



Spray drying of organic strawberry extract

Organik çilek özütünün püskürtmeli kurutulması

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ABSTRACT

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Key Words: Encapsulation, Response surface methodology, Spray drying, Strawberry

Öz

Bu çalışmada, püskürtmeli kurutma parametrelerinin organik çilek özütüne etkisi incelenmiştir. Püskürtmeli kurutma koşullarını optimize etmek için tepki yüzey metodolojisi uygulandı. Hava giriş sıcaklığı (120-150°C), besleme karışımındaki özüt kütle yüzdesi (kuru bazda, 15-50%) ve besleme karışımının katı madde miktarı (20-40 °Bx) bağımsız işlem değişkenler ve maltodekstrin kaplama ajansı olarak kullanılmıştır. Operasyon verimliliği ve fenolik tutunum modelin yanıt değişkenleridir. Optimum sıcaklık, özüt kütle yüzdesi ve besleme karışımının katı madde miktarı sırasıyla 120°C, %23.26 ve 20.00 °Bx olarak bulundu. Optimum koşullarda yanıt değişkenlerinin maksimum seviyeleri %91.95 operasyon verimliliği ve %79.62 fenolik tutunum olarak bulundu. Çilek özüt tozları üretiminde, en önemli değişkenin özüt kütle yüzdesi olduğu bulundu. Sonuç olarak, organik çilek özüt tozu püskürtmeli kurutmayla verimli şekilde üretilebilecegi belirlenmiştir.

Anahtar Kelimeler: Enkapsülasyon, Tepki yüzey metodolojisi, Püskürtmeli kurutma, Çilek

Introduction

Berry fruits have positive and fundamental effect on human health and performance. Among fruits and vegetables, berries are concerned as having the highest anthocyanin content and antioxidant activity (Denev et al., 2010). Berries are regarded as prominent source of extensive range of bioactive compounds like carotenoids;

phenolic compounds involving flavonoids, stilbenes, lignans, tannins and phenolic acids; vitamins, minerals and organic acids (Nile and Park, 2014; Skrovankova et al., 2015). Bioactive compounds have antioxidant, antimicrobial, anti-inflammatory, antineurodegenerative, chemopreventive, antimutagenic, anticancer, anti-diabetic, cardioprotective, anti-proliferative, anti-allergic and hepatoprotective properties (Nile

and Park, 2014). The best dietary sources of bioactive compounds belong to members of some berry families, such as *Ericaceae* (cranberry, blueberry) and *Rosaceae* (raspberry, blackberry, strawberry) (Skrovankova et al., 2015). Strawberry is the fruit of the genus *Fragaria*. Due to the strawberry is acclimated to different environments; it can be cultivated all over the world, intensively in Europe, North America, China and Russian Federation (Skrvankova et al., 2015). The first five countries produce strawberry is as follows: China, USA, Mexico, Egypt and Turkey (FAO, 2018). In Turkey, strawberry is cultivated nearly in all regions especially in Mediterranean, Marmara, Aegean and Black Sea. The extraction of bioactive compounds from various sources is the first and crucial process. It has a prominent and vital role on the final product quality. The quantitative and qualitative analyses performed for bioactive compounds greatly contingent on the choice of appropriate extraction method. Extracts are used to prepare pharmaceuticals, food ingredients, nutraceuticals, dietary supplements, additives, chemical and cosmetic products (Azmir et al., 2013).

The effectiveness of bioactive compounds depends on integrity, stability and bioavailability. However, they are prone to be destructed by physical, chemical and biological factors like heat, light, oxygen, moisture, enzymes, pH and metallic ions. Moreover, polyphenols might be unstable and generally have low bioavailability. Bitterness and astringency also constitute disadvantages. Activity and health benefits of bioactive compounds are limited due to mentioned factors. Delivery methods have been developed to overcome these limitations and among them encapsulation is a promising way. Encapsulated forms provide better stability and protection, mask unpleasant tastes and flavors, enhance bioavailability and promote controlled release (Flores et al., 2016).

Spray drying is one of the earliest and the most commonly applied encapsulation method in food industry. Spray dried encapsulates constitute approximately 80-90% of encapsulated materials.

It is the most common technique used for encapsulation of polyphenols (Mahdavi et al., 2014; Flores et al., 2016). Spray drying is an advantageous method by being economic, flexible, producing high quality products with high yields and stability, and suitable for both thermolabile and heat resistant materials (Mahdavi et al., 2014).

In literature, there are various studies on the encapsulation of bioactive compounds extracted from fruits by spray drying. For instance, grape pomace extract was encapsulated by Tolun et al. (2016); Cagaita fruit extract by Daza et al. (2016); bitter melon extract by Tan et al. (2015); and pomegranate peel extract by Çam et al. (2014) to protect bioactive compounds obtained from fruit sources.

As our best knowledge, this is the first study on spray drying of organic strawberry extract. Therefore, objectives of this research were to investigate the influences of spray drying parameters (air inlet temperature, extract mass percentage and solid content of feed) on operational efficiency and phenolic retention; and to optimize conditions of the spray drying for operational efficiency and phenolic retention by use of response surface methodology.

Materials and Methods

Materials

Organic strawberries (*Fragaria ananassa* Duch., family Rosaceae) harvested in 2015, were obtained from an organic farm, Bursa, Turkey. Strawberries were stored in freezer at -45°C until processing.

Ethanol, Folin-Ciocalteu's phenol reagent, gallic acid, sodium carbonate (Na_2CO_3), sodium sulfate (Na_2SO_4), citric acid, 1,1-diphenyl-2-picrylhydrazyl (DPPH), sodium acetate trihydrate ($\text{C}_2\text{H}_3\text{NaO}_2 \bullet 3\text{H}_2\text{O}$), Iron (III) chloride hexahydrate ($\text{FeCl}_3 \bullet 6\text{H}_2\text{O}$), potassium chloride (KCl), trolox and concentrated HCl were obtained from Sigma-Aldrich (St. Louis, Missouri, USA). 2,4,6-trypyridyl-s-triazine (TPTZ) was bought from Fluka (St. Louis, Missouri, USA). All other reagents and solvents

were of analytical or chromatographic grade. Maltodextrin dextrose equivalent (DE) 8 was obtained from Cargill (Wayzata, Minnesota, USA).

Extraction

Extraction was performed according to the modified methods proposed by Ersus and Yurdagel (2007) and Flores et al. (2014). Briefly, ethanol (containing 1% (m/v) citric acid) was used for extraction as 1:15 fruit to solution ratio. Solvent was added after the crushing of fruits by blender. Extraction was performed on the magnetic stirrer at 600 rpm for 1 hour at 50°C. Beaker was covered with stretch film and aluminum foil for oxygen and light protection. After filtration, extract was concentrated to by a rotary evaporator (Heidolph Instrument GmbH & Co.KG, Schwabach, Germany) operated at 45°C and at 40 rpm. The final average soluble solid content was measured as 60.18 °Bx. Concentrated extract was stored in a freezer at -45°C until further use. Extraction efficiency was calculated according to the retention of phenolic compounds at the end of the extraction process.

Extraction

$$\text{efficiency (\%)} = \frac{\text{Total phenolic content of extract}}{\text{Total phenolic content of fresh fruit}} * 100 \quad (1)$$

Spray drying of strawberry extract

The central composite rotatable design (CCRD) for three independent variables was performed. Independent variables were drying air inlet temperature (120-150°C), extract mass percentage in feed (m/m in dry basis 15-50%), and solid content of feed (20-40 °Bx) (Table 1). Complete design had 20 runs, including six replications of the center points. Dependent variables were operational efficiency and phenolic retention as responses.

Spray drying process was performed in a Büchi B-290 Mini Spray Dryer (Flawil, Switzerland). Maltodextrin (MD) DE 8 was used as encapsulating agent. The feed mixture was prepared with homogenizer (IKA T 18 digital ultra turrax, Germany). Feed flow rate was 3 ml min⁻¹, aspiration was 90%, Qflow was 600 L h⁻¹ and nozzle cleaner was 2. Powders were collected from both cyclone and main chamber. Powders were stored at 4°C in amber glass bottles.

Table 1. Experimental design and results for the three variables studied

Çizelge 1. Deney tasarımları ve çalışılan üç değişken için sonuçlar

Run Set	Air inlet temperature (°C) Hava giriş sıcaklığı (°C)	Extract mass percentage (%) Özüt kütle yüzdesi (%)	Feed mixture (°Bx) Besleme karışımı (Briks)	Operational efficiency (%) Operasyon verimliliği (%)	Phenolic retention (%) Fenolik tutunum (%)
1	120.00	15.00	20.00	88.67	87.19
2	150.00	15.00	20.00	88.83	87.07
3	120.00	50.00	20.00	88.32	74.20
4	150.00	50.00	20.00	66.82	54.70
5	120.00	15.00	40.00	93.06	78.74
6	150.00	15.00	40.00	89.03	77.62
7	120.00	50.00	40.00	83.85	66.23
8	150.00	50.00	40.00	66.21	49.85
9	109.77	32.50	30.00	92.36	76.39
10	160.23	32.50	30.00	92.85	72.26
11	135.00	3.07	30.00	85.72	64.60
12	135.00	61.93	30.00	36.85	28.02
13	135.00	32.50	13.18	88.20	70.09
14	135.00	32.50	46.82	87.79	66.28
15	135.00	32.50	30.00	93.08	66.81
16	135.00	32.50	30.00	92.38	67.69
17	135.00	32.50	30.00	92.65	69.35
18	135.00	32.50	30.00	91.40	73.75
19	135.00	32.50	30.00	91.95	73.62
20	135.00	32.50	30.00	92.96	73.71

Extraction of bioactive materials from powders

Firstly, ethanol: water 60:40 (v/v) containing 0.5% citric acid (m/v) was heated to about 50°C. Then, 1 g of powder was taken and completed to 15 mL with this solvent and shaken for 5 min. After that, the solution was filtered. Obtained extracts were used for total phenolic content (TPC), total anthocyanin content (ACN) and antioxidant activity analyses.

Analyses

Characterization of fresh strawberry, extract and powder

Total soluble solids (°Bx) of samples were measured by use of refractometer (PTR 46, Optical Activity Limited, UK). The moisture content of samples was determined by the oven method according to AOAC (1995). Hygroscopicities of powders were determined according to the method used by Cai and Corke (2000). Solubility of strawberry extract powders was determined according to the procedure used by Fazaeli et al. (2012). Densities were determined according to the method proposed by Goula et al. (2004). Packed bulk density was calculated from the weight of powder contained in the cylinder after being tapped by hand on a bench 50 times from the height of 10 cm. The glass transition temperature (Tg) of the powders was determined according to the method used by Santhalakshmy et al. (2015).

The ratio of weight of the resulting powder and consumed feed mixture on dry basis were used to determine spray drying operational efficiency (yield) and expressed as % operational efficiency.

Operational

$$\text{efficiency (\%)} = \frac{\text{Dry solid mass of product (powder)}}{\text{Dry solid mass of feed}} * 100 \quad (2)$$

Color measurements, L* (lightness/darkness), a* (redness/greenness) and b* (yellowness/blueness), of the fresh strawberry, extract and powder samples were carried out using a HunterLab Colorflex (A60-1010-615 Model

Colorimeter, HunterLab, Reston, VA) according to CIELAB system. The instrument was standardized for each measurement with a black and a white tile ($L_0 = 93.01$, $a_0 = -1.11$ and $b_0 = 1.30$). Measurements were done at Daylight Color (D65/10*).

Determination of bioactive properties

Determination of total phenolic content

Folin-Ciocalteu method was used to determine total phenolic content according to the method proposed by Singleton et al. (1999). Extracts (450 µL) were mixed with 2.25 mL of Folin-Ciocalteu reagent, previously diluted with distilled water (1:9, v/v). After 3 minutes of shaking at room temperature, 1.8 mL of sodium carbonate solution (75 g/L) was added to the samples. Then samples were left in dark for 2 hours at room temperature to react. Phenolic compounds were detected spectrophotometrically (Optima SP 3000 nano, Japan) at a wavelength of 760 nm. Gallic acid solutions (450 µL) in the concentration range of 10-100 µg/mL were prepared and subjected to the above reactions to plot calibration curve. TPC values were expressed as mg gallic acid equivalents (GAE) per g of dry sample.

Determination of total anthocyanin content

The pH-differential method described by Lee et al. (2005) was used to determine anthocyanin content. Firstly, two buffer systems: sodium acetate buffer, pH 4.5 (0.4 M) and potassium chloride buffer, pH 1.0 (0.025 M) were prepared. Diluted extracts volume of 0.5 mL were transferred to a tube and made up to 7.5 mL with corresponding buffer. Then the absorbance was measured at 515 and 700 nm against blank (distilled water). The absorbances (A) of the diluted extracts were calculated according to Equation 3.

$$A = (A_{515} - A_{700})_{pH1.0} - (A_{515} - A_{700})_{pH4.5} \quad (3)$$

The concentration of anthocyanins in samples was evaluated using the following formula:

$$\text{Total anthocyanins content } \left(\frac{\text{mg}}{\text{L}}\right) = \frac{A * \text{MW} * \text{DF} * 1000}{\varepsilon * 1} \quad (4)$$

where, cyanidin-3-glucoside molecular weight (MW = 449.2 g/mol), DF is dilution factor and the molar absorptivity ($\varepsilon = 26\,900\text{ L/mol.cm}$) constants were used. Total anthocyanin contents of samples were denoted as g of cyanidin-3-glucoside equivalents per kg dry sample.

Determination of DPPH radical scavenging activity

The DPPH radical scavenging activity of samples was determined according to the method which was adapted from Brandwilliams et al. (1995). To a 2500 μL of a 89.7 $\mu\text{mol/L}$ (final absorption adjusted to 0.800 ± 0.010 AU at 517 nm) DPPH radical ethanolic solution and 500 μL of extract or blank (ethanol) were added. All mixtures were left in dark and absorbance was measured at 517 nm against a 95 % pure ethanol blank after 1 hour reaction time. The DPPH radical scavenging activity of samples was calculated according to the equation below and reported as EC₅₀ values.

$$\% \text{DPPH radical scavenging activity} = \left(1 - \left[\frac{A_{\text{sample}}}{A_{\text{blank}}}\right]\right) * 100 \quad (5)$$

Determination of ferric reducing antioxidant power

The ferric reducing antioxidant power of samples was measured according to the method of Benzie and Strain (1996) with some modifications. The constituents of FRAP solution were sodium acetate buffer (300 mM, pH3.6), 10 ml of 1,3,5-tri (2-pyridyl)-2,4,6-triazine (TPTZ) solution (10 mM TPTZ in 40 mM HCl) and 20 mM iron (III) chloride solution in 10:1:1 (v/v/v) ratios, respectively. The FRAP solution was prepared and held at 37°C during experiment. 100 μL of calibration solution, blank or sample were mixed with 3.0 ml of FRAP solution, left in dark for 10 mins. Absorbances of samples were measured at 593 nm, using ethanol as blank. Trolox solutions (100 μL) in the concentration range of 0-500 $\mu\text{mol L}^{-1}$ ethanol were prepared to draw calibration curve.

All measurements were made in triplicate.

Statistical analyses

Obtained data were analyzed statistically by use of RSM (Stat-Ease, Design-Expert software, version 7). The analysis of variance, determination of the regression coefficients, modelling and optimization, and drawing of three-dimensional graphs were carried out by use of RSM. Validation of the model was done by evaluating the coefficient of determination (R^2) and Fisher test value (F-Value). The level of significance for all tests was set at 95% confidence level.

Spray drying process parameters were optimized by using desirability function of response surface methodology in order to obtain strawberry extract powders with maximum operational efficiency and phenolic retention.

Results and discussion

Properties of fresh fruits and extracts

Properties of fresh strawberries and extracts are given in Table 2. According to analysis, fresh strawberry had 8.85 °Bx. Similar results have also been reported in literature by Kadioğlu et al. (2011). Color values of whole fresh strawberry are given in Table 2. Terefe et al. (2009) measured skin color of strawberries as $L^* = 37.8$, $a^* = 25.1$ and $b^* = 7.28$. Color values are different from our study. It may be caused by sample preparation methods. Whole strawberries were blended and used for color measurement in this study. TPC result of this study is compatible with the values given in literature. TPC of fresh strawberries was reported between 20.71 mg 100 g^{-1} FW by Muradoglu et al. (2011) and 443.4 mg 100 g^{-1} FW by Szajdek and Borowska (2008). Görgüç et al. (2019) reported TPC values of fresh strawberries between 169.6-245.1 mg GAE 100 g^{-1} for different strawberry cultivars. Higher phenolic content value of that study is near to the values obtained in this strawberry study. TPC of fresh strawberries was reported between 96.5 and 142 mg GAE 100 g^{-1} during storage after ultrasound washing (Görgüç et al., 2019b). The content and variety of phenolic compounds are dependent on factors

like species, variety, genotype, region, harvesting time, preharvest and postharvest conditions, maturity, environmental factors, agricultural methods, pH, storage time and conditions (Szajdek and Borowska, 2008; Skrovankova et al., 2015). Differences and similarities of the results between literature and this study could depend on these factors as well as extraction procedures (Bakowska-Barczak and Kolodziejczyk, 2011). Similar results about ACN of fresh strawberries have been reported in literature. ACN of fresh strawberries was reported between 6-102 mg C3G 100 g⁻¹ FW by Howard and Hager (2007). Cultivar type, environmental factors (light, temperature), agricultural methods and pH affect occurrence of anthocyanins (Skrvankova et al.,

2015). Factors affecting stability and color of anthocyanins are structure and concentration, temperature, pH, metallic ions, light, self-association, presence of copigments, enzyme, oxygen, sugar, ascorbic acid and degradation products of them, proteins and sulphur dioxide (Ersus and Yurdagel, 2007). It was found that EC₅₀ value of fresh strawberries as 1.20 mg soluble solids mL⁻¹. Factors that influence the antioxidant capacity of fruits and their derived products are chemical structure and content of the antioxidants, pre and post-harvest factors, and processing factors. Different components in plant extracts contribute differently to their total antioxidant ability (Zou et al., 2016).

Table 2. Properties of fresh strawberry and strawberry extract

Çizelge 2. Taze çilek ve çilek özütünün özelliklerini

Parameter Parametre	Fresh strawberry Taze çilek	Strawberry extract Çilek özübü
Soluble solids (°Bx)	8.85 ± 0.05	60.18 ± 0.03
L*	32.12 ± 0.21	3.99 ± 0.08
a*	38.97 ± 0.75	23.68 ± 0.27
b*	24.41 ± 0.47	6.65 ± 0.11
Total phenolic content (mg GAE/100 g FW, mg GAE/g soluble solids, mg GAE/g dry sample)	281.82 ± 28.92 31.84 ± 3.27 29.02 ± 2.98	244.31 ± 17.58 13.57 ± 0.98 13.39 ± 0.97
Total anthocyanin content (mg C3G/100 g FW, g C3G/kg soluble solids, G C3G/kg dry sample)	33.96 ± 2.35 3.84 ± 0.27 3.49 ± 0.24	20.09 ± 0.24 1.12 ± 0.01 1.1 ± 0.01
Ferric reducing antioxidant power (µmoles TE/g soluble solids, µmoles TE/g dry sample)	236.02 ± 14.55 215.12 ± 13.27	96.58 ± 3.19 95.26 ± 3.14

Extraction efficiency was calculated as 86.69% (Equation 1). Fernandes et al. (2012) found TPC of strawberry extracts as 81 and 108 µM GAE g⁻¹ FW and ACN as 18 and 29 µM g⁻¹ FW. EC₅₀ value of strawberry extract was 1.70 mg soluble solids mL⁻¹ in this study. In literature, ferric reducing antioxidant power was found between 12.9-31.7 µM TE g⁻¹ FW and DPPH value was between 6.4-20.1 µM TE g⁻¹ FW by Fernandes et al. (2012). Bioactive contents (TPC, ACN and antioxidant activity) of strawberry extracts depend on the factors that are affecting fresh fruit contents in addition to the extraction conditions (Bakowska-Barczak and Kolodziejczyk, 2011)

Effects of independent variables on responses of strawberry extract powders

Operational efficiency (yield)

Operational efficiency (yield) of strawberry extract powders changed between 36.85 and 93.08%. The lowest operational efficiency was obtained at the highest extract mass percentage (61.93%). This is in good agreement with the pomegranate juice study made by Horuz et al. (2012). They obtained the lowest operational efficiency at the highest juice percentage in the feed mixture.

According to the results of statistical analyses made by RSM, quadratic model was found to be significant ($p<0.05$) and was well to describe

operational efficiency. For operational efficiency, backward elimination gave more suitable statistical values for model. Air inlet temperature (A), linear (B) and quadratic (B^2) effects of extract mass percentage and interaction between

temperature and extract mass percentage (AxB) had negative and significant ($p<0.05$) effects on operational efficiency. Effect of feed solid content (C) was not significant (Equation 6, Table 3) ($p>0.05$) (Figure 1b).

$$\text{Operational efficiency (\%)} = 92.19 - 3.09 * A - 10.00 * B - 4.41 * A * B - 10.32 * B^2 \quad (6)$$

where, A is air inlet temperature ($^{\circ}\text{C}$) and B is extract mass percentage (%).

Table 3. Analysis of variance table and estimated coefficients for operational efficiency

Çizelge 3. Operasyon verimliliği için ANOVA tablosu ve hesaplanmış katsayılar

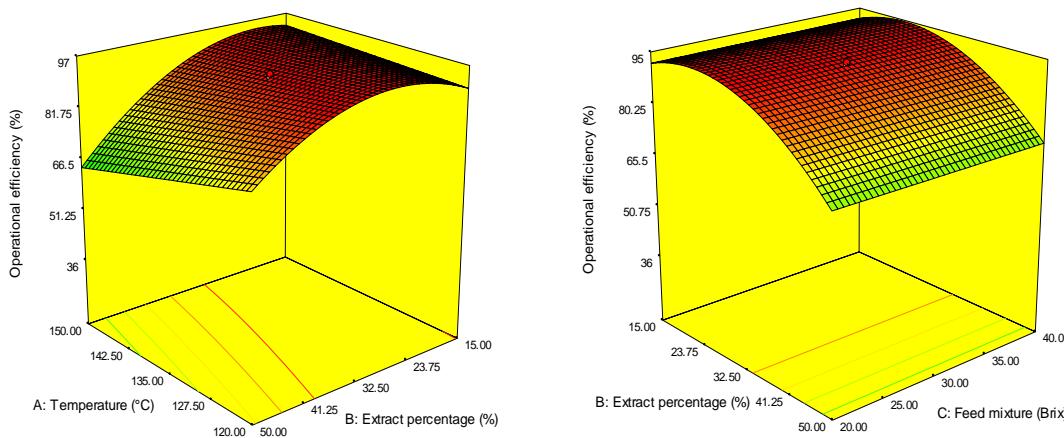
Source	Coefficients	Sum of Squares	df	Mean Square	F Value	p-value Prob>F
Model		3213.47	4	803.37	32.15	< 0.0001
Intercept	92.19					
Linear						
Air inlet temperature (A)	-3.09	130.40	1	130.40	5.22	0.0373
Extract mass percentage (B)	-10.00	1365.54	1	1365.54	54.65	< 0.0001
Interaction						
A*B	-4.41	155.50	1	155.50	6.22	0.0248
Quadratic						
B^2	-10.32	1562.03	1	1562.03	62.52	< 0.0001
Residual		374.77	15	24.98		
Lack of Fit		372.74	10	37.27	91.59	< 0.0001
Pure Error		2.03	5	0.41		
Cor Total		3588.25	19			

$R^2=0.8956$, adj $R^2=0.8677$, pred $R^2=0.6741$

Figure 1a shows 3D response surface plot of air inlet temperature and extract mass percentage effects on operational efficiency of powders at 30 °Bx feed solid content. Increase in extract mass percentage and temperature caused to decrease in operational efficiency (Figure 1a). Effect of extract mass percentage was greater than that of temperature (Equation 6, Table 3). These are in agreement with the pomegranate juice study made by Horuz et al. (2012).

Increase in extract percentage (decrease in maltodextrin percentage) caused a sharp decrease in operational efficiency (Figure 1a). Some other researchers also found that

operational efficiency increased with increasing percentage of encapsulating agent. Gong et al. (2018) found operational efficiency between 15-56.5% for strawberry concentrate powder. In addition to the direct relation between encapsulating agent amount and operational efficiency, they observed operational efficiency changed depending on composition of encapsulating agent. Their values are lower than the values found in this study. A possible reason for this difference is that they only collected powder from product collection vessel while powder was also collected from the main chamber in this study.



(a)

(b)

Figure 1. Effect of air inlet temperature and extract mass percentage at 30 °Bx (a); and extract mass percentage and solid content of feed at 135°C (b) on operational efficiency of strawberry extract powders.

Şekil 1. Çilek özüt tozlarının operasyon verimliliği üzerine (a) 30 Briks'te hava giriş sıcaklığı ve özüt kütle yüzdesinin etkisi; ve (b) 135°C'de özüt kütle yüzdesi ve besleme karışımı katı madde miktarının etkisi.

Moreover, Igual et al. (2014) found that the most significant factor for yield was the percentage of encapsulating agent. Besides, getting the lowest yield at the highest extract percentage is an expected case because encapsulating agent provides protection and prevents sticking to the wall of spray dryer. Sticking causes low product yields, operating problems and carrier agents prevent stickiness of product by increasing Tg of product during spray drying. Maltodextrins have high Tg values and therefore increase the Tg of feed mixture (Vardin and Yasar, 2012).

In addition to the percentage of encapsulating agent, inlet air temperature could also influence the operational efficiency. Vardin and Yasar (2012) and Jafari et al. (2017) observed inverse relation between inlet temperature and operational efficiency like in this study. It was stated by Fazaeli et al. (2012) that low yield due to the sticking problems can be occurred at higher drying temperature above their glass transition temperatures.

Phenolic retention

Retention of bioactive compounds (such as phenolic) is a measure of encapsulation efficiency (Tan et al., 2015). In this study, statistical analyses were made to describe phenolic retention by RSM. It showed that quadratic model was found to be significant ($p<0.05$). Linear (B) and quadratic

(B^2) effects of extract mass percentage on phenolic retention were found to be significant ($p<0.05$) and negative (Equation 7, Table 4). In literature, Igual et al. (2014) and Bazaria and Kumar (2016) found inverse relation between active material mass percentage and phenolic retention as in this study.

Phenolic

$$\text{retention (\%)} = 70.52 - 10.78 * B - 6.66 * B^2 \quad (7)$$

where, B is extract mass percentage (%).

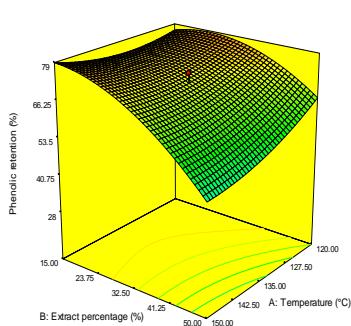
Effects of air inlet temperature and extract mass percentage on phenolic retention of powders are shown in Figure 2a. Increase in extract mass percentage caused to sharp decrease in phenolic retention (Figures 2a and 2b) while increase in solid content of feed did not affect it (Figures 2b and 2c). As shown in Table 1, phenolic retention of powders changed between 28.02 and 87.19%. In literature, phenolic retention was reported as 43.14-87.53% in yerba mate extracts by Nunes et al. (2015); 93.31% in sour cherry by Garofulić et al. (2017) and 81.20% in strawberry by Farias-Cervantes et al. (2018). In this study, the lowest phenolic retention was obtained at the highest extract mass percentage (61.93%). Change of soluble solid content of feed did not affect phenolic retention (Figure 2b).

Table 4. Analysis of variance table and estimated coefficients for phenolic retention

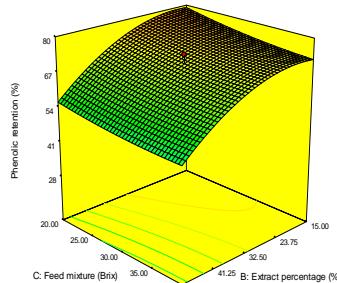
Çizelge 4. Fenolik tutunum için ANOVA tablosu ve hesaplanmış katsayılar

Source	Coefficients	Sum of Squares	df	Mean Square	F Value	p-value Prob>F
Model		2872.76	9	319.20	7.88	0.0017
Intercept	70.52					
Linear						
Air inlet temperature (A)	-3.23	142.25	1	142.25	3.51	0.0905
Extract mass percentage (B)	-10.78	1585.97	1	1585.97	39.13	< 0.0001
Feed mixture (C)	-2.72	100.91	1	100.91	2.49	0.1457
Interaction						
A*B	-4.33	150.05	1	150.05	3.70	0.0832
A*C	0.26	0.56	1	0.56	0.014	0.9090
B*C	0.63	3.22	1	3.22	0.079	0.7839
Quadratic						
A ²	3.24	151.40	1	151.40	3.74	0.0821
B ²	-6.66	640.16	1	640.16	15.80	0.0026
C ²	1.07	16.51	1	16.51	0.41	0.5376
Residual		405.28	10	40.53		
Lack of Fit		352.50	5	70.50	6.68	0.0287
Pure Error		52.78	5	10.56		
Cor Total		3278.03	19			

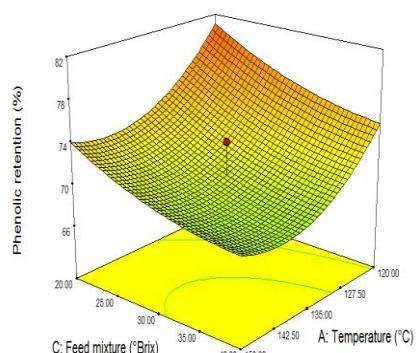
R²=0.8764, adj R²=0.7651, pred R²=0.1633



(a)



(b)



(c)

Figure 2. Effect of extract mass percentage and air inlet temperature at 30 °Bx (a); extract mass percentage and solid content of feed at 135°C (b); temperature and solid content of feed at 32.5 % extract (c) on phenolic retention of strawberry extract powders.

Şekil 2. Çilek özüt tozlarının fenolik tutunuşunu üzerine (a) 30 Briks'te özüt kütle yüzdesi ve hava giriş sıcaklığının etkisi; (b) 135°C'de özüt kütle yüzdesi ve besleme karışımı katı madde miktarının etkisi; (c) % 32,5 özüt içeriğinde sıcaklık ve besleme karışımı katı madde miktarının etkisi

Fang and Bhandari (2011) stated that high retention values suggesting spray drying was a satisfactory technique for encapsulation of heat labile phenolic compounds. Effect of temperature was found to be insignificant for spray drying of strawberry extract possibly because of high amount of MD. It was stated by Garofulić et al. (2017) that when the amount of encapsulating agent was high, the effect of temperature was annulled. Feguš et al. (2015) found no correlation

between inlet air temperature and antioxidant activity for strawberry concentrate powders as in this study. On the other hand, higher inlet temperatures may cause higher degradation of bioactive compounds. Kha et al. (2010) observed that higher inlet air temperature caused decrease in bioactive retention and antioxidant activity in Gac fruit aril powders. Kha et al. (2010) stated that the main reasons for these findings are thermal degradation and oxidation. Çam et al.

(2014) stated that higher number of encapsulating agents provides better protection, retention and longer stability. Igual et al. (2014) stated that for bioactive retention the most significant factor was the amount of encapsulating agent. Garofulić et al. (2017) found that the effect of encapsulating agent percentage is very important because of the role of encapsulating agent on protection of active materials.

Optimization

Optimization of spray drying conditions was performed achieving the highest operational efficiency and phenolic retention. The desirability function of the response surface is shown in Figure 3. Desirability was found to be 0.963. Desirability was high for low extract mass percentage and low air inlet temperature values. Extract mass percentage had greater effect on desirability than air inlet temperature. The optimum conditions were 120.00°C air inlet temperature, 23.26% extract and 20.00 °Bx feed solid content.

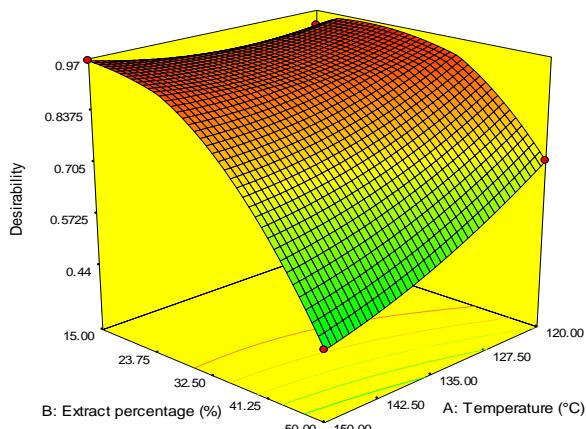


Figure 3. Desirability plot as a function of extract mass percentage and air inlet temperature for strawberry extract powders at 20 °Bx

Sekil 3. Özüt kütle yüzdesi ve hava giriş sıcaklığının fonksiyonu olarak çilek özüt tozları için 20 Briks'te istenirlik çizimi

At optimum conditions, outlet temperatures of powders were recorded between 63-66°C. For

operational efficiency predicted and experimental values were 95.36 and 91.95%, respectively. For phenolic retention predicted and experimental values were 82.92 and 79.62%, respectively. The experimental results were close to the predicted ones. It can be concluded that response surface methodology model was satisfactory.

Characterization of strawberry extract powder obtained at optimum conditions

Moisture content of strawberry extract powder at optimum conditions was calculated as $4.21 \pm 0.14\%$ (Table 5). Gong et al. (2018) reported moisture content of strawberry powders between 4.52 and 4.92%. Researchers reported that moisture content could be affected by inlet and outlet temperatures (Santhalakshmy et al., 2015) and percentage of encapsulating agent (Horuz et al., 2012).

Hygroscopicity was found as 43.51 g moisture 100 g^{-1} dry solids. Samples became like a toffee at the end of the experiment. In literature, hygroscopicity values were reported in the range of 24.6-69.4 g moisture 100 g^{-1} dry solids for betacyanin powders by Cai and Corke (2000); 17.83-20.47 g moisture 100 g^{-1} dry solids for yerba mate extract powders by Nunes et al. (2015). Santhalakshmy et al. (2015) reported that difference between hygroscopicity values could be arisen from nature of samples, difference in procedures, MD percentage; inlet air temperature and moisture content.

Table 5. Properties of strawberry extract powder obtained at optimum conditions

Çizelge 5. Optimum koşullarda elde edilen çilek özüt tozunun özelliklerini

Parameter Parametre	Value Değer
Moisture content (%)	4.21 ± 0.14
Hygroscopicity (g moisture 100 g^{-1} dry solids)	43.51
Solubility (%)	98.30 ± 0.62
Bulk density (g mL^{-1})	0.29
Packed density (g mL^{-1})	0.57
Glass transition temperature (°C)	89.66 ± 1.95
L^*	81.79 ± 0.09
a^*	24.53 ± 0.10
b^*	9.71 ± 0.09

Solubility of strawberry extract powder was $98.30 \pm 0.62\%$ (Table 5). It can be concluded that powders obtained in this study were highly soluble in water. Solubility was found about 87% for black mulberry juice powder by Fazaeli et al. (2012) and between 87.70-97.80% for cagaita extract powder by Daza et al. (2016). Horuz et al. (2012) and Nunes et al. (2015) stated that solubility was affected by temperature and MD percentage.

Bulk and packed density of powder were 0.286 g mL^{-1} and 0.571 g mL^{-1} , respectively. Gong et al. (2018) reported bulk density of powders between 0.45 and 0.55 g mL^{-1} . Bulk density of powders was affected by several operational conditions like vibration, temperature and moisture content (Fazaeli et al., 2012; Vardin and Yasar, 2012) and composition of encapsulating agent (Gong et al., 2018).

Glass transition temperature was found to be $89.66 \pm 1.95^\circ\text{C}$. Glass transition temperature of strawberry concentrate powder was reported between 32.60 and 38.39°C by Gong et al. (2018). They stated that glass transition temperature of powder was significantly affected by composition of ingredients. Can Karaca et al. (2016) observed direct relation between Tg and MD percentages for sour cherry concentrate.

L^* , a^* and b^* values were 81.79 ± 0.09 , 24.53 ± 0.10 and 9.71 ± 0.09 . L^* , a^* , b^* values in strawberry concentrate powder were found as 53.8, 22.8 and 6.4, respectively by Feguš et al. (2015). Color of powders could be affected by degree of caramelization, moisture content, inlet

temperature (Vardin and Yasar, 2012; Feguš et al., 2015); DE of MD (Ersus and Yurdagel, 2007); and percentage of encapsulating agent (Nunes et al., 2015; Daza et al., 2016). High temperatures can cause degradation in colored bioactive compounds and therefore cause change in color.

Bioactive properties of strawberry extract powder

Total phenolic content

TPC is $2.71 \pm 0.05 \text{ mg GAE g}^{-1}$ dry powder (in extract basis $10.81 \pm 0.20 \text{ mg GAE g}^{-1}$ extract soluble solids, in fresh fruit basis $21.99 \pm 0.40 \text{ mg GAE g}^{-1}$ fresh fruit soluble solids) (Table 6). Type and percentage of encapsulating agent directly influence retention of bioactive compounds and encapsulation efficiency. Inlet air temperature influences the amount of phenolic compounds. Phenolics are heat labile and they can be damaged at high temperatures. Flores et al. (2016) stated that TPC was affected by extraction solvent. Sample (juice or pomace) and processing conditions affect results. Initial TPC of fresh fruit also affects the final value. Mentioned factors also influence ACN and antioxidant activities.

Total anthocyanin content

ACN is $0.28 \pm 0.02 \text{ g C3G kg}^{-1}$ dry powder (in extract basis $1.10 \pm 0.07 \text{ g C3G kg}^{-1}$ extract soluble solids, in fresh fruit basis $2.23 \pm 0.15 \text{ g C3G kg}^{-1}$ fresh fruit soluble solids). Extraction and processing procedures, type of the encapsulating agent and percentage, inlet air temperature and initial ACN value of fresh strawberry could affect ACN of powder.

Table 6. Bioactive properties of strawberry extract powder obtained at optimum conditions

Çizelge 6. Optimum koşullarda elde edilen çilek özüt tozunun biyoaktif özellikleri

Parameter Parametre	Value Değer
Total phenolic content (mg GAE g^{-1} dry powder, mg GAE g^{-1} extract soluble solids, mg GAE g^{-1} fresh fruit soluble solids)	2.71 ± 0.05 10.81 ± 0.20 21.99 ± 0.40
Total anthocyanin content (g C3G kg^{-1} dry powder, g C3G kg^{-1} extract soluble solids, g C3G kg^{-1} fresh fruit soluble solids)	0.28 ± 0.02 1.10 ± 0.07 2.23 ± 0.15
$\text{EC}_{50}(\text{mg soluble solids mL}^{-1})$	10.76
Ferric reducing antioxidant power ($\mu\text{moles TE g}^{-1}$ dry powder, $\mu\text{moles TE g}^{-1}$ extract soluble solids, $\mu\text{moles TE g}^{-1}$ fresh fruit soluble solids)	31.20 ± 1.32 124.29 ± 5.28 252.76 ± 10.73

DPPH radical scavenging activity

DPPH free radical scavenging activity EC₅₀ value of powder was calculated as 10.76 mg soluble solids mL⁻¹ for strawberry extract powder (Table 6). DPPH radical scavenging activity of strawberry powders reported between 20.65-29.78 µM TE 100 g⁻¹ sample by Farias-Cervantes et al. (2018).

Ferric reducing antioxidant power

Ferric reducing antioxidant power result of this powder is 31.20 ± 1.32 µmoles TE g⁻¹ dry powder (in extract basis 124.29 ± 5.28 µmoles TE g⁻¹ extract soluble solids, in fresh fruit basis 252.76 ± 10.73 µmoles TE g⁻¹ fresh fruit soluble solids). Farias-Cervantes et al. (2018) reported FRAP antioxidant power of strawberry powders between 181.35-223.08 µM TE 100 g⁻¹ sample. They observed that when phenolic retention is greater antioxidant activity is also greater. Factors affecting TPC, ACN also influence antioxidant activities because all of these are bioactive properties.

Conclusion

In spray drying of strawberry extract, inlet air temperature and extract mass percentage showed significant ($p<0.05$) effects on operational efficiency. Extract mass percentage had also significant ($p<0.05$) effect on phenolic retention. The most effective independent variable on all responses was extract mass percentage. Increase in extract mass percentage affected all responses inversely.

The extract powders can be used as food supplements and functional food ingredients and they can be incorporated into different type of foods. They can provide nutrient, color and taste to the food products.

As a result, it can be concluded that encapsulation of phenolic compounds by use of spray drying and maltodextrin encapsulating agent could be an effective way to produce and preserve strawberry extract powders because of high operational efficiency and phenolic retention values.

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Conflict of Interest: The authors declare that they have no conflict of interest.

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