Biblid: 1821-4487 (2013) 17; 1; p 20-23
UDK: 66.046

Original Scientific Paper
Originalni naučni rad

APPLICATION OF RESPONSE SURFACE METHODOLOGY IN THE DEVELOPMENT OF NEW HEAT-STABLE FRUIT FILLING'S COMPOSITION

APLIKACIJA METODE ODZIVA POVRŠINE U RAZVOJU NOVIH TERMOSTABILNIH KOMPOZICIJA VOĆNIH UKUSA

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ABSTRACT

The research was designed in aim to study the influence of amylopectin potato starch on heat-stable, physicochemical, sensory and rheological characteristics of fruit fillings on the basis of Response Surface Methodology (RSM). The fruit fillings' samples were prepared locally from apple puree, sugar, amylopectin potato starch and citric acid. After a short-term storage they were put through a series of physicochemical, sensory and rheological tests, including standard bakery test for evaluating the heat-stability after baking in the oven at a temperature of 220 °C for 20 minutes. The regression polynomial equations and response surface plots were represented in the research in order to visualize the influence of two main factors – soluble solids and amylopectin starch content – on heat-stable and rheological properties of the fruit fillings.

Key words: *amylopectin starch, fruit filling, thermal stability.*

REZIME

Istraživanje je osmišljeno u cilju da se ispita uticaj amilopektin krompirovog skroba na termičku stabilnost, fizičko-hemijske, senzorne i reološke karakteristike voćnih punila na osnovu metodologije odzivne površine (RSM). Uzorci voćnih punila su pripremljeni od jabukovog pirea, šećera, amilopektin krompirovog skroba i limunske kiseline. Nakon kratkoročnog skladištenja izloženi su nizu fizičko-hemijskih, senzornih i reoloških testova, uključujući i standardni pekarski test za procenu toplotne stabilnost nakon pečenja u rerni na temperaturi od 220 °C za 20 minuta. Regresione jednačine, polinomi i ravan odzivne površine predstavljeni su u istraživanju da bi se vizuelno prikazao uticaj dva ključna faktora - rastvorljive suve materije i amilopektin skroba - na toplotu stabilnim i reološke osobine voćnih punila.

Ključne reči: *amilopektin skrob, voćno punjenje, termička stabilnost.*

INTRODUCTION

Bakery and confectionary products with natural fruit fillings are a worldwide favorite type of meals, well accepted among consumers of all ages. Nowadays one of the significant issues related to the filled bakery products (doughnuts, muffins, puff pastries, croissants, pies and buns) consists in thermal instability (physicochemical and sensory degradation, boil-out and water releasing otherwise called "syneresis", etc.) of the used fruit fillings. Besides the requirement to high heat-stability, fruit fillings should also possess a number of textural parameters which are essential for overall sensory acceptance. Each parameter is influenced by a quantity and quality of certain compound presented it the whole system. Hardness is directly depended on percentage of hydrocolloids and soluble solids, while thick consistency and chewiness on eating are mostly related to molecular weight of the complex carbohydrates. Heat-stability is a function of stabilizator content and state, as well as soluble solids content and type of raw materials. In large-scale manufacturing, significant boil-out during baking was removed by using gelatin (*Schrieber and Gareis, 2007*), various gums (gellan, xantan, and guar) and also a large quantity of starches in fruit fillings' formulations. The major disadvantage of these solutions is that fruit compositions prepared for fillings on the basis of gums or natural ordinary starches experience severe syneresis after prolonged storage, and, thus, can be considered heat-stable only if they are applied directly after the preparation. As long as wide groups of consumers try to avoid gelatin as a hydrolyzed form of collagen (protein of animal origin) due to the health concerns, religious reasons or diet preferences, it is not so profitable to use this food additive as heat-stabilization agent for fruit fillings. To replace gelatin, it is feasible to utilize various types of natural polymers, depending on the final soluble solids of fruit fillings. Natural amylopectin potato starch was selected as possible replacer of gums and gelatin for heat-stable fruit fillings' development due to its excellent thermal and freeze-thaw stability and also capacity to bind water without moisture loss or syneresis. Amylopectin starch is a widely used ingredient in food industry for its numerous technologically important properties (thickening, texturizing, water binding, etc.) and, therefore, can be also used in heat-stable fruit fillings' development. The main goal of this research was to study the influence of amylopectin potato starch on heat-stable, physicochemical, sensory and rheological parameters of fruit fillings within a wide range of soluble solids – from 40 to 70 °Brix by using Response Surface Methodology (RSM) as one of the most commonly used techniques of experimental design.

MATERIAL AND METHOD

Raw materials

Sugar was purchased at a local supermarket (Chisinau, Republic of Moldova). Apple aseptic puree was manufactured at the canning plant "Conserv-E" (Chisinau, Republic of Moldova). Citric acid solution (50%) was prepared locally in the Laboratory of Functional Foods of the Practical Scientific Institute of Horticulture and Food Industry (Republic of Moldova). Amylopectin potato starch type Eliane BC-160 was kindly supplied by the Trading House AVERS (Sankt-Peterburg, Russian Federation).

Fruit fillings preparation

Sucrose was initially mixed with apple puree and heated up to complete dissolution. This prior apple-sugar mix served as the basis for four different fruit fillings' formulations presented in the study. In order to prepare fruit filling's samples within a large diapason of soluble solids, some water was added to the mixture for obtaining fillings with low soluble solids and respectively, in case of high soluble solids the initial mixture was concentrated up to requested dry matter. At a temperature of more than 80 °C, dry amylopectin potato starch was added to the apple blend according to the four different formulations and followed by intensive mixing. After obtaining a homogeneous mixture, citric acid was added to the final apple filling's composition.

All fruit fillings' samples were hot bottled in glass bottles properly sterilized, leaving a space for the formation of vacuum after closing. After cooling, the flasks were sealed and stored for two days in the refrigerator for subsequent physicochemical, rheological, and sensory analyses.

Physicochemical and sensory analysis

The physicochemical, rheological and sensory analysis of the finished fruit fillings were conducted at the Laboratory of Functional Foods of the Practical Scientific Institute of Horticulture and Food Industry of the Republic of Moldova.

The soluble solids of the prepared fruit fillings were measured using benchtop refractometer ABBE and expressed in °Brix. The pH was determined by dint of potentiometric method, introducing the electrode directly into the fruit fillings. Fruit fillings' water activity was measured by using portable Novasina water activity analyzer ms1 at a temperature of 26 ºC.

The rheological behavior of the initial fruit fillings was investigated through experimental measurements at the rotational rheometer Rheotest RV2 at a temperature of 30 °C and shear rate s^{-1}.

Sensory analysis of the fruit fillings' samples after baking was carried out by 10 randomly selected members of the panel. Each of the key sensory parameters (taste, color, appearance, flavor and texture) was estimated by a numerical estimation, ranging between 1 (for extremely bad characteristics) and 5 (excellent characteristics). Average value based on estimates of each characteristic was calculated to receive overall evaluation of the sample.

Determination of heat-stability

The fruit fillings samples were put through a standard bakery test to determine their thermal stability as follows: a specific amount of prepared fruit filling was given into a base of special filter paper named "Blue ribbon" with a diameter of 120 mm by a metal ring with defined geometry (50 mm diameter and 10 mm height) and then was baked under exactly fixed conditions: at a temperature of and 220 °C for 20 minutes (*Herbstreith & Fox KG*). The bakery index was determined by measuring the sample diameter before and after baking established by placing a line across the sample and calculating by using the following formula:

$$BI = 100 - \frac{D_2 - D_1}{D_2} \cdot 100 \quad (1)$$

where: BI – bakery index, %; D_1 – average sample diameter before baking, mm; D_2 – average sample diameter after baking, mm.

Diameter of the filling sample before baking is 50 mm, because it's the diameter of the metal ring used in bakery test. For measuring the sample diameter depending on its shape from two to four lines were drawn, and the average was calculated.

Statistical analysis

Response Surface Methodology (RSM) was applied to determine the optimal quantity of amylopectin starch introduced into fruit filling's composition for attributing high thermostability to the final product. It was established that mainly soluble solids and percentage of stabilizer affect fruit fillings' thermal stability and rheological behavior (at the same high temperature and for the same baking duration). Thus, only percentage of amylopectin starch and soluble solids content as independent variables were used in two-level factorial design. The levels of these variables were set at: 0.1 (or "-1" in coded form) and 1.0 (or "+1" in coded form) for percentage of amylopectin starch and 40 (or "-1" in coded form) and 70 (or "+1" in coded form) for soluble solids, °Brix. The heat-stability of fruit fillings as one of the response variables was expressed through the bakery index (BI, units). There was also introduced a control sample as zero point in the design of experiments. All experiments adjusted by the design planned in coded and encoded form of process variables, were conducted randomly. The results obtained through application of response surface methodology were verified by conducting the validation experiments under the optimized conditions of all the factors. The adequacy of the regression equations was evaluated by the F-test for analysis of variance (ANOVA) using statistical package STATISTICA v.6. In order to visualize the influence of all the factors on the response variables 3D surface plots were drawn by using MATCAD v.15

RESULTS AND DISCUSSION

Basic physicochemical indicators of the fruit fillings prepared with amylopectin starch are presented in Table 1.

Table 1. Physicochemical indicators of the fruit fillings prepared with amylopectin starch

Product name	pH	$a_{w\ t=26ºC}$	Soluble solids after baking, ºBrix
Fruit filling with 1 % starch and 40 ºBrix	2.9	0.952	67
Fruit filling with 1 % starch and 70 ºBrix	2.9	0.740	75
Fruit filling with 10 % starch and 40 ºBrix	3.1	0.916	72
Fruit filling with 10 % starch and 70 ºBrix	3.1	0.690	85
Fruit filling with 5 % starch and 40 ºBrix	3.0	0.932	69

After baking at a temperature of and 220 °C for 20 minutes the fruit filling samples were estimated for basic sensory parameters. The statistical analysis of sensory scores revealed that no significant difference was found ($p>0.05$) before and after baking for the taste, color, appearance, flavor and texture of all fruit fillings prepared with high percentage of amylopectin starch (10 %). However, the overall acceptability of these fruit fillings samples with high content of amylopectin starch can not be considered good because of the high-adhesive, viscous and tough texture. Sensory score for the taste, color, flavor and overall acceptability of the fruit fillings prepared with both low and high soluble sol-

ids and low content of amylopectin starch (1%) after baking process was significantly different from initial values ($p<0.05$). Mean values of the fruit fillings' organoleptic parameters are shown in Table 2, indicating low acceptability of the product.

Table 2. Organoleptic parameters of the fruit fillings prepared with amylopectin starch

Product name	Average sensory scores				
	taste	color	appearance	flavor	texture
Fruit filling with 1% starch and 40 ºBrix	4.5	5.0	4.5	4.0	4.0
Fruit filling with 1% starch and 70 ºBrix	4.0	4.0	4.0	3.5	4.0
Fruit filling with 10% starch and 40 ºBrix	3.0	5.0	3.0	4.0	3.0
Fruit filling with 10% starch and 70 ºBrix	2.5	3.0	2.5	3.5	2.0

However, quality characteristics of the fruit fillings analyzed under laboratory conditions have demonstrated that they meet the international food standard CODEX STAN 296-2009 FOR JAMS, JELLIES AND MARMALADES.

The elaboration of heat-stable fruit fillings was realized through design expert software package STATISTICA v.6. The design of experiments with independent and dependent variables in coded and encoded form is reported in Table 3.

Table 3. 2^k design matrix for fruit fillings development on the basis of amylopectin starch

No	X_1 starch content, %		X_2 soluble solids, °Brix		Y_1 bakery index expressing hermal stability	Y_2 viscosity, Pa·s
	Coded values	Encoded values	Coded values	Encoded values		
1	1	10	-1	40	90.9	164.6
2	1	10	1	70	100.0	479.4
3	-1	1	-1	40	55.6	18.9
4	-1	1	1	70	40.0	64.7
5	0	5	0	40	70.9	182.4

After experimental data processing, the following regression equations (2 and 3) describing fruit fillings heat-stability and viscosity in terms of actual values were obtained:

$$BI = 76.03 + 0.27 \cdot A - 0.61 \cdot SS + 0.09 \cdot A \cdot SS \quad (2)$$

$$V = 67.21 + 30.38 \cdot A + 0.46 \cdot SU + 1.06 \cdot A \cdot SU - 5.34 \cdot A^2 \quad (3)$$

where: BI – bakery index, units; V – viscosity, Pa·s; A – amylopectin starch content, %; SS – soluble solids, %.

F-test for analysis of variance (ANOVA) using statistical package STATISTICA v.6 has shown that the derived models were statistically significant. The validation experiments' data closely agreed to the predicted values of the developed models with acceptable percentage errors. The response surface plots of the polynomial equations represented above have been plotted using MATCAD v.15 as a function of two variables i.e. soluble solids and amylopectin starch content (Figures 1, 2) in order to visualize their common effect on the response variables.

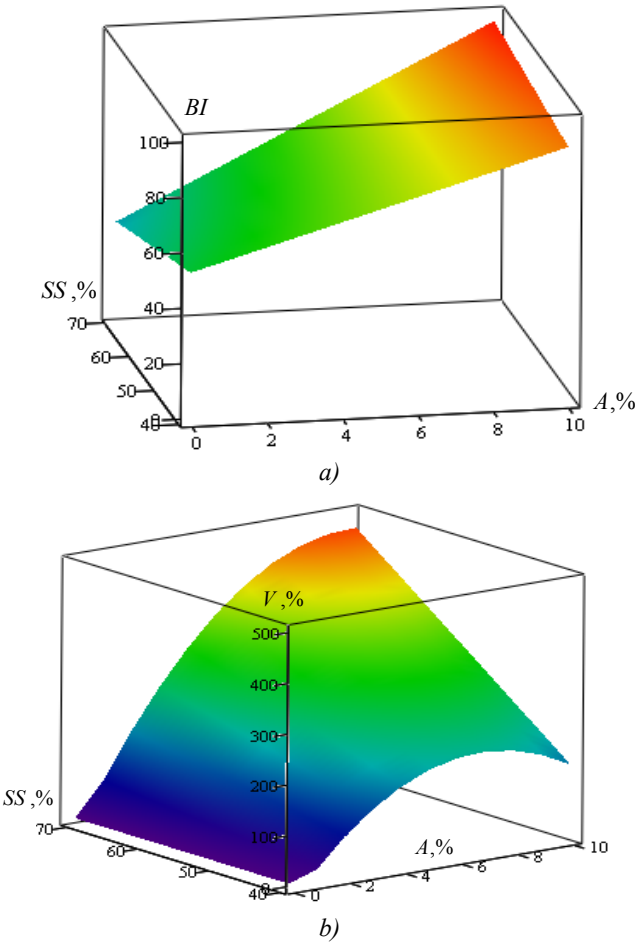

Figure 1. 3D surface plots: Effect of amylopectin starch concentration and soluble solids on bakery index (a) and viscosity (b) of fruit fillings

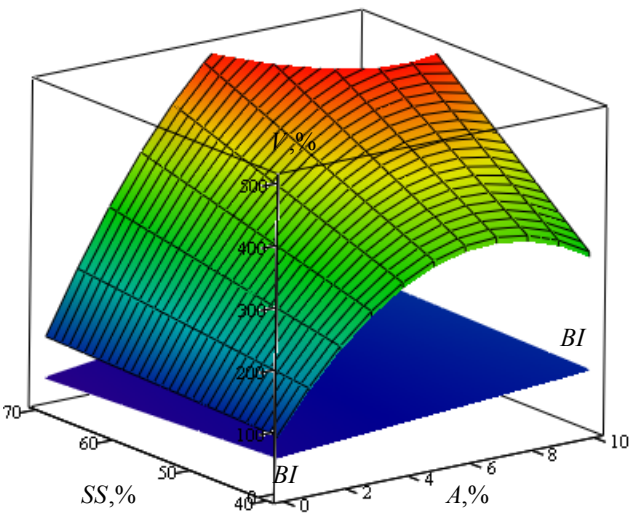

Figure 2. The dependence of amylopectin starch content and soluble solids on both bakery index and viscosity of fruit fillings

Analyzing the validation experiments, it is evident that the fruit fillings samples prepared with high content of amylopectin starch (>5 %) didn't become burnt or caramelized and maintained their original shape and volume. The taste and aroma of the fresh apple puree was also well preserved. However, the con-

sistency of these fruit fillings was too much sticky, gooey and viscous, that considerably deteriorated the overall acceptability of the product.

CONCLUSION

Response surface methodology was applied for determining the optimal percentage of amylopectin potato starch added to attribute high thermo-stability to fruit filling's composition. There were obtained two regression equations in terms of actual factors describing the influence of soluble solids and amylopectin starch content on fruit filling's heat-stability and viscosity. The adequacy of these equations performed by the F-test for analysis of variance using statistical package STATISTICA v.6 has shown that all two models were statistically significant. According to the present study it was also revealed that amylopectin potato starch would be more advantageous try to use in combination with another stabilizer (by finding their common synergetic effect) for fruit fillings in order to reduce its big doses that lead to highly viscous compositions, while maintaining high heat-stable, sensory and textural characteristics.

REFERENCES

Box, G., Hunter, J., Hunter, W. (2005). Statistics for Experimenters: Design, Innovation, and Discovery. John Wiley & Sons, New York.

Goupy, J., Creighton, L. (2007). Introduction to Design of Experiments with JMP Examples. SAS Institute Inc., Cary, NC, USA.

Herbstreith & Fox KG. Recipe. Product. Pectins in Fruit Preparations for Bakery Products: http://www.herbstreith-fox.de

Peleg, M. (2006). On fundamental issues in texture evaluation and texturization – A view. Food Hydrocolloids, 20, 405-414.

Schrieber, R., Gareis, H. (2007). Gelatine handbook. Wiley-VCH Verlag, Weinheim, Germany.

Szczesniak, A. (2002). Texture is a sensory property. Food Quality and Preference, 13, 215-225.

Codex Stan 296-2009 for Jams, Jellies and Marmalades: http://www.codexalimentarius.org/

Received: 17.02.2013.. Accepted: 25.03.2013.