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OPTIMIZATION OF GARLIC (*ALLIUM SATIVUM* L.) VACUUM DRYING PROCESS BY RESPONSE SURFACE METHODOLOGY (RSM)

OPTIMIZACIJA SUŠENJA BELOG LUKA (*ALLIUM SATIVUM* L.) U VAKUUMU KORIŠĆENJEM METODE ODZIVNIH POVRŠINA (RESPONSE SURFACE METHODOLOGY, RSM)

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ABSTRACT

*The objective of this work was to optimize the vacuum drying of garlic in order to preserve good physical properties. Fresh garlic (Allium sativum*L.) *was dried by vacuum drying process under different drying conditions. Three level, three variable, Box-Behnken experimental design (BBD) with response surface methodology (RSM) was used for optimization of drying process in terms of physical properties (moisture content, water activity, total colour change and shear force). Heat surface temperature (48 – 78 °C), airpressure (30 – 330 mbar) and drying time (3 – 11 h) were investigated as independent variables. Experimental results were fitted to a second-order polynomial model, where multiple regression analysis and analysis of variance were used to determine fitness of the model and optimal conditions for investigated responses. Contour plots were generated from the mathematical model. The optimal conditions for vacuum drying and simultaneous optimization of all responses (physical properties of dried garlic) were heat surfacetemperature of 78 °C, airpressure of 30 mbar and drying time of 11 h. Chemical parameters (total phenols content and antioxidant activity) were investigated as significant quality indicators of dried garlic samples. Fresh garlic was also dried by lyophilization and conventional drying in order to compare quality of samples dried by different techniques. It could be concluded that vacuum drying results in dried products with good physical properties, which are similar to sample dried by lyophilization and significantly better than convective dried sample.*

***Key words**: garlic (Allium sativum*L.), *vacuum drying, physical and chemical properties, optimization, response surface methodology (RSM).*

REZIME

Cilj ovog istraživanja bio je optimizacija procesa vakuum sušenja belog luka u pogledu što boljeg očuvanja njegovih osnovnih fizičkih i hemijskih karakteristika. Svež beli luk (Allium sativum L.*) osušen je u vakuum sušari pri različitim uslovima, liofilizacijom i konvencionalnim sušenjem u cilju poređenja kvaliteta uzoraka osušenih različitim tehnikama sušenja. Za optimizaciju procesa vakuum sušenja, u pogledu fizičkih karakteristika (sadržaj vlage, aktivnost vode, ukupna promena boje, tekstura), korišćeni su Box-Behnken dizajn (BBD) i metoda odzivne površine (Response Surface Methodology, RSM). Kao nezavisne promenjive korišćeni su sledeći parametri: temperatura grejne ploče(48 – 78 °C), pritisak vazduha(30 – 330 mbar) i vreme sušenja (3 – 11 h). Rezultati su fitovani polinomnim modelom drugog reda i multipla regresija i analiza varijanse su korišćeni za određivanje adekvatnosti modela i optimalnih uslova za ispitivane odzive. Konturni dijagrami su generisani na osnovu matematičkih modela. Kao optimalni uslovi za vakuum sušenje belog luka dobijeni su: temperatura grejne ploče 78 °C, pritisak vazduha30 mbar i vreme sušenja 11 h. Hemijske karakteristike (sadržaj ukupnih fenola i antioksidativni kapacitet) su dodatno ispitivani kao značajni parametri kvaliteta osušenog belog luka. Kvalitet belog luka osušenog vakuum sušenjem, u pogledu fizičkih karakteristika, bio je sličan uzorcima osušenim liofilizacijom i značajno bolji u odnosu na uzorke osušene konvencionalnim sušenjem.*

***Ključne reči**: beli luk (Allium sativum* L.*), vakuum sušenje, fizičke i hemijske karakteristike, optimizacija, RSM.*

INTRODUCTION

Garlic (*Allium sativum*L.), one of the main 'bulbs' of *Allium* family, is very common in traditional Serbian cuisines and households. Garlic is known to possess a huge variety of important biological functions for human health, such as cardioprotective (*Rahman and Lowe, 2006; Gorinstein et al., 2007*), anticancer (*Tsubura et al., 2011; Patel et al., 2004*) and antimicrobial activity (*AnkriandMirelman, 1999; Kim et al., 2013*). Many of these biological effects are related to the thiosulfinates, volatile sulfur compounds, typical of the *Allium* plants, which are also responsible of their characteristic pungent aroma and taste. However, main components of garlic dry matter are carbohydrates, in the range of 28.7 % and 63.8 %, proteins in the range of 5.3 % and 8.4 %, fats in the range of 0.1 % and 0.2 % and organosulfur organic sulphur compounds in the range of 0.087 % and 0.214 % (*Artık and Poyrazoğlu, 1994*). Garlic is rich in vitamins and minerals and because of these qualities and characteristics garlic has been used both in food nutrition and for the treatment of many diseases (*Lanzotti, 2006*.). Garlic is also prominent in the field of scientific research, precisely because of its phenolic content and antioxidative properties.

Garlic is used fresh, frozen, or in dried form as an ingredient of precooked foods and instant convenience foods including sauces, gravies and soups. This use of garlic has led to a sharp increase in the demand of dried garlic (*Sharma and Prasad, 2006*). Dehydration of garlic is considered to be one of the best means of its preservation since dried garlic take much less storage space and also have longer shelf-life than fresh or frozen garlic (*Cui et al., 2003*). It is well known that the quality of a dehydrated food product is strongly affected by drying methods and drying processes (*Yang and Atallah, 1985; Krokida et al., 2000*). It is also known that conventional drying is commonly used type of drying, but in recent years vacuum drying has been investigated as a potential method for obtaining high-quality dried product (*Ursachi and Segali, 2009*). Moreover, lower airpressure used in vacuum drying process, can additionally shorten the drying time because lowered airpressure induces faster evaporation of water from the material at reduced temperature, which directly influence on good quality of dried products (*Figiel, 2009*). These conditions of vacuum drying process allow drying of thermal sensitive types of fruits and vegetables and reduce negative chemical reactions which support preserving important bioactive compounds during dehydration process (*Joshi et al., 2011*). Influence of different drying techniques on quality indicators of garlic were also investigated in studies by *Cui et al. (2003), Figiel (2009), Calín-Sánchez (2014) and Sharma (2006)*. In study by Calín-Sánchez (2014) was presented significant influence of drying process on total phenolic compounds and antioxidant capacity. However, total phenolic compounds significantly decreased, while dried garlic samples showed significantly higher antioxidant capacity compared with fresh garlic slices (*Calín-Sánchez* et al., *2014).*

Each unit operation in industrial production demands optimization study, since it directly affects quality and yield of the final product, as well as reduction and minimization of operational cost of the same process. Commonly used optimization studies were usually performed using one-factor-at-a-time approach, where influence of independent variables on responses were investigated one by one, keeping all other independent variables at constant values. Time and money consumption are recognized as main drawbacks of this approach, since some experiments are quite expensive and their number is often limited. Moreover, interactions between process parameters are ignored and hence, the chances of obtaining the true optimum conditions are dubious. Therefore, response surface methodology (RSM) was developed as alternative approach in order to overcome these disadvantages. This methodology represents a collection of statistical and mathematical techniques and is often used for development, improvement and optimization of various processes, where certain response is influenced by several variables (*Baş and Boyaci, 2007; Bezerra et al., 2008*). Determination of independent variables interaction effects on investigated response and generation of the predictive model equations which are usable within the investigated experimental range, are the designated advantages of the RSM (*Myers et al., 2009*). Moreover, RSM has recently been successfully utilized for optimization of vacuum drying (*Šumić et al., 2013; Šumić et al., 2015; Šumić et al., 2016*).

Basic objective of this work was to optimize vacuum drying process of Garlic (*Allium sativum*L.). Response surface methodology (RSM) was used for optimization of vacuum drying parameters and three level, three variables, experimental design with heat surface temperature, air pressure and drying time, as independent variables. Moreover, garlic was dried by lyophilization and conventional drying in order to compare quality of samples dried by different techniques. Influence of vacuum drying parameters on physical and chemical properties of dried product (moisture content, water activity, total colour change and shear force, total phenols content and antioxidant activity) has been investigated.

MATERIAL AND METHOD

Sample

Garlic (*Allium sativum*L.) was collected near Begeč (Serbia) in June (2015). After purchasing, samples were frozen and stored at -20 °C until drying, in order to insure the initial properties of raw samples and prevent potential deteriorative processes in the samples during the experiment.

Chemicals

1.1-Diphenyl-2-picryl-hydrazyl-hydrate (DPPH) was purchased from Sigma Aldrich (Germany); Folin–Ciocalteu reagent and gallic acid were purchased from Merck (Germany). All other chemicals and reagents were of analytical reagent grade.

Drying procedure

Drying was performed in a vacuum dryer prototype, described in detail in *Šumić et al. (2013)*. Experimental drying facility is composed of a cylindrical vacuum chamber, vacuum pump (which provides air pressure in the chamber of 2 mbar) and condensate collector. Samples were chopped into small pieces (between 40 and 50 mm^3). Samples were exposed to direct contact with heat surface and thus they are dried with both conduction and convection. Heat surface temperature, air pressure in vacuum chamber and the change in product weight during the drying processwere controlled and registered by the drying procedure control system (PLC). Sample size was kept constant (approximately 200 g) for each experiment. Weight loss was recorded in 10 minute intervals and drying was continued until no mass change was detected (final moisture content in equilibrium). Drying runs were performed at heat surfacetemperatures from 48 to 78 °C and airpressures from 30 to 330 mbar, according to the central composite rotatable design experimental design given in Table 1. Drying times ranged from 3 to 11 h, depending on working conditions (heat surfacetemperature and airpressure).

Design of experiments

Box-Behnken experimental design consisted of seventeen experimental runs with five replicates at the central point, with three numeric independent variables on three levels. Heat surface temperature, airpressure and drying time were chosen as the most designated vacuum drying parameters. Therefore, their influence on different responses has been determined. Vacuum drying was performed at different heat surfacetemperatures (T, 48 – 78 ˚C), air pressure (p, 30 – 330 mbar) and drying time (t, 3 – 11 h), while all other factors were held constant for each experimental run. Drying parameters were normalized as coded variables, so they can affect the response more evenly and the units of the parameters are irrelevant (*Bas and Boyaci, 2007*).

Table 1. Box-Behnken experimental domain with uncoded and coded values of vacuum drying parameters

Variable	Coded levels		
	-1	0	1
	Natural levels		
Heat surface temperature [°C]	48	63	78
Air pressure [mbar]	30	180	330
Drying time [h]	3	7	11

The response variables were fitted to a second-order polynomial model (Eq. (1)):

$$Y = \beta_0 + \sum_{i=1}^{2} \beta_i X_i + \sum_{i=1}^{2} \beta_{ii} X_i^2 + \sum_{i<j=1}^{2} \beta_{ij} X_i X_j \qquad (1)$$

where Y represents the response variable (moisture content, a_w value, total colour change and shear force), X_i and X_j are the independent variables affecting the response, and β_0, β_i, β_{ii} and β_{ij}, are the regression coefficients for intercept, linear, quadratic and interaction terms. Analysis of variance (ANOVA) was used in order to evaluate model adequacy and determine regression coefficients and statistical significance. Statistical analysis was performed using suitable software for RSM, Design-Expert v.7 Trial (Stat-Ease, Minneapolis, MN, USA). The results were statistically tested at the significance level of p = 0.05. The adequacy of the model was evaluated by the coefficient of determination (R^2) and model p-value. Contour plots were generated and drawn by using the function of two factors, keeping the third factor constant.

Moisture content

Moisture content was determined by drying the samples at 105 °C until constant weight. Experiments were replicated three times for statistical purpose.

Water activity

Water activity was determined by placing approximately 2.5 g of chopped and dried garlic in the sample holder of a TESTO 650 (Germany) a_w-meter, at 25 °C. a_w values were recorded after equilibration.

Total colour change

The CIE $L^*a^*b^*$colour coordinates were measured by using MINOLTA Chroma Meter CR-400 (Minolta Co., Ltd., Osaka, Japan). The surface colour of all samples was measured in terms of *L* (degree of lightness), *a* (degree of redness and greenness) and *b* (degree of yellowness and blueness). Total colour change between raw garlic sample (L_0^*, $a_0^*$and b_0^*) and dried garlic samples (L^*, a^* and b^*) was determined according to Equation

$$\Delta E = \sqrt{[(L_0^* - L^*)^2 + (\alpha_0^* - \alpha^*)^2 + (b_0^* - b^*)^2]_0} \qquad (2)$$

Samples were placed on the measure head of Chroma Meter and measurements of colour were performed for all prepared samples. A standard white colour was used for calibration and experiments were replicated five times for statistical purpose.

Shear force

Instrumental texture measurements were performed using a Texture Analyser (TE32, Stable Micro Systems, UK). The shearing force of dried garlic was measured, using a knife blade. TA settings were the following: test speed – 0.5 mm/s; distance – 10 mm; load cell – 250 kg.

Total phenols content

Dried garlic samples were ground in a blender before the extraction. 5.0 g of ground sample was transferred to a volumetric flask and 50 mL of methanol, as extraction solvent, was added. Extraction was carried out for 24 h at the room temperature. The obtained extract was filtered under vacuum. Prepared extracts were placed into a glass bottles and stored to prevent oxidative damage until analysis. The content of total phenols compounds in methanolic extracts was determined by Folin-Ciocalteu procedure (*Singleton and Rossi, 1965; Kähkönen et al., 1999*). Absorbance was measured at 750 nm (6300 Spectrophotometer, Jenway, UK). Content of total phenols has been expressed as mg of gallic acid equivalent per 100 g of dry weight of dried garlic (mg GAE/100 g DW). Experiments were replicated three times and results are expressed as mean values.

DPPH assay

Previously prepared phenolic extract was used for DPPH assay. Free radical scavenging activity of samples was determined using DPPH assay, previously described by Espín et al. (2000). A certain volume of diluted sample was mixed with 95 % methanol and 90 µM 1.1-diphenyl-2-picryl-hydrazyl (DPPH) in order to obtain different final concentrations. After incubation on room temperature for 60 min, the absorbance was measured at 515 nm and result was expressed as radical scavenging capacity (RSC, %). Antioxidant activity was further expressed as inhibition concentration at 50 % of RSC value (IC_{50}). IC_{50} represents the concentration of test solution required to obtain 50 % of radical scavenging capacity, expressed as mg per mL. All experiments were performed in triplicate, and results are expressed as mean values.

RESULTS AND DISCUSSION

Vacuum drying process possesses the advantages in terms of both lower airpressure in vacuum chamber and lower heat surfacetemperature and it can improve energy efficiency and product quality. However, lower airpressure means lower oxygen content, which causes reduction of oxidative processes in products during vacuum drying process. Drying time and properties of raw material are also important and adaptable parameters that influence on quality of dried products during vacuum drying process. Basic objective of vacuum drying process is preserving primary characteristics of raw material, which considers reducing negative influences of drying on quality indicators of dried product. Experimentally observed results of physical properties of vacuum dried garlic obtained in designed experiments using Box-Behnken experimental design, as well as results of the fresh sample, convective dried sample and lyophilized sample, were presented in Table 2. Method of least square (MLS) was used for calculation of regression coefficients and their corresponding *p*-values were presented in Table 3. Influence of each term was described as statistically significant ($p< 0.05$) and insignificant ($p> 0.05$). Fitness of the applied quadratic model on experimental results has been determined by coefficient of multiple determination (R^2) and *p*-value for the model, obtained by analysis of variance (ANOVA) (Table 4). According to particularly high R^2 for the moisture content and a_w value (0.971 and 0.964, respectively), good fitness between experimental results and predicted model has been suggested. On the other hand, in case of total colour change and shear force, R^2 has been lower (0.842 and 0.895, respectively). Complete information about the model fitness was provided by the *p*-value for the model which has been <0.05 for all physical properties (Table 4). This suggested that second-order polynomial model represented a good approximation of experimental results in case of each investigated response. Therefore, regression equations obtained by RSM could be successfully used for optimization as predictors of these responses within investigated experimental domain.

Table 2. Box-Behnken experimental design with vacuum drying conditions and experimentally observed physical properties of dried samples

Run	Drying parameters			Physical properties			
	Heat surface temp. [°C]	Air press. [mbar]	Drying time [h]	Moist. content [%]	Water activity	ΔE	Shear force [10^{-2} N]
1	48	180	3	50.14	0.923	9.32	453.94
2	63	30	11	5.51	0.529	10.63	2059.19
3	63	180	7	12.70	0.666	13.91	3861.38
4	63	30	3	12.28	0.693	12.35	3474.48
5	48	180	11	15.65	0.748	18.28	1054.63
6	63	180	7	14.17	0.662	12.26	2658.50
7	63	330	3	43.88	0.913	11.24	759.49
8	48	30	7	9.58	0.512	10.96	2160.86
9	63	180	7	14.99	0.679	10.53	1953.16
10	63	180	7	13.06	0.612	14.73	3773.01
11	78	30	7	5.12	0.341	13.84	1977.29
12	78	330	7	11.77	0.548	15.18	3211.63
13	48	330	7	28.88	0.888	15.05	462.70
14	78	180	3	20.21	0.800	17.21	730.03
15	63	180	7	11.77	0.615	13.13	2941.15
16	78	180	11	5.52	0.409	15.51	2092.57
17	63	330	11	10.94	0.606	13.44	4754.23
L[a]	-30	0.01	72	1.13	0.124	11.53	1016.63
C[b]	78	1000	8	7.23	0.515	20.60	2375.55
F[c]	-	-	-	68.57	0.953	-	-

[a] *lyophilized sample*
[b] *convective dried sample*
[c] *fresh sample*

Table 3. Corresponding p-values of linear, interaction and quadratic terms of regression coefficients, obtained for selected response variables

Term	Response			
	Moisture content	a_w value	ΔE	Shear force
Linear				
X_1	0.0003	0.0002	0.0862	0.0674
X_2	0.0003	0.0003	0.1236	0.7952
X_3	< 0.0001	0.0001	0.0993	0.0391
Interaction				
X_{12}	0.0958	0.1211	0.3716	0.0541
X_{13}	0.0196	0.0474	0.0077	0.5671
X_{23}	0.0053	0.1792	0.2158	0.0037
Quadratic				
X_{11}	0.1469	0.4624	0.0300	0.0029
X_{22}	0.2285	0.0464	0.1158	0.3681
X_{33}	0.0035	0.0048	0.9313	0.1062

Table 4.Analysis of variance (ANOVA) of the fitted models for moisture content, a_w value, total colour change and shear force

Source	Sum of squares	DF	Mean square	F – value	p – value
Moisture content					
Model	2517.70	9	279.74	25.88	0.0001
Residual	75.66	7	10.81		
Lack of fit	69.29	3	23.10	14.52	0.0129
Pure error	6.36	4	1.59		
Total	2593.36	16			
R^2 = 0.971					
a_w value					
Model	0.43	9	0.047	20.60	0.0003
Residual	0.016	7	2.295×10^{-3}		
Lack of fit	0.012	3	4.069×10^{-3}	4.22	0.0991
Pure error	3.859×10^{-3}	4	9.647×10^{-4}		
Total	0.44	16			
R^2 = 0.964					
Total colour change					
Model	77.25	9	8.58	4.14	0.0373
Residual	14.53	7	2.08		
Lack of fit	8.04	3	2.68	1.65	0.3123
Pure error	6.48	4	1.62		
Total	91.77	16			
R^2 = 0.842					
Shear force					
Model	2.405×10^7	9	2.672×10^6	6.64	0.0103
Residual	2.816×10^6	7	4.023×10^5		
Lack of fit	2.678×10^5	3	89274.85	0.14	0.9309
Pure error	2.548×10^6	4	6.371×10^5		
Total	2.687×10^7	16			
R^2 = 0.895					

Physical properties of dried garlic

Moisture content

Moisture content is one of the most important quality indicators of dried products and reducing its initial value in fresh sample is considered as primary task of drying process. Moisture content reduction influences directly and positively on reduction of microorganisms growth. This means that shelf life of dried product could be prolonged by drying, since microorganisms are the main cause of food spoilage. Moisture content in fresh garlic sample was 68.57 % and such particularly high moisture content prevents its long shelf life. This result is similar with the result obtained in study by *Cui et al. (2003)*, where it is reported that initial moisture content of garlic sample was 65.40 %. Depending on different drying conditions, moisture content in vacuum dried samples varied from 5.12 to 50.14 %. The highest moisture content was observed at 48 °C, 180 mbar and 3 h, while the lowest moisture content was obtained at 78 °C, 30 mbar and 7 h. It could be seen that higher heat surfacetemperature, lower airpressure and longer drying time caused better evaporation of water, which results in lower moisture content of dried product. Moisture content was also determined in garlic sample dried by lyophilization (-30 °C, 0.01 mbar and 72 h) and in conventional dried sample (78 °C, atmospheric pressure and 8 h). The obtained results show that the lowest moisture content in vacuum dried samples was higher than moisture content obtained in lyophilized sample (1.13 %) and was also lower comparing to moisture content observed in convective dried garlic sample (7.23 %). Further, the lowest moisture content obtained in vacuum dried samples was similar to results obtained in study by *Cui et al. (2003)*, 4.20, 4.50 and 4.70 % in samples dried by freeze-, hot-air drying and combination of microwave-vacuum and air drying, respectively.

According to *p*-values from the Table 3, all linear terms of independent variables, heat surfacetemperature-drying time and air pressure-drying time interaction and quadratic term of drying time exhibited significant influence on moisture content. Negative linear effects of heat surfacetemperature and drying time and positive effect of airpressure was rather expected and since prolonged drying with increased heat surfacetemperature and reduced airpressure in vacuum chamberprovides faster heat and mass transfer, which results in low moisture content in dried samples. These effects could be also observed in Figure 1.a. Positive influence of quadratic term drying time indicated asymptotic behaviour of decrease of moisture content. Negative effect of airpressure-drying time interaction suggested that moisture content would be rather low in samples drying at higher level of these two variables. Therefore, drying time could be designated as the most influential parameter affecting moisture content.

Water activity

Microorganism growth mostly depends on the amount of available water, so drying process results in dried product with particularly good microbiological stability. In study by *Rockland and Nishi (1980)* is shown that tight water activity (a_w) range is related with changes in colour, texture, and acceptability of dried products. Therefore, dried fruits should be kept in a_w range of 0.45–0.54 for freeze-dried samples and between 0.46 and 0.63 for convective dried samples (*Šumić et al., 2013*). Microorganisms usually grow best between a_w 0.98 and 0.99. The most microbes stop their growth at a_w less than 0.90, while some fungi stop their growth only at a_w as low as 0.62 (*Raimbault, 1998*). a_w in vacuum dried samples varied from 0.341 (78 °C, 30 mbar and 7 h) and 0.923 (48 °C, 180 mbar and 3 h). Maximum and minimum values of a_w were observed on same experimental points as the maximum and minimum values of moisture content, which is rather expected because these two parameters are in direct correlation. As in the case of moisture content, the lowest a_w value in vacuum dried samples was higher comparing to a_w obtained in lyophilized garlic sample (0.124) and lower than a_w observed in convective dried garlic sample (0.515). However, the highest a_w in vacuum dried samples was similar with a_w in fresh garlic sample (0.953), which means that quality of this sample was reduced and its shelf-life was decreased.

Statistical analysis showed that linear terms of all independent variables, heat surfacetemperature-drying time interaction and quadratic terms of airpressure and drying time exhibited significant influence (p<0.05) on water activity (Table 3). Influence of similar independent variables was observed for moisture content in dried garlic, which suggested that correlation of these responses (moisture content and water activity) exists in vacuum dried garlic samples.

Positive correlation between these responses was confirmed since linear terms of vacuumdrying parameters exhibited same influence on moisture content and a_w (negative – heat surfacetemperature and drying time and positive – airpressure). Negative effect of heat surfacetemperature-drying time interaction suggested that samples dried at higher level of these variables would have particularly low a_w (Figure 1.b).

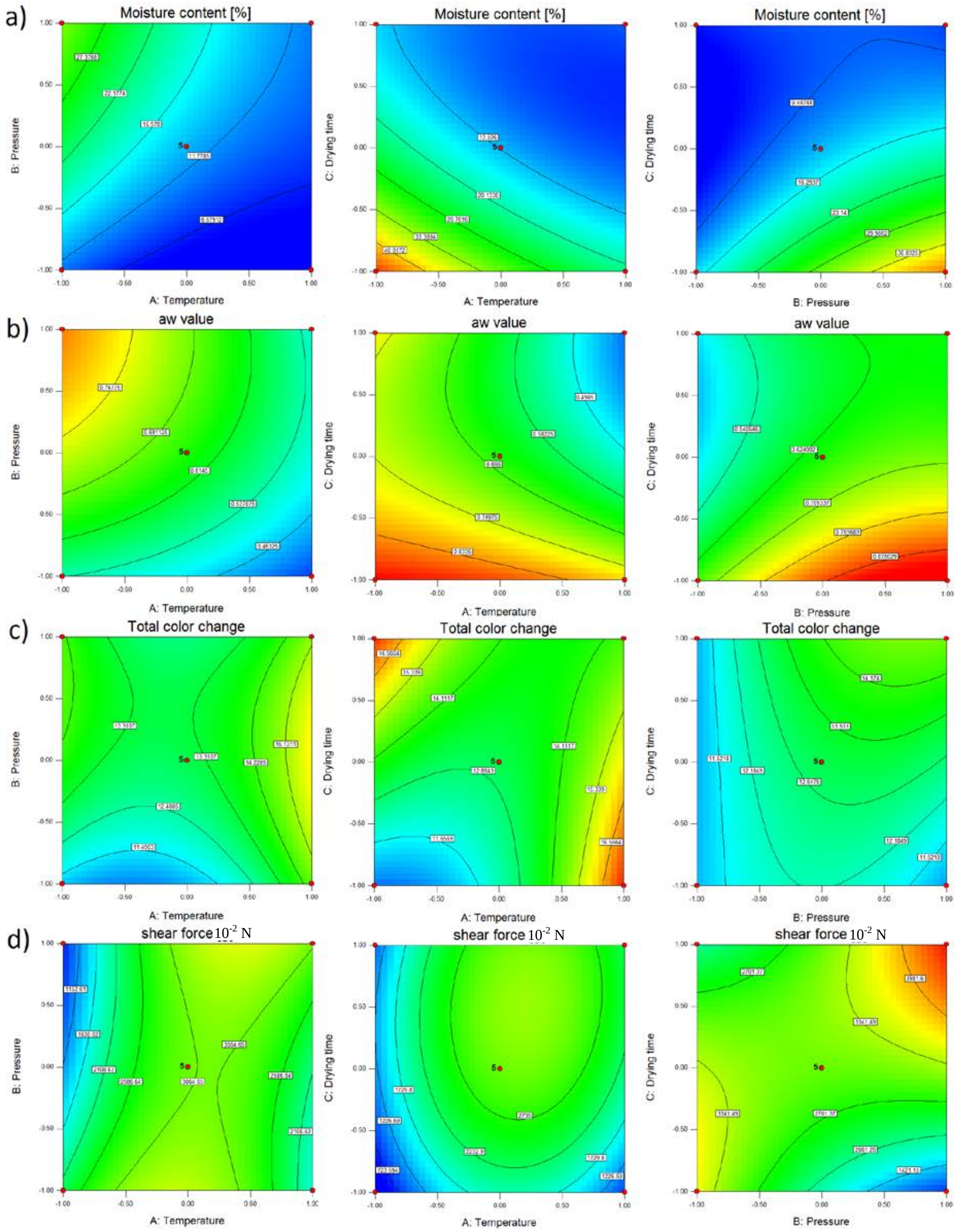

Fig. 1. Response surface contour plots showing combined effects of vacuum drying parameters (heat surfacetemperature, airpressure and drying time) on: a) moisture content, b) a_w value, c) total colour change and d) shear force

Total colour change

The eye catching appearance of food causes good first impressions and can also serve as a quality guide for consumers (*Muengkaew et al., 2016*). Accordingly, colour of dried products has huge impact on costumer acceptability, which also means that colour directly influence on commercial value of dried products (*Radojčin et al., 2010*). It is desirable for dried products to have very small colour changes, comparing to fresh sample and to keep intensive and uniform colour during drying (*Nieto-Sandoval et al., 1999*). Total colour change, ΔE, of garlic samples, was between 9.32 and 18.28. The lowest ΔE was obtained in sample dried on particularly mild vacuum drying conditions (48 °C, 180 mbar and 3 h). Since these conditions include lowest applied heat surfacetemperature and drying time, it was rather expected for this sample to have lower total colour change. However, this sample had rather low quality due to particularly high moisture content (50.14). The highest ΔE was observed in sample also dried at 48 °C and 180 mbar, but drying time was significantly higher (11 h) which caused darkening of the sample. The lowest ΔE in vacuum dried samples was lower comparing to ΔE in lyophilized sample (11.53) and the highest ΔE in vacuum dried samples was lower comparing to ΔE in convectional dried sample (20.60).Total colour change founded in study by *Cui et al. (2003)* were 28.80, 33.96, and 30.27 in samples dried by freeze-, hot-air drying and combination of microwave-vacuum and air drying, respectively. These results are significantly higher comparing to results observed in this study. Further, results in this study are more similar with results founded in study by *Figiel (2009)*, where total colour change was between 5.80 and 12.28 in vacuum-microwave dried samples.

Statistical indicators (Table 3) showed that only heat surfacetemperature-drying time interaction and quadratic effect of heat surfacetemperature exhibited significant influence on total colour change. Positive quadratic influence ofheat surface temperature was rather expected since increased heat surfacetemperature causes alteration degradation reactions in the garlic samples. Negative influence ofheat surface temperature-drying time interaction suggested that alteration in garlic sample would be reduced when prolonged drying time was applied. Therefore, it is suggested that chemical reactions that caused chemical reactions happened at the beginning of the drying process. Effects of vacuum drying parameters on total colour change could be also observed on Figure 1.c.

Shear force

Different types of food processing cause different state food transitions, since raw materials are subjected to external stress. During drying process, influenced by high temperatures, cell walls undergo modifications in terms of their macrostructure and microstructure properties (*Kunzen et al., 1999; Radojčin et al., 2015*). Shear force is also important quality indicator in terms of costumer acceptability (*Babić et al., 2007*), so it is not desirable for drying process to result in too brittle and firm dried products. Shear force in vacuum dried samples varied from 453.94 to $4754.23 \cdot 10^{-2}$ N. The lowest shear force was measured in sample dried at 48 °C, 180 mbar and 3 h which was expected, since this sample has the highest moisture content which makes the sample tender and sticky. The highest shear force was observed in sample dried at 63 °C, 330 mbar and 11 h. It could be seen that higher heat surfacetemperature and longer drying time cause fragility and brittleness of vacuum dried sample which is not desirable for consumer acceptability. Shear force in lyophilized sample was $1016.63 \cdot 10^{-2}$ N while in samples dried by convectional drying was twice as high, $2375.55 \cdot 10^{-2}$ N. Values of shear force obtained in study by *Cui et al. (2003)* was 3268.50, 3437.50 and $3383.40 \cdot 10^{-2}$ N in samples dried by freeze-, hot-air drying and combination of microwave-vacuum and air drying, respectively and these values are in the range of results obtained in this study.

According to results from Table 3, linear term of drying time, airpressure-drying time interaction and quadratic term of heat surfacetemperature exhibited significant influence on shear force ($p<0.05$), which was negative in all three cases. This indicated that shear force will significantly increase when all three variables are on their higher levels and that drying time was the most influential variable which affected this response.

Chemical properties of dried garlic

Phenolic compounds

Total phenolic content could be used as good quality indicator of dried products, since different parameters of drying (especially highheat surface temperature) causes their reduction (*Tomás-Barberán and Espin, 2001; Mikulić-Petkovsek et al., 2015*). Physical properties of dried products, such as colour, taste and flavour, also depend on phenolic content and it is desirable to preserve this compounds as much as possible (*Šumić et al., 2013*). Total phenolic content (TP) in fresh garlic sample was 84.49 mg GAE/100 g DW and similar TP (79.7 mg GAE/100 g DW) was presented in study by *Calín-Sánchez (2014)*. Loss of TP in vacuum dried samples was in the range from 35.57 to 91.17 %. These results are similar with results presented in study by *Calín-Sánchez (2014)*, where loss of TP in garlic samples, dried by a combined convective and vacuum-microwave drying, were between 29.61 and 83.56 %. The highest loss of TP was obtained at 63 °C, 180 mbar and 7 h, while the lowest loss of TP was obtained at 63 °C, 330 mbar and 3 h (Figure 2). It could be seen that lower airpressure i.e. higher oxygen content during drying process reduce TP and also that longer drying time influenced negatively on TP.

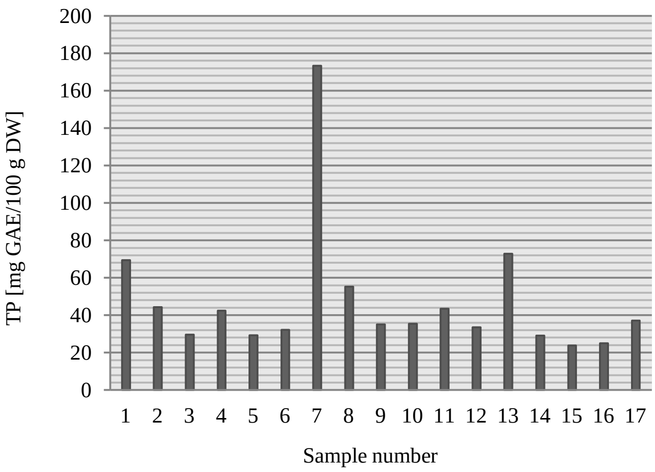

Fig. 2. Total phenols content (TP) observed in vacuum dried garlic

Antioxidative properties

Garlic, through its antioxidant activities, has been reported to provide protection against free radical damage in the human body. Phenolic compounds, secondary plant metabolites which possess antimicrobial, antiviral and anti-inflammatory properties are directly and positively correlated with the antioxidant properties of dried products (*Ignat et al., 2011; Mitić et al., 2011*). Long drying time, high heat surfacetemperature and airpressure used during drying process influence negatively on reduction of phenolic compounds and thus, antioxidative properties present one more significant quality indicator of dried products. IC_{50} values in vacuum dried samples varied from 0.951 and 6.302 (Figure 3). The lowest antioxidant activity, i.e. the highest IC_{50}, was obtained in sample dried at 63 °C, 180 mbar and 7 h, since high level ofheat surface temperature was applied (Figure 3), proposing that high heat surfacetemperature caused antioxidant inactivation. Further, the highest antioxidant activity was observed on 48 °C, 330 mbar and 7 h, suggesting that reduced heat surfacetemperature preserves antioxidants in dried garlic sample.

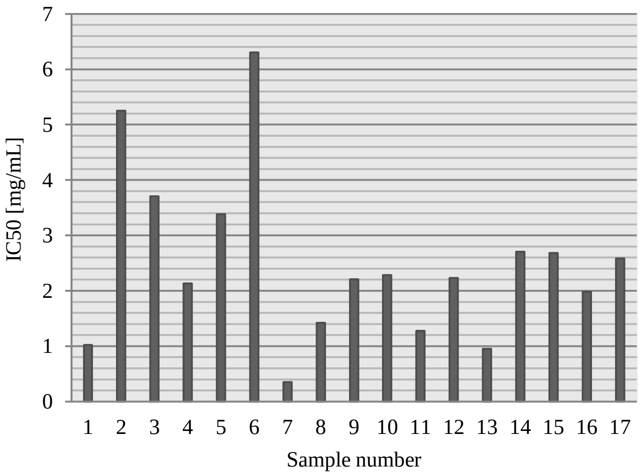

Fig. 3. Antioxidant activity expressed as IC_{50} value observed in vacuum dried garlic

CONCLUSION

Due to satisfactory statistical parameters (R^2 and CV) and analysis of variance (ANOVA) for the model and lack-of-fit testing, it could be concluded that second-order polynomial model provided adequate mathematical description of investigated physical properties (moisture content, water activity, total colour change and shear force) during vacuum drying process. Accordingly, RSM could be successfully used for simultaneous optimization of all physical properties. Optimal conditions for all response variables were heat surface temperature of 78 °C, air pressure of 30 mbar and drying time of 11 h, while generated model predicted obtaining of dried garlic with following physical properties: moisture content 8.73 %, water activity 0.326, total colour change 11.95 and shear force 598.32 $\cdot 10^{-2}$ N.

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