SİDAS MEDYA
Akademik Gıda®
ISSN Online: 2148-015X
https://dergipark.org.tr/tr/pub/akademik-gida

Akademik Gıda 22(3) (2024) 167-178, DOI: 10.24323/akademik-gida.1603502

Research Paper / Araştırma Makalesi

Optimization of Spray Drying Parameters and Wall Material Composition of Juniper (*Juniperus drupacea* L.) Extract Using Response Surface Methodology

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Received (Geliş Tarihi): 26.08.2024, Accepted (Kabul Tarihi): 19.11.2024
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ABSTRACT

The aim of this study was to produce a juniper extract powder rich in some bioactive and volatile components such as phenolics, α-pinene and d-limonene with a high yield. For this purpose, the juniper extract, which can be used in various food formulations, was spray-dried under optimized conditions. In this optimization, inlet air temperature (120°C - 180°C) in the spray drying process and the carrier composition were selected as independent variables, while dependent variables included drying efficiency and the total phenolic (TPC), and α-pinene contents of the extract powder. Response surface methodology was used to maximize product yield, TPC and volatile levels, especially α-pinene. The optimum inlet air temperature and carrier ratio were 180ºC and 15 g gum Arabic (GA) per 100 mL extract, respectively. The highest powder yield (37.92%), TPC (9.91 mg GAE/g dm powder) and α-pinene content (peak area 1.3×10^7) were obtained under the optimum conditions while the bulk and compressed bulk densities, TPC and antioxidant activity of the extract powder were 0.39±0.01 g/cm^3 and 0.51±0.02 g/cm^3, 9.89±0.27 g gallic acid equivalent (GAE)/100 g (dm) and 4.12±0.14 g Trolox® equivalent antioxidant activity (TEAC)/100 g dm, respectively. The particle size of the powder produced under optimum conditions ranged from 1.09 to 22.39 µm. Fifteen volatiles in both juniper extract and the reconstituted form of the extract powder were identified, and the major components of juniper extract were d-limonene, α-pinene and γ-muurolene.

Keywords: Juniper, Spray drying, Optimization, TPC and volatile components

Ardıç (*Juniperus drupacea* L.) Ekstrakt Tozu Üretiminde Taşıyıcı Bileşimi ve Püskürtmeli Kurutma Parametrelerinin Yanıt Yüzey Yöntemiyle Optimizasyonu

ÖZ

Bu çalışmada, fenolikler, α-pinen, d-limonen gibi bazı biyoaktif ve uçucu bileşenler bakımından zengin ve yüksek toz verime sahip ardıç (andız) ekstrakt tozu üretimi amaçlanmıştır. Bu amaçla farklı gıda formülasyonlarında kullanılabilir nitelikte toz ürün optimize edilmiş bir koşulda püskürtmeli kurutucuda kurutulmuştur. Bu optimizasyonda taşıyıcı bileşimi ve püskürtmeli kurutucuya giriş havası sıcaklığı bağımsız değişken; kurutma verimi, toplam fenolik madde ve α-pinen içerikleri bağımlı değişkenler olarak seçilmiştir. Ürün verimini, toplam fenolik madde içeriği ve uçucu bileşen seviyeleri, özellikle de α-pinen içeriğini maksimize etmek için yanıt yüzeyi yöntemi kullanılmıştır. Optimum kurutucu giriş havası sıcaklığı ve taşıyıcı oranı sırasıyla 180ºC ve 15 g Arap zamkı/100 mL ekstrakt olarak belirlenmiştir. Optimum koşullarda gerçekleştirilen kurutma işleminde en yüksek toz verimi (%37.92), toplam fenolik madde içeriği

(9.91 mg GAE/g toz) ile α-pinen içeriği (pik alanı 1.3×10^7) elde edilirken tozun yığın ve sıkıştırılmış yığın yoğunlukları ile toplam fenolik madde içeriği ve antioksidan aktivitesi sırasıyla 0.39±0.01 ve 0.51±0.02 g/cm^3, 9.89±0.27 g gallik asit eşdeğeri (GAE) /100 g (km) ve 4.12±0.14 g Trolox® eşdeğeri antioksidan aktivite (TEAC) /100 g km olarak belirlenmiştir. Optimum koşullarda üretilen tozun partikül boyutu dağılımı 1.09 ile 22.39 µm arasında değişmiştir. Hem andız ekstraktında hem de sulandırılmış tozda on beş uçucu bileşen tanımlanırken andız ekstraktının ana bileşenleri d-limonene, α-pinene ve γ-muurolene olmuştur.

Anahtar Kelimeler: Andız, Püskürterek kurutma, Optimizasyon, TPC ve uçucu bileşenler

INTRODUCTION

Juniper (*Juniperus drupacea* (Labill) Antoine and Kotschy) is a perennial tree that is distributed in the Mediterranean countries at altitudes of 600 – 1750 m and constitutes 4% of Türkiye's forests [1-3]. The berries of junipers contain a high amount of reducing sugar (22%), sucrose (11%), oil (4%), protein (2.5%), minerals (2.5%), and essential oils (0.38%) [4-6]. In addition, the fruit extracts of junipers which is mostly used in the production of pekmez [7-9], contain many phenolics such as protocatechuic acid, gallic acid, *p*-hydroxybenzoic acid, catechin, tyrosol, rutin and *m*-coumaric acid, [1, 10] and terpenoids such as α-pinene, camphor and thymol [11].

Juniper berries are commonly used in the form of molasses after extraction, however, there are limited uses in the form of tar and, essential oils for various medical and cosmetic purposes [10-15]. The extract of Juniper berries shows significant antimicrobial effects against some pathogens and molds, especially due to its volatile components. [11, 16, 17]. This extract is widely used in the treatment of helminth infections, stomach pain, hemorrhoids, and diarrhea [7, 18-21]. It has also antioxidant, anticarcinogenic, anti-inflammatory [22], antiarthritic [23], antiviral [24], antitumor [25] and antihyperglycemic [26] effects.

Ripe fruits are unsuitable for direct consumption or addition in food formulations due to their hard texture and extremely astringent taste. For this reason, it can be considered as an alternative that juniper fruit extract can be turned into powder with innovative food processing methods and can be used as a natural food additive to add functionality and enrichment to different products. Powdering of juniper extract, either directly or with carriers, provides various advantages such as increased chemical and microbiological stability due to the decrease in moisture content and water activity (a_w) and decreasing transportation costs by reducing weight and volume [27-29]. The addition of a carrier is a common practice in spray drying of extracts or juices. In this application, both the product yield is increased, and the active ingredients are better preserved.

Interest in the use of fruit and fruit extracts in powder form is increasing. There are various on the use of extract powder in a number of foodstuffs about this subject [30-34]. However, to the best of our knowledge, no study has been found on the optimization of the spray drying conditions of juniper berry extract.

Various processes are applied to obtain the product in powder form; however, spray drying, one of these processes, is frequently used in the food and pharmaceutical industries to convert the liquid product into powders. Spray-dried powders have lower water activity, are easier to process, and are stable against environmental factors such as heat, oxygen, light, etc. [35]. The quality of the powders depends on the feeding solution properties, the contact patterns of the hot air with the droplets in the drying chamber, the type of atomizer, also the temperature, pressure, and flow rate of air in the dryer [36]. Solutions such as extracts and fruit juices with high sugar content can be transformed to powders with lower volume and longer shelf life using this method, but problems with sticking to the spray cylinder occur when drying such solutions. Feed solutions containing low molecular weight sugars and high concentrations of organic acids are the solutions where this problem is most commonly encountered. Sugars and organic acids in such feeding solutions reduce the temperature of glass transition and cause it to stick to inner surface of the drying chamber, resulting in reduced efficiency [37]. By adding carriers such as maltodextrin (MD), β-cyclodextrin (BCD), modified starches and gum arabic (GA) to the solution, this problem can be avoided, and it may be possible to protect sensitive components in foods [38].

The main goal of this study was to produce TPC and α-pinene rich juniper extract powders with high production yield for use in the food industry, by optimizing the inlet air temperature of the spray drying process and the carrier composition. For this purpose, 15 different powders containing MD, GA and BCD at different ratios and inlet temperatures of 100-180°C were prepared according to an optimal (combined) experimental design obtained from the Design Expert 13.0 statistical program. Juniper extract powder produced under optimum conditions was evaluated for quality characteristics.

MATERIALS AND METHODS

Materials

The ripe fruits of juniper trees were obtained from around the same local plantation (920 m altitude) around Gümüşdamla Village, Akseki (Antalya, Türkiye) in November 2020. Akseki is the most intensive juniper plantation in Türkiye. The GA (Alfasol, Istanbul, Türkiye) MD (9 DE) and BCD (Smart Kimya, Izmir, Türkiye), were procured from a local company in Türkiye. The other chemicals (Sigma-Aldrich, Darmstadt, Germany) used in the analyses were purchased from a local distributor.

Experimental Design

Optimization was carried out using response surface methodology with the software of Design Expert 13.0 (Stat-Ease Inc., Minneapolis, MN, USA). An optimal (Combined) experimental design was used to plan the experiments and identify optimum variables, with fifteen treatments carried out for optimization. The independent variables selected were wall material composition and inlet air temperature (120-180°C). The carrier composition is composed of a mixture of MD and GA with a fixed proportion of BCD (1 g/100 mL) in extracts. MD:GA ratio, as determined from preliminary experiments, was adjusted so that its total in the extract was 15 g/100 mL extract. The dependent variables as response selected for optimization were product yield and α-pinene peak area.

Preparation of Juniper Extract

The ripe, brownish fruits were harvested from trees. They were then crushed with a hammer and placed in deionized water (1:4, w/w) for about 3 days until the soluble solids of extract reached 15 °Bx. The extraction process was performed in a jacketed vessel (İldam, Kahramankazan, Ankara, Türkiye) at room temperature with continuous stirring using an overhead propeller type stirrer (Jeio Tech Lab companion MSD-0420, Yuseong-gu, Daejeon, South Korea). The final mixture was filtered through cheesecloth to obtain a particle-free extract [4].

Spray Drying

The mixture of MD/GA (total of 15 g) and 1 g BCD were added to 100 mL of extracts as indicated in the experimental design to give a total weight of 16 g. The obtained solution was then homogenized using an Ultra-Turrax (IKA T25 Digital, Staufen, Germany) at rotation speed of 15,000 rpm for 5 min to ensure uniformity and then spray dried.

In this study, a mini spray dryer (Buchi, B-290, Flawil, Switzerland) equipped with a dual fluid pneumatic nozzle with a diameter of 0.5 mm was co-currently used with compressed air at a flow rate of 600 L/h. The heated air flow rate was maintained at 30 m^3/h and the drying air inlet temperatures of the drying processes were set to the experimental temperature. The feed solution was pumped at an ambient temperature by a peristaltic pump after conditioning. The flow rate was regulated to provide an exact outlet temperature of 80±5°C, corresponding to 240 - 640 mL/h, depending on the soluble solids of the feed solution. During the drying process, the solutions were continuously stirred with a magnetic stirrer at 25°C. At the end of the drying experiments, the powders collected in the collection vessel were transferred to sealed bottles and stored at 4°C for subsequent analyses.

Product Yield

The product yield (PY), calculated using the weight of the solid in the feed solution and the weight of the powder obtained from the collection vessel, was determined gravimetrically as described in Equation (1).

$$PY = \frac{\text{weight of collected powder (g)}}{\text{weight of solid in feed liquid (g)}} \times 100 \quad (1)$$

Moisture Content and Water Activity

The moisture content of samples was determined gravimetrically using a moisture analyzer (Kern DBS, Balingen, Germany) by running samples at 105 °C until constant weight was reached, as described in the instrument manual. Meanwhile, the water activity (a_w) of samples was determined at 25 °C using a a_w analyzer (Aqualab 4TE: Decagon Devices, Pullman, WA, USA).

Color Values

The L* (lightness - darkness), a* (redness - greenness) and b* (yellowness - blueness) color parameters of the samples were determined with a tristimulus colorimeter (CR 400, Konica Minolta Corp., Tokyo, Japan) calibrated with a standard plate (L = 97.02, a* = 0.08, b* = 1.75). The color parameters of the samples (with a minimum sample height of 1 cm) were measured individually in the instrument's special holder for powder samples by placing them on the head of the light source [30,53]. Hue angle, chroma and browning index (BI) values were determined using Equations (2-5).

$$Hue\ angle = \frac{180}{\pi}\ x\ arctan\frac{b^*}{a^*} \quad (2)$$

$$Chroma = \sqrt{b^{*2}+a^{*2}} \quad (3)$$

$$BI = \frac{100(x-0.31)}{0.17} \quad (4)$$

$$x = \frac{a^*+1.75L^*}{5.645L^*+(a^*-3.012b^*)} \quad (5)$$

Solubility

The solubility values were determined according to a previous study [39]. Exactly 0.50 g of the samples were dissolved in 50 mL of distilled water and stirred at 600 rpm for 5 minutes using a magnetic stirrer (Jeio Tech Lab companion, MS-32M, Yuseong-gu, Daejeon, South Korea). Then, the slurry was centrifuged (Sigma 3 K-18, Osterode am Harz, Germany) at 3000g for 5 min. The supernatant (25 mL) was transferred to a Petri dish and kept at 70°C until a constant weight was reached. The percentage solubility (%) was determined from the measurements of water-soluble samples to total samples.

Bulk and Tapped Density

The bulk density (ρ_b) and tapped density (ρ_t) of the samples were determined according to the methods used [40]. The bulk density was determined by measuring the weight and the volume of the powder and then proportioning them to each other. Similarly, the tapped density was determined by measuring the volume of 2 g of powder after tapping on a hard surface until the volume remained constant. The results are expressed in units of g/cm^3. Carr index and Hausner ratio values were calculated by using Equations (6) and

(7) with the values of bulk density and tapped bulk density.

$$CI = \frac{\rho_t - \rho_b}{\rho_t} \quad (6)$$

$$HR = \frac{\rho_t}{\rho_b} \quad (7)$$

Total Phenolic Content

Phenolic substances were extracted from samples according to a method used in a previous study [41] with slight modifications. Specifically, 0.5 grams of the samples were dissolved in 9.5 mL of 80% aqueous methanol solution and homogenized with a homogenizer (Ultraturrax, T 25 IKA Labortechnic, Staufen, Germany) for 10 minutes. The extraction process was performed for 2 hours on an orbital shaking water bath (GFL 1092, Burgwedel, Germany) operating at 150 rpm, set at 50°C. The extracts cooled to room temperature were filtered through filter paper (Whatman No. 42) to obtain clear extracts.

The TPC of clear extracts was determined using the Folin–Ciocalteu method reported in a previous work [42]. The volume of 0.5 mL extract was added into the solutions of 2.5 mL of 0.2 N Folin–Ciocalteu reagent and 2 mL of a 75 g/L Na_2CO_3. The mixture was incubated at 50°C for 5 minutes, then allowed to cool to room temperature. The absorbance of the final mixture was recorded with a spectrophotometer (Shimadzu UV Vis 160 A, Kyoto, Japan) at a wavelength of 760 nm. The aqueous methanol solution (80%) was used as the blank. The results were expressed as milligrams of gallic acid per gram of the powders.

DPPH Radical Scavenging Activity

The radical scavenging activity (%) of the powders were assessed using the DPPH radical inhibition method (1,1-diphenyl-2-picrylhydrazyl, Sigma-Aldrich Chemie, Steinheim, Germany) [43]. In this procedure, 950 µL of a 60 µM DPPH solution was mixed with 50 µL of the diluted extract and allowed to incubate in a dark place at ambient temperature for 30 minutes. The initial absorbance of the DPPH solution was measured against methanol at 516 nm. After incubation for 30 minutes, absorbance of the samples was recorded and the differences in absorbance were calculated relative to the DPPH solution. The antioxidant activity of the samples was then determined based on these absorbance differences. The results were calculated as grams of Trolox equivalent per 100 grams of dry matter (g TEAC/100 g dm).

The percent inhibition of DPPH radicals by each extract was calculated using the following Equation (8).

$$\textit{Radical Scavenging Activity}\ (\%) = [\ Abs_{blank} - (Abs_{sample} / Abs_{blank})\ ] * 100 \quad (8)$$

Volatile Composition

Volatile component analyses of *Juniper extract* powder, feeding solution and rehydrate powder were conducted using the GC/MS (QP 2000, Shimadzu, Kyoto, Japan) with the solid phase microextraction (SPME) method by using an autosampler (Combi PAL, CTC Analytics, Zwingen, Switzerland).

Volatile compounds were extracted from the dissolved powders. The powders were rehydrated to a final concentration of 20°Bx. Exact amount of 2 g rehydrated powders were transferred into 20 mL screw-cap vials and they were sealed with PTFE septum. Then the reconstituted samples were incubated in the vials at 40°C for 15 minutes while being agitated at 250 rpm. During the incubation, a fiber coated with 50/30 µm DVB/CAR/PDMS (Supelco, Pennsylvania, USA) was used to absorb the volatile compounds. After incubation, the fiber was desorbed in the injection port of the GC for 10 minutes at 200°C, with a split ratio of 1:5. Separation of volatile compounds was carried out using a fused silica column (TRB5-MS, 30 m x 0.25 mm i.d. x 0.25 µm film thickness). Helium (99.99% purity) was used as the carrier gas at a constant flow rate of 1.78 mL/min. Temperature of the column was programmed to hold at 40°C for 3 min, then increase to 100°C at 4°C/min (held for 3 min), and then to 200°C at 10°C/min (held for 3 min). The ion source, injection port, and detector temperatures were maintained at 200°C, 200°C, and 275°C, respectively. The MS conditions were set to electron impact (EI) at 70 eV, with scanning range of 30–500 m/z and scanning rate of 769 scans/s. Volatile compounds were identified by comparing mass spectral data with the Wiley 10 and NIST libraries, as well as retention indices [11, 44].

Particle Size

The particle size of the powder was assessed using the light scattering technique with a particle size analyzer (Mastersizer 2000 instrument, Malvern, Worcestershire, UK). This instrument was equipped with a liquid dispersion unit called Hydro 2000S. 2-Propanol was used as a dispersant to disperse the spray-dried powder. The particle size measurements were conducted with this dispersion at a temperature of 25°C. The refractive index of the particles was 1.52, while the dispersant refractive index was 1.39. The particle size analysis yielded two parameters: the surface mean diameter (D_{32}) and the volume mean diameter (D_{43}). Additionally, the particle size distribution of the powder was assessed using the span, calculated according to Equations (9-11) [45, 52]

$$D_{32} = \frac{\Sigma n_i d_i^3}{\Sigma n_i d_i^2} \quad (9)$$

$$D_{43} = \frac{\Sigma n_i d_i^4}{\Sigma n_i d_i^3} \quad (10)$$

$$Span = \frac{d_{90} - d_{10}}{d_{50}} \quad (11)$$

Where n_i is the number of particles of diameter d_i and d_{90}, d_{50}, and d_{10} are the equivalent volume diameters at 90%, 50%, and 10% cumulative volumes, respectively.

Statistical Analyses

The experiments were carried out in duplicates. All analyses were performed in triplicates. The results were evaluated in statistical analyses in an optimal (Combined) experimental design, while the response variables were estimated by a polynomial Equation (11) given below:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 + \beta_{133} x_1 x_3 x_3 + \beta_{233} x_2 x_3 x_3 \quad (12)$$

where x_1, x_2, and x_3 are independent variables corresponding to MD, GA and the inlet air temperature. Y and β are the optimized dependent variables and constant of coefficient, respectively. The values of probability (*p*) were used to determine the significance and effective levels of the model. Regression analyses were performed using Design Expert 13.0 (Stat-Ease Inc., Minneapolis, MN, USA) software to determine the coefficient of determination (R^2), analysis of variance (ANOVA), F-tests (lack of fit). The response surface methodology (RSM) was carried out according to the desirability function as given in following Equation (13):

$$D = (d_1 \times d_2 \times \ldots \times d_m)^{1/m} \quad (13)$$

where, m is the number of responses and d is the desirability of individuals. Individual desirability (d) evaluates how the settings optimize a single response while composite desirability (D) evaluates how the settings optimize a set of responses overall. The desirability functions were estimated as the product yield and α -pinene were maximum. The optimum points were determined by considering the highest values among the calculated desirability functions.

RESULTS and DISCUSSION

Optimum Drying Conditions for Juniper Extract

The juniper extract was dried with the mixtures of carrier materials to produce juniper extract powder with high phenolic content, α-pinene content and product yield. The ratio of the MD, GA, and optimum inlet air temperature were selected as independent variables. BCD proportion was fixed at 1 g/100 mL for each extract due to solubility concern. In the optimization experiments, it was decided to use a total of 15 g/100 mL of carrier materials, as the combinations of MD and GA in the proportions determined by the program (Table 1). The limit values of the independent variables were determined as regard to literature review and some preliminary tests. Three-dimensional surface graphs illustrating binary interactions of the variables were generated using Design Expert 13.0 software.

Table 1. The levels of factors and their effect on response variables (product yield, α–pinene area and total phenolic contents) used in the optimization of spray drying conditions

Run*	MD* (g/100 mL extract)	GA (g/100 mL extract)	Inlet Temperature (°C)	PY (%)	α-pinene (Peak area × 10^6)	TPC (mg GAE /g dm)
1	11.25	3.75	165	35.44	6.67	9.09
2	7.50	7.50	180	39.39	5.30	9.89
3	7.50	7.50	150	30.67	6.69	9.14
4	0.00	15.00	120	31.22	12.00	9.95
5	15.00	0.00	150	27.12	4.06	8.81
6	0.00	15.00	180	35.82	14.14	10.39
7	0.00	15.00	120	28.90	9.94	9.87
8	3.75	11.25	135	34.09	6.11	9.42
9	15.00	0.00	180	33.48	1.25	8.20
10	15.00	0.00	180	34.24	2.83	8.46
11	11.25	3.75	135	34.86	3.03	8.95
12	7.50	7.50	120	33.96	7.41	8.93
13	15.00	0.00	120	33.95	2.18	8.52
14	0.00	15.00	150	26.31	5.43	9.32
15	0.00	15.00	180	38.81	1.33	10.00

* For all runs, the amount of β-cyclodextrin was 1 g per 100 mL extract. **MD: Maltodextrin, GA: Gum Arabic, PY: Product Yield, TPC: Total Phenolic Content

The impact of the three variables on the response is depicted in Figures 1-3. According to the statistical analysis presented in Table 2; MD x AG, AG x T_{inlet} and T_{inlet} x T_{inlet} interactions were identified as the significant factors ($P < 0.05$) affecting the product yield. As seen in Figure 2, a saddle formation is present in the graph. The saddle peak is observed in the region where the ratios of the carriers in the mixture are close to each other, but the gum Arabic is relatively higher at the drying temperature of 180°C. This may be related to the shorter drying time of the particles in the drying chamber and the fact that mixtures containing GA adhered less to the glass drying chamber of the unit.

Table 2. The result of response surface methodology analysis for spray drying optimization

Legends		Product Yield (%) KCV model × KCV model		α - Pinene (area) Linear × Quadratic		TPC (mg/g) Linear × Quadratic	
		Coefficients	*P*	Coefficients	*P*	Coefficients	*P*
B_0	Constant	0	0.0045*	0	0.0001*	0	0.0002*
B_1	MD	28.42		4.68×10^6		8.89	
B_2	AG	27.98		6.01×10^6		9.41	
B_{12}	MD × AG	18.21	0.0064*				
B_{13}	MD × T_{inlet}	0.11	0.9225	-8.51×10^4	0.9149	0.0231	0.8628
B_{23}	AG × T_{inlet}	3.59	0.0048*	1.11×10^6	0.1345	0.2231	0.0794
B_{33}	T_{inlet} × T_{inlet}	5.41	0.0023*				
B_{133}	MD × T_{inlet} × T_{inlet}			2.79×10^6	0.0780	-0.4445	0.0914
B_{233}	AG × T_{inlet} × T_{inlet}			6.15×10^6	0.0016*	0.6852	0.0163*
	Lack of Fit		0.2973		0.3002		0.3264
R^2	Adjusted R^2	0.8106	0.7053	0.9187	0.8735	0.9112	0.8619
A.P.	Predicted R^2	9.2523	0.5427	12.6573	0.8173	12.5024	0.7652

*Values with *P*<0.05 are statistically significant. (A.P.: Adequate precision)

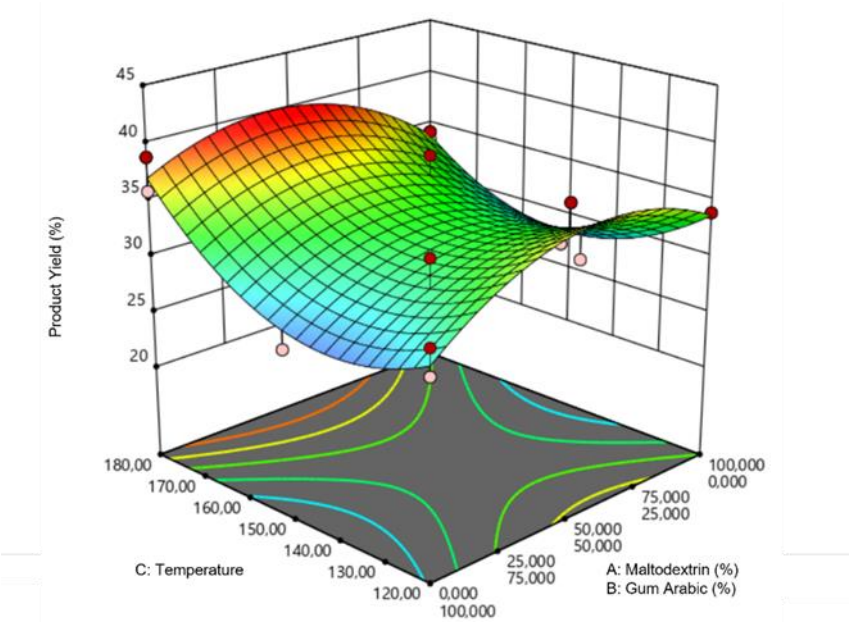

Figure 1. The effect of temperature, maltodextrin and gum *Arabic* amounts on product yield

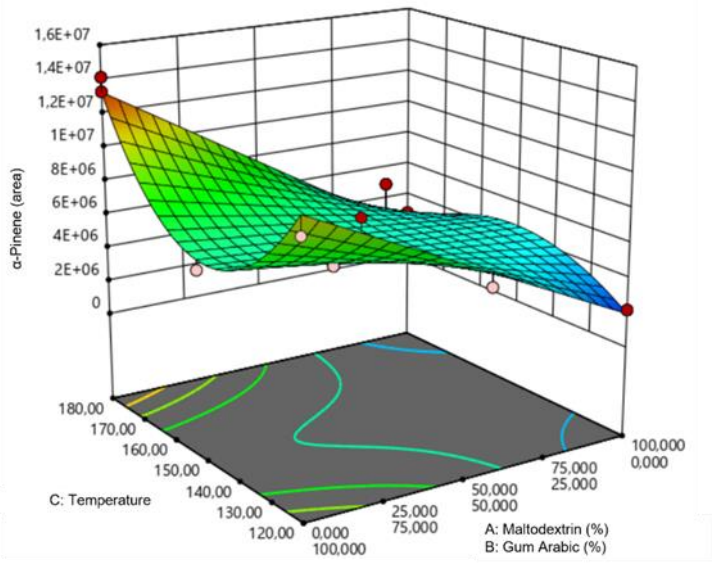

Figure 2. The effect of temperature, maltodextrin and gum Arabic amounts on α-pinene area

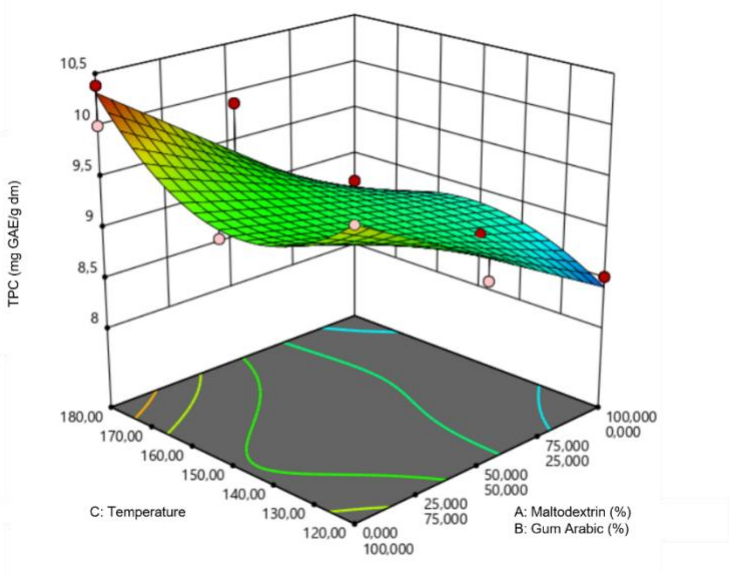

Figure 3. The effect of temperature, *maltodextrin* and *gum Arabic* amounts on total phenolic contents

According to the statistical analysis presented in Table 2, the interaction of MD × AG × T_{Inlet} was determined as the significant factors (P<0.05) affecting the α-pinene area. As seen in three-dimensional surface graphics illustrated in Figure 2, the α-pinene area increased as the amount of gum Arabic increased. In terms of temperature changes, an inverted hump formed in the graph; α-pinene area, which was at a minimum at the midpoint, increased with both an increase and a decrease in temperature. The increase in temperature boosted the α-pinene area more significantly. It is evaluated that this is due to the reduction in the loss of volatile components caused by the shortened drying time in the drying chamber with the increase in temperature.

The effects of independent variables on the TPC of the powder can be clearly seen in three-dimensional surface graph (Figure 3). The TPCs of the powder increased with increasing GA in the carrier mixture, but decreased with increasing MD. However, the effect of the carrier composition on the TPC was also associated with the drying temperature. Indeed, at drying temperatures around midpoint, the TPC of the powder decreased with higher AG content in the carriers, but it increased with the lower AG content. This interaction of AG × T_{Inlet} × T_{Inlet} was determined to be significant (p<0.05) on the TPC content (Table 2).

Optimized Spray Drying Conditions

Spray drying temperature and carrier composition were optimized to achieve maximum yield, α-pinene area and TPC. The optimum inlet air temperature, and the ratios of MD and GA were estimated as 180°C, 0 and 15 g/100 mL extract, respectively with a maximum soluble amount of BCD (1 g/100 mL extract). Using this condition, PY, α-pinene area and TPC were theoretically estimated as 36.89%, 13267352 and 10,32 mg/g dm, respectively. The theoretical results were experimentally verified and the difference between the results of the optimized dependent variables was below 6% (Table 3).

Table 3. Theoretical optimal spray drying conditions and experimental validation

	MD* (g/100 mL extract)	GA (g/100 mL extract)	T_{Inlet} (°C)	PY (%)	α – Pinene (peak area)	TPC (mg/g dm)
Theoretical	0	15	180	36.892	13.27×10^6	10.317
Experimental	0	15	180	35.284	12.49×10^6	9.886
Error (%)				4.557	5.890	4.178

*MD: Maltodextrin, GA: Gum Arabic, PY: Product Yield, TPC: Total Phenolic Content

Powder Analysis

Moisture Content and Water Activity

The moisture content and a_w of the juniper extract powder produced through the spray drying process were determined as 0.24±0.01 and 4.44±0.33 g/100 g, respectively (Table 4). Similar moisture content and a_w results of spray-dried fruit powders were reported in range of 2–5% and 0.2–0.6, respectively [46]. Based on the classification reviewed by [34], food powders are considered hygienic if they have a moisture content of less than 10% and a a_w of less than 0.6. Therefore, the current moisture content and a_w results of the juniper extract powder correspond to a stable and hygienic product.

Color Values

The color of many products mostly changes by thermal food processes, and they are simply characterized by L*, a*, b*, Hue Angle, and Chroma values. In this respect, the color properties of the juniper extract powder were measured as 87.15±0.30 for L*, 1.25±0.19

for a*, 16.32±0.85 for b*, 85.65±0.43° for hue angle, and 16.37±0.86 for chroma (Table 4). These color parameters indicate a light, slightly reddish yellow with moderate saturation. It is close to a pure and pale yellow but has a hint of red which makes it warm. The high lightness and moderate chroma of the color give it a vivid but not overwhelming appearance.

Table 4. Quality properties of juniper extract powder

Quality properties (unit)		Values
Product yield (%)		35.28±3.85
Moisture content (%)		4.44±0.33
Water activity (a_w)		0.24±0.01
Color values	L*	87.15±0.30
	a*	1.25±0.19
	b*	16.32±0.85
	h°	85.65±0.43
	C*	16.37±0.86
Browning index		21.34±1.42
Solubility (%)		78.30±0.54
Bulk density (g/cm^3)		0.39±0.01
Taped density (g/cm^3)		0.51±0.02
Carr index		23.81±0.81
Hausner ratio		1.31±0.01
Particle distribution	Span	2.15±0.05
	D_{43}	11.30±0.30
	D_{32}	3.49±0.15
HMF (mg/kg)		21.37±0.61
TPC (g GAE/100 g dm)		9.89±0.27
Antioxidant activity		4.12±0.35

Solubility

The water solubility value of juniper extract powder was determined to be 78.3% which indicates moderate solubility characteristic in water (Table 4). This result is in agreement with a number of previous work which indicated that the solubility is related to the use of 100% GA [33, 47].

Solubility is a critical quality parameter of powdered products that can significantly affect the reconstitution behavior of food powders. High solubility is particularly desired in food powders, especially for instant products. This property is primarily influenced by the main constituents, moisture content and particle size of the powders [48]. Previous studies reported that fruit powders produced by spray drying have high or moderate solubilities with the solubility values ranging from 70% to 99% [49, 50].

Bulk and Tapped Density

The bulk and tapped densities of the obtained juniper extract powders were determined as 0.39±0.01 g/cm^3 and 0.51±0.02 g/m^3, respectively (Table 4). In previous studies on spray drying of different raw materials, bulk densities were calculated between 0.20 - 0.60 g/cm^3 [49, 51-53]. The results of the present study are consistent with the findings of several previous studies. As these parameters affect the storage, stability and flow behavior of powdered products, they are important for food powders.

TPC

The TPC of the juniper extract powder was determined as 9.89±0.27 g GAE/100 g dm (Table 4). This result is relatively higher than that of a previous study (4.81 g GAE/100 g extract) performed on extract of juniper by [10], [54]. The higher value of the TPC result is most likely attributed to the calculation in dry matter. It may also be related to the origin of the fruits, harvesting time and the processing conditions of the powder.

DPPH Radical Scavenging Activity

The DPPH radical scavenging activity method relies on the reduction of the DPPH radical by antioxidants that donate hydrogen and electrons. The antioxidant activity of juniper extract powder was determined to be 4.12 g of TEAC per 100 grams of dry matter. These antioxidant compounds determining the DPPH radical scavenging activity can mitigate the detrimental effects of free radicals in cellular systems [55].

Volatile Composition

The juniper extract powder produced in optimized condition were tested in characteristic volatile compounds to compare feeding solution, reconstituted powder and powder. Fifteen volatile compounds were identified for both extract and reconstituted powder. d-limonene, α-pinene and γ–muurolene were determined to as the main components of the extract. These main components were also determined as the main volatile components of the reconstitute powder, but their peak areas percentage were determined to be lower in comparison to the feeding extract. The optimized encapsulation process performed by the spray drying process enabled to keep 80.98% of the volatile component. The results obtained from the reconstituted samples showed that about 20% of the total volatiles were lost during the drying process. It is noteworthy that the highest encapsulation among the identified volatile

components could be provided in the β-pinene component, followed by α-selinene, d-limonene, β-myrcene and α-pinene. On the contrary, the highest losses among the volatile components were determined in α-muurolene, α-copaene and δ-cadinene. The volatile release from powder form of samples was 0.53%, indicating that an effective encapsulation was achieved. In terms of volatile release from the powders, the other components were not detected in the headspace of the samples.

Particle Size Index

The particle sizes of the juniper extract powder were measured and calculated as both volume-weighted mean representing the average diameter of a sphere with equivalent volume, and surface-weighted mean representing the average diameter of a sphere with equivalent surface area. The particle sizes were determined in range between 1.091±0.21 and 22.390±0.40 µm (Figure 4). The surface mean diameter (D_{32}), volume mean diameter (D_{43}), and span values of the powder were calculated as 3.495±0.15 µm, 11.298±0.30 µm, and 2.150±0.05, respectively (Table 4).

The particle size values were mostly influenced by wall and core materials composition, type of atomizer and spray drying conditions. The particle size of the juniper extract powder obtained in this study were significantly smaller than those reported in a previous study [56] performed on spray dried berry.

Table 5. Volatile composition of feeding solution, powder (aroma gain) and powder (aroma release)

RI TRB5-MS	Compounds	Feeding solution		Powder (Volatile Gain)		Powder (Volatile Release)	
		Area	%	Area	%	Area	%
910-925	α-pinene	3.22×10^7	24.30	2.56×10^7	19.30	1.01×10^5	0.08
952	β- pinene	1.86×10^6	1.41	1.74×10^6	1.32	-	
968	β- myrcene	8.15×10^6	6.15	6.54×10^6	4.94	-	
1007	D- limonene	5.31×10^7	40.08	4.72×10^7	35.62	-	
1321-1349	α- cubebene	4.55×10^5	0.34	7.06×10^5	0.53	-	
1343-1391	ylangene	1.65×10^6	1.24	8.86×10^5	0.67	-	
1349-1401	α-copaene	7.03×10^6	5.30	3.74×10^6	2.82	5.52×10^4	0.04
1391-1445	β- ylangene	4.65×10^5	0.35	7.46×10^5	0.56	-	
1401-1448	β- cubebene	6.32×10^5	0.48	8.83×10^5	0.67	-	
1427-1452	α- humulene	2.12×10^5	0.16	3.90×10^5	0.29	-	
1468-1491	α - selinene	3.57×10^5	0.27	3.30×10^5	0.25	-	
1452-1468	α- amorphene	3.57×10^5	0.27	2.74×10^5	0.21	-	
1448-1485	γ - muurolene	1.49×10^7	11.27	1.15×10^7	8.68	3.59×10^5	0.27
1471-1493	α- muurolene	2.78×10^6	2.10	1.45×10^6	1.09	-	
1485-1508	δ- cadinene	8.34×10^6	6.19	5.35×10^6	4.04	1.92×10^5	0.15
	Total	1.33×10^8	100.00	1.07×10^8	80.98	7.07×10^5	0.53

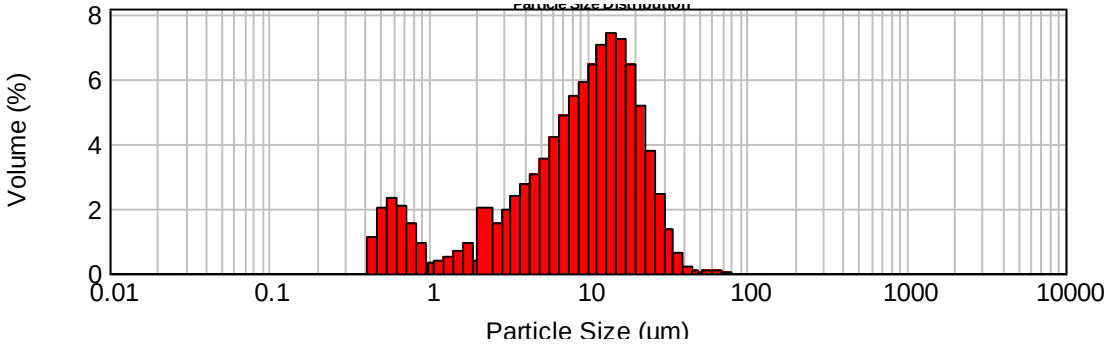

Figure 4. Particle distribution of juniper extract powder

CONCLUSION

In this study, for the first time, the production conditions of high-yield juniper extract powders rich in some bioactive and volatile components such as phenolics and α-pinene were optimized using spray drying. The sample using only GA as a wall material and powdered at an inlet temperature of 180°C maximized the responses obtained. The optimized spray drying encapsulation process enabled 80.98% of the volatile component to be retained. The volatile release in the final powder was 0.53%, indicating that an effective encapsulation was achieved. Fifteen volatile compounds were identified for both extract and reconstituted powder. d-limonene, α-pinene and γ–muurolene were determined to as the main components of the extract. Considering the physicochemical properties, juniper extract powder produced under optimized spray-drying conditions can enrich many food formulations in terms of flavor and bioactive components. Nevertheless, further studies should be carried out to improve particle distribution and increase product yield.

ACKNOWLEDGEMENTS

The authors thank Akdeniz University (Antalya, Türkiye) for the use of the laboratory and the Council of Higher Education of the Republic of Turkey 100/2000 program for financial support.

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