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SCIENTIFIC PAPER

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OPTIMIZATION OF THE BALL MILL PROCESSING PARAMETERS IN THE FAT FILLING PRODUCTION

Article Highlights

- Influence of the ball mill processing parameters on fat filling quality and energy consumption
- Optimization of agitator shaft speed and milling time in fat filling production
- Physical and sensory properties of fat filling are mostly influenced by agitator shaft speed
- Milling energy consumption is mostly influenced by milling time
- Optimization - maximum agitator shaft speed and 30-min milling time

Abstract

The aim of this study was to determine the effect of the main milling variables, i.e., agitator shaft speed (50, 75 and 100%, which is 25, 37.5 and 50 rpm) and milling time (30, 45 and 60 min) on physical and sensory properties of fat filling, as well as on energy consumption during the production in a laboratory ball mill. Within the response surface method, the face centered central composite design is used. A response surface regression analysis for responses was performed and a full quadratic model was fitted to the experimental data. It is shown that agitator shaft speed had the most significant influence on physical properties (particle size distribution, rheological and textural propertes) and sensory characteristics of fat filling, while the milling energy consumption is highly influenced by milling time with contribution 55.4%, followed by agitator shaft speed (40.04%). The model obtained by regression analyses was used to perform the optimization of processing parameters in order to provide the combination of agitator shaft speed and milling time that cost less energy while at the same time do not compromise the quality of the fat filling. Optimization of production of fat filling in a laboratory ball mill would imply the maximum agitator shaft speed and 30-min milling time.

Keywords: fat filling, ball mill milling variables, physical properties, sensory characteristics, optimization.

Fat fillings contain high amount of fat (30–40%) which presents a continuous phase and determines the consistency of the filling. Confectionary products with fat filling can melt faster or slower during the consumption, creating the overall sensory impression [1]. Milling is the next necessary step for obtaining the optimal particle size distribution and thereby physical characteristics and appropriate sensory quality of confectionery suspensions of solids in fat phase [2]. The particle size reduction takes place in a five-roll mill and very often with pre-refining in a three-roll refiner. On the other hand, presently the refining of cocoa spreads, fat fillings and even chocolate mass very often takes place in a ball mill [3]. The mass and the balls are additionally agitated by a shaft with arms, operating at a variable rotation speed. During refining, the mass can also be recycled through the ball mill, thus going through a thick layer of balls, which are kept in continuous movement and forced to bounce against each other [4]. The investigation of

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Fišteš *et al.* [5] showed that power requirements and energy consumption of the ball mill depend on processing parameters. Alamprese *et al.* [6] investigated the optimization of working conditions of a ball mill used for chocolate refining process in order to reduce refining time and energy consumption without loss of quality characteristics of the final product. Investigations of Yeganehzad *et al.* [7] and Zarić *et al.* [8] showed that the refining time in a ball mill had a significant effect on rheological properties, particle size, hardness and sensory properties of chocolate.

Having in mind that improving energy efficiency as well as energy saving represents one of major problems in production processes [9], the papers of Scanlon and Lamb [10] and Holdich [11] showed that the ball mill processing parameters have a significant effect on energy consumption and the quality characteristics of the obtained product. However, relatively little research has been published considering the use of ball mill for fat filling production.

This research investigated the interaction effects and optimization of main ball milling variables (agitator shaft speed and milling time) on physical and sensory properties of fat filling, as well as on energy consumption during the fat filling production in a laboratory ball.

EXPERIMENTAL

Materials

Fat filling mass, refined by a 3-roll mill in industrial conditions, consisted of powdered sugar (Crvenka JSC, Serbia), cocoa powder (Centroproizvod JSC, Serbia), soy flour (Sojaprotein JSC, Serbia), milk powder (Imlek JSC, Serbia), and vegetable fat (Dijamant JSC, Serbia). Fat characteristics - fatty acid composition, solid fat content at different temperatures and thermal properties are given in our previous research [12]. Native soybean lecithin (Victoriaoil JSC, Serbia) was used as an emulsifier. The composition of fat filling included: 50% of powdered sugar, 30% of vegetable fat, 7% of cocoa powder, 5.5% of soy flour, 7% of milk powder, 0.5% of lecithin.

Process method

Research included the influence and optimization of processing parameters: agitator shaft speed and milling on physical and sensory characteristics of fat filling produced in a ball mill as well as on milling energy consumption. Selected input factors and responses are listed in Table 1. The extreme levels of the input factors were selected on the basis of previous experiments and the technical limitations of the equipment.

Methods

Fat filling production. At the beginning of production, fat and lecithin were homogenized in a laboratory ball mill (Mašino Produkt, Serbia) at 40 °C for 5 min. Then the fat filling mass was added, and the production of fat filling included defined agitation shaft speed (50, 75, and 100%, or 25, 37.5 and 50 rpm) and milling time (30, 45, and 60 min) for each agitation speed. After the chosen milling time, cream samples were added into sterile plastic cups and capped with plastic lids.

The capacity of laboratory ball mill is 5 kg. It constitutes of a double-jacket cylinder, 0.25 m in diameter and 0.31 m in height and a stirring group. The vertical shaft with horizontal arms, while rotating, puts the steel balls (9.1 mm diameter) in movement. The ball mill is equipped with a temperature control sys-

Table 1. Variables and levels in the experimental design

Input factor	Levels		
	Low (-1)	Medium (0)	High (1)
A: agitator shaft speed, rpm	25	37.5	50
B: milling time, min	30	45	60
Dependent responses			
R1: Particle size parameter *d*(0.1), µm			
R2: Particle size parameter *d*(0.5), µm			
R3: Particle size parameter *d*(0.9), µm			
R4: Hardness, kg			
R5: Work of shearing, kg s			
R6: Thixotropic curve area, Pa/s			
R7: Casson yield stress, Pa			
R8: Casson viscosity, Pa s			
R9: Sensory analysis			
R10: Energy consumption, J/kg			

tem made up of a water jacket equipped with temperature sensors and thermo-regulators controlled by electric board. The maximum temperature is 100 °C, with deviation ±1 °C.

Particle size distribution. The influence of milling variables on particle size distribution in fat filling samples was determined by a Mastersizer 2000 laser diffraction particle size analyzer equipped with a Hydro 2000 μP dispersion unit (Malvern Instruments, England). Fat filling sample was dispersed in sunflower oil at ambient temperature (20±2 °C) and added until adequate obscuration was obtained (10-20%). The results were quantified as volume-based particle size distribution, using Mastersizer 2000 Software. All measurements were performed in triplicate. Obtained particle size distribution parameters included following parameters: *d*(0.5) - mass median diameter of the volume of distribution, indicating that 50% of the sample has a size smaller than that value, whereas 50% has a larger size; *d*(0.1) - indicating that that 10% of the sample mass are particles in sizes smaller than that value; and parameter *d*(0.9) – indicates that 90% are smaller and 10% are larger than that value [13].

Rheological properties. Rheological properties of fat filling samples were determined by a Rheo Stress 600 rotational rheometer (Haake, Germany). The tests were carried out at 40 °C using a concentric cylinder system (sensor Z20 DIN). The shear rate was increased from 0 to 60 s^{-1} within a period time of 180 s, then was kept constant at maximum speed of 60 s^{-1} for 60 s and after that was reduced from 60 to 0 s^{-1}, within 180 s [14].

Textural characteristics. Textural characteristics of fat samples were analyzed using a Texture Analyzer TA.XT Plus (Stable Micro System, UK). The hardness and work of shearing were determined by penetration at ambient temperature of 22±2 °C, according to method Chocolate Spread – SPRD2_SR_PRJ. Each sample was placed into the cone sample holder and pressed down in order to eliminate air pockets. Any excess of sample was scraped off with a knife. The filled cone sample holder was then put in the base holder and 45° cone probe with the diameter of 38 mm was used to penetrate the samples at 3 mm/s.

Sensory analysis. Sensory analysis was made using the scoring procedure described in Popov-Raljić and Laličić-Petronijević [15] with some modifications. A group of 10 experienced panelists, 6 women and 4 men, ages 28 to 45, who had been trained to evaluate the sensory properties of fat filling [16,17], evaluated the following quality parameters using the scores from 1 to 5: appearance (color, brightness, surface), texture (structure, firmness), chewing, smell and taste. The obtained scores of these parameters were multiplied by a weight coefficient (1.0, 0.8, 0.6, 0.6, 1.0) respectively (Table 2).

The category of quality was defined based on the total number of points (<11.2, unacceptable; 11.2--13.1, acceptable; 13.2-15.1, good; 15.2-17.5, very good; 17.6-20, excellent). The fat filling samples were analysed seven days after their stabilization. Cookie samples were plates labeled with three-digit codes from a random number table and served to panelists on white plastic. Evaluation was performed in sensory laboratory of the Faculty of Technology Novi Sad, in partitioned booths, illuminated with fluorescent lights [18].

The milling energy consumption. The milling energy consumption, E [J/kg], was calculated using Eq. (1):

$$E = \frac{Pt}{m} \quad (1)$$

where *P* [W] is power, *m* [kg] is the mass of fat filling (5 kg) and *t* [s] is the time of the milling run determined by the chronometer. Power readings were determined using the Network recorder MC750/UMC750 (Iskra MIS, Slovenia) connected to the ball mill.

Statistical analysis. The main research aims were determination of influences of input factors and their interaction on output factors and optimization of process. According design of experiment (DOE) and response surface method (RSM) within it were used for statistical analysis. Since significant variations at points with extreme values were expected, face centered central composite design with three central points was chosen for analysis.

Influence of 2 input factors: A – agitator shaft speed; B – milling time in a laboratory ball mill on particle size distribution (responses R1-R3), textural characteristics (responses R4 and R5), rheological properties (responses R6-R8), sensory analysis of fat filling (response R9), and energy consumption (R10) was studied.

By specified design, 11 runs were determined and all results were expressed as mean of triplicate measures. Regression analysis was performed, where a full quadratic model was used:

$$R = \beta_0 + \beta_1 A + \beta_2 B + \beta_{12} AB + \beta_{11} A^2 + \beta_{22} B^2 \quad (2)$$

where R is the measured response; β_i and $\beta_{i,j}$, $i,j \in \{0,1,2\}$ are regression coefficients; A, B are the coded levels of input factors, the term AB represents interactions of input factors, while A^2 and B^2 represent

Table 2. Sensory evaluation of the chocolate quality using the scoring procedure

Basic sensory properties	Score	Weight coefficient	Description of the evaluated property
Appearance	5	0.6	Smooth, bright surface; irreproachable color
color, brightness,	4		Insignificant deviation of color; smooth, bright surface
surface	3		Lower quality color; insufficient bright surface; air bubbles (a small number); insignificant packaging demage
	2		Atypical color; matte surface or less separation of oil phase on the surface; a large number of air bubbles; less damage of the packaging
	1		Appearance of gray-white spots on the surface; completely matte surface or separation of oil phase on the surface; packaging demage
Texture	5	0.8	Homogeneous, smooth, structure; appropriate firmness; soft, spreadable consistency
Structure, firmnes	4		Insignificant deviation of firmness and consistency; homogeneous, smooth, structure
	3		Appearance of air bubbles in the mass; firmness and consistency inappropriate; homogeneous, smooth, structure
	2		Grainy structure; inadequate firmnes; low spreadability; insignificant deviation of homogenous structure
	1		Rough grainy structure; inadequate firmnes; low spreadability; unhomogenous structure
Chewig and other textural properties	5	1.0	Appropriate chewiness; melting in the mouth
	4		Slower melting; good chewiness, spreadiness
	3		Average chewiness; spreadiness; weak sandiness
	2		Slow melting; sandiness; stickiness
	1		Slow melting; heavy sandiness; stickiness
Smell	5	0.6	Appropriate; rounded; aromatic; resistant for some period of time
	4		Appropriate; poorer rounded; aromatic
	3		Appropriate; poor rounded; weakly aromatic
	2		Not appropriate; sourish; staled
	1		Foreign odor; sour; staled; mouldy
Taste	5	1.0	Appropriate; rounded; aromatic; resistant for some period of time
	4		Appropriate; less rounded; aromatic
	3		Poorly rounded; poorly aromatic
	2		Sourish; not rounded
	1		Foreign taste; sour; bitter

quadratic terms. The adequacy of the obtained model was confirmed by R^2 coefficient and lack of fit value.

Significance of input factors and their interaction in the observed model were determined by statistical method of analyses of variance (ANOVA), where sum of squares were used to calculate the corresponding contributions. Using 5% level of significance, a factor is considered to affect the response if the p value is less than 0.05.

Constrained optimization procedure maximizes desirability function [21]:

$$D = \left(d_1 d_2 \ldots d_n\right)^{\frac{1}{n}}$$

where d_i, $i \in \{1,2,\ldots,n\}$, are individual desirability functions associated to constrains on responses R_i.

The analyses were carried out using Statistica 12 (Stratosoft, USA) and Design-Expert 10 (trial version).

RESULTS AND DISCUSSION

The influence of main milling variables on physical properties and sensory characteristics of fat filling, and milling energy consumption are shown in Table 3.

The input factors, agitator shaft speed (A) and milling time (B) affected both energy consumption and all quality characteristics of fat filling produced in a laboratory ball mill. Table 4 represents regression coefficients for responses R1-R9, as well as appropriate R^2 coefficients.

Physical and sensory properties of fat filling. Figure 1a shows the curve of particle size distribution

Table 3. The influence of input factors on dependent responses

Run	Input factor		Dependent response									
	A	*B*	*R*1	*R*2	*R*3	*R*4	*R*5	*R*6	*R*7	*R*8	*R*9	*R*10
3	25	30	3.42	20.96	81.92	5.96	5.23	2987	40.25	4.78	11.77	122.98
5	25	45	3.16	14.85	53.07	6.38	5.27	2699	41.09	4.57	12.66	207.30
7	25	60	3.21	14.44	48.31	6.98	5.93	2071	43.28	2.78	14.50	292.40
11	37.5	30	3.26	12.98	44.12	19.13	18.34	4918	41.25	6.58	12.1	182.00
4	37.5	45	2.92	12.74	43.28	18.36	17.59	4057	43.76	7.89	14.84	285.4
6	37.5	45	2.79	12.26	42.48	19.65	19.32	3862	40.24	8.59	14.35	273.6
9	37.5	45	2.99	13.15	44.09	18.06	16.86	4215	45.15	7.26	15.36	294.80
2	37.5	60	2.98	12.44	42.02	19.53	20.57	4548	53.63	8.67	17.37	399.70
8	50	30	3.06	12.75	46.87	23.44	22.81	2192	30.16	10.21	16.53	247.60
1	50	45	2.78	10.55	36.73	24.85	23.39	2950	37.75	12.75	16.28	408.20
10	50	60	2.84	10.82	35.89	24.98	24.52	3230	40.77	14.76	16.86	568.90

Table 4. Regression equation coefficients for responses R1-R10

Coefficient	*R*1		*R*2		*R*3		*R*4		*R*5	
β_0	2.9121	*p*	12.285	*p*	41.313	*p*	18.883	*p*	18.184	*p*
β_1	-0.185	0.0013	-2.688	0.0039	-10.64	0.0104	8.9917	< 0.0001	9.0483	< 0.0001
β_2	-0.118	0.0087	-1.498	0.0369	-7.782	0.0330	0.4933	0.1410	0.7733	0.1027
β_{12}	-0.002	0.9455	1.1475	0.1377	5.6575	0.1435	0.13	0.7224	0.2525	0.6178
β_{11}	0.0397	0.4047	1.0618	0.2502	6.2234	0.1896	-3.557	0.0004	-4.246	0.0009
β_{22}	0.1897	0.0074	1.0718	0.2463	4.3934	0.3330	0.1584	0.7304	0.8795	0.2006
R^2	0.9434		0.8923		0.8653		0.9954		0.9917	
Lack of Fit	0.9432		0.0697		0.1025		0.7439		0.8417	
Coefficient	*R*6		*R*7		*R*8		*R*9		*R*10	
β_0	4232.6	*p*	44.144	*p*	7.9637	*p*	14.722	*p*	285.87	*p*
β_1	102.5	0.5108	-2.657	0.0774	4.265	< 0.0001	1.8233	0.0085	100.34	< 0.0001
β_2	-41.33	0.7868	4.3367	0.0152	0.7733	0.0125	1.355	0.0263	118.07	< 0.0001
β_{12}	488.5	0.0401	1.895	0.2531	1.6375	0.0012	-0.65	0.2763	37.97	0.0006
β_{11}	-1690	0.0006	-6.364	0.0182	0.6208	0.1035	-0.059	0.9332	19.976	0.0249
β_{22}	218.42	0.3722	1.6558	0.4104	-0.414	0.2420	0.2061	0.7703	3.0763	0.6463
R^2	0.9304		0.8633		0.9902		0.8527		0.9967	
Lack of Fit	0.1452		0.4110		0.8495		0.1321		0.5899	

in fat filling after milling on 3 roll mill where parameters *d*(0.1), *d*(0.5) and *d*(0.9) have the following values: 5.18, 45,40 and 110,77 µm, respectively. Milling in laboratory ball mill resulted in decreasing of all particle size parameters and more uniform distribution, getting the appearance of Gaussian curve distribution.

Regarding particle size distribution, increasing the agitator shaft speed generally decreased parameters *d*(0.1), *d*(0.5), and *d*(0.9) in fat filling samples, as also confirmed by negative value of equation coefficient β_1. The example of the influence of agitator shaft speed on particle size distribution is given in Figure 1c. It was also evident that increasing the milling time observed at each particular agitator shaft speed generally decreased all particle size parameters, as confirmed by negative value of β_2 and shown in Figure 1b presenting particle size distribution of fat filling produced under maximum agitator shaft speed and each applied milling time.

Analyzing all samples, the parameter *d*(0.1) (response *R*1) ranged from 2.78 to 3.42 µm, meaning that 10% of the volume distribution of the samples was smaller than the particular d(0.1) value. The 50% of the volume distribution in all samples was smaller than 20.96 µm, which was the highest value for *d*(0.5) achieved in fat filling sample produced with agitator shaft speed of 25 rpm and minimum milling time. The parameter *d*(0.9) indicated that 90% of the volume distribution of all the samples was smaller than 81.92

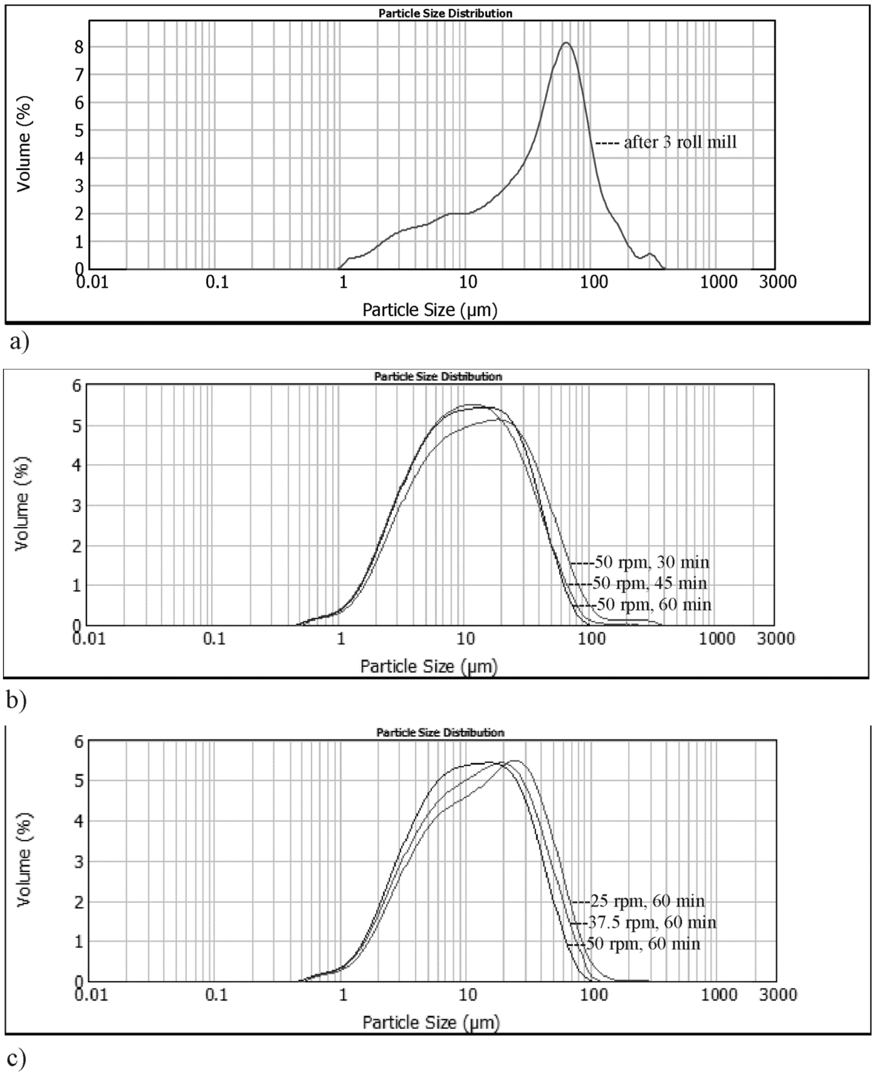

Figure 1. Influence of: a) 3 roll mill pre-refiner; b) milling time observed at maximum agitator shaft speed; c) agitator shaft speed observed at maximum milling time on particle size distribution of fat filling.

µm, which is the value observed in sample with minimal applied agitator shaft speed and milling time.

A fat filling mass, like a cream mass, has a non-uniform particle size distribution and it exhibits thixotropic properties characterized by a plastic flow and yield stress [19]. Decreasing particle size parameters, while increasing the agitator shaft speed and milling time in the laboratory ball mill affected the textural and rheological properties of fat filling in terms of increasing the hardness and Casson viscosity. Figure 2a represents the influence of agitator shaft speed on rheological properties of fat filling produced at maximum milling time, while the influence of milling time on rheological properties observed at maximum agitator shaft speed is shown on Figure 2b.

Afoakwa *et al.* [20] investigated the effects of the particle size distribution and the composition on the rheological properties of dark chocolate, where increase in particle sizes resulted in a decrease in Casson plastic viscosity due to an increased number of particles, and points of contact between them. In this case, the decrease in particle size distribution contributed to higher specific surface area of particles and due to more compact system with higher values of textural parameters and Casson viscosity. The positive values of regression coefficients β_1 and β_2 for responses *R*4 and *R*5 confirm that the increase of main milling variables also increased the values of hardnes and work of shearing of fat filling. It is also obvious that increasing the milling time influenced increasing the values of thixotropic curve area and Casson viscosity while increasing the agitator shaft speed increased Casson yield stress and Casson viscosity.

Affecting textural and rheological properties, particle size distribution also contributed to some

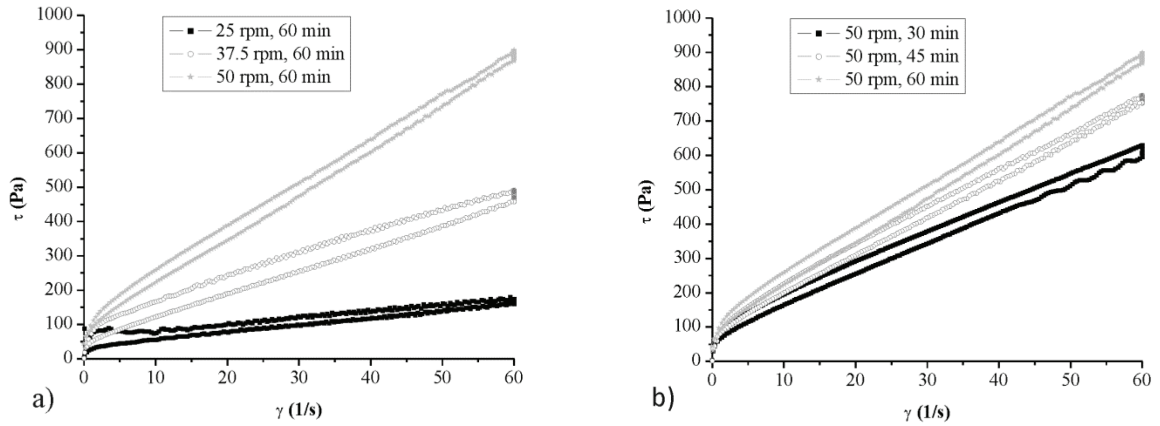

Figure 2. Influence of agitator shaft speed and milling time on rheological properties of fat filling: a) the influence of agitator shaft speed observed at maximum milling time; b) the influence of milling time observed at maximum agitator shaft speed.

sensory properties of fat filling. All fat filling samples were well scored by the panelists 24 h after production (Figure 3).

All fat filling samples had intrinsic color of cocoa 24 h after production, without the presence of white and gray color on the surface. However, the samples produced under maximum agitator shaft speed as well as sample produced uder agitator shaft speed of 37.5 rpm and maximum milling time had a slightly darker color probably due to a higher specific surface area of particles, which contributed to higher sensory scores. On the other hand, lower specific surface area of particles in samples milled under minimum agitator shaft speed and agitator shaft speed of 37.5 rpm and milling time of 30 and 50 min have not contributed to fat phase separation on the fat filling surface, but affected chewiness causing a grainy feeling in the mouth while eating. Also, the consistency of samples produced under minimum agitator shaft speed, especially the sample milled for 30 min, were grainy and inhomogeneous. Furthermore, free fatty phase worsened the smell and taste of those samples. Increasing the milling time on 45 and 60 min improved sensory characteristics of fat filling samples produced under minimal gitator shaft speed where sample milled for 60 min belongs to category of quality – good. Sample milled under agitator shaft speed of 37.5 rpm and maximum milling time had the most aromatic smell and was the least steaky while chewing, compared to all other samples. It is classified in quality category – very good, fallowed by fat filling samples produced under maximum agitator shaft speed that also classified in quality category – very good.

The positive values of coeeficients β_1 and β_2 for response $R9$ indicate improving of sensory characteristics by increasing the main milling parameters. On the other hand, the positive values of those coeffi-

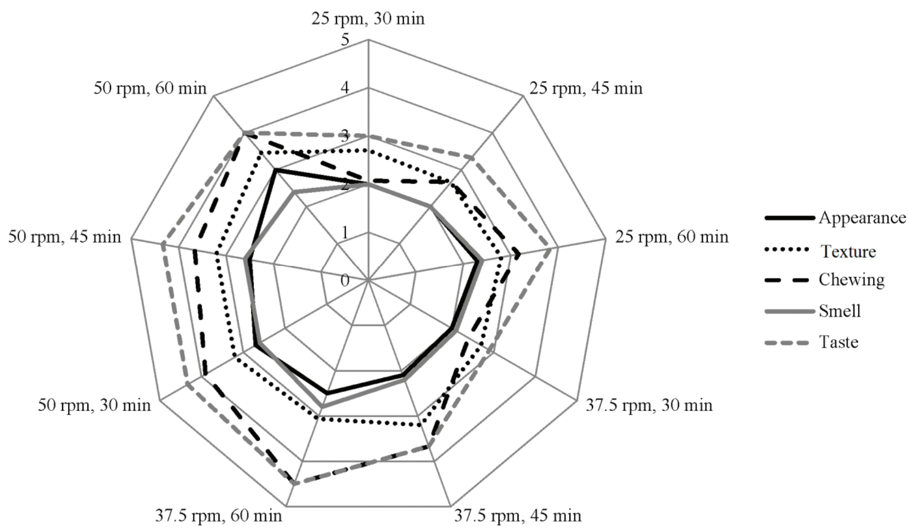

Figure 3. Weight scores of basic sensory properties of fat filling.

cients for response *R*10 certainly indicate higher energy consumption when increasing agitator shaft speed and milling time.

The contribution of input factors on dependent responses is shown on Figure 4.

Increasing the agitator shaft speed had the highest influence on decreasing all particle size parameters with the highest impact on parameter *d*(0.5) with 63.89%. The milling time had less pronounced influence on particle size parameters, compared to agitator shaft speed.

Regarding textural characteristics agitator shaft speed had the contribution of even 93.52% on hardness and 90.52% on work of shearing, while milling time had minimal influence of less than 1%. Thixotropic curve area and Casson viscosity were also the most affected by the agitator shaft speed with the contribution of 86.30% of quadratic term on thixotropic curve area and linear 87.41% on Casson viscosity. On the other hand, both input factors have statistically significant influence on Casson yield stress.

Sensory characteristics of fat filling were most affected by agitator shaft speed (60.87%) followed by milling time (33.62%) where increasing both input factors increased sensory quality.

Energy consumption. Power requirements are highly influenced by the agitator shaft speed. Previous studies of Fišteš *et al.* [5] showed that every increase of agitator shaft speed of the laboratory ball mill led to a statistically significant increase in power requirements. Alamprese *et al.* [6] also stated that the energy consumption of ball mill increased proportionally to the increase in speed and refining time. The results obtained in this study also showed that the both investigated milling parameters have a significant influence on energy consumption.

Although the highest values of milling energy consumption were obtained under maximum agitator shaft speed, the ANOVA under selected design showed that milling time had higher contribution (55.4%) on energy consumption in comparison to agitator shaft speed (40.04%). In terms of reducing

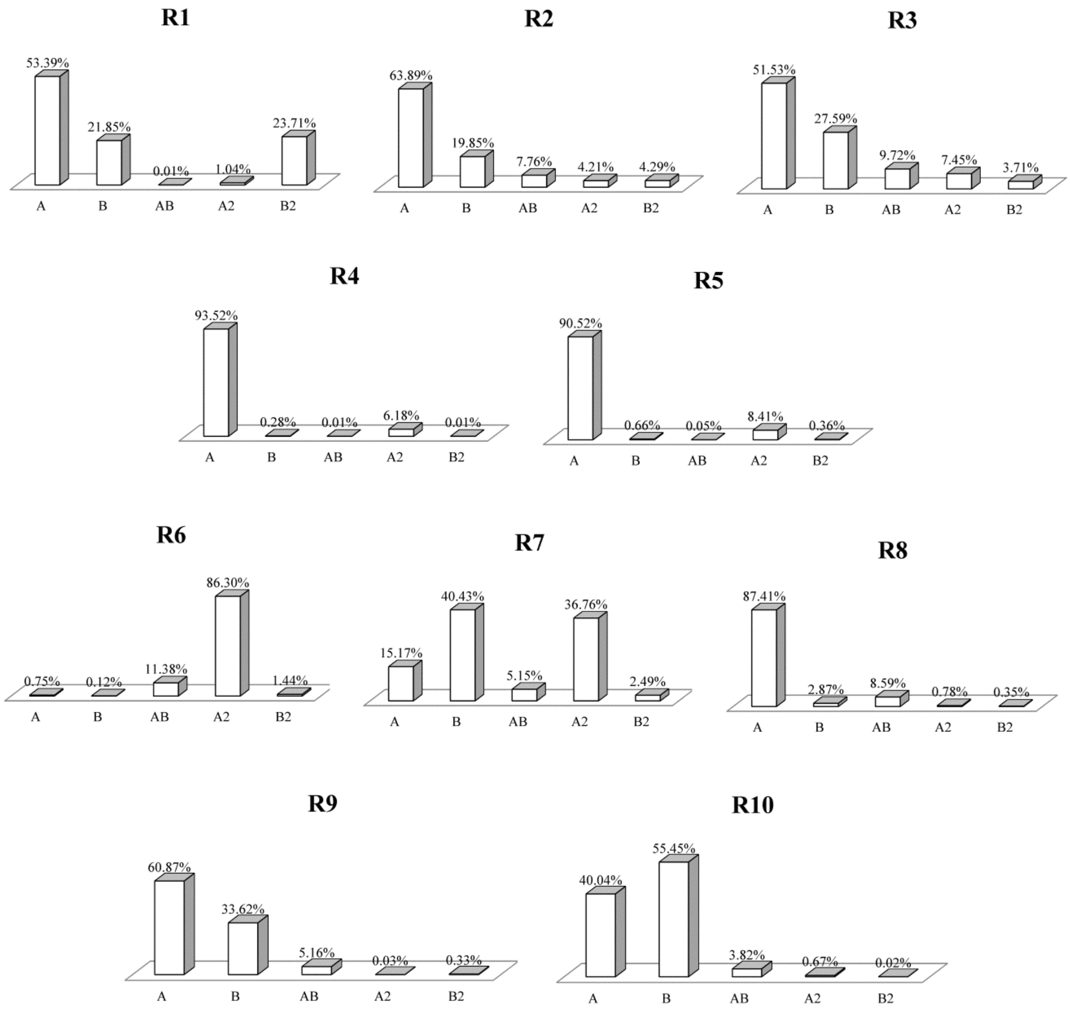

Figure 4. The contribution of input factors on dependent responses.

the energy consumption of the process the milling time should be kept at minimum. Lucisano *et al.* [4] showed that prolonged reefing time could even become a problem because of significant and undesirable reduction in size. Also, with shorter retention time the amount of product that can be processed during a certain period of time is increasing, therefore the capacity of ball mill is also increasing. From the energy efficiency point of view, the agitator shaft speed should be run at lowest possible speed to meet the product quality requirements.

Optimization. Model obtained by regression analyses is used to perform the optimization of processing parameters in order to find the optimal combination of input factors such that *R*3, *R*6, *R*7 and *R*10 have minimal, and *R*9 as it possible higher value. The recommended combination included maximal value of A and minimal value of *B*, while values of responses are given in Table 5.

Table 5. Optimization of processing parameters on quality characteristics of fat filling and milling energy consumption

Cond.	*A*	*B*	*R*1	*R*2	*R*3	*R*4	*R*5	*R*6	*R*7	*R*8	*R*9	*R*10	D
	In range	In range	None	None	Min	None	None	Min	Min	None	Max	Min	
Sol. 1	50	30	3.08	12.08	43.42	23.85	22.84	2416.31	30.55	10.02	15.99	253.22	0.826
Sol. 2	50	27.02	3.18	12.62	45.76	23.80	23.02	2422.86	30.03	9.36	15.94	223.60	0.831

With this input values, using regression coefficients (Table 4), the responses *R*3 (43.42), *R*6 (2416), *R*7 (30.55) and *R*10 (253.2) are obtained within agiator shaft speed of 50 rpm and milling time of 30 min as optimal.

CONCLUSION

The main objective of the study was to determine the effect of agitator shaft speed and milling time on quality of fat filling produced in a laboratory ball mill, as well as to optimize the ball milling parameters in order to reduce energy consumption without compromising the quality of the product.

High values of the determination coefficients (R^2, 0.86-0.99) indicated that the application of fitted full quadratic model for describing experimental data by means of the theoretical curve was justified. Equation coefficients indicated that the increase of both agitator shaft speed and milling time actually increased the degree of particle size reduction in fat filling, which increased the hardness and Casson viscosity and, at the same time, improved the sensory characteristics of fat filling. On the other hand, the increase of milling variables contributed to higher energy consumption in fat filling production.

The model obtained by regression analyses showed that maximum agiator shaft speed and minimum milling time are found as optimal in fat filling production, where high desirability value of 0.8246 provides significance of the proposed solution.

REFERENCES

[1] B. Pajin, Đ. Karlović, R. Omorjan, V. Sovilj, D. Antić, Eur. J. Lipid Sci. Tech. **109** (2007) 1203-1207

[2] V. Glicerina, F. Balestra, M. Dalla Rosa, S. Romani, J. Food Eng. **145** (2015) 45-50

[3] B. Pajin, D. Zarić, Lj. Dokić, Z. Šereš, D. Šoronja-Simović, R. Omorjan, I. Lončarević, Acta Period. Technol. **42** (2011) 101-110

[4] M. Lucisano, E. Casiraghi, M. Mariotti, Eur. Food Res. Technol. **223** (2006) 797-802

[5] A. Fišteš, D. Rakić, B. Pajin, Lj. Dokić, I. Nikolić, Chem. Ind. **67** (2013) 747-751

[6] C. Alamprese, L. Datei, Q. Semeraro, J. Food Eng. **83** (2007) 629-636

[7] S. Yeganehzad, M. Mazaheri-Tehrani, M. Mohebbi, M.B. Habibi Najafi, Z. Baratian, J. Agr. Sci. Tech. **15** (2013) 125-135

[8] D. Zarić, B. Pajin, I. Lončarević, D. Šoronja-Simović, Z. Šereš, Acta Period. Technol. **43** (2012) 139-148

[9] M. Todorović, D. Živković, M. Mančić, G. Ilić, Chem. Ind. Chem. Eng. Q. **20** (2014) 511-521

[10] M.G. Scanlon, J. Lamb, J. Mater. Sci. **30** (1995) 2577-2583

[11] R.G. Holdich, Fundamentals of Particle Technology; Midland Information Technology and Publishing: Leicestershire, 2002, p.115

[12] I. Lončarević, B. Pajin, R. Omorjan, A. Torbica, D. Zarić, J. Maksimović, J. ŠvarcGajić, J. Texture Stud. **44** (2013) 450-458

[13] Z. Stojanović, S. Marković, Tech.- New Mater. **21** (2012) 11-20

[14] IOCCC 2000. Viscosity of Cocoa and Chocolate Products - Analitycal Method 46. Available from CAOBISCO, rue Defacqz 1, B-1000 Bruxelles, 2000

[15] J. Popov-Raljić, J. Laličić-Petronijević, Sensors **9** (2009) 1996-2016.

[16] ISO 8586-1 (1993)

[17] ISO 8586-2 (2008)

[18] ISO 8589 (2007)

[19] I. Lončarević, B. Pajin, J. Petrović, D. Zarić, M. Sakač, A. Torbica, D.M. Lloyd, R.Omorjan, J. Food Eng. **171** (2016) 67-77

[20] E.O. Afoakwa, P. Alistair, M. Fowler, Eur. Food Res. Technol. **226** (2008) 1259-1268

[21] R.H. Myers, D.C. Montgomery, Response Surface Methodology - Process and Product Optimization Using Designed Experiments, 2nd ed., John Wiley & Sons Inc., Toronto, 2008.

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NAUČNI RAD

OPTIMIZACIJA PROCESNIH PARAMETARA KUGLIČNOG MLINA U PROIZVODNJI MASNOG PUNJENJA

Cilj ovog rada bio je da se ispita uticaj procesnih parametara - brzine obrtanja mešača (25, 37,5 i 50 rpm) i vremena mlevenja (30, 40 i 50 min) na potrošnju energije kao i fizičke i senzorske karakteristike masnog punjenja, proizvedenog u laboratorijskom kugličnom mlinu. U okviru metode odzivne površine korišćen je "face centered composite" dizajn. Izvršena je regresiona analiza odzivne površine gde su su eksperimentalno dobijeni podaci fitovani potpunim kvadratnim modelom. Rezultati su pokazali da brzina obrtanja mešača ima najveći uticaj na fizičke karakteristike (raspodelu veličina čestica, reološke i teksturalne karakteristike) kao i na senzorske osobine masnog punjenja. S druge strane, na potrošnju energije prilikom proizvodnje masnog punjenja u kugličnom mlinu najveći uticaj ima vreme mlevenja (55.4%) dok brzina obrtanja mešača ima uticaj od 40.04%. Model dobijen regresionom analizom korišćen je za optimizaciju procesnih parametara kako bi se dobila kombinacija brzine obrtanja mešača i vremena mlevenja koja obezbeđuje minimalnu potrošnju energije bez narušavanja kvaliteta masnog punjenja. U cilju optimizacije proizvodnje masnog punjenja u kugličnom mlinu neophodno je primeniti maksimalnu brzinu obrtanja mešača i minimalno vreme mlevenja.

Ključne reči: masno punjenje, parametri mlevenja, fizičke karakteristike, senzorne karakteristike, optimizacija.