

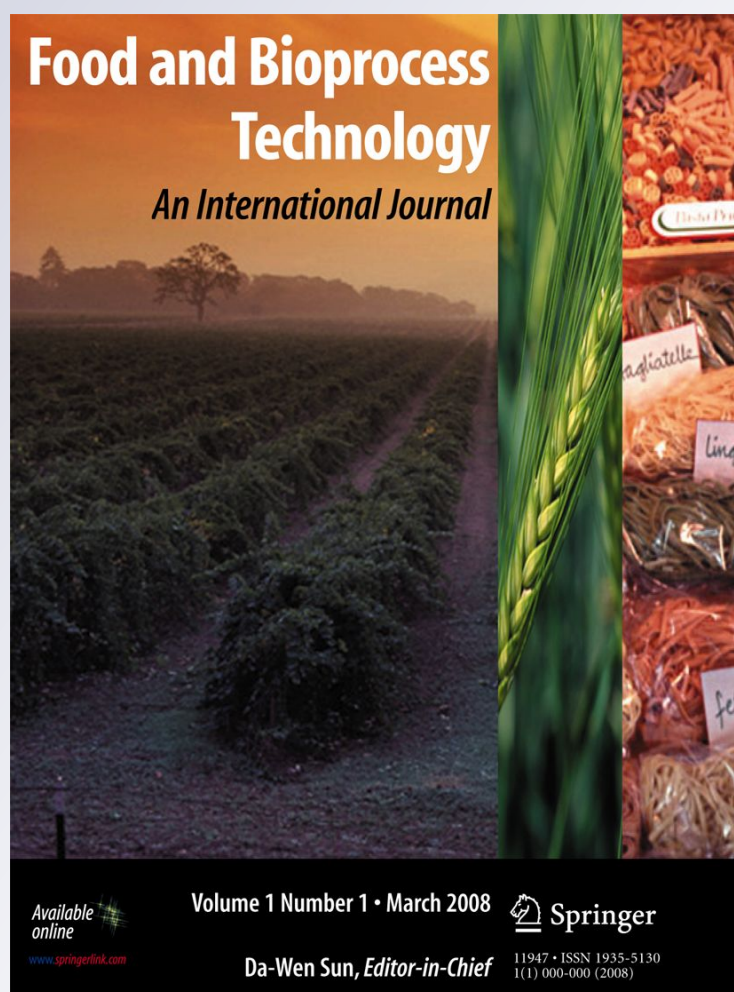
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Optimization of Basic Ingredient Combination for Sandwich Cookie Filling Using Response Surface Methodology

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Abstract An optimum formulation for a cookie sweet cream filling was selected using response surface methodology (RSM). The formulation included corn starch, powdered sugar, unflavored gelatin, cocoa butter, and distilled water. The effects of the amount of starch (0–30 %), sugar (40–70 %), gelatin solution (5/55 g water) (0–12 %), and cocoa butter (18–30 %) on the water activity and the textural characteristics (firmness, elasticity, relaxation time, adhesiveness, and cohesiveness) of cookie fillings were investigated. Significant regression models, which explained the effects of different percentages of these ingredients on all response variables, were determined. Based on the results, the formulation for the production of a cookie filling with acceptable water activity value and desired texture qualities was obtained by incorporating 23.16 % corn starch, 46.84 % powdered sugar, 0.87 % gelatin solution, and 29.13 % cocoa butter.

Keywords Cookie filling · Water activity · Response surface methodology · Composite food

Introduction

The increasing consumer demand for good nutritional ready-to-eat foods with long shelf lives has initiated the development of numerous composite foods associating a dry crispy compartment with a soft filling, e.g., filled cookies.

Moisture migration is a common problem in many composite food systems where components of high/low water activity (a_w) are adjacent (Guillard et al. 2002). Water diffuses from the “wet” phase to the “dry” phase leading to irreversible changes in the organoleptic and microbiological quality of the composite food and to a reduction of its shelf life (Labuza and Hyman 1998; Le Meste et al. 2002).

The concept of water activity introduced by Scott (1957) is the most useful expression of the water availability for microbial growth (Lacey and Magan 1991) and enzyme activity (Acker 1963). It is recommended to prevent the migration of water to maintain the water activity of the filling to be less than or equal to the casing material.

Cookies are hygroscopic, and when they take up moisture, they expand, and separation may occur at the cookie cream interface. The moisture diffusion is affected by the fat content of food products. Fat, dispersed into the food material, hinders the migration of water in the solid matrix of the product (Roca et al. 2006).

In sweet creams, the major ingredients are sugar and fat. The sugar should be in a powdered form with few, if any, large crystals. The smaller the crystal size, the more quickly the sugar dissolves in the mouth. However, there must be a balance as the finer the particle size, the more fat is required to give a desired consistency for cream sandwiching (Manley 2000a). Other ingredients such as starches, water, and gelling agents are commonly found in filling recipes. Corn, potato, and rice starches have been used as fillers and cream “dryers,” and a small addition of water is made when a considerable

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increase in consistency is desired (Manley 2000b). According to Manley (2000a), the use of fats with steep melting curves along with gelling agents helps the cream filling in the sandwich cookie to be firm at ambient temperature. This is from the point of view of maintaining the sandwich cookie in the shape that is desired and so that as the cookie is broken or bitten, the cream does not squeeze out. As the cookie is chewed, the fat in the cream should melt rapidly to release the sugar and other ingredients. Therefore, it would be desirable to develop a cookie filling with a low water activity and appropriate textural properties to be manipulated both in manufacture and during end use.

The effectiveness of response surface methodology (RSM) in optimization of ingredient levels, formulations, and processing conditions in food technology from raw to final products such as snack food (Thakur and Saxena 2000), biscuit dough (Gallagher et al. 2003), and traditional baked cassava cake (Gan et al. 2007) have been documented by different researchers. RSM consists of a group of mathematical and statistical procedures that can be used to study the relationships between one or more responses (dependent variables) and factors (independent variables) (Murphy et al. 2003).

The objective of this study was to investigate the effects of the main natural ingredients on the water activity values and the textural characteristics of a sandwich cookie filling, and to optimize the formulations of the filling in order to obtain a product with similar characteristics to commercial ones by using RSM.

Materials and Methods

Materials

Ingredients used for the filling preparation were food grade and/or GRAS. The cream formulation included powdered sugar (10×, Dos Anclas®, Argentina), unflavored gelatin (Royal®, Kraft Foods Inc., Brazil), corn starch (Maizena Duryea®, Unilever de Argentina S.A.), cocoa butter (Parafarm®, Saporiti S.A.C.I.F.I.A., Argentina), and distilled water. Samples of five commercial cookie fillings were analyzed to establish reference values for the evaluated parameters. The commercial fillings used corresponded to different types according to Manley (2005), and their composition is mainly based on sugar, starches, gelling agents, and steep melting curve fats as cocoa butter, lauric fats, or blends made to match the physical characteristics of these.

Cookie Filling Manufacture

All equipments used to prepare the fillings were made of stainless steel and were sanitized according to commonly accepted procedures used in the food industry. All

ingredients were weighed separately. Cocoa butter was melted in a container immersed in a 37 °C thermostatic bath (Haake L, Haake Buchler instruments, Karlsruhe, Germany). Powdered sugar and corn starch were manually mixed in a bowl to constitute the dry mix. Liquid ingredients such as gelatin solution (5/55 g water) and melted cocoa butter were mixed together (wet mix) and added to the dry mix, followed by manual mixing with a spatula until a cream was formed. During the entire procedure, the temperature remained 5 °C above the melting temperature of cocoa butter (MP=32 °C) (Ghotra et al. 2002) to avoid the solidification or crystallization of oils. The samples were stored in glass jars of 150 ml at room temperature (25±2 °C).

Experimental Design

An RSM was used to determine the experimental design and the optimal ingredient levels in formulating cookie filling. A Central Composite Rotatable Design (CCRD) was constructed using the SYSTAT software (SYSTAT, Inc., Evanston, IL) and requires an experiment number according to $N = k^2 + 2k + cp$, where k is the factor number and cp is the replicate number of the central point. Thus, the design included 11 experiments and is adopted by adding three central points and four axial points to 2^2 full factorial design. The center runs provide a means for estimating the experimental error and a measure of lack of fit. The axial points were added to the factorial design to provide for estimation of curvature of the model.

The factors studied were the dry mix composition (corn starch, 0–30 % of filling, and powdered sugar, 40–70 % of filling) (x_1) and the wet mix composition (gelatin solution, 0–12 %, and cocoa butter, 18–30 %) (x_2). In all cases, the ratio between liquid (gelatin solution and melted cocoa butter) and solid (starch and powdered sugar) ingredients was 7:3. The formulations for filling cream samples and coded levels of the CCRD corresponding to the dry mix and wet mix are given in Table 1.

The responses measured were adhesiveness (y_1), cohesiveness (y_2), firmness (y_3), elasticity (y_4), relaxation time (y_5), and water activity (y_6). Since the exact nature of the true function(s) between each response and the input factors is either unknown or too complex, these functions were approximated by second-order polynomials (Floros and Chinnan 1988):

$$y_n = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 \quad (1)$$

where β_0 is the value of the fixed response (y_n ; $n=1, 2, 3, 4, 5, 6$) at the central point of the experiment, that is the point (0,0); β_1 and β_2 are the linear regression terms, β_{11} and β_{22} are the quadratic regression terms, and β_{12} is the interaction regression term.

Table 1 Cream-filling formulations and coded values of the central composite rotatable design arrangement

Trial	Coded levels x_1	Dry mix		Coded levels x_2	Wet mix	
		Corn starch ^a	Powdered sugar ^a		Gelatin solution (5/55 g) ^a	Cocoa butter ^a
1	-1.0000	4.40	65.60	-1.0000	1.76	28.24
2	-1.0000	4.40	65.60	1.0000	10.24	19.76
3	1.0000	25.60	44.40	-1.0000	1.76	28.24
4	1.0000	25.60	44.40	1.0000	10.24	19.76
5	-1.4142	0.00	70.00	0.0000	6.00	24.00
6	1.4142	30.00	40.00	0.0000	6.00	24.00
7	0.0000	15.00	55.00	-1.4142	0.00	30.00
8	0.0000	15.00	55.00	1.4142	12.00	18.00
9	0.0000	15.00	55.00	0.0000	6.00	24.00
10	0.0000	15.00	55.00	0.0000	6.00	24.00
11	0.0000	15.00	55.00	0.0000	6.00	24.00

^aData in percent w/w based on filling weight

Determination of Water Activity

Water activity (a_w) of both commercial and experimental filling samples was determined in duplicate using a water activity measurement device, AquaLab Serie 3 (Decagon Devices, Inc., Pullman, WA), at 25 °C.

Texture Measurement

Textural analysis was performed on the cylinder-shaped filling by introducing the filling cream into a 22×60-mm glass tube and cutting the molded sample in sections 20 mm in length. The equipment used was a TA.XT2i Texture Analyzer (Stable Micro Systems Ltd., England) in the compression mode, interfaced with a computer using the software supplied by Texture Technologies Corp. Compression was exerted by an aluminum cylindrical probe with a flat section (75 mm in diameter) at a displacement speed of 2 mm/s. Filling firmness was defined as the force F_0 (in newtons) measured at 20 % (4 mm) compression. This compression was maintained for 20 min, and the force F_{20} exerted on the probe was measured. Filling elasticity was calculated as F_{20}/F_0 , and relaxation time τ (in minutes) was taken as the time at which $F = (F_0 + F_{20})/2$ (Lupano et al. 1992; Peleg 1979).

Texture profile analysis (Bourne 1978) was performed on filling samples with two compression cycles at 20 % compression. The displacement speed was 2 mm/s, and the time elapsed between two cycles was 5 s. Filling adhesiveness (J) was calculated as the negative force area obtained after the first compression cycle, representing the work necessary to pull the compressing plunger away from the sample. Filling cohesiveness was calculated as the ratio of the positive force area during the second compression to that during the first compression ($A2/A1$) (Bourne 1978). For each combination

and for the commercial fillings, the average of four determinations was calculated.

Optimization of the Filling Formulations

Maximization, minimization, and the search for the target value of the polynomials thus fitted were performed by the desirability function method, and mapping of the fitted responses was achieved using the SYSTAT software (SYSTAT, Inc., Evanston, IL).

During a process of optimization, usually several response variables are to be optimized. Some of these variables are to be maximized, some are to be minimized, and others must achieve a certain value. In many cases, these responses are competing, and improving one response may have an opposite effect on another one. If the optimal values for each response are localized in different regions, it will be more difficult to find the conditions that simultaneously satisfy all responses. This problem could be solved through the use of a desirability function (Murphy et al. 2005), a multicriteria methodology applicable when various responses have to be considered at the same time and it is necessary to find optimal compromises between the total numbers of responses taken into account.

This methodology is initially based on constructing a desirability function for each individual response ($d_n=f(y_n)$). Through the individual functions, the specifications that each response must fulfill can be introduced. The scale of the individual desirability function ranges between $d_n=0$, for a completely undesirable response, and $d_n=1$, for a fully desired response, above which further improvements would have no importance. The method finds the combination of factors that provides the most desirable response values.

Depending on whether a particular response (y_n) is to be maximized, minimized, or must attain a particular value,

different desirability functions can be used (Derringer and Suich 1980). Let $y_{n,\min}$, $y_{n,\max}$, and $y_{n,\text{tar}}$ be the lower, upper, and target values, respectively, that are desired for the response y_n , with $y_{n,\min}$, $y_{n,\max}$, and $y_{n,\text{tar}}$. Three desirability functions were defined to calculate the maximum (Eq. 2), minimum (Eq. 3), and the target value (Eq. 4) as optimum formulations.

$$d_n = \begin{cases} 0 & ; \quad y_{n,\min} > y_n \\ \frac{(y_n - y_{n,\min})}{(y_{n,\max} - y_{n,\min})} & ; \quad y_{n,\min} \leq y_n \leq y_{n,\max} \\ 1 & ; \quad y_n > y_{n,\max} \end{cases} \quad (2)$$

$$d_n = \begin{cases} 1 & ; \quad y_{n,\min} > y_n \\ \frac{(y_n - y_{n,\max})}{(y_{n,\min} - y_{n,\max})} & ; \quad y_{n,\min} \leq y_n \leq y_{n,\max} \\ 0 & ; \quad y_n > y_{n,\max} \end{cases} \quad (3)$$

$$d_n = \begin{cases} 0 & ; \quad y_{n,\min} > y_n \\ \frac{(y_n - y_{n,\min})}{(y_{n,\text{tar}} - y_{n,\min})} & ; \quad y_{n,\min} \leq y_n \leq y_{n,\text{tar}} \\ \frac{(y_n - y_{n,\max})}{(y_{n,\text{tar}} - y_{n,\max})} & ; \quad y_{n,\text{tar}} \leq y_n \leq y_{n,\max} \end{cases} \quad (4)$$

In general, the desirability of each response must be higher than zero as a necessary condition to establish the global system desirability (D). The global desirability was calculated as the geometric mean of the individual desirability values for each response variable (Derringer and Suich 1980; Harrington 1965). The final global desirability is then computed as follows:

$$D = (d_1^{v_1} \cdot d_2^{v_2} \cdot d_3^{v_3} \dots d_k^{v_k})^{1/\sum v_n} = \left(\prod_{n=1}^k d_n^{v_n} \right)^{1/\sum v_n} \quad (5)$$

where v_n is a number indicating the relative importance of the n th response, which might typically be an integer in the range of 1–5, with 5 indicating the greatest importance and 1 indicating the least. Thus, the simultaneous optimization process is reduced to find the levels of factors that demonstrate the maximum overall desirability.

Statistical Analysis

An analysis of variance (ANOVA) was conducted separately on the dependent variables studied considering each formulation as a level in a one-way factorial design. For simultaneous pairwise comparisons, least significant differences or Fisher test was chosen. Significance was judged by determining the probability level that the F statistic calculated from the data is less than 5 %. All statistical procedures were computed using the SYSTAT software (SYSTAT, Inc., Evanston, IL). Experimental data were reported as mean values.

Results and Discussion

Commercial Cookie Filling Properties

The water activity and textural attributes such as firmness, elasticity, relaxation time, adhesiveness, and cohesiveness for five commercial samples of cookie fillings are shown in Table 2. The range of values obtained was taken as reference values in the desirability functions to estimate the optimal filling formulation.

Model Fitting from RSM

Experimental results for the response variables of cookie fillings are shown in Table 3. The independent and dependent variables were fitted to the second-order model equation and examined for goodness of fit. The analysis of variance was performed to determine the lack of fit and the significance of the linear, quadratic, and interaction effects of the independent variables on each response (Table 4). The ANOVA showed that lack of fit was not significant for all the response surface models at 95 % confidence level. The lack of fit test is a measure of the failure of a model to represent data in the experimental domain at which points were not included in the regression (Varnalis et al. 2004). On the other hand, R^2 and adj- R^2 were calculated to check the model adequacy. A high proportion of variability ($R^2 > 0.96$) in the response models was obtained (Table 4). However, a large value of R^2 does not always imply that the regression model is a good one. Adding a variable to the model will always increase R^2 , regardless of whether the additional variable is statistically significant or not. Thus, it is preferred to use an adj- R^2 to evaluate the model adequacy, and it should be over 90 %. Table 4 shows that R^2 and adj- R^2 values for the models did not differ dramatically indicating non-significant terms have not been included in the model. The regression equation coefficients of the proposed models for each response are given in Table 5.

Effect of the Dry Mixture and Wet Mixture Composition

The effect of different amounts of dry (corn starch and powdered sugar) and wet (gelatin solution and melted cocoa butter) ingredients on the a_w value and textural attributes (adhesiveness, cohesiveness, firmness, elasticity, and relaxation time) of cookie fillings are reported by the coefficients of the second-order polynomials (Table 5). To aid visualization, the three-dimensional plots of the response surfaces are shown in Figs. 1, 2, 3, 4, 5, and 6. From Figs. 1, 2, 3, 4, 5, and 6, it is clear that water activity and textural attributes were affected by the amount of all ingredients.

Table 5 showed that a_w was affected by the composition of the wet mix used, with positive linear and negative quadratic effects at $P \leq 0.001$ and 0.05, respectively. These

Table 2 Averages and standard deviation results for adhesiveness, cohesiveness, firmness, elasticity, relaxation time, and water activity (a_w) of commercial samples of cookie fillings

Filling type ^a	Bourbon	Mint	Custard	Lemon puff	Chocolate
Adhesiveness (N·m)	0.369±0.009a	0.061±0.002d	0.211±0.010b	0.009±0.003c	0.050±0.006d
Cohesiveness	1.035±0.019d	1.440±0.023b	1.283±0.008c	1.544±0.034a	1.237±0.014c
Firmness (N)	116.047±0.407a	96.531±0.509b	119.611±0.321a	73.213±0.335d	90.853±0.029c
Elasticity	0.187±0.001c	0.178±0.005d	0.184±0.003d	0.309±0.009a	0.273±0.013b
Relaxation time (min)	1.220±0.047c	1.260±0.050c	1.460±0.094b	1.720±0.036a	1.864±0.057a
a_w (25 °C)	0.390±0.002d	0.407±0.007c	0.451±0.000b	0.415±0.001c	0.476±0.003a

Different letters within the same row indicate significant differences ($P<0.05$)

^a According to Manley (2005)

results indicated that a_w increased continuously as the gelatin solution/cocoa butter ratio was increased (Fig. 1 and Table 1). That is, a_w increased when the water solution increased, but the rate of increment was lower at higher levels of wet mixture composition. On the other hand, the linear effect of dry mix composition on a_w values was negative at $P\leq 0.05$, and it is observed that for a given wet mix composition, the fillings enriched by sugar always presented higher a_w values than starchy fillings.

The linear effects of dry and wet mix composition on adhesiveness were positive at $P\leq 0.05$ and negative at $P\leq 0.001$, respectively, and the response variable had a curvilinear pattern owing to a significant negative interaction term ($P\leq 0.01$). Thus, the adhesiveness depends mainly on the wet mix composition and on the interactions between the dry and wet mixture composition. Adhesiveness represents the work necessary to overcome the attractive forces between the sample surface and the surface of other materials (Bourne 2002), as the plunger probe. Adhesiveness allows the cookie–cream

interface to key into the cookie shells when a sandwich cookie is made (Manley 2005). The wet mix composition did not affect the adhesiveness of the filling without starch and with the higher sugar content (Fig. 2). On the other hand, the adhesiveness increased with starch content in fillings with higher cocoa butter content, but decreased with starch content in fillings with higher gelatin solution content. The behavior observed in Fig. 2 could be attributed to the ability of gelatin to form gel structures, trapping starch and sucrose molecules inside them and preventing them from adhering to the surface of the probe. Be warned that if the fat content of the cream is too low, there may be a problem with splitting of the cream from the biscuit shells. This is because there are not enough fat crystals at the biscuit–cream interface to key into the biscuit shell when the fat was cooled (Manley 2005).

The variation in the proportion of ingredients in the dry and wet mixtures showed similar effects on the cohesiveness and elasticity (Table 5). Both response variables showed a linear

Table 3 Averages and standard deviation of the experimental results for the response variables of cookie fillings

Trial	Responses					
	Adhesiveness (N·m)	Cohesiveness	Firmness (N)	Elasticity	Relaxation time (min)	a_w (25 °C)
1	0.062±0.002b	1.075±0.003c	35.876±1.052e	0.147±0.005d	0.747±0.025bc	0.740±0.031cd
2	0.032±0.003d	0.615±0.009g	13.186±0.698g	0.072±0.009f	0.215±0.006g	0.903±0.027a
3	0.120±0.004a	1.315±0.017a	175.768±0.677b	0.268±0.010a	1.647±0.030a	0.686±0.040d
4	0.021±0.004e	1.045±0.002cd	107.197±0.572c	0.189±0.009c	0.725±0.039cd	0.835±0.022b
5	0.050±0.004c	0.820±0.009f	11.500±0.714gh	0.089±0.009ef	0.460±0.035f	0.834±0.006b
6	0.062±0.002b	1.205±0.009b	200.020±3.217a	0.276±0.017a	1.140±0.035b	0.748±0.006c
7	0.121±0.004a	1.090±0.000c	84.000±1.475d	0.235±0.015b	1.131±0.030b	0.600±0.004e
8	0.008±0.001f	0.630±0.023g	9.188±0.683h	0.105±0.004e	0.303±0.005fg	0.902±0.007a
9	0.064±0.002b	0.985±0.037de	35.000±0.726ef	0.174±0.005cd	0.573±0.025e	0.841±0.002b
10	0.060±0.004b	0.957±0.037e	32.000±1.681f	0.172±0.004cd	0.580±0.019e	0.820±0.014b
11	0.062±0.004b	0.983±0.040de	37.000±0.774e	0.176±0.007c	0.630±0.052de	0.850±0.011ab

Different letters within the same column indicate significant differences ($P<0.05$)

Table 4 ANOVA showing the linear, quadratic, interaction, and the lack of fit of the response variables

Source of variation	Df	Response variables											
		Adhesiveness (N·m)		Cohesiveness		Firmness (N)		Elasticity		Relaxation time (min)		a_w (25 °C)	
		Sss	F	Sss	F	Sss	F	Sss	F	Sss	F	Sss	F
Regression	5	0.012	49.125*	0.445	30.574*	19.674	16.608*	0.046	147.390*	1.698	35.122*	0.084	29.030*
Linear	2	0.011	109.923*	0.423	71.011*	18.871	33.811*	0.046	367.158*	1.263	80.900*	0.076	65.279*
Square	2	0.000	0.914 ns	0.023	3.909 ns	35.181	74.265*	0.000	1.286 ns	0.101	4.942 ns	0.008	7.251***
Interaction	1	0.001	23.949**	0.009	3.033 ns	26.267	9.889***	0.000	0.064 ns	0.177	3.925 ns	0.000	0.091 ns
Residual error	5	0.000		0.015		53.217		0.000		0.010		0.001	
Lack of fit	3	0.000	20.042 ns	0.014	19.658 ns	84.473	13.338 ns	0.000	19.252 ns	0.046	16.014 ns	0.002	3.448 ns
Pure error	2	0.000		0.000		6.333		0.000		0.002		0.000	
Total error	10	0.012		0.470		44.456		0.046		1.747		0.087	
R^2 (%)		98.0		96.8		99.4		99.3		97.2		96.7	
R^2 adj (%)		96.0		93.7		98.8		98.9		94.5		93.3	

* significant at $P \leq 0.001$; ** significant at $P \leq 0.01$; *** significant at $P \leq 0.05$

ns not significant, Sss sequential sum of squares, Df degree of freedom, F ratio of variance estimates

dependence with the dry and wet mix compositions (Figs. 3 and 4). The cohesiveness and elasticity increased with starch content and with cocoa butter content (Table 1). As cohesiveness is a direct function of the work needed to overcome internal bonds of the material (Steffe 1996) according to these results, the starch and the cocoa butter would be the main components that maintain the structure of the filling. The lowest values of elasticity and cohesiveness were obtained at higher sugar contents, suggesting that the sugar molecules may interfere with the formation of a filling structure.

The regression model (Table 5) showed that the composition of the dry mixture had positive linear ($P \leq 0.001$) and quadratic ($P \leq 0.05$) effects on the filling firmness and relaxation time. It can be observed from Figs. 5 and 6 that both response variables were significantly increased at larger dry mix composition coded values, which correspond to higher starch and lower powdered sugar concentration, according to Table 1. The curve in Fig. 6 showed that the relaxation time of the samples increases rapidly

with the decrease in the amount of gelatin solution and the increase in the amount of cocoa butter in the formulation. Otherwise, a negative linear effect ($P \leq 0.001$) of the wet mix composition on the filling firmness was found, based on which higher amounts of gelatin solution and lower amounts of cocoa butter cause the decrease in firmness. Results also indicated that the interaction term on firmness ($P \leq 0.05$) was negative; thus, the firmness was dependent on both of these factors.

The starch and, in a lower degree, the cocoa butter increased the firmness of the filling and its relaxation time. The relaxation time is related with the viscous behavior of the samples; that is, higher relaxation times are associated with solid-like behaviors.

Optimization of the Cookie Filling Formulation

The global system desirability value (Eq. 5) would be as higher as optimal the combination of the different

Table 5 Estimated regression coefficients of the fitted second-order polynomial for the response variable

Trial	Estimated coefficients					
	Adhesiveness (N·m)	Cohesiveness	Firmness (N)	Elasticity	Relaxation time (min)	a_w (25 °C)
β_0	0.062*	0.975*	34.666*	0.174*	0.594*	0.837*
β_1	0.008**	0.152*	62.564*	0.063*	0.296*	-0.030**
β_2	-0.036*	-0.173*	-24.633*	-0.042*	-0.328*	0.092*
β_{11}	-0.003 ns	0.038 ns	37.254*	0.002 ns	0.122*	-0.018 ns
β_{22}	0.001 ns	-0.038 ns	7.672 ns	-0.004 ns	0.080 ns	-0.038**
β_{12}	-0.017***	0.047 ns	-11.470**	0.000 ns	-0.097 ns	-0.004 ns

*significant at $P \leq 0.001$; ** significant at $P \leq 0.05$; ***significant at $P \leq 0.01$

ns not significant

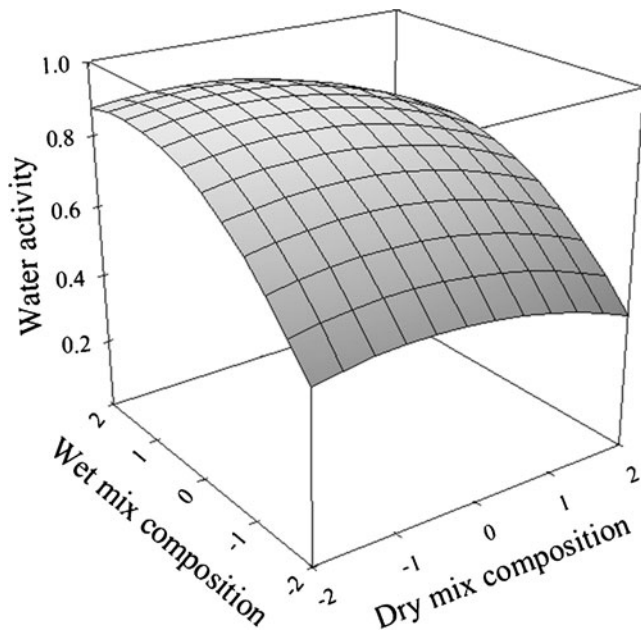


Fig. 1 Response surface plot of the effects of the wet and dry mix composition on the water activity of cookie filling

performance could be. Criteria for optimization and the prediction values of response variables are shown in Table 6.

It was found that optimal ingredient levels were obtained by incorporating 23.16 % of corn starch, 46.84 % of powdered sugar, 0.87 % of gelatin solution (5/55 g water), and 29.13 % of cocoa butter (in percent based on filling weight). Global desirability (D) obtained was of 0.702 (Eq. 5).

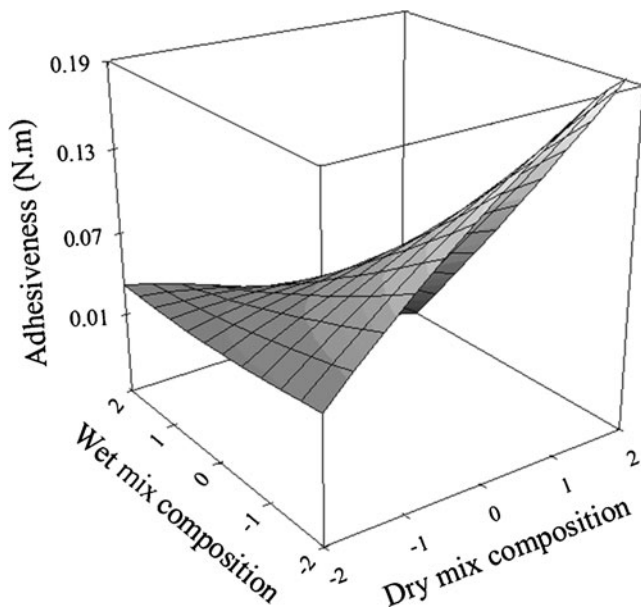


Fig. 2 Response surface plot of the effects of the wet and dry mix composition on the adhesiveness of cookie filling

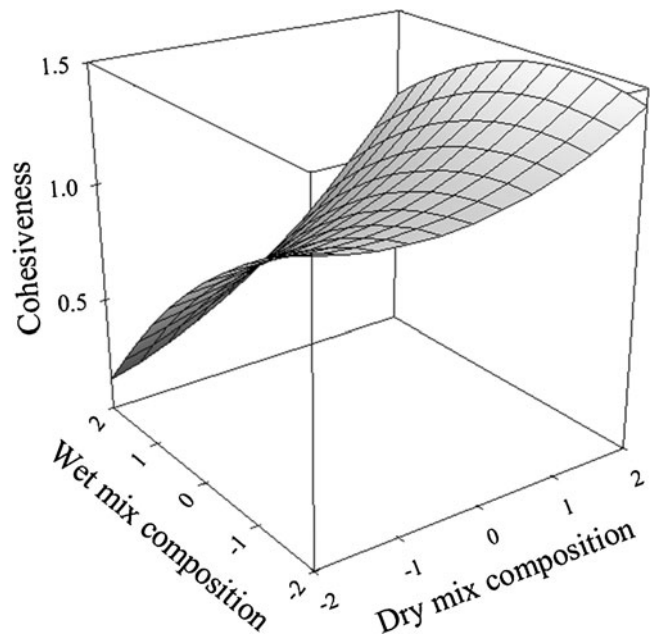


Fig. 3 Response surface plot of the effects of the wet and dry mix composition on the cohesiveness of cookie filling

Desirability functions were developed for the following criteria: minimum water activity and firmness, maximum adhesiveness, and cohesiveness, elasticity, and relaxation time values similar to those found in commercial cookie fillings. The lower and upper values in the desirability function (Eq. 4) for cohesiveness, elasticity, and relaxation time were established as the lowest and highest values obtained for those responses in commercial fillings (Table 2), while the average value for each response was considered as the target value (cohesiveness,

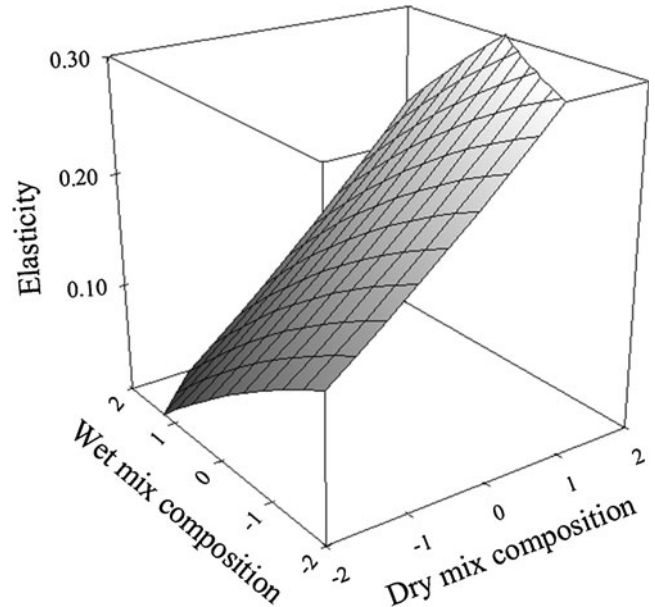


Fig. 4 Response surface plot of the effects of the wet and dry mix composition on the elasticity of cookie filling

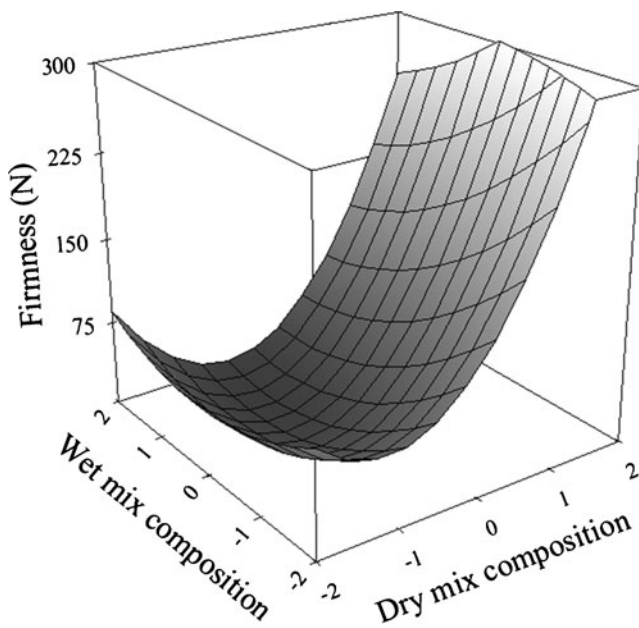


Fig. 5 Response surface plot of the effects of the wet and dry mix composition on the firmness of cookie filling

1.300 ± 0.087 ; elasticity, 0.260 ± 0.027 ; and relaxation time, 1.505 ± 0.126 min). The relaxation time is related to the viscous behavior and therefore related to the spreadability of the filling.

Since filling adhesiveness is one of the main parameters to achieve satisfactory keying of the cream filling to the shells of filled sandwich cookies (Manley 2000b), it was sought to maximize adhesiveness by establishing the average value for commercial filling adhesiveness (0.140 ± 0.067 N·m) as the target value in the desirability function (Eq. 2).

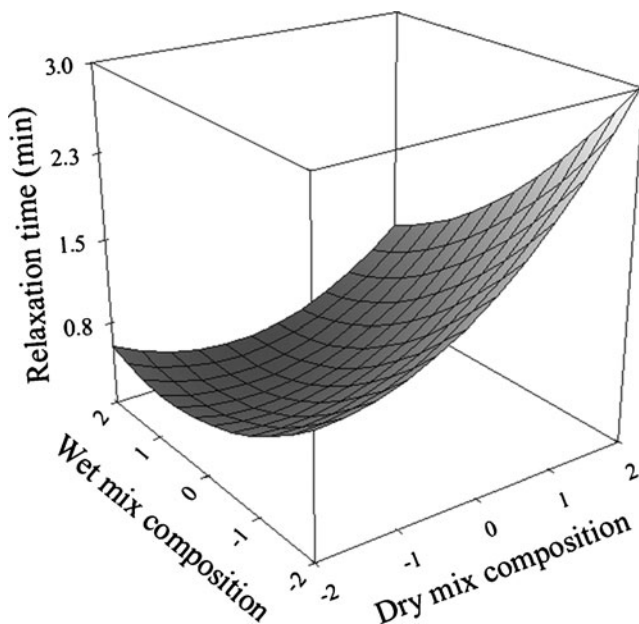


Fig. 6 Response surface plot of the effects of the wet and dry mix composition on the relaxation time of cookie filling

Table 6 Parameters for the analysis of the desirability function (d_n)

	Minimum value	Target value	Maximum value	Predicted value	(d_n)
Adhesiveness	0.099	0.140	–	0.127	0.687
Cohesiveness	1.100	1.300	1.500	1.224	0.622
Firmness	–	155.000	160.000	156.500	0.707
Elasticity	0.180	0.260	0.309	0.278	0.630
Relaxation time	1.200	1.505	1.900	1.497	0.991
Water activity	–	0.550	0.690	0.630	0.632

No maximum value is required to be specified for (Eq. 2), since if the predicted value exceeds the target value, a fully desired response would be achieved ($d_n=1$).

An optimum formulation of the cookie filling is presented as a filling with a low water activity and a low firmness, simultaneously, *inter alia*.

According to the proposed model for water activity (Table 5), the lowest a_w value achievable within the range of concentrations set out is 0.550. This value was used as target value for the minimization of water activity (Eq. 3), and if the predicted value is lower than the target value, the desirability function reaches its highest value.

Due to the opposite behavior among the water activity and the firmness of elaborated fillings, the optimal values for each response are localized in different regions. Consequently, in order to find the conditions that simultaneously satisfy all responses, a higher target value (155 N) in the desirability function for firmness (Eq. 3) was accepted.

A low a_w filling is desirable because the control of the initial moisture content and moisture migration is critical to the quality and safety of a multi-domain system (Labuza and Hyman 1998) such as filled sandwich cookies. In Table 6, it has been set that the optimal formulation of the filling has a predicted value for firmness of 156.5 N, a value significantly higher than those reported for commercial samples in Table 2. This optimum formulation was acceptable since the increase in gelatin solution (5/55 g water) concentration, besides lowering the filling firmness, causes the undesirable effect of increasing the water activity. The a_w cannot be offset by the addition of starch because although starch has a negative linear effect on a_w , it also has positive linear and quadratic effects on the filling firmness (Table 5). Due to the opposite behavior of these two response variables, the predicted value for firmness derived from the analysis of the desirability function was accepted in favor to maintain a low a_w .

The fat and vegetable oil blends commonly used in commercial fillings present lower melting points than cocoa butter (O'Brien 2004), because the cocoa butter melting curve is very sharp due principally to the low spread of triglycerides naturally present. A factor which affects the firmness of a fat-based product is the degree of plasticity of

the fat. Fats which have been mechanically agitated as they cool have small free crystals and are said to be plasticized. Fats which cool passively from liquid are much firmer at ambient temperature because the crystals have grown together in an interlocked form (Manley 2005). In addition, the higher the fat content, the firmer will be the cream under these conditions. This could explain the higher firmness of the elaborated fillings observed over the commercial samples.

Conclusions

RSM was a useful tool to model the influence of basic ingredients such as corn starch, powdered sugar, gelatin solution, and cocoa butter on cookie filling properties. The results also suggested that by modifying the proportion of these ingredients, a large range of variation may be obtained. Based on the desirability function execution, a basic formulation similar to those presented by commercial cookie sweet cream fillings could be obtained by the incorporation of 23.16 % corn starch, 46.84 % powdered sugar, 0.87 % gelatin solution (5/55 g water), and 29.13 % cocoa butter.

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