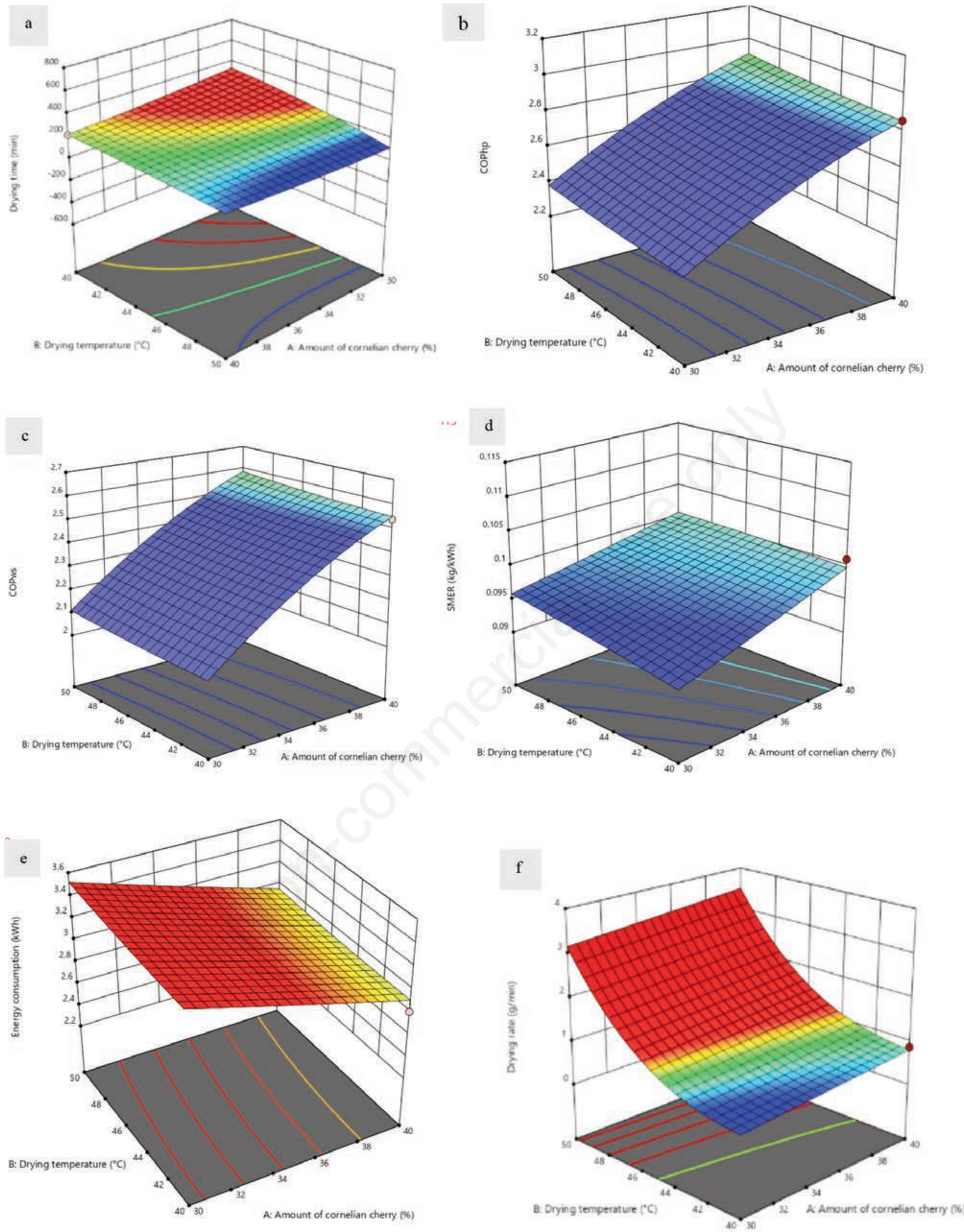
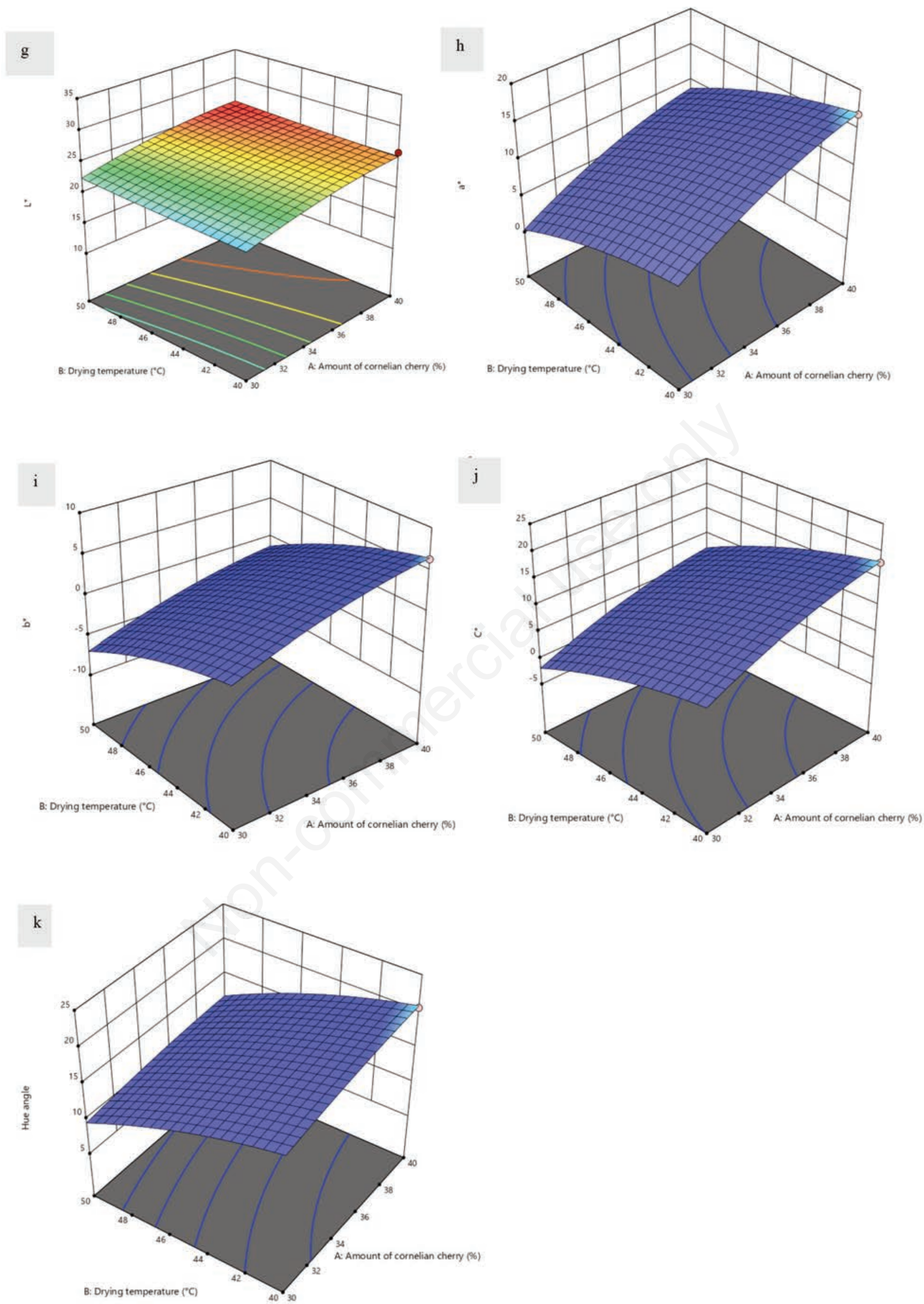


Figure 1. 3D surface graphs showing the effects of the amount of cornelian cherry and drying temperature on drying time (**a**); coefficient of the performance of heat pump (**b**); coefficient of the performance of the whole system (**c**); specific moisture extracted ratio (**d**); total energy consumption (**e**); drying rate (**f**); *L** value (**g**); *a** value (**h**); *b** value (**i**); *C** value (**j**); *h°* value (**k**).


g
L*
B: Drying temperature (°C)
A: Amount of cornelian cherry (%)
h
a*
B: Drying temperature (°C)
A: Amount of cornelian cherry (%)
i
b*
B: Drying temperature (°C)
A: Amount of cornelian cherry (%)
j
C*
B: Drying temperature (°C)
A: Amount of cornelian cherry (%)
k
Hue angle
B: Drying temperature (°C)
A: Amount of cornelian cherry (%)

pulp concentration and drying temperature caused a reduction in the COP_{ws} (Figure 1c). Additionally, it was observed from this figure that cornelian cherry pulp concentration has a significant effect ($p<0.05$), whereas drying temperature's influence was nonsignificant ($p>0.05$) on COP values (Table 2). Like our findings, Coşkun *et al*. (2017) obtained an increment in COP values when drying temperatures rose from 35°C to 45°C for tomato slices dried by HPD. Moreover, Şevik *et al*. (2013) observed a similar trend for heat pump drying of mushrooms.

Specific moisture extracted ratio

$$SMER(Y_4) = 0.0971 + 0.0032\ X_1 + 0.0012\ X_2 - 0.0008\ X_1X_2 + 0.0001\ X_1^2 - 0.0002\ X_2^2 \quad (23)$$

Linear terms of both cornelian cherry pulp concentration and drying temperature positively affected SMER value. However, it was negatively influenced by interaction terms of cornelian cherry pulp concentration, drying temperature, and quadratic terms of drying temperature. The increase of cornelian cherry pulp concentration and drying temperature increased the SMER value (Figure 1d). The highest SMER value was obtained as 0.113 from Run 8 at 52.07°C drying temperature and 35% cornelian cherry pulp concentration, while the lowest value was obtained as 0.095 from Run 7 at 37.92°C drying temperature and 35% cornelian cherry pulp concentration (Table 3). This increment in SMER values was associated with obtaining higher drying rates at higher drying temperatures by Coşkun *et al*. (2017).

Total energy consumption

$$\text{Total energy consumption } (Y_5) = 3.19 - 0.2746\ X_1 + 0.0267\ X_2 - 0.0042\ X_1X_2 + 0.0065\ X_1^2 + 0.0120\ X_2^2 \quad (24)$$

According to Equation 24, linear terms of cornelian cherry pulp concentration and interaction terms of cornelian cherry pulp concentration and drying temperature had negative effects; at the same time, drying temperature and quadratic terms of both cornelian cherry pulp concentration and drying temperature had positive effect on total energy consumption ($p<0.05$). Figure 1e showed that the increase in cornelian cherry pulp concentration, independent of the drying temperature, reduced energy consumption.

Drying rate

$$\text{Drying rate } (Y_6) = 1.24 + 0.1626\ X_1 + 1.20\ X_2 + 0.0083\ X_1X_2 + 0.7521\ X_2^2 - 0.0006\ X_1^2X_2 + 0.1389\ X_2^3 \quad (25)$$

According to Equation 25, linear terms of cornelian cherry pulp concentration (X_1) and drying temperature (X_2) had a positive effect on the drying rate. Besides, the significantly positive effect of the interaction terms of cornelian cherry pulp concentration and drying temperature (X_1X_2), and quadratic (X_2^2) and cubic (X_2^3) terms of drying temperature were observed. However, drying was negatively influenced by the interaction between the quadratic term of cornelian cherry pulp concentration and drying temperature ($X_1^2X_2$). The 3D surface graph of drying rate shows that when the drying temperature and cornelian cherry pulp concentration were increased, the highest drying rate was observed (Figure 1f).

Colour values

L value*

$$L^* (Y_7) = 24.51 + 1.83\ X_1 + 0.4456\ X_2 - 0.7586\ X_1^2 + 0.1063\ X_2^2 \quad (26)$$

From Equation 26, linear terms of cornelian cherry pulp concentration and drying temperature positively affected the *L** value. The quadratic term of cornelian cherry pulp concentration had a negative effect, whereas drying temperature had a positive effect on the *L** value ($p<0.05$). From the 3D graphical representation (Figure 1g), when cornelian cherry pulp concentration and drying temperature were increased, the *L** value was also increased. When the maximum *L** value was found in Run 2 (40% cornelian cherry pulp, drying at 40°C), the minimum *L** value was found in

Table 3. The results of analysed responses for cornelian cherry-capia pepper leather.

Run	Drying temp. (°C)	Cornelian cherry pulp concentration (%)	Drying time (min)	COP_{hp}	COPsys	SMER	Total energy consumption (kWh)	Drying rate (g/min)	*L**	*a**	*b**	*C**	*h*°	D_{eff} (m^2/s)
1	40	30	220	2.862	2.539	0.099	2.857	0.862	25.13±0.1	17.36±3.77	7.42±2.71	18.90±4.54	22.68±2.90	1.149×10^{-9}
2	40	40	216	2.857	2.538	0.101	2.811	0.874	25.92±0.07	16.89±0.90	6.35±0.47	18.04±1.01	20.59±0.55	1.142×10^{-9}
3	50	30	172	2.969	2.672	0.108	2.463	1.113	22.98±2.95	17.47±2.09	7.39±0.95	18.97±2.29	22.92±0.45	1.285×10^{-9}
4	50	40	164	2.975	2.674	0.107	2.400	1.148	24.19±1.73	16.19±2.07	6.03±0.83	17.28±2.23	20.40±0.27	1.318×10^{-9}
5	45	27.92	200	3.002	2.683	0.103	2.82	0.615	25.00±1.58	18.31±2.77	7.69±1.73	19.86±3.22	22.62±1.40	1.214×10^{-9}
6	45	42.07	192	2.975	2.667	0.104	2.728	1.044	23.70±0.46	16.91±0.69	6.21±0.28	18.02±0.75	20.16±0.11	1.259×10^{-9}
7	37.92	35	256	2.785	2.493	0.095	3.158	0.754	24.65±1.28	17.28±1.86	7.15±0.98	18.70±2.09	22.43±0.67	1.026×10^{-9}
8	52.07	35	156	2.957	2.664	0.113	2.368	1.238	20.59±1.01	16.61±1.50	6.90±0.76	18.99±1.64	22.53±0.39	1.52×10^{-9}
9	45	35	192	2.925	2.622	0.103	2.784	1.001	21.82±1.37	15.96±2.47	6.66±1.12	17.30±2.71	22.60±0.38	1.246×10^{-9}
10	45	35	188	2.921	2.626	0.103	2.724	1.022	24.70±1.12	19.49±4.25	8.64±2.89	21.34±5.07	23.54±2.28	1.240×10^{-9}
11	45	35	192	2.948	2.643	0.104	2.757	1.007	24.61±2.00	15.94±1.46	6.45±0.80	17.19±1.67	22.01±0.64	1.207×10^{-9}
12	45	35	172	3.022	2.696	0.105	2.388	1.090	25.13±0.16	17.36±3.77	7.42±2.71	18.90±4.54	22.68±2.90	1.279×10^{-9}
13	45	35	176	3.004	2.682	0.104	2.464	1.061	25.92±0.07	16.89±0.90	6.35±0.47	18.04±1.01	20.59±0.55	1.246×10^{-9}

COP_{hp}, coefficient of the performance of heat pump; COP_{sys}; SMER, specific moisture extracted ratio; *L**; *a**; *b**; *C**, Chroma; *h*°, hue angle; D_{eff}, effective moisture diffusivity.

Run 8 (35% cornelian cherry pulp, drying at 52.07°C). Sengul *et al.* (2010) reported that the L^* value of cornelian cherry leather was 30.32. Our results were similar to the results (26.94±0.97) of Ünver (2019).

a value*

$$a^* (Y_8) = 10.71 + 4.52 X_1 - 3.85 X_2 - 0.2025 X_1X_2 - 1.28 X_1^2 - 0.9478 X_2^2 \quad (27)$$

According to Equation 27, only the linear term of cornelian cherry pulp concentration had a positive effect on a^* value ($p<0.05$). It can be seen from Figure 1h that the highest a^* value was observed when the cornelian cherry pulp concentration was increased, and the drying temperature was reduced. However, the increment in drying temperature, and the decrement in cornelian cherry pulp concentration caused the lowest a^* value. While, Ünver (2019) determined a^* value of cornelian cherry leather as 9.78±2.63, Sengul *et al.* (2010), measured this value as 11.50. Our results were higher than both researchers' results.

b value*

$$b^* (Y_9) = 1.34 + 2.95 X_1 - 3.76 X_2 - 0.0725 X_1X_2 - 0.7961 X_1^2 - 0.8339 X_2^2 \quad (28)$$

Similar to the a^* value, only linear terms of cornelian cherry pulp concentration had a positive effect on the b^* value ($p<0.05$). Figure 1i demonstrated that cornelian cherry pulp concentration had favourable, but the drying temperature had a negative effect on the b^* value. While the maximum b^* value was found in Run 10 (35% cornelian cherry pulp, dried at 45°C), the minimum b^* value was found in Run 4 (40% cornelian cherry pulp, dried at 50°C). b^* value of cornelian cherry leather reported by Ünver (2019) (3.62±0.55) was lower than our results. Whereas the b^* value reported by Sengul *et al.* (2010) (6.64) was similar to our results.

C (Chroma) value*

$$C^* (Y_{10}) = 10.52 + 4.77 X_1 - 5.26 X_2 - 0.2075 X_1X_2 - 1.31 X_1^2 - 1.26 X_2^2 \quad (29)$$

Although cornelian cherry pulp concentration had a positive effect on chroma value, linear terms of drying temperature and quadratic terms of both cornelian cherry pulp concentration and drying temperature negatively affected it. Moreover, the C^* value was negatively influenced by the interaction term of cornelian cherry pulp concentration – drying temperature. When cornelian cherry pulp concentration increased and drying temperature decreased, the maximum C^* value was obtained (Figure 1j). Ünver (2019) determined the C* value of cornelian cherry leather as 10.45±2.63.

h° value

$$\text{Hue angle } (h°) (Y_{11}) = 16.36 + 1.24 X_1 - 4.41 X_2 - 0.1075 X_1X_2 - 0.3586 X_1^2 - 0.9037 X_2^2 \quad (30)$$

According to Equation 30, only the linear term of cornelian cherry pulp concentration had a positive effect on the $h°$ value. Linear terms of drying temperature had the highest negative impact on hue angle ($p<0.05$). The highest $h°$ value was observed when the cornelian cherry pulp concentration increased and the drying temperature was reduced (Figure 1k). However when minimum cornelian cherry pulp concentration was used and maximum drying temperature was applied, the $h°$ value was the lowest. When the maximum hue angle value was found in Run 13 (35% cornelian cherry pulp, drying at 45°C), the minimum $h°$ value was found in Run 6 (42.07% cornelian cherry, drying at 45°C). Similar to our results, Ünver (2019) measured the $h°$ value of cornelian cherry leather as 20.96±3.32. 3D surface graphs of all colour values except L^* showed completely blue. This might be because most of the values obtained are close to the minimum level and below the optimum point. Similar to our findings, completely yellow 3D surface figures were also obtained by Turkmen Erol *et al*. (2022).

Hydroxymethylfurfural

HMF (5-hydroxymethylfurfural) is a heterocyclic compound derived from furan. Thermal treatments are the main factor in the formation of HMF in foods. In addition, high sugar content, particularly fructose, organic acid content, long term storage cause the formation of HMF. HMF may be a risk factor for human health because it could turn into 5-sulfooxymethyl-2-furaldehyde. 5-sulfooxymethyl-2-furaldehyde compound presents mutagenic action.

Table 4. Criteria for optimisation for process conditions.

Name	Goal	Lower limit	Upper limit	Predicted condition	Experimental condition
Drying time	Minimise	157	256	172.79	205.00
COP_{hp}	Maximise	2.79	3.02	2.98	2.98
COP_{ws}	Maximise	2.49	2.69	2.67	2.58
SMER	Maximise	0.09	0.11	0.11	0.10
Energy consumption	Minimise	2.37	3.16	2.58	2.87
Drying rate	None	0.62	2	1.07	1.05
L^*	Maximise	20.59	24	23.92	23.46
a^*	Maximise	16.19	19.49	19.05	19.23
b^*	Is in range	6.03	8.64	8.39	8.13
C^*	Is in range	17.28	30	20.95	21.23
Hue angle ($h°$)	Is in range	20.16	23.78	23.21	22.94
Composite desirability = 0.846					

COP_{hp}, coefficient of the performance of heat pump; COP_{ws}, coefficient of the performance of the whole system; SMER, specific moisture extracted ratio; L^*; a^*; b^*; C^*, Chroma.

Foods containing high amounts of HMF are reported as cytotoxic, neurotoxic, genotoxic and cancerogenic (Dzugan *et al.,* 2021). Consequently, heat-treated dried foods are critically important regarding their HMF content. Tontul and Topuz (2017) investigated pomegranate leather by four different drying methods [Hot air drying (50, 60, 70°C), microwave-assisted hot air drying (90 W; 50, 60, 70°C), microwave-assisted hot air drying (180 W; 50, 60, 70°C) and refractance window drying (90, 95, 98°C)]. They were determined to have HMF content in the 142.8 – 626.11 mg/kg db range. However, the maximum limit of HMF content in fruit leather is 50 mg/kg according to Turkish Standards Institute (Tontul and Topuz, 2017). In this study, HMF was not determined in cornelian cherry-capia pepper leather (Figure 2). The reason for this could be using lower temperatures for drying and being of leathers as added sugar-free.

Optimisation of levels of independent variables

The independent variables optimised to obtain quality characteristics of cornelian cherry-capia pepper leather, giving equal importance to the effect of all responses on the final product. The used criteria, experimental and predicted responses were given in Table 4. The realised RSM model suggested 9 solutions for optimum conditions. The optimal condition for all responses with composite desirability of 0.846 was: 47.419% cornelian cherry pulp concentration (X_1) and 33.574°C drying temperature (X_2).

Effective moisture diffusivity

D_{eff} values determined for each run were shown in Table 3. D_{eff} values of cornelian cherry-capia pepper leather were in the range of $1.026\times 10^{-9} – 1.532\times 10^{-9}$ m^{2}s^{-1}. This range is between $10^{-11} – 10^{-6}$ m^2/s, the range where the moisture diffusion coefficient usually changes in the drying of food materials (Surendhar *et al.,* 2019). The increment in drying temperature enhanced the D_{eff} value of leather samples. Higher drying temperatures lead to higher heating energy, and thus water molecules with increased activity cause high moisture diffusion (Phahom *et al.,* 2021). D_{eff} values of the different drying parameters of carrot + tomato, carrot + red pepper and carrot pestil were calculated between $1.96\times10^{-8} – 2.19\times10^{-7}$, $2.45\times10^{-8} – 9.93\times10^{-8}$, $1.90\times10^{-8} – 2.72\times10^{-8}$ m^2/s respectively (Özkan Karabacak, 2021). D_{eff} values of jackfruit leather were calculated in the range of 3.25×10^{-10} and 1.0062×10^{-9} m^2/s (Chowdhury *et al.,* 2011). Yılmaz *et al.* (2017), produced pomegranate leather using vacuum, cabinet and open-air drying. They found D_{eff} values between 3.1×10^{-9} and 52.6×10^{-9} m^2/s. According to this research, drying temperature and D_{eff} value have a positive correlation. Moreover, Jaya and Das (2003) investigated mango leather production by vacuum drying. The authors also reported that D_{eff} value increased with increasing drying temperature.

Thin-layer drying models

The drying behaviour of cornelian cherry-capia pepper leather with optimal conditions 47.419% cornelian cherry pulp concentra-

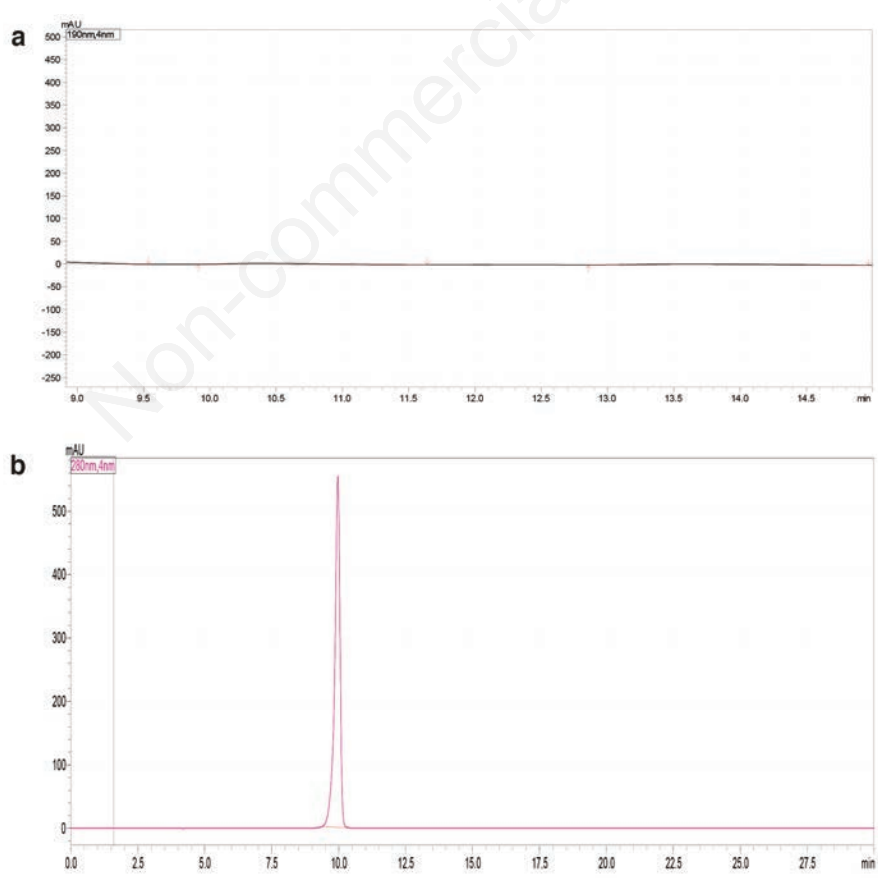


Figure 2. High-performance liquid chromatography chromatogram of hydroxymethylfurfural in leather samples from cornelian cherry and capia pepper (**a**) and 50 ppm hydroxymethylfurfural standard (**b**) (retention time: 9.32 min).

tion (X_1), 33.574°C drying temperature (X_2) acquired with Central Composite Design, was evaluated with seven different thin layer drying models. The statistical parameters based on R^2, RMSE and χ^2 values ranged from 0.8267 to 0.9845, 0.004087 to 0.035626 and 0.000853 to 0.066247, respectively.

The most suitable model to represent the drying characteristics of cornelian cherry-capia pepper leather was chosen as Page and Modified Page models. Suna and Özkan-Karabacak (2019) investigated the effects of microwave (90 W and 180 W), hot air (60 and 70°C) and vacuum (60 and 70°C with 200 and 300 mbar) drying methods on drying characteristics of medlar fruit leather and reported the best-fitted models as Page and Modified Page. Alam and Gupta (2014), produced grape leather using by convective dryer system and the two-term exponential model was determined to be the best model by them. Süfer *et al.* (2017) dried onion by using convective drying (50, 60 and 70°C), vacuum drying (50, 60 and 70°C) and microwave drying (80, 240 and 400 MPa). Fitting the experimental data into 13 thin-layer drying models resulted in the Sigmoid model as the most appropriate model for all investigated drying processes. Diffusion coefficients measured between 1.962×10^{-9} and 1.372×10^{-8} m^2/s for convective, 9.757×10^{-9} and 1.723×10^{-8} m^2/s for vacuum and 3.193×10^{-8} and 9.139×10^{-7} m^2/s for microwave drying.

Conclusions

In this study, cornelian cherry-capia pepper leather production with a heat pump dryer was optimised using RSM. The independent variables (drying time, COP_{hp}, COP_{ws}, SMER, energy consumption, drying rate and colour values) of the process parameters optimised by Central Composite Design. In optimal conditions, the experimental values of drying time, COP_{hp}, COP_{ws}, SMER, energy consumption, drying rate, L^*, a^*, b^*, C^* and $h°$ values were 205 min, 2.98, 2.58, 0.5, 2.87 kWh, 1.05 g/min, 23.46, 19.23, 8.13, 21.23 and 22.94, respectively. Higher drying temperatures resulted in higher D_{eff} values. The experimental data of the optimal condition were modelled with seven different thin-layer drying models. It was determined that the most fitted models were Page and Modified Page in optimal conditions of independent variables (cornelian cherry pulp concentration: 47.419% and drying temperature: 33.574°C). As a result of this study, leather with high functional properties and free of HMF was produced. In addition, heat pump drying is an important alternative drying process for leather production with lower energy requirements.

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