Modeling and optimization of the spray drying parameters for soapwort (*Gypsophila* Sp.) extract

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Abstract In this study, the effects of spray drying parameters as feed soluble solid content, inlet air temperature and outlet air temperature on spray drying of soapwort (*Gypsophila arrostii*) extract have been examined. Central composite design was used with 15–30 °Bx, 110–160 °C and 50–80 °C for feed soluble solid content, inlet air temperature and outlet air temperature, respectively. Moisture content, water activity, tapped density, total saponin content and foam volume of the obtained powders were measured as responses of the experimental plan. The spray drying parameters were optimized by considering saponin content and foam volume by response surface methodology. According to the numerical optimization, optimum feed soluble solid content, inlet and outlet air temperatures were 15 °Bx, 160 °C and 67 °C respectively with a desirability factor of 0.822. After verification at optimum conditions, the error percentages were found to be 5.96% and 2.15% for saponin content and foam volume respectively.

Keywords Spray drying · Response surface methodology (RSM) · Maltodextrin · Soapwort extract · Saponin

Introduction

Soapwort (*Gypsophila arrostii*) is a plant that grows naturally in Southern Asia. The roots of soapwort plant contain high concentration of saponin, which is a glycoside. Traditionally, the roots of soapwort have been used for the preparation of remedies against coughs and ailments of the upper respiratory system and also for the production of a variety of other products such as cleaning chemicals, film emulsion, in confectionery as a stabilizer of froth and as an ingredient in fire extinguishers (Çelik et al., 2006). The aqueous extract of soapwort root has widely used in Turkish halva and delight production in Turkey because of its high saponin concentration. In halva production, soapwort root extract is used to give characteristic properties to halva by whitening the syrup, to prevent the separation of sesame oil by acting as an emulsifier, to improve the textural properties and to increase the volume. Also the foam obtained after whipping soapwort root extract gives softness and desired texture to white-foamy Turkish delight (Sultan delight) (Çelik et al., 2006; Ceyhun et al., 2010; Guneser and Zorba, 2014; Ozdikicierler et al., 2014). Some industrial producers prepare aqueous extract of soapwort root and supply this to halva or delight producers. Due to high microbial sensitivity of this extract, some microbial preservatives such as sodium benzoate and potassium sorbate are used to enhance the durability of the soapwort extract. For this reason, reducing the water activity by drying of soapwort extract presents an opportunity to produce a stable product without the addition of these kind of chemical preservatives.

Most of the plant extracts needs further treatments for storage due to their high sugar concentration, moisture content, respiration rate and nutritional aspects. In food science and technology, the drying of a food is a

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widespread operation both for the preservation of foods and production of food materials, this is also true for the plant extracts with desired quality characteristics. The production of extract powders by using spray drying operation has become more popular in last decade in food industry (Bringas-Lantigua et al., 2011; Jafari et al., 2017). Spray drying is the transformation of liquids into powders; by simply atomizing them in a hot gas stream to instantaneously obtain the powders. Microencapsulation by spray drying of various plant juices and extracts, is one of the mostly used encapsulation methods due to the wide availability of the equipment, low processing costs, the possibility of using a wide variety of coating material and good final product stability (Gharsallaoui et al., 2007; Rocha et al., 2009). There exists several recent studies in literature related with spray drying of plant juices and extracts (Abadio et al., 2004; Cano-Chauca et al., 2005; Chegini and Ghobadian, 2005; Papadakis et al., 2006; Quek et al., 2007; Jangam and Thorat, 2010; Mestry et al., 2011; Boonchu and Utama-Ang, 2013; Bicudo et al., 2015; Ramírez et al., 2015; Saikia et al., 2015; Cortés-Rojas et al., 2015; Muzaffar and Kumar, 2015). Many different coating materials are being used for microencapsulation with spray drying. These coating materials dissolved in feed prior to spray drying operation. Coating materials which are generally used for encapsulation are maltodextrin, modified starch, protein conjugates, gums and pectin (Gharsallaoui et al., 2007; Jafari et al., 2017). The coating material covers the material to be encapsulated and acts as a physical barrier to oxygen and light to ensure protection from chemical and enzymatic destruction (Saikia et al., 2015). Among all coating materials, maltodextrin is largely considered due to its better water solubility which contributes to significant reduction of the viscosity of feed which is important for better atomization of the feed (Pierucci et al., 2007). Utilization of low dextrose equivalent maltodextrin in spray drying, ensure the production of encapsulated plant extract powders that have lower moisture content (Ersus and Yurdagel, 2007).

Response surface methodology (RSM) is a statistical methodology which is used for optimization, modeling and analysis of experimental data simultaneously that includes different variables with the help of several experimental design systems that has been successfully used for developing, improving and optimizing in different systems (Pishgar-Komleh et al., 2012; Ramírez et al., 2015). The application of RSM consists of these stages; determination of the multi-response parameters and effective levels, selection of experimental design, prediction and verification of the model equations, generating response surfaces and determination of optimum conditions (Balasubramani et al., 2015).

Because of the industrial usage of soapwort extract and its foam, optimization of soapwort extract powder production parameters, became a noteworthy subject for scientific and industrial aspects. In this manner, our study was focused on determining the feasibility of microencapsulation of soapwort extract using spray drying operation and modeling the effects of spray drying process parameters into polynomial equations. Feed soluble solid content ($^{\circ}\text{Bx}$), inlet air temperature ($^{\circ}\text{C}$) and outlet air temperature ($^{\circ}\text{C}$) were selected as process factors while moisture content, water activity, tapped density, total saponin content and foam volume of the powders were selected as responses. Process factors were optimized in order to obtain a soapwort extract powder having high saponin content and good foam producing property by using RSM.

Materials and methods

Soapwort roots were supplied from a Turkish commercial soapwort extract producer in grounded form, which is suitable for extraction. Soapwort root extract was obtained by sequential boiling of soapwort roots with water in seven extraction stages. For each extraction stage, soapwort root:water ratio was kept as 1:5 and extractions was continued until $\frac{1}{4}$ of water (by volume) had been vaporized. Finally, extracts obtained from all extraction stages were mixed and concentrated to 10 $^{\circ}\text{Bx}$ in an evaporator. Then, the final extracts were filled into glass bottles and stored at $-25 \pm 1^{\circ}\text{C}$ until spray drying experiments. Low dextrose equivalent maltodextrin (Dextrose Equivalence of 10–12) was obtained from a local distributor (Smart Chemicals, İzmir/Turkey). All chemicals were of analytical reagent grade with high purity (Merck, Darmstadt, Germany).

Spray drying operation

Prior to spray drying process, maltodextrin was added to the extracts according to experimental design to increase the soluble solid content of the extract to desired values of soluble solid contents (Ersus and Yurdagel, 2007; Özdikicierler et al., 2014). The samples were spray dried with a spray dryer (Niro-Atomizer Mobile Minor, Soeborg, Denmark) at different inlet air and outlet air temperatures. Face Central Composite Experimental Design was used for spray drying operations. Upper and lower limits of feed soluble solid content, inlet air and outlet air temperature levels are given in Table 1. During spray drying process, the air pressure of atomizer was applied at a constant speed of 296 kPa. For each combination of inlet air temperature and feed soluble solid content, the feed flow rate was adjusted to attain the desired outlet air temperature. After

Table 1 Levels of factors used in the experimental design

Factor	Independent variables	Variable levels		
		− 1*	0*	+ 1*
X ₁ (A)	Feed soluble solid content (°Bx)	15	22.5	30
X ₂ (B)	Inlet air temperature (°C)	110	135	160
X ₃ (C)	Outlet air temperature (°C)	50	65	80

* − 1, 0 and + 1 are factor codes

spray drying operation, soapwort extract powders were placed in well-sealed glass jars and stored in a dry and dark place until further analyses.

Analytical techniques

Moisture content, water activity, tapped density, total saponin content and foam volume were determined in triplicate for the powdered samples.

Moisture content of the samples was determined by using AOAC method 920.151 and water activity (a_w) was determined with a water activity measurement device (Testo – AG 350XL, Testo Inc, Lenzkirch, Germany) at a constant room temperature of 22 ± 3 °C.

Tapped density of the samples was determined by placing 20 g of powder in a 100 mL graduated cylinder and measuring the volume after the sample was gently dropped 120 times onto a rubber mat from a height of 15 cm (Chegini and Ghobadian, 2005).

Modified methods described in previous studies were used for total saponin content determination (Ceyhun Sezgin and Artik, 2010; Chen et al., 2013). Powder samples were reconstituted with water to initial feed soluble solid content (°Bx) prior to analysis. Reconstituted samples were extracted with 50 mL diethyl ether and solvent phase discarded. After diethyl ether extraction, saponins were extracted using n-butanol (5×15 mL) and n-butanol was evaporated using a rotary evaporator. Residue was mixed with diethyl ether:methanol mixture (1:1). The mixture was centrifuged (4000 g, 20 min) and supernatant (solvent phase) was discarded. Precipitate was dried in desiccator, transferred to a 25 mL volumetric flask and made up to the mark with methanol. The solution was filtered through a 0.45 mm Millipore filter before HPLC analysis. The HPLC system consisted of a binary pump (Agilent 1200, Agilent, Germany), an auto sampler and a DAD detector (Agilent 1200 Series G1315B DAD, Agilent, Germany) at 210 nm for all analyses. Chromatographic separation was carried out on an Altima C18 column (4.6×250 mm, 5 mm). The mobile phase consisted of acetonitrile (A) and water using the following gradient program: 0–10 min, 65.0%A, 10–20 min, 65.0–85.0%A, 20–30 min, 85%A. Saponin

standard (Merck Lot no: 558255) was used for quantification of saponins. Results were given as percentage of saponin in the sample.

For the determination of the foam volume, powder samples were used in reconstituted form. To exclude the confounding effect of differences in the soluble solid contents of the samples on foam formation and to ensure a reliable comparison, all powder samples were reconstituted to 10 °Bx. The reconstituted extracts were treated for five minutes with Ultra Turrax T25 (IKA, Alberta, USA) at 15,000 rpm in 140 mL volumetric glass and foam volume was directly measured.

Statistical analysis and evaluation

The experimental plan was developed by using Face Central Composite Experimental design according to RSM (Myers et al., 2011). Design Expert[®] for Windows version 7.0 (StatEase, Inc., Minneapolis, MN, USA) was used for the statistical evaluation and modeling with a second-order polynomial equation to determine the coefficients of the response model as well as their standard errors and significance. Limits of the independent variables which are given in Table 1, were determined after a series of preliminary experiments.

The response models with all quadratic terms included were similar to the basic equation shown in Eq. (1).

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_{i < j} \beta_{ij} X_i X_j \quad (1)$$

where Y is the predicted response, X_i , X_j represent the levels of the factors according to Table 1 and β_i , β_j represent coefficient estimates with β_0 having the role of a scaling constant. The backward substitution method was used to develop models after the addition of all quadratic terms to the equation. Models were evaluated in detail using PRESS, regression coefficient (r^2), and adequate precision (A-Pres) values. Repeatability and reproducibility of the developed models were discussed by using the coefficient of variances (CV). A model having a CV value lower than 10, has better repeatability and reproducibility (Chen et al., 2012).

For the verification of the optimization process, additional spray drying operations were performed at optimum spray drying conditions. On the other hand, optimized responses were predicted at 11 different process conditions which were not included in the experimental design and spray drying operations were performed at these process conditions. All predicted values were compared with actual values via difference percentage (Error %) between predicted and actual values. The formula of error (%) was given in Eq. (2).

$$\text{Error (\%)} = (\text{EV} - \text{PV}) \times 100/\text{EV} \quad (2)$$

where EV is the experimental value obtained from the spray drying operations, PV is the predicted value which is calculated from optimization model (Boonchu and Utama-Ang, 2013).

Results and discussion

Statistical evaluation and modeling of the results

The experimental results revealed that soapwort extract was effectively dried by using a spray drier. We obtained powder samples from collector showing an effective drying. Powders had acceptable values for all the analyzed properties. These preliminary observations showed that spray drying parameters were determined in a suitable range for the study.

The results of the measurements on moisture content, water activity, tapped density, total saponin content and foam volume are shown in Table 2.

According to the results, all the powder samples had relatively low moisture levels desired for a powder product. The highest moisture level was observed as 5.23% (run 6)

while lowest moisture content was 1.14% (run 11) among all powder samples. No cloaking were observed among samples. Microbial growth, especially growth of molds is a limiting factor for storage of powder product. It's a well-known fact that the water activity (a_w) level of a reconstitutable powder product should be lower than 0.6 in order to maintain a good prevention against any microbial activity (Ray and Arun, 2013). In our study water activity (a_w) levels of powder samples varied between 0.054 (run 5) and 0.338 (run 15) showing prospect for a high resistance against microbial growth. A previous study reported that a_w levels of spray dried grape pomace extract powders were determined between 0.15 and 0.31 (Boonchu and Utama-Ang, 2013). Also another study indicated that a_w levels of spray dried sumac extract powders were varied between 0.157 and 0.215 (Caliskan and Dirim 2013). These results which are about a_w levels of spray dried plant extract powders are in agreement with our results. The tapped density is an increased bulk density attained after mechanically tapping a container containing the powder sample. Tapped density and bulk density are informative values for powder products and higher values of these properties are expected (Jafari et al., 2017). Tapped density values varied between 375.6 kg/m³ (run 1) and 610.7 kg/m³ (run 6) for powder samples obtained in our study. The

Table 2 Results of spray drying operations

Runs	A-feed soluble solid content (°Bx)	B-inlet air temperature (°C)	C-outlet air temperature (°C)	Moisture (%)	a_w	Tapped density (kg/m ³)	Total saponin content (%)	Foam volume (ml)
1	15	160	80	1.77	0.100	375.6	7.07	97.0
2	22.5	135	65	2.16	0.141	470.6	4.35	100.0
3	15	160	50	3.76	0.237	379.1	6.98	98.5
4	22.5	135	80	1.73	0.098	425.5	4.60	101.3
5	30	110	80	1.24	0.054	516.1	2.88	83.3
6	30	160	50	5.23	0.285	610.7	2.85	84.5
7	15	135	65	2.62	0.180	430.1	7.44	95.0
8	22.5	135	65	2.52	0.138	476.2	4.52	97.0
9	15	110	50	4.36	0.253	500.0	7.34	83.5
10	30	135	65	3.00	0.160	540.5	3.04	79.0
11	15	110	80	1.14	0.094	462.4	7.18	95.3
12	22.5	135	65	3.38	0.183	479.0	4.39	96.8
13	22.5	135	65	2.94	0.174	476.2	4.47	94.3
14	22.5	135	65	2.32	0.144	459.8	4.15	101.5
15	22.5	135	50	5.07	0.338	526.3	4.30	94.0
16	22.5	110	65	1.46	0.111	500.0	4.66	97.5
17	22.5	160	65	2.86	0.127	470.6	4.75	105.3
18	30	110	50	5.20	0.299	571.4	2.88	77.5
19	22.5	135	65	2.68	0.159	481.9	4.43	90.0
20	30	160	80	1.24	0.095	503.1	3.12	84.0

importance of foam forming capacity of a reconstituted soapwort extract powder was described under introduction. For the determination of foam forming capacity, in order to exclude the effect of differences in the soluble solid contents of the samples on foam formation and to ensure a reliable comparison all powder samples were reconstituted to 10 °Bx. The measured foam volume values varied between 105.3 mL (run 17) to 77.5 mL (run 18) in our study.

Results given in Table 2 were used in order to generate mathematical models showing the relationship between responses and process parameters. For every response, initially all quadratic terms were added to model equation and insignificant terms ($\alpha = 0.1$) were excluded to develop the models using backward substitution method. Main independent variables of the study (feed soluble solid content, inlet air temperature and outlet air temperature) were kept in the models to maintain logical expression capability for the system behavior by the developed models. Analysis of variance (ANOVA), estimated coefficients of coded factors, PRESS, CV, r^2 and A-Press values of each model were shown in Table 3.

According to Table 3, p-values of all models were significant and lack of fit values were insignificant at a significance level (α) of 0.05. Adequate precision values of models were higher than “4” which shows models have adequate signals and reasonable for this experimental design (Myers et al., 2011). The repeatability and reproducibility of the developed models were evaluated by considering CV values. Models of tapped density, total saponin content and foam volume had smaller CV values than 10 (3.0, 3.6, 3.6, respectively), thus those models were found to be more repeatable and reproducible than models of moisture content and water activity (CV values are 14.4 and 11.5, respectively) (Balasubramani et al., 2015). The developed mathematical models with actual factors for all responses were shown in Table 4.

The response surface graphs of each mathematical models were given in Fig. 1 by using all statistical effective independent variables of each response according to ANOVA given in Table 3.

Moisture content and water activity are very important parameters not only for flocking attitude but also for microbial activity aspects of a powdered food product. According to results given in Table 3, both moisture content and water activity were affected from outlet air temperature of the spray drier. Similar outcome was previously reported in elsewhere (Simon-Brown et al., 2016). As the outlet air temperature increased, moisture level and water activity significantly decreased. Since the temperature of the final powder product is directly dependent on the outlet air temperature, low moisture powders can be obtained in spray drying operations at higher outlet air temperatures

(Ozdikicierler et al., 2014; Da Costa et al., 2015). Since water activity of a food product is dependent on moisture content, models of moisture content and water activity have same terms. Similar relationship between outlet air temperature and moisture content was emphasized in a previous study that focused on spray drying of *B.pilosa* extracts. Researchers also indicated that agglomeration may occur at powder products with a high moisture content (Cortés-Rojas et al. 2015). Some previous studies also discussed the interaction effect of feed flow rate and inlet air temperature on moisture and water activity levels of powders (Roccia et al., 2014; Muzaffar and Kumar, 2015). In these studies researchers reported that an increase in inlet air temperature or decrease in feed flow rate lead a decrease in moisture content and water activity of final powder products. It is a well-known fact that there is a relationship between outlet temperature and feed flow rate and inlet air temperature in spray drying operations such as increase of feed flow rate causes a decrease in outlet temperature for constant inlet air temperature. Similarly outlet air temperature increases with inlet air temperature at constant feed flow rate (Ozdikicierler et al., 2014). In our study, outlet air temperature kept constant as inlet air temperature changed during spray drying operations. Therefore, although effect of inlet air temperature on the moisture content and water activity has seemed to be directly proportional according to the positive constants for the models given in Table 3, the effects were statistically insignificant. Although feed soluble solid content was not effective on moisture content or water activity in our results, it was indicated that increment in coating material content of feed caused an increase in moisture content of spray dried powder products in some previous studies. Researchers of the studies found that, coating materials with higher molecular weight such as starch, prevented vaporizing of water during spray drying with an increase in moisture content of final product (De Barros Fernandes et al., 2014; Da Costa et al., 2015).

Tapped density, which is a function of particle size and shape, is an important factor for the aspects of handling, storage, transportation and flow characteristics. Especially higher tapped densities can be preferred due to high compressibility of the powder. According to results obtained in this study showed that feed soluble solid content, inlet air temperature and outlet air temperature are effective on tapped density of the soapwort extract powder during spray drying. It can be followed from the graphs showed in Fig. 1 that increments in inlet air and outlet temperature leads to production of soapwort extract powder with low tapped densities. Tapped density values increased with the increase in the feed soluble solid content. Interaction effect was determined between feed soluble solid content and inlet air temperature. In a previous study it has indicated

Table 3 ANOVA, estimated regression coefficients and model validity values for all responses

Source	Est coeff	SoS	df	MSq	F-value	<i>p</i> value	PRESS	CV	<i>r</i> ²	A-Pres
<i>Moisture content (%)</i>										
Model	4.3823	30.6899	6	5.1150	30.609	< 0.0001*	5.5	14.4	0.934	18
A	0.2277	0.5112	1	0.5112	3.059	0.1038				
B	0.2110	0.2151	1	0.2151	1.287	0.2771				
C	− 0.4819	27.1673	1	27.1673	162.572	< 0.0001*				
A × C	− 0.0030	0.9354	1	0.9354	5.598	0.0342*				
B ²	− 0.0008	0.7217	1	0.7217	4.319	0.0581				
C ²	0.0034	1.8593	1	1.8593	11.126	0.0054*				
Residual		2.1724	13	0.1671						
Lack of fit		1.1950	8	0.1494	0.764	0.6506				
Pure error		0.9774	5	0.1955						
Total		32.8623	19							
<i>Water activity (<i>a_w</i>)</i>										
Model	0.2278	0.1079	6	0.0180	48.121	< 0.0001*	0.013	11.5	0.957	22
A	0.0104	0.0001	1	0.0001	0.225	0.6431				
B	0.0178	0.0001	1	0.0001	0.291	0.5984				
C	− 0.0366	0.0943	1	0.0943	252.329	< 0.0001*				
A × C	− 0.0002	0.0024	1	0.0024	6.464	0.0245*				
B ²	− 0.0001	0.0053	1	0.0053	14.308	0.0023*				
C ²	0.0003	0.0108	1	0.0108	28.934	0.0001*				
Residual		0.0049	13	0.0004						
Lack of fit		0.0031	8	0.0004	1.106	0.4773				
Pure error		0.0018	5	0.0004						
Total		0.1127	19							
<i>Tapped density (kg/m³)</i>										
Model	930.8830	58,546.2909	6	9757.7151	47.896	< 0.0001*	11,980.5	3.0	0.957	28
A	− 14.1825	35,362.1437	1	35,362.1437	173.575	< 0.0001*				
B	− 4.3528	4444.9990	1	4444.9990	21.818	0.0004*				
C	1.0110	9287.7501	1	9287.7501	45.589	< 0.0001*				
A × B	0.1560	6842.6308	1	6842.6308	33.587	< 0.0001*				
A × C	− 0.1352	1851.6530	1	1851.6530	9.089	0.0100*				
A ²	0.2188	757.1143	1	757.1143	3.716	0.0760				
Residual		2648.4696	13	203.7284						
Lack of fit		2336.4881	8	292.0610	4.681	0.0529				
Pure error		311.9815	5	62.3963						
Total		61,194.7605	19							
<i>Total saponin content (%)</i>										
Model	20.2753	47.2061	9	5.2451	183.050	< 0.0001*	1.1	3.6	0.994	38
A	− 0.8814	45.1011	1	45.1011	1573.984	< 0.0001*				
B	− 0.0684	0.0028	1	0.0028	0.099	0.7600				
C	0.0568	0.0250	1	0.0250	0.873	0.3721				
A × B	0.0005	0.0571	1	0.0571	1.993	0.1884				
A × C	0.0004	0.0143	1	0.0143	0.498	0.4967				
B × C	0.0002	0.0338	1	0.0338	1.180	0.3028				
A ²	0.0114	1.1311	1	1.1311	39.474	< 0.0001*				
B ²	0.0002	0.0316	1	0.0316	1.102	0.3185				
C ²	− 0.0007	0.0600	1	0.0600	2.094	0.1785				
Residual		0.2865	10	0.0287						

Table 3 continued

Source	Est coeff	SoS	df	MSq	F-value	p value	PRESS	CV	r ²	A-Pres
Lack of Fit		0.2026	5	0.0405	2.415	0.1776				
Pure Error		0.0839	5	0.0168						
Total		47.4927	19							
<i>Foam volume (ml)</i>										
Model	− 58.3822	1075.7845	5	215.1569	19.469	< 0.0001*	276.2	3.6	0.874	15
A	7.1797	373.0156	1	373.0156	33.753	< 0.0001*				
B	0.5528	103.5231	1	103.5231	9.367	0.0085*				
C	1.0330	52.0981	1	52.0981	4.714	0.0476*				
B × C	− 0.0065	47.8976	1	47.8976	4.334	0.0562				
A ²	− 0.1776	499.2503	1	499.2503	45.175	< 0.0001*				
Residual		154.7208	14	11.0515						
Lack of Fit		69.8875	9	7.7653	0.458	0.8545				
Pure Error		84.8333	5	16.9667						
Total		1230.5053	19							

A: Feed soluble solid content (°Bx). B: Inlet air temperature (°C). C: Outlet air temperature (°C), Est Coeff: Estimated coefficients of factor codes, SoS: Sum of squares, df: Degrees of freedom, MSq: Mean square, CV: Coefficient of variance, A-Pres: Adequate precision. *Statistically significant at a significance level (α) of 0.05

that spray dried powders with high tapped density has low flow ability, which is an undesired situation for powder products (Cortés-Rojas et al. 2015). On the other hand, researchers of another study investigating the effects of feed soluble solid content on microencapsulation kinetics of rosemary extract, indicated that powder products having high tapped density could be contained in smaller packages which was a useful property (De Barros Fernandes et al., 2014). This determination clarified that tapped density is a functional property and depended on the usage purposes of the powder product.

Saponins are a group of naturally occurring plant glycosides, characterized by their strong foam-forming properties in aqueous solution (Man et al., 2010). Saponin is a functional substance of soapwort extract for industrial usage aspects. In Turkish halva production, saponins establish the desired structure and stabilize the sesame oil in the halva mixture. Intense stirring or whipping of soapwort extract causes saponins to form into foams, which is a significant ingredient of Sultan Delights. Therefore, both saponin content and foam forming capacity are desired characteristics of soapwort extract powder for industry. According to the results given in Table 3 and response surface graphs shown in Fig. 1, saponin content is inversely proportional with feed soluble solid content whereas saponin content of the soapwort extract powder increased as the feed soluble solid content decreased. Since the feed soluble solid content of soapwort extracts were regulated with added maltodextrin, as maltodextrin proportion was increased in soapwort extracts, saponin ratio was decreased. A previous study concerning spray drying

of ginger (*Zingiber officinale*) extract indicated that inlet air temperature is effective on functional component of the feed. If the functional component is volatile, increase in inlet air temperature cause a significant decrease in this component (Jangam and Thorat, 2010). Since saponins are known to be resistant to high temperatures (Olvera-Garcia et al., 2014), both inlet air and outlet temperatures did not have a significant effect on saponin content of spray dried soapwort extract powders.

Results of this study showed that the foam volume of the reconstituted soapwort powder was dependent on all process parameters. When F-values were compared, the most significant effect on foam volume was feed soluble solid content. According to the response surface graphs given in Fig. 1, the maximum foam volume was obtained from soapwort extracts having 22.5 °Bx of feed soluble solid content. On the other hand, foam volume increased as the inlet air and outlet temperatures increased.

Numerical optimization, verification of optimum point and estimated points

Numerical optimization was applied to determine optimum feed soluble solid content, inlet air temperature and outlet air temperature values for spray drying of soapwort extract. Optimization criteria were selected based on different parameters including product-quality related specific attributes as total saponin content (%) and foam volume (mL). Although moisture content and water activity are important responses for dried food products in terms of microbial aspects, models of those responses did not included in

Table 4 Mathematical models moisture content, water activity, tapped density, total saponin content and foam volume with actual factors

Model	Equation*
Model 1.	Moisture Content (%) = $4.38227 * A + 0.22772 * B + 0.21103 * C - 0.00304 * B * C - 0.00076 * B^2 + 0.00339 * C^2$
Model 2.	Water Activity (a_w) = $0.22777 + 0.01047 * A + 0.01779 * B - 0.03658 * C - 0.00015 * A * C - 0.00006 * B^2 + 0.00026 * C^2$
Model 3.	Tapped Density ($\frac{\text{kg}}{\text{m}^3}$) = $930.88300 - 14.18250 * A - 4.35285 * B + 1.01102 * C + 0.15598 * A * B - 0.13523 * A * C + 0.21876 * A^2$
Model 4.	Total Saponin Content (%) = $20.27526 - 0.88145 * A - 0.88145 * B - 0.06837 * C + 0.00045 * A * B + 0.00037 * A * C + 0.00017 * B * C + 0.001140 * A^2 + 0.00017 * B^2 - 0.00065 * C^2$
Model 5.	Foam Volume (ml) = $-58.38221 + 7.17967 * A + 0.55282 * B + 1.03304 * C - 0.00652 * B * C - 0.17764 * A^2$

*Letters A, B and C indicates Feed soluble solid content (°Bx), Inlet air temperature (°C), Outlet air temperature (°C) respectively

numerical optimization since CV values of these models found to be low. Moreover, moisture content and water activity levels were relatively low and all powder samples obtained from spray drying trials were considered as safe for microbial aspects. Despite high tapped density for powder products could be a desired feature in terms of handling and storage, powders having low tapped density has better flow ability. Therefore, the tapped density considered as a preferable option for powder products, was also not included in numerical optimization.

Major aims of optimization were maximizing the total saponin content and foam volume. According to the numerical optimization results, optimum feed soluble solid content, inlet air and outlet temperature are 15 °Bx, 160 °C and 67 °C, respectively with a desirability factor of 0.822.

In this study, soapwort extract powder was produced by drying soapwort extract at determined optimum spray drying conditions. In addition, saponin contents and foam volumes at eleven different process conditions, which were not present in experimental design, were predicted using Design Expert software and spray drying trials were performed at these conditions for verification. Predicted points were verified by comparing them with the experimental values obtained at these conditions and these results were displayed in Table 5.

As can be seen in Table 5, one optimum condition and eleven random conditions were predicted and validation experiments were performed at these conditions. At optimum condition, saponin content and foam volume were predicted as 6.81% and 95 mL respectively. Saponin content and foam volume of soapwort extract powders obtained from the verification trial performed under optimum conditions were 7.24% and 97.1 mL, respectively. According to the results of the verification spray drying trial at optimum conditions, the error percentages were found to be 5.96% and 2.15% for saponin content and foam volume respectively.

In order to compare predicted values and experimental values, average difference (Error %) was calculated. All error % values calculated in our study was lower than 15% (Table 5) for both optimum point and predicted points which means that optimization can be considered as valid and acceptable (Myers et al., 2011). According to a previous study, low average difference between predicted and experimental results indicates the robustness of the experimental design (Jagadish et al., 2017). Moreover, desirability factor of numerical optimization is relatively high (0.822). Desirability factors between 1 and 0.8 are considered as “very good” and shows that numerical optimization model highly meets the goals of the optimization (Myers et al., 2011).

In conclusion, the effects of the feed soluble solid content, inlet air temperature and outlet air temperature on

Fig. 1 Response surface plots of moisture content, water activity, tapped density, total saponin content and foam volume

Table 5 Predicted and experimental values of saponin content and foam volume at optimum condition and predicted points

Feed soluble solid content (°Bx)	Inlet air temperature (°C)	Outlet air temperature (°C)	Predicted values		Experimental values		Error (%) in Saponin content	Error (%) in foam volume
			Saponin content (%)	Foam volume (ml)	Saponin content (%)	Foam volume (ml)		
Optimum conditions								
15	160	67	6.81	95.0	7.24	97.1	5.96	2.15
Point prediction								
15	110	65	6.83	97.0	7.44	90.6	8.25	7.01
15	135	50	6.84	97.0	7.08	91.6	3.39	5.91
15	135	80	7.22	99.0	7.09	96.1	1.76	2.97
22.5	110	50	4.40	99.0	4.46	89.8	1.36	10.24
22.5	110	80	4.26	99.5	4.43	99.3	3.87	0.24
22.5	160	50	4.28	100.5	4.30	101.1	0.40	0.62
22.5	160	80	4.84	98.5	4.53	100.8	6.98	2.28
30	110	65	2.67	82.5	3.02	78.4	11.73	5.19
30	135	50	2.85	81.0	2.75	79.4	3.81	2.06
30	135	80	3.01	80.5	2.93	83.9	2.76	4.09
30	160	65	2.72	82.5	3.16	84.9	13.93	2.79

moisture content, water activity, tapped density, total saponin content and foam volume of spray dried soapwort extract was investigated in this study. The models were developed using backward substitution method and were evaluated in terms of their CV and adequate precision levels, and total saponin content, foam volume were selected for numerical optimization. According to the results obtained from this study, moisture content and water activity of soapwort extract powder were inversely proportional with outlet air temperature of spray drier. In general, moisture content and water activity of all powder samples were in the safety range mentioned for powder products to consider the samples as safe for long storage periods in microbial aspects. Tapped density was affected from all main variables as well as feed soluble solid content and inlet air temperature interaction. Soapwort extract powders with high tapped density values were obtained at low inlet air and outlet temperatures and high feed soluble solid contents. Saponins, which are functional components of soapwort extract, was found to be inversely proportional with feed soluble solid content. Also it was found that saponins are not effected from elevated temperatures of spray drying. Foam volume was only effected from soluble solid content of feed. Maximum foam volume was measured at 22.5 °Bx of feed soluble solid content in our study. Production of soapwort powders with the highest saponin contents and foam volumes could be possible at 15 °Bx feed soluble solid content, 160 °C inlet air temperature 67 °C outlet temperature according to numerical optimization. Validation showed that all difference percentages (Error %) between predicted and experimental values were lower than 15% showing that numerical optimization of spray drying operation process parameters was valid and reliable.

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