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Research Paper / Araştırma Makalesi

Optimization of Spray Drying Encapsulation of Bioactive Compounds from Organic Blueberry Extract

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ABSTRACT

In this study, the effects of spray drying parameters on organic blueberry extract were investigated. High amounts of bioactive compounds were extracted from blueberry by solvent extraction. Response surface methodology was applied for the optimization of spray drying conditions. Extract mass percentage of feed mixture (m/m in dry basis 15-50%), air inlet temperature (120-150°C) and solid content of feed (20-40°Brix) were independent variables. Operational efficiency (yield) and phenolic retention were responses. Maltodextrin was used as an encapsulating agent. The optimum extract mass percentage, temperature and solid feed content were estimated as 19.51% (m/m) extract, 120°C and 20.03°Brix, respectively. The maximum levels of responses under optimum conditions were obtained as operational efficiency of 91.20% and phenolic retention of 87.12%. It was found that the most important variable for bioactive compound retention was the extract mass percentage. Encapsulated powder had 3.19% moisture content, and contained 5.54 mg gallic acid equivalents (GAE), 1.52 mg cyanidin-3-glucoside (C3G), and 46.41 µmol Trolox equivalents (TE) per gram dry powder. DPPH free radical scavenging activity value (EC_{50}) of powder was 8.14 mg soluble solids/mL. Bioactive powder obtained could be considered as a possible functional food ingredient. In conclusion, blueberry extract powder could be efficiently produced by spray drying.

Keywords: Spray drying, Encapsulation, Blueberry, Response surface methodology

Organik Yaban Mersini Ekstraktından Elde Edilen Biyoaktif Bileşiklerin Püskürtmeli Kurutmayla Enkapsülasyonu

ÖZ

Bu çalışmada, püskürtmeli kurutma parametrelerinin organik yaban mersini ekstraktına etkileri incelenmiştir. Solvent ekstraksiyonu ile yaban mersininden yüksek miktarda biyoaktif bileşik ekstrakte edildi. Püskürtmeli kurutma koşullarının optimizasyonu için yanıt yüzey metodolojisi uygulandı. Besleme karışımının ekstrakt kütle yüzdesi (kuru bazda, 15-50%), hava giriş sıcaklığı (120-150°C) ve besleme karışımının katı madde miktarı (20-40 Brix) bağımsız işlem değişkenleridir. Operasyon verimliliği ve fenolik tutunum modelin yanıt değişkenleridir. Kaplama ajanı olarak maltodekstrin kullanıldı. Optimum ekstrakt kütle yüzdesi, sıcaklık ve besleme karışımının katı madde miktarı sırasıyla 19.51%, 120°C ve 20.03 Briks olarak belirlendi. Optimum şartlar altında yanıt değişkenlerinin maksimum seviyeleri %91.20 operasyon verimliliği ve %87.12 fenolik tutunum olarak bulundu. Biyoaktif bileşiklerin tutunumunda en önemli değişken ekstrakt kütle yüzdesi olarak bulundu. Enkapsüle edilmiş toz %3.19 nem içeriğine sahipti ve kuru tozun gramı başına 5.54 mg gallik asit eşdeğeri (GAE), 1.52 mg siyanidin-3-glukozit (C3G) ve 46.41 µmol Troloks eşdeğeri (TE) içeriyordu. Tozun DPPH serbest radikal yakalama aktivitesi değeri (EC_{50}) 8.14 mg çözünür katı/mL olarak bulundu. Elde edilen biyoaktif toz muhtemel fonksiyonel gıda bileşenidir. Sonuç olarak, yaban mersini ekstraktı tozu püskürtmeli kurutmayla verimli şekilde üretilebilmiştir.

Anahtar Kelimeler: Püskürtmeli kurutma, Enkapsülasyon, Yaban mersini, Tepki yüzey metodolojisi

INTRODUCTION

The best sources of bioactive compounds are members of *Ericaceae* (cranberry, blueberry) and *Rosaceae* (raspberry, blackberry, strawberry) berry families [1, 2]. Blueberry has a high level of phenolic compounds such as flavonols and anthocyanidins [3]. Bioactive compounds like phenolics have antimicrobial, antioxidant, antitumor activities and neuro protective, antidiabetic, anti-inflammatory, antiallergic and cardioprotective properties [4]. The first five countries producing blueberry are as follows USA, Canada, Mexico, Poland and Germany [5].

The extraction of bioactive compounds from their sources has a critical effect on the final product properties. The analyses made for bioactive compounds largely depend on the selection of suitable extraction procedure [6]. In literature, there are various studies on the encapsulation of bioactive compounds extracted from blueberry by spray drying. For instance, blueberry concentrate was encapsulated by Atacan and Yanık [7]; blueberry by-products by Ma and Dolan [8]; blueberry extract by Jiménez-Aguilar et al. [1]; blueberry juice and extract by Turan et al. [9]; and blueberry pomace extract by Flores et al. [10].

Despite their extensive health benefits, bioactive compounds can be easily degraded with exposure to light, heat, oxygen, moisture, and pH change. They might be unstable during processing, distribution, storage, and also in the gastrointestinal system (pH, enzymes and presence of other nutrients). Activity and positive health effects of bioactive components are limited because of mentioned factors. Encapsulation is an efficient way to overcome these limitations. Most of the encapsulation methods provide better stability, protection and retention, mask unpleasant flavors and tastes, enhance bioavailability and solubility and promote controlled release [11-13]. Encapsulation by spray drying is one of the most widespread encapsulation techniques in the food industry for the protection of functional compounds. Spray drying is a favorable process due to being flexible, economical, producing high-quality products with high operational efficiency and stability [14, 15]. In this study, encapsulation was applied to protect the bioactive compounds of blueberry by spray drying.

The objectives of this study were to investigate the effects of spray drying parameters (extract mass percentage, solid content of feed and air inlet temperature) on phenolic retention and operational efficiency; and to optimize spray drying conditions for phenolic retention and operational efficiency by using of response surface methodology.

MATERIALS and METHODS

Materials

Organic blueberries (*Vaccinium corymbosum,* Bluecrop variety) harvested in 2015 were purchased from an organic farm in Trabzon, Turkey. Blueberries were stored in a freezer at -45°C until use. Ethanol, gallic acid, Folin–Ciocalteu reagent, 1,1-diphenyl-2-picrylhydrazyl (DPPH), sodium carbonate (Na_2CO_3), citric acid, sodium sulfate (Na_2SO_4), sodium acetate trihydrate ($C_2H_3NaO_2 \cdot 3H_2O$), potassium chloride (KCl), Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), concentrated HCl and Trolox® were purchased from Sigma-Aldrich (St. Louis, Missouri, USA). 2,4,6-tripyridyl-s-triazine (TPTZ) was obtained from Fluka (St. Louis, Missouri, USA). All of the other solvents and reagents were of analytical or chromatographic grade. Maltodextrin (MD) dextrose equivalence (DE) 8 was purchased from Cargill (Wayzata, Minnesota, USA).

Extraction

Extraction was applied according to literature with some modifications [16, 10]. Ethanol:distilled water ratios (v/v) between 100:0 and 62.5:37.5 were examined about total extraction efficacy, and 65:35 (v/v) gave the highest total phenolic content (TPC) and used for further extraction. Citric acid was added as 0.5% in mass:volume ratio. Fruit:solvent ratio was 1:15. Berries were crushed approximately with 300 mL solvent in a blender (8011ES Model HGB2WTS3, Waring Commercial, Torrington, USA) for 2 minutes. After crushing, the extraction was performed with solvent by magnetic stirrer at 50°C and 600 rpm for 45 minutes. Beaker was wrapped with stretch film and aluminum foil. After filtration, the solvent was evaporated by a rotary evaporator (Heidolph Instrument Gmbh & Co.KG, Schwabach, Germany) at 45°C and 40 rpm. The blueberry extract was concentrated to 50.86°Brix and stored in the freezer at -45°C until use. Extraction efficiency was evaluated according to the retention of phenolic compounds after extraction (Equation 1).

$$Extraction\ efficiency\ (\%) = \frac{Total\ phenolic\ content\ of\ extract}{Total\ phenolic\ content\ of\ fresh\ fruit} x100 \quad (1)$$

Spray Drying of Blueberry Extract

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

Spray drying was performed by using of a Büchi B-290 Mini Spray Dryer (Flawil, Switzerland). The encapsulating agent was maltodextrin DE 8 and it had 5% moisture. For the feed mixture preparation, a homogenizer (IKA T 18 digital ultra-turrax, Germany) was used. For every experiment, 50 g of feed solution

was prepared. The feed flow rate was adjusted as 3 mL/min (10% pump rate), nozzle cleaner was 2, Qflow was 4 cm (600 L/h) and aspiration was 90%. Powders were collected and immediately sealed to prevent subsequent moisture uptake. Powders were stored in amber glass bottles at 4°C.

Table 1. Process variables and experimental responses in the central composite rotatable design for three independent variables

Independent variables	Code	Independent variable levels					Responses
		-1.68	-1	0	1	1.68	
Air inlet temperature (°C)	A	109.77	120	135	150	160.23	Operational efficiency (%)
Extract mass percentage of feed (%)	B	3.07	15	32.5	50	61.93	
Solid content of the feed (°Brix)	C	13.18	20	30	40	46.82	Phenolic retention (%)

Extraction of Bioactive Materials from Powders

Ethanol:distilled water 60:40 (v/v) solution which was containing 0.5% citric acid (m/v) heated to 50°C. This solvent was then added to the powders at 1:15 (m/v) ratio and mixed for 5 minutes at 22000 rpm. After that, the solution was filtered with traditional pillow cloth. Clarified extracts were used for total anthocyanin content (TAC), total phenolic content (TPC) and antioxidant activity determinations.

Analyses

Characterization of fresh blueberry, extract and powder

Total soluble solids (°Brix) of samples were determined by a refractometer (PTR 46, Optical Activity Limited, Cambridgeshire, UK). Blueberry extracts were diluted with distilled water at 1:10 (m/v) ratio before measurement, and total soluble solids of fresh blueberries were measured after crushing with a commercial blender at room temperature. The oven method, according to AOAC [17] was used for moisture content determination. For fresh fruits, 2 g, for extracts, 1.5 g, for powders, 1 g of samples were weighed and dried at 105°C in the oven until constant weight attain for 4 hours.

For the hygroscopicity experiment, 2 g of powders were weighed and placed into an airtight plastic container containing a beaker filled with a saturated Na_2SO_4 solution (81% RH). After one week, hygroscopic moisture was denoted as g of moisture per 100 g dry solids [18]. The method proposed by Fazaeli et al. [19] was used to determine the solubility of powders. Determination of densities was made according to the procedure reported by Goula et al. [20] with some modifications. Two g of powder was transferred into the 50 mL graduated cylinder. The bulk density was evaluated by dividing the weight of the powder by the volume occupied in the cylinder. Packed bulk density was evaluated from the mass of powder contained in the cylinder after being tapped on a bench 50 times from a 10 cm height. The glass transition temperature (Tg) of the powders was determined using a differential scanning calorimeter (DSC-6, Perkin Elmer, Waltham, USA) [21]. All measurements were made in triplicate.

The ratio of the mass of the powder to the mass of feed mixture on dry basis was used to calculate the operational efficiency (yield) of spray drying and denoted as % operational efficiency (Equation 2).

$$Operational\ efficiency\ (\%) = \frac{Dry\ solid\ mass\ of\ product\ (powder)}{Dry\ solid\ mass\ of\ feed} x100 \quad (2)$$

Color measurements of the fresh blueberry, extract and powder were performed using a HunterLab Colorflex (A60-1010-615 Model Colorimeter, HunterLab, Reston, VA) according to CIELAB system. The color values were denoted as L* (lightness/darkness), a* (redness/greenness) and b* (yellowness/blueness), respectively. Measurements were obtained at Daylight Color (D65/10*).

Determination of Bioactive Properties

Total Phenolic Content

Total phenolic content assay was carried out using the Folin-Ciocalteu reagent, according to the method of Singleton et al. [22]. TPC values were expressed as mg gallic acid equivalents (GAE) per g of dry matter.

Total Anthocyanin Content

The pH-differential method proposed by Lee et al. [23] was applied to determine the anthocyanin content of fresh berries, extracts and powders. Total anthocyanin contents of samples were expressed as g of cyanidin-3-glucoside equivalents (C3G) per kg dry matter.

DPPH Radical Scavenging Activity

The DPPH radical scavenging activity of samples was determined according to the method of Brand-Williams et al. [24]. The DPPH radical scavenging activity of samples was calculated according to the equation below:

$$\%\ DPPH \bullet\ scavenging\ activity = \left(1 - \left[\frac{A_{sample}}{A_{blank}}\right]\right) x100$$

where A_{sample} is the absorbance of sample containing DPPH solution, A_{blank} is the absorbance of DPPH solution without sample solution at 517 nm. Different sample concentrations were used in order to obtain antiradical curves for calculating the EC_{50} values. The results were expressed as DPPH free radical scavenging activity EC_{50} value, the concentration of the sample required to scavenge 50% of the radical.

Ferric Reducing Antioxidant Power

The method adapted from Benzie and Strain [25] was used to determine the ferric reducing antioxidant power of samples. Ferric reducing antioxidant power results were expressed as µmoles Trolox equivalents (TE) per g of dry matter.
All measurements were made in triplicate.

Statistical Analyses

Obtained data were statistically analyzed by use of RSM (Stat-Ease, Design-Expert software, version 7). Determination of the regression coefficients, the analysis of variance, modelling and optimization, and three-dimensional graphs were obtained using RSM. Optimization of spray drying parameters was performed by using the desirability function of RSM to obtain blueberry extract powders with maximum phenolic retention (Equation 3) and operational efficiency.

$$Phenolic\ retention\ (\%) = \frac{TPC\ of\ obtained\ powder}{TPC\ of\ used\ extract\ (TPC\ of\ feed\ solution)} x100 \quad (3)$$

RESULTS and DISCUSSION

Characterization of Fresh Fruit and Its Extract

The properties of fresh blueberries and blueberry extracts are presented in Table 2. The total phenolic content of fresh blueberries was reported between 46.24-585 mg GAE/100 g fresh weight (FW) [26, 27, 28]. TPC of fresh blueberries in this study was in this range. The content of phenolic compounds in plants are dependent on some intrinsic (species, genus, cultivars) and extrinsic (environmental, agronomic, handling and storage) factors and also on the method of extraction [29, 30].

TAC result of fresh blueberry is given in Table 2. TAC of fresh blueberries was reported as 120 mg/100 g FW for bluecrop variety [31]. TAC result was compatible with the range in the literature. Total anthocyanin content varies depending on plant species and cultivars, extrinsic factors such as temperature, light and altitude, chemical structure, temperature, pH, light intensity, oxygen, solvents, presence of enzymes and metallic ions [32, 33].

EC_{50} value of fresh blueberries was found as 0.66 mg soluble solids/mL in this study. Content and chemical structure of the antioxidants, pre- and post-harvest conditions, and processing factors affect the antioxidant capacity of fruits and fruit products [34].

Table 2. Properties of fresh blueberry and blueberry extract

Parameter	Fresh blueberry	Blueberry extract
Soluble solids (Brix)	9.90±0.06	50.86±0.04
L*	8.44±0.17	0.93±0.08
a*	16.83±0.56	3.00±0.38
b*	2.04±0.32	0.36±0.36
Total anthocyanin content (mg C3G/100 g FW,	118.37±5.48	93.20±2.80
g C3G/kg soluble solids,	11.96±0.55	7.25±0.22
g C3G/kg dry matter)	8.81±0.41	7.33±0.22
Total phenolic content (mg GAE/100 g FW,	467.31±46.23	375.13±7.89
mg GAE/g soluble solids,	47.20±4.67	29.16±0.61
mg GAE/g dry matter)	34.77±3.44	28.83±0.60
Ferric reducing antioxidant power (µmol TE/g soluble solids,	301.29±12.42	185.54±2.58
µmol TE/g dry matter)	409.02±16.86	187.66±2.61

Extraction efficiency was calculated as 80.3% for the blueberry extract. In literature, Tatar Turan et al. [12] found TPC of extract as 11.041 mg GAE/g dry matter. It is lower than the TPC value of this study, which was 28.83 mg GAE/g dry matter. The total anthocyanin content of blueberry extracts was reported as 65.1 mg C3G/kg dry matter by Tatar Turan et al. [12]. FRAP value was reported as 318.24 µmol TE/kg dry matter [12]. The values of TPC, TAC and FRAP found by Tatar Turan et al. [12] were lower than the results of this study. This difference can mainly be caused by their encapsulating agent percentage was lower than this study. EC_{50} value of blueberry extract was found as 0.88 mg soluble solids/mL in this study.

Influences of Independent Variables on Responses of Blueberry Extract Powders

Operational Efficiency (Yield)

The operational efficiency is one of the main process parameters in spray drying. The operational efficiency of blueberry extract powders was determined between 70.86 and 94.24% (Table 3). The minimum operational efficiency was obtained at the maximum extract mass percentage (61.93%).

The statistical analyses showed that the quadratic model was significant and was well used to describe the

operational efficiency of blueberry extract powder ($p<0.05$). On operational efficiency, linear effect of extract mass percentage (B), the interaction effect between extract mass percentage and solid content of feed (BxC) and quadratic effect of extract mass percentage (B^2) were significant and negative ($p<0.05$) (Equation 4). The increase in extract mass percentage caused a decrease in operational efficiency (Figures 1a and 1b).

$$Operational\ efficiency\ (\%) = 92.87 - 4.57xB - 1.51xBxC - 5.58xB^2 \quad (4)$$

where B is extract mass percentage (%) and C is solid content of feed (Brix).

Other factors were not significant ($p>0.05$). It was stated that at low feed solid levels, the effects of inlet and outlet temperatures on operational efficiency were not important [31-35]. As shown in Figure 1a, the increase or decrease of air inlet temperature did not affect operational efficiency. Similarly, Horuz et al. [36] reported that drying temperature did not significantly affect operational efficiency. From Figure 1b it can be observed that any change in solid content of feed did not affect operational efficiency.

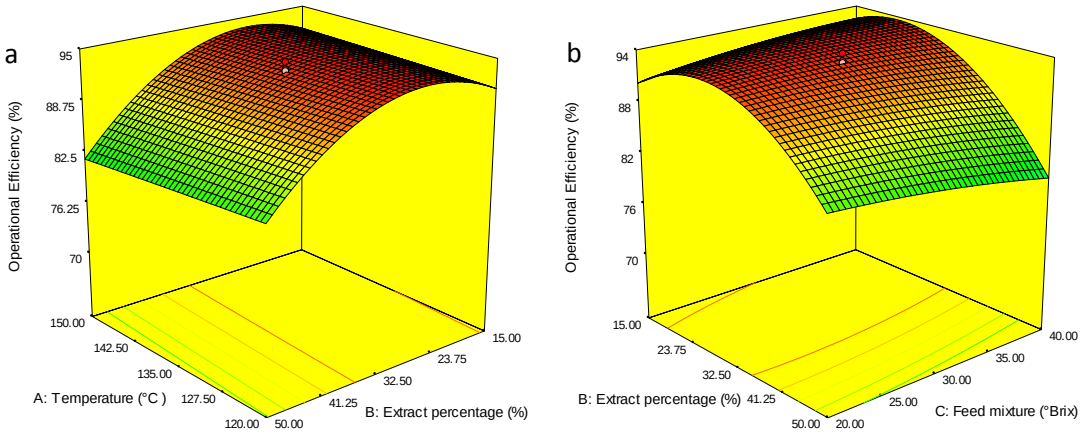

Figure 1. Influence of extract mass percentage and air inlet temperature at 30°Brix (a); solid content of feed and extract mass percentage at 135°C (b) on the operational efficiency of blueberry extract powders

There were comparable studies in literature such as spray drying of blueberry concentrate [7] and blueberry by-products [8]. Atacan and Yanık [7] reported operational efficiency between 8.45-79.76%, and a decrease in concentrate content (increase in MD content) caused to increase in operational efficiency in agreement with this blueberry extract study. Four different levels of extract containing feed solutions (5-50%) were spray dried and operational efficiencies were reported between 62.08 and 90.32% by Ma and Dolan [8].

In conclusion, the amount and type of encapsulating agent [37, 38], inlet air temperature [35, 39], DE of MD [38-40] and where you collect powders from (if other parts were used in addition to the product collection vessel or not) and operating conditions like aspiration rate could affect operational efficiency [41].

Phenolic Retention

Retention of bioactive compounds is a good indicator of encapsulation efficiency. Phenolic retention of blueberry extract powders varied between 56.62 and 94.18% (Table 3). Maximum phenolic retention was obtained at the lowest extract mass percentage (3.07%) and minimum phenolic retention was obtained at the highest extract mass percentage (61.93%).

A linear model was found to be significant and was well to describe phenolic retention of blueberry extract powder ($p<0.05$). Extract mass percentage had a significant linear and negative effect on phenolic retention of powder ($p<0.05$). An increase in extract mass percentage decreased phenolic retention (Equation 5, Figure 2a and 2b).

$$Phenolic\ retention\ (\%) = 72.54 - 7.46xB \quad (5)$$

where B is extract mass percentage (%).

Influences of other independent variables such as air inlet temperature and solid content of feed were found to be insignificant ($p>0.05$). As can be seen in Figure 2a, temperature change did not affect the phenolic retention. Figure 2b shows that increase or decrease of feed brix did not influence phenolic retention. In the study made by Ma and Dolan [8], phenolic retention for blueberry changed between 76-89%, and the highest phenolic retention was determined at the lowest extract mass percentage like in this study. Moreover, the highest retention in antioxidant activity and anthocyanidin content was observed at the lowest extract level (5%). There was significantly greater retention of bioactive compounds with increased maltodextrin levels showing a protective effect of MD on

the bioactive compounds within spray drying. Atacan and Yanık [7] stated the range of phenolic retention as 8-89%, and inverse relation between blueberry concentrate percentage and phenolic retention in agreement with this study. Phenolic retentions reported between 73.2 and 95.1% by Jiménez-Aguilar et al. [1] for spray drying of blueberry extract. Turan et al. [9] encapsulated bioactive compounds of blueberry juice and extract, and retentions of phenolic contents were reported as 54% and 65% for juice and extract powders, respectively. Anthocyanin retention changed between 25-85% for blueberry juice and extract powders in the study of Tatar Turan et al. [12]. In literature, there were comparable results with this study. In conclusion, possible differences could be from the type of encapsulating agents and their amount, inlet and outlet air temperatures, dextrose equivalence of maltodextrin, microstructure, and total soluble solids in feed.

Table 3. Experimental design of spray drying of blueberry extract and results for the three variables studied

Run	Air inlet temperature (°C)	Extract mass percentage (%)	Solid content of feed (°Brix)	Operational efficiency (%)	Phenolic retention (%)
1	120.00	15.00	20.00	91.09	77.94
2	150.00	15.00	20.00	90.15	71.87
3	120.00	50.00	20.00	86.79	69.10
4	150.00	50.00	20.00	80.47	63.38
5	120.00	15.00	40.00	92.47	73.57
6	150.00	15.00	40.00	92.11	70.46
7	120.00	50.00	40.00	79.17	58.41
8	150.00	50.00	40.00	79.37	64.27
9	109.77	32.50	30.00	92.21	77.13
10	160.23	32.50	30.00	94.24	74.67
11	135.00	3.07	30.00	84.16	94.18
12	135.00	61.93	30.00	70.86	56.62
13	135.00	32.50	13.18	91.87	79.23
14	135.00	32.50	46.82	91.90	76.12
15	135.00	32.50	30.00	92.82	71.05
16	135.00	32.50	30.00	92.46	79.63
17	135.00	32.50	30.00	91.95	69.11
18	135.00	32.50	30.00	93.92	72.05
19	135.00	32.50	30.00	93.07	76.16
20	135.00	32.50	30.00	92.85	75.93

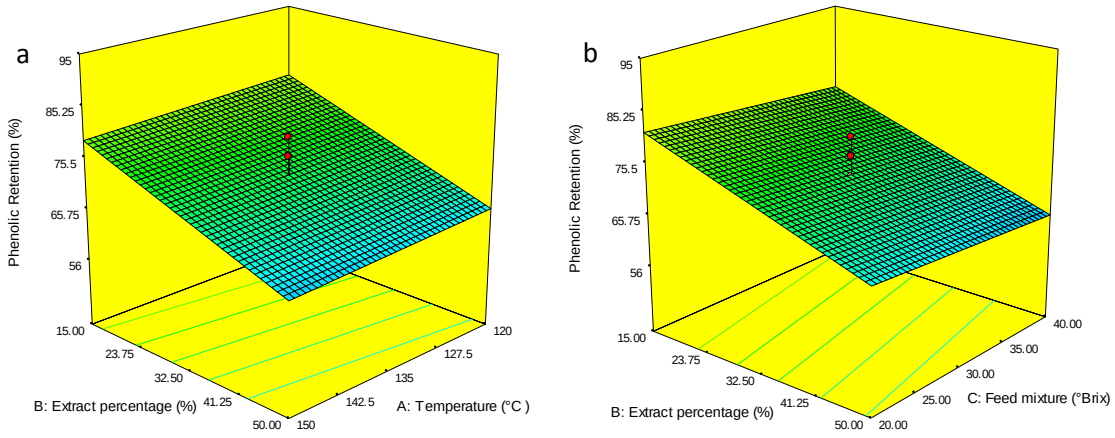

Figure 2. Influence of air inlet temperature and extract mass percentage at 30 Brix (a); solid content of feed and extract mass percentage at 135°C (b) on phenolic retention of blueberry extract powders.

Optimization

Optimization of spray drying conditions was carried out based on achieving the highest phenolic retention and operational efficiency. The desirability function of the response surface is demonstrated in Figure 3. Desirability was high for low air inlet temperature and the extract mass percentage values. Effect of extract mass percentage on desirability had higher than that of the air inlet temperature. Desirability was 0.771 for blueberry powders. The optimum conditions were 19.51% extract, 120.00°C air inlet temperature and 20.03°Brix.

Predicted and experimental values were 92.66 and 91.20%, respectively for operational efficiency and

80.56 and 87.12%, respectively for phenolic retention. The experimental and predicted values were close to each other. It can be concluded that the response surface methodology model was satisfactory.

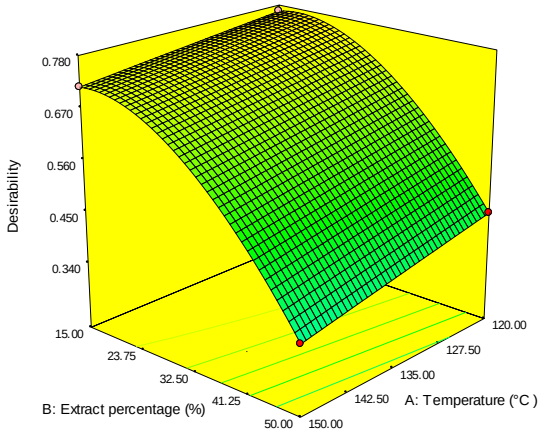

Figure 3. Desirability function response surface plot as a function of air inlet temperature and extract mass percentage for blueberry extract powders at 20.03 Brix

Characterization of Blueberry Extract Powder Obtained at Optimum Conditions

The moisture content of blueberry extract powder was evaluated as 3.19±0.19%. Similar results were obtained by Atacan and Yanık [7], that found the moisture content of blueberry concentrate powder as 3.51%. Ma and Dolan [8] found moisture contents of blueberry extract powders in the range of 6.84-8.08%. Flores et al. [10] reported the moisture content of blueberry pomace extract powders as 5%. Moisture content could be influenced by several factors like inlet and outlet temperatures, feed flow rate [1, 21], type of the encapsulating agent and its percentage [42, 43] and DE of MD [44].

Hygroscopicity is the ability of a material to absorb moisture from the environment and it is important due to its influence on stability [38]. Hygroscopicity of powder was found as 41.99 g moisture/100 g dry solids. In literature, hygroscopicity was determined as 10.4 g moisture/100 g powder by Correia et al. [45] for blueberry pomace extract by using soy protein isolate as encapsulating agent. Difference between hygroscopicity values could be caused by the difference in encapsulating agents.

The solubility of blueberry extract powder was 96.27±2.75%. Atacan and Yanık [7] found the solubility of blueberry concentrate powder as 97.2%. This was close to the solubility result of this study. It could be concluded that the bioactive blueberry powder produced in this study was highly water-soluble. High solubility implies that these powders could easily and efficiently be reconstituted in water to utilize the bioactive compounds in aqueous systems [37-39]. MD percentage and temperature could affect the solubility [43].

The bulk density of blueberry extract powder was 0.268 g/mL. The packed density of blueberry extract powders was 0.502 g/mL. Atacan and Yanık [7] reported bulk density as 0.4 g/mL and packed density 0.74 g/mL for blueberry concentrate powder; Turan et al. [9] reported bulk densities of blueberry extract powder as 0.197 g/mL. Several factors and operational conditions like drying temperature, applying vibration, moisture content and particle size and size range could influence the bulk density of powders [19, 36, 40].

Glass transition temperature (Tg) was determined as 88.92±1.99°C. Atacan and Yanık [7] calculated the glass transition temperature of blueberry concentrate powder as 85.6°C. It is observed that Tg values increased with increasing MD percentages by Fang and Bhandari [37] for bayberry juice and Can Karaca et al. [46] for sour cherry juice concentrate.

L*, a* and b* values were 60.64±0.08, 38.31±0.03 and -4.87±0.03 for powder. Jiménez-Aguilar et al. [1] found L*, a*, b* values of spray dried blueberry extract powders between 35.80-39.48, 32.88-34.46 and 3.06-5.89 by dissolving powders in water, respectively. The difference between the literature and values of this study could be that the powder was dissolved in water in that study. This caused a dilution and gave different color values. Percentage of the encapsulating agent, moisture content and inlet air temperature could affect the color of powders [21, 40].

Bioactive Properties of Blueberry Extract Powder

Total Phenolic Content

TPC result of this study is 5.54±0.20 mg GAE/g dry powder (in extract basis 25.39±0.93 mg GAE/g extract soluble solids, in fresh fruit basis 32.99±1.21 mg GAE/g fresh fruit soluble solids), and in literature, similar results were reported. Ma and Dolan [8] calculated the total

phenolic content of powders between 24.61 and 33.53 mg GAE/g blueberry solids. Jiménez-Aguilar et al. [1] calculated TPC of powders between 18.24 and 23.69 mg GAE/g soluble solids of blueberry extract by using mesquite gum as encapsulating agent and 96% ethanol as extraction solvent. TPC result obtained in this study was higher than those values. This difference could be the result of difference in extraction procedures and used encapsulating agent. Mentioned factors also influence TAC and antioxidant activity.

Turan et al. [9] reported TPC of blueberry extract powder as 1089.7 mg GAE/100 g dry powder. In the same study, TPC was reported as 1517.63 mg GAE/100 g dry powder for extract powder using an ultrasonic nozzle. These TPC values were more significant than that obtained in this study. The amount of encapsulating agent was the possible reason of this difference when the conventional nozzle was used. They used 10% (w/w) encapsulating agent in the feed solution. This directly increased the extract amount; therefore, TPC increased in the feed solution. In this study amount of encapsulating agent was 80.49%. In addition, this difference could be the result of the difference in the TPC of fresh berries. It could also be due to the effects of extraction methods for fresh fruit and bioactive powder compounds. Furthermore, the type of encapsulating agent could cause differences in TPC results; they used a mixture of MD and arabic gum for powders obtained from the extract. Mentioned factors (type and amount of encapsulating agent, TPC of fresh berries, and extraction methods) also affect TAC and the antioxidant activity of powders. Flores et al. [10] reported TPC of blueberry pomace extract powder as 2.83 mg GAE/g powder which was lower than the TPC value of this study. This could be due to the extraction of bioactive compounds from the pomace obtained after juice processing. Some phenolics and other bioactive compounds could be transferred to the juice part. Encapsulating agent was whey protein isolate, extraction solvent was 80% ethanol and air inlet temperature was 160°C in that study. Type of encapsulating agent directly affects the phenolic retention, and solvent determines the extracted amount of phenolic compounds. High temperature could cause degradation of bioactive compounds and reduction in phenolic content. Flores et al. [47] found TPC of blueberry anthocyanin extract powder between 0.57-0.87 mg GAE/g product which was lower than the TPC result of this study. Encapsulating agents were gum Arabic and whey protein isolate, and fresh fruits were extracted with acetone, ethanol and methanol while powders were extracted with deionized water in that study. Differences in encapsulating agents and extraction conditions could cause difference in phenolic content values. Flores et al. [46-48] found TPC of blueberry extract powders as 34.7 mg GAE/g sample with the encapsulating agent ethanol:water:whey protein isolate miscible fluid, and air inlet temperature was 160°C. Tatar Turan et al. [12] studied with ultrasonic nozzle and found TPC as 5.152 mg GAE/g dry powder for extract. The ultrasonic nozzle may provide better protection to bioactive compounds; hence, they might obtain a higher TPC value than this study. In addition, they used encapsulating agent as about 10% (m/m). This could cause to have high bioactive content in the feed solution. The mentioned factors influencing TPC also affect TAC and antioxidant activity because these factors and operational conditions affect the bioactive contents of samples.

Total Anthocyanin Content

TAC result of this study was 1.52±0.09 g C3G/kg dry powder (in extract basis 6.98±0.42 g C3G/kg extract soluble solids, in fresh fruit basis 9.10±0.54 g C3G/kg fresh fruit soluble solids). Jiménez-Aguilar et al. [1] reported total anthocyanin contents of powders between 11.98-15.7 mg C3G/g soluble solids of blueberry extract. This value was higher than the TAC result of this study. Differences in extraction procedures, type of encapsulating agent, initial TAC value of fresh berry could be possible reasons for this difference.

Flores et al. [10] reported TAC of blueberry pomace extract powder as 1.32 mg C3G/g powder which was near the result of this study. Flores et al. [47] calculated TAC of blueberry anthocyanin extract powder between 0.11-0.34 mg C3G/g powder which is lower than the TAC result of this study. This difference could be caused by used encapsulating agents which were Arabic gum and whey protein isolate; used extraction solvents which were ethanol, methanol and acetone and 160°C air inlet temperature. Type of encapsulating agent directly affects the anthocyanin retention and solvent determines the extracted amount of anthocyanins. High temperature could cause degradation of bioactive compounds and reduction in anthocyanin content. Tatar Turan et al. [12] found TAC as 15.176 mg C3G/g dry powder for extract by use of ultrasonic nozzle. The value of that study was higher than the TAC result of this study, and this can be caused by also amount of encapsulating agent (as about 10% (m/m)). This could cause to have high bioactive content in the feed solution.

DPPH Radical Scavenging Activity

DPPH free radical scavenging activity value (EC_{50}) of powder was evaluated as 8.14 mg soluble solids/mL for blueberry extract powder. Atacan and Yanık [7] found the EC_{50} value of blueberry concentrate powder as 38.6 mg/mL. This result indicated that blueberry extract powder has better DPPH radical scavenging activity than concentrate powder. The possible reason of this activity difference could be that the extract had higher antioxidant content than that of the concentrate.

Ferric Reducing Antioxidant Power

Ferric reducing antioxidant power result of this study is 46.41±3.16 µmoles TE/g dry powder (in extract basis 212.87±14.49 µmoles TE/g extract soluble solids, in fresh fruit basis 276.59±18.82 µmoles TE/g fresh fruit soluble solids).

Tatar Turan et al. [12] found ferric reducing powers as 214.468 µmoles TE/g dry powder for extract by use of

ultrasonic nozzle. This value more significant than the FRAP value of this study. The main reason may be the percentage of the encapsulating agent (as about 10% (m/m)). The feed solution contained about 90% (m/m) blueberry extract in that study; therefore, it had a high amount of bioactive compounds with antioxidant activity.

CONCLUSION

In spray drying of blueberry extract, the only significant independent variable was the extract mass percentage on all responses. It was the most influential independent variable on all responses ($p<0.05$). An increase in extract mass percentage (it means that a decrease of encapsulating agent percentage) affected all responses inversely. The optimum conditions obtained from spray drying were 120°C inlet air temperature, 19.51% extract, 20.03 Brix for blueberry. Maximum levels of responses at optimal conditions obtained as phenolic retention of 87.12% and operational efficiency of 91.20%. These extract powders can be utilized as functional food ingredients and food supplements, and they can be added to different types of food products. They could provide nutrients, taste, and color to the foods. Therefore, it can be concluded that spray drying encapsulation of phenolic compounds by using maltodextrin could be an effective way to produce and preserve blueberry extract powders by reason of high phenolic retention and operational efficiency values.

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