



## FORMULATION OPTIMIZATION OF A FUNCTIONAL COOKIE USING RESPONSE SURFACE METHODOLOGY

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### Abstract

A functional cookie containing oligofructose, dietary fibre and lower calorie, fat and sugar contents was produced using Response Surface Methodology (RSM). Instant N-Oil II was used as a fat replacer, while Raftilose<sup>®</sup>P95 was used as a sugar substitute with the addition of fructose to enhance sweetness. Selection of the optimal formulation was based on caloric content. An optimized formulation, V1, was produced from the model  $Y = 4927.70 - 152.34X_1 - 155.42X_3 + 104.20X_3^2 + 151.71X_3^3 - 95.08X_3^4$ , where Instant N-Oil II replaced 30% of butter and 24.4%, w/w (30.5g) fructose replaced 40.0%, w/w (50.0g) sucrose. Two additional optimized formulations, S1 and S2, were proposed which contained the same ingredients as V1, but both have an addition of 19.0%, w/w (23.8g) Raftilose<sup>®</sup>P95. Also, S2 had a higher fat replacement level (42%). The standard formulation received significantly higher scores ( $P < 0.05$ ) in the sensory evaluation conducted. However, when health benefits of the functional cookies were explained to the panels, the majority of them chose S1 which, with 19.04% fat, 8.62% fructose and 0.74% sucrose, had significantly lower fat and sucrose levels and higher fructose content than the standard formulation.

*Keywords : functional foods, optimization, Response Surface Methodology*

### Abstrak

Kaedah Respons Permukaan telah digunakan untuk menghasilkan biskut berfungsi yang mengandungi oligofruktosa, gentian diet dan kandungan kalori, lemak dan gula yang lebih rendah berbanding biskut biasa. Instant N-Oil II telah digunakan sebagai pengganti lemak dan Raftilose<sup>®</sup>P95 digunakan sebagai pengganti gula. Fruktosa digunakan untuk memperkuatkan rasa manis. Pengoptimuman formulasi dilakukan berdasarkan kandungan kalori biskut yang terhasil. Formulasi optimum, V1 dihasilkan daripada model  $Y = 4927.70 - 152.34X_1 - 155.42X_3 + 104.20X_3^2 +$

$151.71X_3^3 - 95.08X_3^4$  di mana, Instant N-Oil II menggantikan 30% mentega dan 24.4%, b/b (30.5g) fruktosa menggantikan 40.0%, b/b (50.0 g) sukrosa. Dua formulasi tambahan telah dicadangkan berdasarkan model tersebut, iaitu S1 dan S2. Kedua-dua formulasi ini mengandungi ramuan yang sama seperti V1, tetapi juga mengandungi 19.0%, b/b (23.8 g) Raftilose ®P95. S2 juga mempunyai paras penggantian lemak yang lebih tinggi iaitu sebanyak 42%. Formulasi piawai menerima skor yang lebih tinggi ( $P < 0.05$ ) dalam ujian penilaian sensori. Walaubagaimanapun, selepas ciri-ciri positif biskut berfungsi telah dihuraikan kepada ahli panel, majoritinya telah memilih biskut S1. Biskut S1 ini yang mengandungi 19.04% lemak, 8.62% fruktosa dan 0.74% sukrosa mempunyai paras lemak dan sukrosa yang lebih rendah dan paras fruktosa yang lebih tinggi berbanding biskut formulasi piawai.

*Kata kunci : Makanan berfungsi, pengoptimuman, Kaedah Respons Permukaan*

## **1 Introduction**

The current consumer interest in fat, sugar and caloric reduction has led to the development of alternative sweeteners, fat replacers and functional foods. The demand for functional food amongst consumers is increasing due to greater awareness of health issues. Functional foods are foods that are similar to conventional foods, consumed as part of a regular diet and can demonstrate physiological benefits or reduce the risk of chronic diseases beyond basic nutritional functions. As such, functional foods contain specific ingredients that can enhance a specific physical or mental function. Foods typically targeted for fat and sugar reduction include baked products, frozen desserts, butter and margarine, meat products and snack foods. In recent years, oligofructose has gaining popularity due to its soluble dietary fibre content and prebiotic properties. In fact, combinations of oligofructose and nutritive sweeteners such as fructose are increasingly being applied successfully in sugar-reduced food products. In this study, the commercially-available Raftilose ®P95 was used as both sugar replacer and source of oligofructose. According to ORAFIT Food Ingredients, it possesses prebiotic characteristics and is a good source of dietary fibre. However, as it contributes only 30% sweetness compared with sucrose, fructose was used to enhance sweetness in this study. The food-grade maltodextrin, Instant N-Oil II which contributes only 1.2 kcal/g was used as a fat replacer.

Response Surface Methodology (RSM) is a collection of statistical techniques for designing experiments, building models, evaluating the effects of the factors and searching for optimal conditions of factors for desirable responses (Myers 1976; Montgomery 1991). In this study, RSM

was used to produce a functional cookie with lower fat and sucrose levels and higher fructose level compared to a conventional cookie by incorporating fat and sugar replacers.

## 2 Materials and Methods

### 2.1 Standard and test cookie formulations

A standard butter cookie was obtained using the ingredients shown in Table 1 (Chang 2002). Butter and castor sugar were mixed with a mixer (ELBA Model ECM948) at Speed 1 for 4 min and then at Speed 4 for another 4 min. Condensed milk and egg yolk were added followed by salt and vanilla essence. Low-gluten flour, cornflour and mixed cereals were then folded in. Cookies were shaped by hand into flattened balls weighing approximately 11 g each and baked at 160°C for 25 min.

TABLE 1 Composition of the standard butter cookie (Chang 2002)

Component	Weight (g)	% w/w*
Butter	75.0	60.0
Castor sugar	50.0	40.0
Condensed milk	9.0	7.2
Egg yolk	8.0	6.4
Vanilla essence	1.0	0.8
Salt	0.5	0.4
Low gluten flour	100.0	80.0
Cornflour	25.0	20.0
Mixed cereals	8.0	6.4

\*Percentage of each component is based on total weight of flour

A rotatable central composite design was applied to optimize the cookie formulation (Table 2). The experiment consisted of 8 factorial runs, 6 axial runs and 6 center runs. The 3 independent variables were Instant N-Oil II ( $X_1$ , % w/w of butter replaced in the standard formulation), Raftilose @P95 ( $X_2$ , % w/w of flour used in the standard formulation) and fructose ( $X_3$ , % w/w of flour used in the standard formulation). Each variable was set at 5 levels and a total of 20 experiments were designed whereby Formulation 15, namely the centre-point formulation, was repeated 6 times. Each experiment was performed in duplicates and the average caloric content (kcal/g) was taken as the response.

To set up a statistical model, we let  $Y$  denote caloric values measured as kcal/g and we determined coded factor levels as follows:  $X_1 = (\text{Instant N-Oil II} - 30/11.9)$ ,  $X_2 = (\text{Raftilose @P95} - 19/4.2)$  and  $X_3 = (\text{Fructose} - 23/3)$ . Table 3 shows the coded and actual levels of factors used in this

study. Preliminary tests were conducted to obtain levels of factors that are capable of producing a cookie with acceptable characteristics, ie. crispy, non-soggy, non-sticky. For each factor, a conventional level which was determined from these preliminary tests was set to zero as a coded level. Treatment combinations and observed responses are shown in Table 2.

Test formulations were prepared by mixing Raftilose<sup>®</sup>P95 and fructose with butter at Speed 1 for 2 min. Instant N-Oil II was used as a 30% solution in this study. Water was added to Instant N-Oil II at the ratio of fat replacer : water (30 : 70) and mixed with the sugar replacer and fructose at Speed 4 for 4 min. Subsequent steps involving addition of condensed milk, egg yolk, salt, vanilla essences and flours were similar to those of the standard formulation.

TABLE 2 Treatment combinations and responses

Formulation	Factor			Response, $Y^b$
	$X_1^a$ (% , w/w)	$X_2$ (% , w/w)	$X_3$ (% , w/w)	
1	18.1 <sup>c</sup> (-1) <sup>d</sup>	14.8 (-1)	20.0 (-1)	5045
2	18.1 (-1)	14.8 (-1)	26.0 (1)	5095
3	18.1 (-1)	23.2 (1)	20.0 (-1)	5090
4	18.1 (-1)	23.2 (1)	26.0 (1)	5100
5	41.9 (1)	14.8 (-1)	20.0 (-1)	4808
6	41.9 (1)	14.8 (-1)	26.0 (1)	4776
7	41.9 (1)	23.2 (1)	20.0 (-1)	4817
8	41.9 (1)	23.2 (1)	26.0 (1)	4771
9	50.0 (1.632)	19.0 (0)	23.0 (0)	4667
10	10.0 (-1.632)	19.0 (0)	23.0 (0)	5220
11	30.0 (0)	26.0 (1.632)	23.0 (0)	4974
12	30.0 (0)	12.0 (-1.632)	23.0 (0)	4893
13	30.0 (0)	19.0 (0)	28.0 (1.632)	4922
14	30.0 (0)	19.0 (0)	18.0 (-1.632)	5001
15 <sup>e</sup>	30.0 (0)	19.0 (0)	23.0 (0)	4920

<sup>a</sup>  $X_1$ = Instant N-Oil II (% , w/w of fat replacement);  $X_2$ , = Raftilose<sup>®</sup>P95 (% , w/w of flour used in the standard formulation);  $X_3$  = fructose (% , w/w of flour used in the standard formulation)

<sup>b</sup> kcal/g

<sup>c</sup> Actual levels of factors

<sup>d</sup>(-1.632), (-1), (0) (1), (1.632) are coded levels of factors

<sup>e</sup> Formulation 15 was repeated 6 times

TABLE 3 Coded and actual levels of factors used in this study

Factor	Symbol	Actual factor level at coded factor level of :				
		-1.682	-1	0	1	1.682
Instant N-Oil II (%, w/w) <sup>a</sup>	$X_1$	10.0	18.1	30.0	41.9	50.0
Raftilose®P95 (%, w/w) <sup>b</sup>	$X_2$	12.0	14.8	19.0	23.2	26.0
Fructose (%, w/w) <sup>b</sup>	$X_3$	18.0	20.0	23.0	26.0	28.0

<sup>a</sup> % (w/w) replacement of fat used in the standard formulation

<sup>b</sup> % (w/w) of flour used in the standard formulation

## 2.2 Analytical Determinations

Energy contents of the cookies were determined with IKA Calorimetersystem C4000 and caloric values are expressed in kcal/g. Sugars were determined using AOAC Method 997.20 and 982.14 (AOAC 1990). The fat content of the samples was determined by extraction with hexane using Soxhlet apparatus (Tecator Soxtec System HT 1043 Extraction Unit, Sweden).

## 2.3 Sensory Evaluation

Fifty panelists (ages 20–55) both male and female assessed the sensory attributes of cookie samples. A 7-point hedonic scale ranging from 1 to 7, where 1 = extremely dislike and 7 = extremely like was used to evaluate acceptability of sample (colour, sweetness, butter taste, crispiness, overall acceptability). Data were subjected to analysis of variance (ANOVA) using the SAS software and differences among the means were compared using Duncan's Multiple Range test. A significance level of 0.05 was chosen.

## 2.4 Experimental design and statistical analysis

A central composite design was used to allocate treatment combinations in this experiment and the actual levels corresponding to the coded levels are shown in Table 3. In our regression model, the response variable was kcal/g and candidates for explanatory variables were linear, interaction, quadratic, cubic and quartic terms of coded levels of the factors tested. The  $\alpha$ -level at which every term in the selected model should be significant was set as 0.05. Optimum conditions were found through SAS data-step programming.

### 3 Results and Discussion

#### 3.1 Developing a regression model

A second-order polynomial regression model containing 3 linear, 3 quadratic and 3 interaction terms was employed by using the RSREG procedure of SAS/STAT. This model was found to be insignificant ( $P = 0.1040$ ) with  $r^2 = 0.6753$  and coefficient of variation = 3.9747. Moreover, its lack of fit was significant ( $P < 0.0001$ ). This indicates that it may be necessary to include higher order terms in the regression model as the second-order model is not able to represent the experimental data accurately. Since each factor had five levels, up to quartic terms could be included in the model (Box & Draper 1982).

Thus, variable selection techniques were used in attempts to find a better model. The maximum  $r^2$  improvement technique and forward variable selection technique were used to select good predictors from the following candidates for model terms:

$$X_1, X_2, X_3, X_1X_2, X_1X_3, X_2X_3, X_1^2, X_2^2, X_3^2, X_1^3, X_2^3, X_3^3, X_1^4, X_2^4, X_3^4.$$

As a result of the variable selection techniques used, the following functional form of this model was obtained:

$$Y = b_0 + b_1X_1 + b_3X_3 + b_{33}X_3^2 + b_{333}X_3^3 + b_{3333}X_3^4 \quad [\text{Eq. 1}]$$

TABLE 4 Analysis of variance in the regression model selected through variable selection

Source of variation	No. of degrees of freedom	Sum of squares	Mean square	F value	P value
Model	5	1142664	228533	173.15	<0.0001
Residual	14	18478	1319.82		
Lack of fit	8	7586	948.25	0.5223	0.8059
Pure error	6	10892	1815.25		
Total	19	1161142			

$$r^2 = 0.9841 \quad \text{coefficient of variation} = 0.5386$$

This fourth-order subset model (Table 4) was significant ( $p < 0.0001$ ) and superior to the second-order full model as it had a larger  $r^2$  ( $0.9841 > 0.6753$ ) and its number of variables was also smaller ( $5 < 9$ ). Its lack of fit was not significant ( $P = 0.8059$ ) and it had a smaller coefficient of variation ( $0.5386 <$

3.9747). The intercept,  $b_0$  was the estimated response at the center point  $(X_1, X_2, X_3)=(0, 0, 0)$ . The fourth-order model is as follows:

$$Y = 4927.70 - 152.34X_1 - 155.42X_3 + 104.20X_3^2 + 151.71X_3^3 - 95.08X_3^4 \quad [\text{Eq. 2}]$$

### 3.2 Finding the optimum point of the factors

The response surface model could be written as:  $Y = b_0 + f_1(X_1) + f_3(X_3)$  [Eq. 3]

where,  $f_1(X_1) = b_1X_1$  and  $f_3(X_3) = f_3(X_3) + f_{33}(X_3^2) + f_{333}(X_3^3) + f_{3333}(X_3^4)$

As  $f_1(X_1)$  is a linear function, it was not optimized but instead was set at its center point value of 30%.  $f_3(X_3)$  was optimized by differentiation. The effect of  $X_2$  was found to be insignificant and as such, Raftilose<sup>®</sup>P95 was not included in the optimized formulation, V1, which consisted of  $(X_1, X_3) = (0, 0.46)$ . These coded levels correspond to actual levels of  $X_1 = 30\%$  and  $X_3 = 24.4\%$ . The estimated optimal response corresponding to the optimum factor levels was 4889 kcal/g. A validation test was conducted to determine the caloric contents of cookies produced from V1, the standard formulation and 2 brands of commercial cookies. V1 cookies contained 5129 kcal/g, which was significantly lower than the standard formulation cookie (5348 kcal/g) and commercial cookies (5339 kcal/g and 5423 kcal/g, respectively).

TABLE 5 Composition of the 3 cookie formulation obtained from optimization of the factors

Formulation	V1	V2	V3
	Weight (g)	Weight (g)	Weight (g)
Butter	52.5	52.5	43.6
Instant N-Oil II	6.8	6.8	9.4
Air	15.7	15.7	22.0
Raftilose <sup>®</sup> P95	-	23.8	23.8
Fructose	30.5	30.5	30.5
Condensed milk	9.0	9.0	9.0
Egg yolk	8.0	8.0	8.0
Vanilla essence	1.0	1.0	1.0
Salt	0.5	0.5	0.5
Low gluten flour	25.0	25.0	25.0
Cornflour	100.0	100.0	100.0
Mixed cereal	8.0	8.0	8.0
Total	258.0	281.8	280.8

Two additional formulations, S1 and S2, were proposed which contained similar ingredients as V1 but also contained  $X_2$ , Raftilose<sup>®</sup>P95 at its centre-point level, ie. 19.0%. In addition, S2 had a higher level of fat replacement, containing 41.9% of  $X_1$  (coded  $X_1$  value = 1). Table 5 shows the composition of formulation V1, S1 and S2. Cookies from four formulations, namely, the standard formulation, V1, S1 and S2 were evaluated by a sensory evaluation panel of 50 using a 7-point hedonic test. Highest sensory scores were recorded for cookies from the standard formulation in all attributes except colour. S1 and V1 received higher scores than S2. When the panelists were asked to choose between cookies from the standard and S1 formulations, they overwhelmingly chose the standard formulation, with only 5 panelists out of 50 choosing S1. However, after they were briefed on the properties of S1, which has significantly lower fat (19.04% <25.86%) and sucrose (0.74% <17.46%) levels as well as contains fructose (8.62%) and oligofructose, 60% of the panelists selected S1 as their cookie of choice. This indicates that health awareness plays an important role in selection of food.

#### 4 Conclusion

Using RSM, two functional cookie formulations were produced, namely, V1 and S1. In V1, which contained 24.4% fructose, 30% fat was replaced by 30% Instant N-Oil II solution. S1 contained 30% Instant N-Oil II, 19.0% Raftilose<sup>®</sup>P95 and 24.4% fructose. The S1 cookie had lower levels of fat and sucrose compared with conventional butter cookies. It also contained fructose and oligofructose.

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