Commercial Spread Optimization Using Experimental Design and Sensory Data

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ABSTRACT: The cost and quality of food products are issues that concern both the consumer and producer. In this research, the process used for the production of a commercial spread was subjected to a statistical experimental design for the purpose of reducing the cost of production while maintaining or improving the sensory quality. Three factors—the amount of oil added (x_1) , the speed of puddling (x_2) , and the temperature treatment (A or B; x_3)—were varied according to a full-factorial design at two levels. The experiments were performed over 2 d, and the factorial design was complemented with three replicates for temperature treatments A and B, which were performed on different days. The products were evaluated with both sensory and physicochemical measurements. Special attention was paid to the hardness of the product since it was permissible to reduce it slightly. In contrast, sensory quality aspects of the product including butter-aroma and off-flavor, as well as other quality properties such as spreadability, shine, and meltability, had to be maintained at the present level or improved. Statistical evaluation of the data showed that it was possible to add high amounts of oil (x_1) without impairing the sensory quality of the product and, hence, reduce the cost of production. The hardness of the product was also slightly reduced when using the high level of oil. In maintaining other sensory qualities such as shine and spreadability at the present levels, the choice of temperature treatment (x_3) was important.

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KEY WORDS: PLS regression, product and process optimization, production cost, sensory evaluation, statistical experimental design.

Both the increasingly intense competition faced by new products and changes in consumer demands have altered the market for spreads and cooking fats. These changes have made it necessary to lower production costs while maintaining or improving the sensory qualities of the products. To improve the quality of an existing product, ingredients and factors in the manufacturing process that affect the outcome of the process must be identified. Clearly, the relevant information for problem solving must be of high quality and reliability, as discussed by Martens and Martens (1), and to make correct decisions, the quality of the input data is important, as well as the tools that are used for interpreting data. Traditional methods for analyzing processes, such as changing a single variable at a time, have limitations; over the years, the use of statistical experimental design (2) in combination with multivariate analysis has increased and proven to be a more efficient approach for product optimization. When using the appropriate statistical experimental design (1–5), the investigated region is thoroughly explored and the odds of finding relevant information are greatly improved. The variables under consideration are varied independently of each other, statistically ensuring that the effect of each variable can be separately measured and estimated by regression methods such as multiple linear regression (MLR) (6) or partial least square regression to latent structures (PLS) (7,8). However, product optimization is not limited to experimental planning and experimental design. The products made according to the experimental design must in some way be analyzed, and the most common way to do this for food products is to obtain a sensory evaluation of the products by a panel of assessors (9). It is important to ensure that the difference between samples, i.e., the treatment effect, is large enough to be detected by the sensory panel. This is best achieved by using statistical experimental design. The relationship between the investigated variables and the sensory quality of the product determined by sensory evaluation is then modeled by multivariate methods such as PLS or MLR. Another common way is to use ANOVA in combination with principal component analysis (10).

The present paper provides an example showing how a statistical experimental design was used in a spread-making process to determine how factors in the process influenced the quality of the final product, especially product sensory qualities. The main objectives were to reduce the cost of production and to investigate whether the sensory quality of the product could be improved by slightly reducing its hardness while maintaining or improving other sensory qualities, such as shine and spreadability.

EXPERIMENTAL PROCEDURES

Statistical experimental design. A full-factorial design for three factors—the amount of added oil (x_1) , the speed of puddling (x_2) , and temperature treatment of the raw material (A) or B; x_3)—was constructed using MODDE, Version 6.0, software (Umetrics Inc., Umeå, Sweden).

The type of oil, whether hydrogenated or vegetable, used for the production of different types of spreads usually affects sensory properties such as hardness more than the amount does. Hydrogenated vegetable oils, compared to vegetable oils, are known to be more stable and to have higher m.p.

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and the experimental nange for each variable								
Variable	Description	Low	High	Range	Type			
X_1	Amount of oil added, %	-1	$+1$	7%	Quantitative			
X_2	Treatment of final product, puddling	-1	$+1$	25 rpm	Quantitative			
X_2	Temperature treatment of raw material		B	Two different treatments, A and B	Qualitative			

TABLE 1 Description of the Factors Varied in the Experiment, the Type of Variable, and the Experimental Range for Each Variable

(11). As discussed by Schmidt *et al.* (12), blends of vegetable oils and fully hydrogenated vegetable oils in various ratios can be used to produce fats with different structures and physical properties, thus making them suitable for the production of different types of margarines. However, in the present study there was no possibility of changing the type of oil used for the production of this specific product. Because of this, the type of oil was not considered as a factor of interest.

In Table 1 the tested variables and their experimental ranges are presented. The specific levels of the *x*-variables are given here only as coded levels for commercial reasons.

The amount of oil used (x_1) , expressed as a percentage, is a quantitative variable that is varied over a range of 7% between the lowest and highest levels. The speed of puddling $(x₂)$ also is a quantitative variable and is varied over a range of 25 rpm. The temperature treatment of the raw material (x_3) is a qualitative variable set at one of two levels, A or B. For treatment A, the raw material is heated and then cooled in several stages over a long period of time. For treatment B no heating occurs; the raw material is directly cooled. Because of the qualitative nature of $x₃$, it is not possible to define an average of temperature treatments A and B.

In Table 2 the experimental design is presented. The resulting data were evaluated using PLS. Three replicates each for temperature treatments A and B, were made to estimate the experimental error.

In experiments involving an operational process, it is not always easy to control the levels of the variables precisely and, as seen in Table 2, some of the experiments did not follow the design exactly. This had two consequences: The actual range for x_1 was 10.4% instead of 7%, and the design was somewhat distorted. The skewness (distortion) of a design is measured by the condition number (7), which shows how close to orthogonal it is. The condition number, or index, is the ratio of the largest and smallest singular values of X (i.e., the eigenvalues of $X'X$), where X is the scaled and centered model matrix. A condition number of 1 indicates an orthogonal design. If there is collinearity between the variables in a design, the condition number of the design is large (>10). The model matrix presented here has a condition number of 1.83 when all responses are fitted in the same model, indicating that the model terms are almost orthogonal. A condition number of 1.83 is acceptable when using PLS or MLR for regression analysis. MLR and PLS give the same results as long as the X-variables are orthogonal, or close to orthogonal, and when only one response is regressed at a time.

Sensory evaluation and chemical composition of the samples. The main focus in this study was to reduce the cost of production. It was also of interest to investigate and evaluate how the experimental factors influenced the sensory properties of the products and how the hardness of the product would be reduced. To do so, standard procedures for sensory evaluation were used. The sensory evaluation of the products was performed in a room especially equipped for sensory analysis. The samples were coded and presented to the eight assessors in a balanced random order, one sample at a time. All 14 samples were evaluated during the same session. To describe the intensity of the different sensory attributes, the assessors were instructed to score the intensity of each attribute on a continuous scale from 0 to 10. Intensities close to 0 or 10 are too weak or too strong, respectively, and an optimal sample has an intensity close to 5 for each attribute, except for off-flavor where the optimal intensity is 0. The assessors were not aware that the optimal intensity was close to 5. Sensory responses are presented in Table 3.

To ensure that the products met the required standards, they were also chemically characterized. These results are

TABLE 2

^aThe first three columns show the original design and the three last columns the actual levels of the variables (in coded form) used. Experiments 9–11 are replicates for temperature treatment A, and experiments 12–14 are replicates for treatment B.

*^b*Original levels for the variables according to a full-factorial design at two levels.

c Actual levels of the variables for each experiment (in coded form) used in the experiments. For a description of variables see Table 1.

Exp. no.	b V_1 Hardness	y_2^b Spreadability	b Y3 Shine	y_4^b Meltability	y_5^b Butter-aroma	y_6^C Off-flavor			
1	5.68	5.60	3.57	5.25	5.67	0.00			
$\overline{2}$	4.55	4.97	4.62	4.88	5.62	0.13			
3	5.98	5.58	3.60	5.47	5.65	0.35			
4	4.42	4.67	4.32	4.85	5.52	0.02			
5	8.22	6.60	1.58	5.73	5.88	0.00			
6	6.13	5.83	0.85	5.33	5.70	0.03			
7	8.05	6.77	0.53	5.63	5.92	0.00			
8	6.42	5.62	0.85	5.23	5.77	0.02			
9	5.12	5.00	3.70	5.18	5.50	0.00			
10	5.48	5.18	4.23	5.10	5.58	0.05			
11	5.00	5.13	4.05	5.05	5.52	0.00			
12	7.32	6.22	1.00	5.48	5.62	0.02			
13	7.35	6.03	1.08	5.65	5.63	0.00			
14	6.80	6.03	1.63	5.45	5.85	0.02			

TABLE 3 Summary of Sensory Evaluation*^a*

^aSix sensory attributes were used to characterize the products. The intensity of each attribute was evaluated by the use of a continuous scale from 0 to 10 and in each case is presented as the average intensity for all assessors.

*b*For $y_1 - y_5$ (hardness, spreadability, shine, meltability, and butter-aroma), an intensity <5 represents too little and >5 too much, i.e., the optimal intensity is close to 5.

 c_{γ_6} : Off-flavor should not be present in the product and the optimal intensity is therefore 0.

presented in Table 4. According to the standards for this product, the fat content (y_8) must be at least 80% and the salt content (y_{10}) must be approximately 1.2% (\pm 0.1%). For the other chemical parameters including iodine number (y_7) , water content (y_0) , and fat content in buttermilk (y_{13}) , there are no restrictions. MV8 (y_{11}) and MV15 (y_{12}) are parameters describing the hardness of the product, as measured with an LFRA Texture Analyser (manufactured by CNS Farnell, Borehamwood, Hertfordshire, England, known formerly as the Stevens Texture Analyser), representing mean values of four or five measurements at 8 and 15°C, respectively.

Regression analysis. The similarities and differences of the

TABLE 4 Summary of Chemical Characterization

responses were simultaneously modeled by an interaction model according to Equation 1, using a partial least square regression to latent structures (PLS):

$$
y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{12} x_1 x_2 + b_{13} x_1 x_3
$$

+ $b_{23} x_2 x_3 + b_{123} x_1 x_2 x_3 + e$ [1]

PLS is used to construct bilinear models between the matrix of predictor variables, **X**, and the matrix of the dependent variables, the responses, **Y**, as follows: $Y = XB + E$. The regression model is then fit to provide information on how the variables in the **X**-matrix affect the responses in the **Y**-matrix, and

a The fat, water, and salt contents of the product were determined by methods SBR International Dairy Federation (IDF) 1C:1987, IDF 10:1960, and IDF 88A:1986, respectively.

*^b*Fat content in buttermilk was determined by method SBR IDF 1C:1987.

c Hardness was measured by an LFRA Texture Analyser (manufactured by CNS Farnell) at two different temperatures, 8 and 15°C. The average stress, measured in grams, derived from four or five measurements for each sample and temperature, is presented in the table as MV8 and MV15.

the regression coefficients, **B**, are estimated using Equation 2, so that the covariance between **X** and **Y** is maximized.

$$
\mathbf{B} = \mathbf{W}(\mathbf{P}'\mathbf{W})^{-1}\mathbf{C}'
$$
 [2]

In Equation 2, **W** represents the weight vector for the original **X**-factors, **P** the loading vector, and **C** the weights for the responses in the **Y**-matrix.

The number of PLS components used in the model is determined by cross-validation (13,14); to determine whether the model is well fitted to the data, the parameters R^2 and Q^2 are estimated. R^2 is a measure of the variation explained by the model, whereas Q^2 describes the predictive ability of the model. These two parameters are calculated according to Equations 3–5:

$$
R^2 = 1 - \frac{\text{SS}_{\text{RES}}}{\text{SS}_{\text{TOT.CORR}}} \tag{3}
$$

$$
Q^2 = 1 - \frac{\text{PRESS}}{\text{SS}_{\text{TOT.CORR}}} \tag{4}
$$

$$
PRESS = \sum (y_{obs} - y_{pred})^2
$$
 [6]

The term PRESS is the predicted error sum of squares determined by cross-validation. When using MODDE the term PRESS is calculated as

$$
PRESS = \sum \frac{(y_{obs} - y_{pred})^2}{(1 - h_i)}
$$
 [6]

where h_i is the *i*th diagonal element of the Hat matrix, $\mathbf{X}(\mathbf{X}'\mathbf{X})^{-1}\mathbf{X}'$.

In addition to R^2 and Q^2 , ANOVA is used. The total variation corrected for the mean, SS_{TOT.CORR}, is decomposed into one part due to the model, SS_{REG} , and the remainder, which is not explained by the model, i.e.*,* the residuals, into another part, SS_{RES} : $SS_{TOT,CORR} = SS_{REG} + SS_{RES}$.

An *F*-test is then applied to confirm whether the variance explained by the regression model is significantly larger than the variance of the residuals. For a significant model, the calculated F_{REG} should be larger than the tabulated F -value (this corresponds to a *P*-value smaller than the preset limit, usually $P = 0.05$),

$$
F_{\text{REG}} = \frac{V_{\text{REG}}}{V_{\text{RES}}} = \frac{\text{SS}_{\text{REG}}/\text{df}_{\text{REG}}}{\text{SS}_{\text{RES}}/\text{df}_{\text{RES}}} \tag{7}
$$

where df_{REG} and df_{RES} refer to the degrees of freedom associated with the regression model and the mean value, respectively. All regression models inevitably contain some degree of error, and the significance of this error, or lack of fit, also can be statistically determined by the use of an *F*-test if there are replicated observations. The residual sum of squares is then further partitioned into lack of fit (SS_{LoF}) and replicate error (SS_{PE}): $SS_{RES} = SS_{LoF} + SS_{PE}$, and the *F*-value is calculated:

$$
F_{\text{LoF}} = \frac{V_{\text{LoF}}}{V_{\text{PE}}} = \frac{SS_{\text{LoF}}/df_{\text{LoF}}}{SS_{\text{PE}}/df_{\text{PE}}}
$$
 [8]

If the model has no significant error, the variances for the lack of fit (V_{LoF}) and the pure error (V_{PE}) belong to the same distribution, so that F_{LoF} is smaller than the tabulated *F*-value.

RESULTS AND DISCUSSION

Comparison of the R^2 and O^2 values for the model fitted according to Equation 1 showed that the model had a slight overfit, i.e., the value of R^2 is more than 0.2 units higher than the value of Q^2 , due to the interaction terms between x_1x_2 , x_2x_3 , and $x_1x_2x_3$ that were classified as nonsignificant. These interaction terms were excluded from the model, and a new model was developed according to Equation 9,

$$
y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{13} x_1 x_3 + e
$$
 [9]

Four PLS components were significant according to crossvalidation, the first and second of which describe 53.4 and 16.3% of the total variance in **X**, respectively.

According to the ANOVA and the corresponding *F*-test, the model for the hardness of the product was considered statistically significant $(P < 0.05)$, and the model error was considered not significant $(P > 0.05)$.

The PLS weight plot for the two first PLS score vectors gives an overview of the relationship between the *x*-variables and the

FIG. 1. Loading scatter plot for the partial least squares (PLS) weights for the first two PLS-dimensions, wc[1] and wc[2], plotted against each other showing how the *x*-variables influence the *y*-variables (responses) and the correlation between responses and between responses and variables. The responses y_1 , y_2 , y_4 , y_5 , y_{11} , and y_{12} form a group and are mainly positively influenced by $x_3(B)$ and negatively influenced by x_1 . Other responses (y_7 , y_{10} , and y_{13}) form another group that is positively influenced by both x_1 and x_3 (B). The interaction term x_1x_3 and the main term $x₂$ are in the center of the plot, indicating that they are less important for the model. $w =$ the PLS weight for the *x*-variables, and $c =$ the weights for the responses.

responses (Fig. 1). The third and fourth PLS components describe 7.2 and 1.3% of the total variation in the data, respectively. The PLS weight plot for these two components provides little further information about the relationship between the variables and the responses, so they are not shown here.

From the plot in Figure 1 one can see that y_1 , y_2 , y_4 , y_5 , y_{11} , and y_{12} have similar weights and thus behave in a similar way. The similarity in behavior of five of these six parameters is not surprising considering the characteristics they represent: hardness (y_1) , spreadability (y_2) , meltability (y_4) , and the hardness parameters $(y_{11}$ and $y_{12})$. The other, less obviously correlated parameter in this group is butter aroma $(y₅)$. This group of responses is mainly positively influenced by x_3 (B) and negatively influenced by x_1 . Members of another group of responses, y_7 , y_{10} and y_{13} , are positively influenced by both x_1 and x_3 (B). The reverse relationship applies to y_3 and y_6 . The interaction term x_1x_3 and the main term x_2 are found in the center of the plot, indicating that they are less important for the model

Table 5 presents the models and the regression coefficients (with confidence intervals) for the variables in the model presented in Equation 9.

The most important response on which to focus is hardness since one of the objectives was to decrease it. It is important to remember that changing the hardness of the product also affects other sensory qualities of the products such as spreadability and meltability because these characteristics are highly correlated with each other. Thus, one has to ensure that other sensory responses are either improved or maintained at their present level while the hardness is decreased. The hardness of the product is mainly influenced by the amount of oil added $(x_1; Fig. 2)$. The temperature treatment of the raw material (x_3) also is shown to be a significant factor, and the speed of puddling (x_2) is shown not to be.

The coefficient plot shows how the variables influence the response, but further analysis is required to determine appropriate levels of the **X**-factors since the objective is to achieve a product with a response near 5 rather than 0 or 10 for all variables except off-flavor. A contour plot of x_1 and x_2 , with x_3 at a constant level, confirms that x_2 (puddling) had no effect on the hardness (Fig. 3). Both x_1 and x_3 had significant effects, and it is possible to find a region with appropriate hardness of the product using either treatment A or treatment B.

Temperature treatment A gave a raw material that was much softer than treatment B. So as to reduce the cost of production, a high level of $x₁$ is desirable. For this reason, temperature treatment B is preferable, since the raw material is then harder and it is possible to use the higher level of x_1 . However, as shown in Table 6, the shininess of the product (characterized by the sensory attribute "shine") did not reach a satisfactory level when using treatment B. An intensity of 1.58 is not sufficient (cutoff level: optimal intensity for this

TABLE 5 Final PLS-Model ($y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{13}x_1x_3 + e$) Showing the Fit of the Model (R^2 and Q^2 values), and the Calculated Regression **Coefficients with Confidence Intervals for Each Attribute***^a*

^aAbbreviations: PLS, partial least squares regression to latent structures; CI, confidence interval; A, number of significant PLS components according to crossvalidation; *R*2, variation described by the model; *Q*2, predictive ability of the model; Spread, spreadability; Butter, butter-aroma; FatBM, fat content in buttermilk; for other abbreviations see Table 4.

TABLE 6

 0.80 0.60 0.40 0.20 0.00 -0.20 -0.40 -0.60 -0.80 $x_1 \cdot x_3(A)$ ×, $\widetilde{\mathcal{A}}$ Ġ, $\mathfrak{g}(\mathsf{B})$ $(36)^{5/4}$

FIG. 2. PLS regression coefficients calculated for the hardness of the product. R^2 and Q^2 for this response are 0.971 and 0.902, respectively. The size of the regression coefficient (bars in the column plot) and the confidence interval (bars on the column plots) are used to determine whether a variable has a significant effect on hardness. A variable with a large, positive regression coefficient tends to increase hardness, making the product harder when used at a high level. In contrast, a variable with a large negative regression coefficient tends to make the product softer when used at a high level. For abbreviation see Figure 1.

FIG. 3. Contour plots for the temperature treatments, A and B (panels [a] and [b], respectively) showing changes in the hardness of the product when x_1 and x_2 are varied between high and low levels (+1 and -1, respectively). The numbers in the plot are the intensities of the attribute (hardness) when the variables x_1 and x_2 are changed from low level to high level. Optimal intensity for hardness is slightly below 5, and both temperature treatments can be used to achieve this. But when using temperature treatment A, a higher level of x_1 can be used, which is desirable.

a The +/− column represents the estimated prediction error. For abbreviations see Table 4.

attribute should be around 5 for an acceptable sample). In contrast, treatment A improved the intensity of the attribute "shine" to an acceptable level. Other responses were quite similar to those observed when treatment B was used.

The conditions in experiments 2 and 4 were identified as the most favorable, and the results were very similar (see Tables 3 and 4) to the predictions for treatment A (Table 6). The difference between experiments 2 and 4 was in the speed of puddling (x_2) , which was at a low level in experiment 2 and at a high level in experiment 4. Using high levels of oil (x_1) decreases the cost of production. The experiments clearly show that it is possible to modify the process in such a way that a high level of x_1 can be used when the raw material has been treated with either temperature treatment A or B. Treatment A is preferable to maintain or improve the sensory qualities of the product. The data also show that experimental design and sensory evaluation work well in combination as has been shown in various previous studies (10,15,16). However, in experiments involving evaluation by a sensory panel, it is important to ensure that the differences generated by the experimental variations are sufficiently large to be detected by the sensory panelists. It is also important to quantify the responses, in this case the sensory attributes, in such a way that the results can be evaluated using regression methods (such as MLR or PLS) or ANOVA, which are commonly used methods for analyzing experimental data in food research.

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