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Development of frozen savoury grilled fish (ikan percik)

(Pembangunan ikan percik sejuk beku)

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Key words: fishery product, frozen savoury grilled fish, coconut milk, mixture experimentation, response surface methodology, optimization

Abstrak

Reka bentuk campuran terbatas empat pembolehubah digunakan untuk menentukan formulasi optimum bagi makanan tradisional iaitu ikan percik. Reka bentuk komposit berputar 2-faktor dan model tertib kedua digunakan untuk menentukan suhu dan masa optimum bagi pemanggangan produk secara mekanikal. Analisis data sensori mendapati terdapat interaksi yang nyata antara keempat-empat bahan utama dalam formulasi. Formulasi optimum mengandungi 50–52% bahan cecair, 14–15.7% bawang, 32–33% santan dan 2.8–3.9% cili kering. Produk yang diproses menggunakan formulasi optimum adalah sah lebih baik. Suhu dan masa optimum bagi proses pemanggangan adalah pada 220–240 °C selama 25–30 minit.

Abstract

A four-variable constrained vertex mixture design was used to determine the optimum formulation for the development of a traditional dish, savoury grilled fish or 'ikan percik'. A 2-factor central composite rotatable design including time and temperature was used to determine the optimum conditions for mechanical grilling and second order models were employed. Multiple Regression analysis of the sensory data indicated a significant interaction between all the four main ingredients used in the formulation. The optimal formulation contained 50–52% water base, 14–15.7% onion base, 32–33% coconut milk and 2.8–3.9% chilli. The optimum product was confirmed to be significantly superior. The optimum grilling conditions were at 220–240 °C for 25–30 minutes.

Introduction

Savoury grilled fish or better known as 'ikan percik' is considered as a special dish in Malaysia. It has a delicate spicy flavour and is usually served with rice and vegetables for meals. Traditionally, the dish is prepared by grilling the fish over smouldering charcoal with occasional basting with a savoury sauce. The sauce is prepared by mixing several kinds of spices, thick coconut milk (santan), salt, sugar and tamarind paste. The taste of the product depends very much on the sauce formulation and the grilling technique. The grilling process needs proper control because the product may get blackened easily. Hence, the preparation of this product is quite tedious and time consuming. Therefore, the

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development of ready-to-use products such as frozen savoury grilled fish will provide both convenience and consistent quality product to our affluent consumers.

Coconut milk, chilli and onions are common ingredients used in local dishes of many countries including the ASEAN region (Edwards et al. 1990). Coconut milk imparts a rich creamy flavour to fish, chicken and meat dishes. Chilli, onions and local spices contribute to the hot and spicy flavour enjoyed by the population of this region. Apart from the hot flavour, chillies also give an attractive red colour to local cuisines such as curry and 'sambal'. Capsaicin, the pungent principle in chilli, is responsible for the characteristic hot, sharp and stinging sensations on the tongue after eating a meal containing chilli. Onion is characterized with a strong penetrating aroma and sweet sulphur-like flavour.

The first step in food product development is to evaluate and efficiently detect critical potential ingredients that affect several sensory properties (Bomio 1994). The classical one-variable-at-a-time experiment had been inadequate because of the inability to find the real overall optima in the multi-ingredients food system. The method also lacks the ability to determine interactions between variables. Various other approaches had been used by many researchers to derive the optimum formulation and process parameters for food products. Alves-Castro et al. (1998) used response surface methodology to develop a cost effective chocolate flavoured drink using soy protein, gelatin and wheat gluten. Prinyawiwatkul et al. (1997) used a three-component constrained mixture design to derive the optimum formulation of extended chicken nuggets containing fermented cowpea and peanut flours. An optimal formulation was derived and the product was rated as acceptable as the control by consumer taste panelists. Chang and Carpenter (1997) used factorial experiments to optimize the quality of frankfurters containing oat bran.

The objectives of this study were to derive the optimum formulation of the sauce for the savoury grilled fish (ikan percik) and to establish the range of optimal grilling times and temperatures for the product. This study was part of the project to develop a frozen microwavable 'ikan percik'. In the development of frozen 'ikan percik', the sauce formulations involved mixture experimentation while response surface methodology was used to establish the optimum grilling condition.

Materials and methods

Materials

Black pomfret (*Parastromateus niger)* weighing 300–350 g each was purchased from a nearby LKIM fish landing complex. Fish were dressed, cleaned, blast-frozen and kept at –25 °C until used within 2 months. Fish were thawed and cut into 2 cm thick fish steaks, weighing 90–110 g each on the day of the processing trials.

Coconut milk powder or 'santan' powder (62% fat), small onions, dried chilli, tamarind paste and other spices were purchased from a local central market and kept in a laboratory walk-in chiller (5 °C) in their original package until used. Small onions, dried chilli and other spices were prepared and cleaned before used.

Preparation of the 'ikan percik' samples

Formulation optimization and verification experimentation The basting sauces were prepared according to the component combinations for each formulation as outlined in *Table 1*. Onions, dried chilli and other spices were blended with water using a Warring heavy-duty commercial blender (USA). Coconut milk powder, the blended spices and the remaining ingredients were then mixed together in a cooking pot and heated to boiling for 10 minutes.

Fish steaks were arranged in a single layer on grilling baskets, and grilled at 220 °C in an electric grill (Euro-grill Model TG 101, Holland) for a total grilling time of 25 min. The baskets with the fish were

Table 1. Composition of savoury grilled fish in a 4-component constrained extreme vertex design evaluated for acceptability of sensory qualities

Formulation number	Type of boundary	Mixture component proportion			
		Water[a] base	Coconut milk powder	Chilli	Onion[b] base
1	Vertex	0.500	0.350	0.001	0.149
2	Vertex	0.500	0.350	0.010	0.140
3	Vertex	0.500	0.300	0.060	0.140
4	Vertex	0.500	0.159	0.001	0.340
5	Vertex	0.500	0.100	0.060	0.340
6	Vertex	0.509	0.350	0.001	0.140
7	Vertex	0.550	0.050	0.060	0.340
8	Vertex	0.609	0.050	0.001	0.340
9	Vertex	0.750	0.050	0.060	0.140
10	Vertex	0.809	0.050	0.001	0.140
11	Face centroid	0.573	0.181	0.025	0.221
12	Face centroid	0.500	0.250	0.030	0.220
13	Face centroid	0.680	0.050	0.030	0.240
14	Face centroid	0.503	0.350	0.004	0.143
15	Face centroid	0.585	0.192	0.001	0.222
16	Face centroid	0.575	0.125	0.060	0.240
17	Face centroid	0.614	0.220	0.026	0.140
18	Overall centroid	0.540	0.090	0.030	0.340
19	Overall centroid	0.573	0.181	0.025	0.221

[a]Contained water and a constant proportion of sugar, salt, shrimp paste and tamarind
[b]Contained onions and a constant proportion of lemon grass, garlic, turmeric and ginger

dipped into the sauce twice during this process, after 5 min and 10 min of grilling. Cooked grilled fish samples were blast-frozen, packed in microwavable containers and stored frozen at –25 °C for product evaluation.

Five samples including three samples using the optimum formulation, one sample using a formulation at the fringe of the optimum region and a sample using traditional recipe were prepared in triplicate *(Table 2)* to verify the quality of the optimum product derived in this study. The moisture and fat content in the sauce were also determined.

Process optimization Samples were prepared using the sauce from the optimum formula and grilled using different time and temperature combinations *(Table 3)*. Samples were evaluated by trained panelists immediately after processing. The moisture and fat contents of all samples were determined.

Sensory evaluation

Formulation optimization and verification experimentation The panelists (n = 25) were research staff of Food Technology Centre of MARDI, who had been trained for general purpose sensory evaluation (Larmond 1977). Evaluation was conducted in individual booths in an air-conditioned sensory evaluation laboratory. Each booth was illuminated with fluorescent light. Panelists were instructed to evaluate all the samples in five seatings within two panel sessions between 1100–1300 h and 1430–1530 h with a 5-min break in between seatings.

The samples were thawed and reheated in a microwave oven. Samples were coded with 3-digits random numbers and served to the panelists after reheating together with

Table 2. Components composition of the savoury grilled fish sauce used in the verification experimentation

Sample code	Description	Percent component composition in the sauce (w/w)			
		Water base	Coconut milk	Chilli	Onion base
OPT	Optimum formulation with coconut milk powder	50	33	3	14
OPT 20%	Optimum formulation with thick fresh coconut milk	50	33	3	14
OPT 10%	Optimum formulation with thin fresh coconut milk	50	33	3	14
FORM 2	Second best formulation (10% fat level)	50.2	31	2.8	16
TRAD	Traditional recipe	Contained 21.8% thick coconut milk, 56.0% water, 2.5% chilli, 11.5% onions, 3.4% crushed lemon grass, 1.4% crushed ginger, 3.4% mixture of salt, sugar, tamarind, shrimp paste and monosodium glutamate			

Table 3. Variables and levels for central composite rotatable design

Process variable	Symbol	Coded variables level				
		–2	–1	0	1	2
Temperature (C)	Y1	150.0	172.5	195.0	217.5	240.0
Time (min)	Y2	20.0	22.5	25.0	27.5	30.0

warm water and white rice. They were asked to evaluate the sample for each of the attributes using an unstructured 5-inch horizontal line labelled at both ends (Stone et al. 1974). The attributes were creaminess intensity and pungency intensity (none to very high), colour, creamy flavour, pungent flavour and overall taste (dislike extremely to like extremely). Panelists indicated their perception by marking a slash on the line provided for each attribute.

Process optimization Sensory tests were conducted at the Sensory Evaluation Laboratory in MARDI Research Station, Kuala Terengganu. Ten panelists were selected from a 15-member sensory panel based on their performances after five 2-h training sessions. During training, sufficient samples that represent under cooked, well-done and over-cooked products were prepared and provided to standardize sensory responses and derive attribute descriptions for 'well-grilled' savoury grilled fish.

Immediately after processing, samples were coded with 3 digits random numbers and served to the panelist together with warm water and white rice to cleanse the palate after each sample. The following attributes derived by consensus during training were rated using an unstructured 5-inch horizontal line test similar to the above: coating thickness (very unsuitable to very suitable), colour (very unacceptable to very acceptable), hardness (hard to soft), juiciness (dry to moist), doneness (not acceptable to very acceptable) and overall acceptance (extremely unacceptable to extremely acceptable).

The panelists ratings of each attribute used in all of the above experimentation were converted to a numerical rating for subsequent analysis by measuring the distance between the left extremity of the

line scale (zero value) to the panelists mark on the line.

Chemical analysis

The moisture, protein, fat, crude fiber, ash, minerals and vitamin were analysed using the AOAC methods (1984) and the laboratory procedures of Tee et al. (1996). All analyses were carried out in duplicate and results were expressed in wet-weight basis.

Experimental design and data analysis for optimization

Four ingredient variables affecting final characteristics of 'ikan percik' were formulated with the constraints on each variable as follows:-

Ingredient	**Lower limit**	**Upper limit**
Water base *(X1)*	0.50	0.809
Coconut milk *(X2)*	0.05	0.35
Chilli *(X3)*	0.001	0.06
Onions *(X4)*	0.14	0.34

In each mixture of the formulation, the proportion of each ingredient is constrained within its lower and upper limit, and the sum of the proportional values of all ingredients must always be 1.0 or 100% ($X1 + X2 + X3 + X4 = 1$).

A total of 19 different formulations as shown in *Table 1* was obtained following the procedure that was first introduced in McLean and Anderson (1966). The 10 formulations located at the vertex were extreme samples which were assumed to be on the fringe of acceptability.

The canonical polynomial model described by Scheffe (1965) was used to evaluate effects of the 4 components on the sensory attributes. The second-degree model was fitted as follows:-

$$Y = b1X1 + b2X2 + b3X3 + b4X4 + b12X1X2 + b13X1X3 + b14X1X4 + b23X2X3 + b24X2X4 + b34X3X4$$

Where Y = predicted response variable,

b1,b2,b3,b4,b12,b13,b14,b23,b24,b34 = corresponding regression estimates for each linear and crossproduct terms

X1 = proportion of water base

X2 = proportion of coconut milk

X3 = proportion of chilli

and *X4* = proportion of onion

Due to the restriction of the mixture design ($X1 + X2 + X3 + X4 = 1$), it was not possible to estimate the intercept (b0). The regression model was not full rank. Stepwise procedure of multiple regression analysis was used to develop prediction equations from the response surface design (SAS Institute, Inc. 1985). The best fitting models for all significant response variables were used to generate graphs that showed trends in attributes. The optimum area was determined by superimposing the acceptable areas (Palomar et al. 1994) from all attributes.

The sensory data recorded in the verification experiment were subjected to analysis of variance (ANOVA) and Duncan's Multiple Range Test (DMRT) to determine differences among samples.

Response Surface Methodology was used to study the simultaneous effect of two process variables, namely temperature *(Y1)* and time *(Y2)* of grilling for 'ikan percik'. Each variable was coded at 5 levels: -2,-1, 0, 1 and 2 *(Table 3)*. Nine combinations of these levels were arranged following the Central Composite Rotatable Design thus allowing for fitting a second-order model (Cochran and Cox 1957). The design points consist of 4 factorial, 4 axial and 4 replicates at the center point with a design radius alpha = 2 (Cornell 1981).

The sensory data recorded were thickness, colour, doneness, hardness, juiciness and overall acceptability which acted as dependent variables. The data were subjected to univariate technique of statistical analysis (ANOVA and t-test) and DMRT to determine differences among samples. A multiple regression model including linear, quadratic and interaction effect was applied to predict any effects of temperature and time on the response

variables. The best-fitting model obtained from the stepwise procedure on significant sensory attributes was used to generate contour plots to show trend in attributes.

Each response attributes was related to the processing parameters by a second-order polynomial equation:

$Y = b0 + b1Y1 + b2Y2 + b11Y1Y1 + b22Y2Y2 + b12Y1Y2$

Where Y = estimated response
$b0, b1, b2, b11, b22, b12$ = equation parameter estimates
$Y1$ = levels of temperature
$Y2$ = levels of time

The estimates in codified units of the model parameters, *b*, of regression were determined. All runs were in a randomised order to minimize any effects of extraneous factors on the observed responses.

Results

Formulation optimization

The mean score for overall acceptability of the 19 samples was 2.90 and ranged from 1.94 to 3.78 with 13 formulations received overall acceptability greater than 2.5 *(Table 4)*. The mean scores for acceptability of pungency, creaminess and colour ranged from 1.97 to 3.62, 2.07 to 3.74 and 1.52 to 4.15, respectively.

The acceptability for creaminess is linearly related to the perceived intensity of creaminess ($R^2 = 0.78$), indicating that panelists preferred a product having a rich creamy coconut milk flavour (*Figure 1)*. However there is a quadratic relationship between the perceived intensity of pungency and its acceptability ($R^2 = 0.83$) as shown in *Figure 2*. The predicted optimum of perceived intensity of pungency was calculated to be 2.76 and the actual overall mean value of 2.86 ± 0.59.

Best-fitting multiple regression models were determined from sensory data to determine optimum levels of the 4 components for optimum formulation

Table 4. Average score for the sensory attributes used to determine optimum region

Formulation number [a]	OA	PA	CA	PI	CI	CLA
1	3.24	2.72	3.25	1.65	4.05	3.37
2	3.64	3.51	3.73	2.01	3.73	4.15
3	3.43	3.17	3.58	3.89	3.41	3.60
4	2.59	2.79	3.03	1.42	3.66	2.90
5	2.21	2.19	2.51	1.33	2.20	3.15
6	2.95	2.86	3.36	1.52	3.57	2.87
7	1.94	1.97	2.07	4.37	2.08	3.37
8	2.40	2.62	2.83	1.49	3.00	1.52
9	2.28	2.24	2.21	4.42	2.11	3.21
10	2.80	3.00	3.14	1.85	2.89	2.80
11	3.68	3.62	3.66	3.38	3.51	3.81
12	3.78	3.61	3.74	3.41	3.70	3.88
13	2.33	2.62	2.56	4.05	2.33	2.62
14	3.08	2.79	3.45	1.18	3.83	3.60
15	2.74	2.50	3.17	1.38	3.59	2.02
16	2.28	2.36	2.66	4.23	2.43	2.67
17	3.67	3.38	3.66	3.55	3.60	3.72
18	2.57	2.81	2.91	4.15	2.87	3.03
19	3.58	3.55	3.63	3.70	3.50	3.69

OA = overall acceptability, PA = pungency acceptability, CLA = colour acceptability, PI = pungency intensity, CA = creaminess acceptability, CI = creaminess intensity.
Scale: 0 = none for intensity, dislike extremely for acceptability.
5 = very high for intensity, like extremely for acceptability.
[a] Formulation numbers correspond to the numbers in *Table 1.*

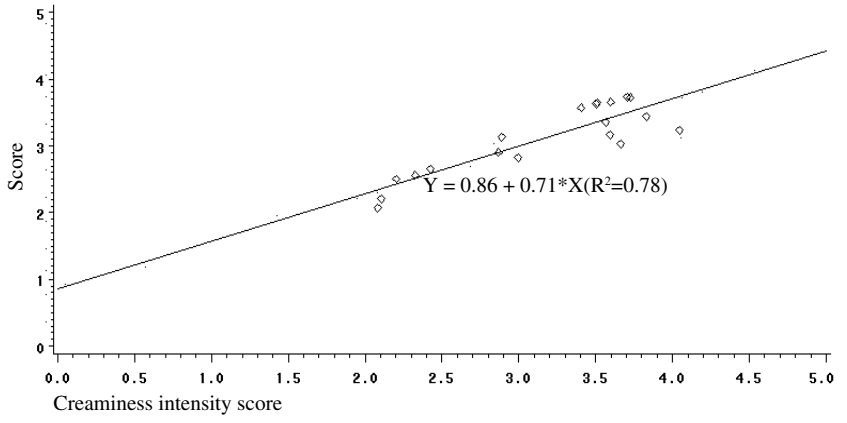


Figure 1. Relationship between creaminess intensity and creaminess acceptability scores

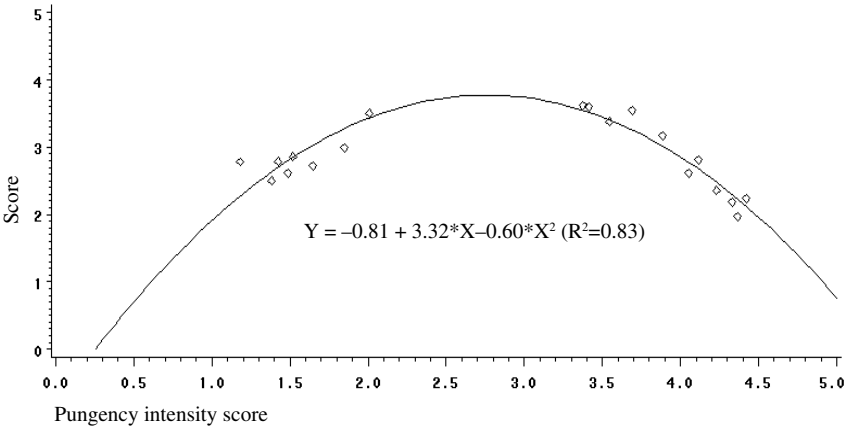


Figure 2. Relationship between pungency intensity and pungency acceptability scores

Table 5. Reduced models to predict the effect of water *(X1)*, coconut milk *(X2)*, chilli *(X3)* and onion *(X4)* on sensory attributes of savoury grilled fish

Sensory attribute	Predictive model
Overall acceptability	$3.12X1 - 625.28X3 + 8.64X1X2 + 644.69X1X3 + 729.40X2X3 + 684.13X3X4$
Pungency acceptability	$5.09X1 - 758.75X3 + 14.97X4 + 790.86X1X3 - 28.10X1X4 + 878.50X2X3 + 782.90X3X4$
Creaminess acceptability	$3.59X1 - 385.70X3 + 3.40X1X2 + 385.71X1X3 + 477.31X2X3 + 17.92X2X4 + 422.91X3X4$
Pungency intensity	$2.31X1 - 1014.35X3 + 1120.64X1X3 + 1117.33X2X3 + 1162.74X3X4$
Creaminess intensity	$2.88X1 - 16.38X3 + 2.05X4 + 6.60X1X2 + 52.36X2X3 + 18.55X2X4$
Colour acceptability	$6.97X1 - 820.18X3 + 28.14X4 + 876.09X1X3 - 60.54X1X4 + 870.78X2X3 + 902.05X3X4$

All models were significant at 1% level and coefficient of determination (R^2) >0.90 for all dependent variables. All variables with a p-value <0.05 were kept in the regression model.

(Table 5). The models were highly significant (p <0.01) with R^2 >0.90 for all dependent variables.

There was a large negative b coefficient in the main effect of *X3* (chilli) for all the sensory equations indicating that increasing the amount of *X3* (chilli) alone would result in lowering all the sensory attribute scores. However, the component had significant positive interaction effect with *X1* (water base), *X2* (coconut milk powder), and *X4* (onion) except for the attribute on perceived creaminess. This showed that *X3* (chilli) blended with the other 3 components can give a synergistic effect and resulted in increasing overall acceptability. On the other hand, *X2* (coconut milk powder) had no linear effect but had high positive interaction effect on *X3* (chilli) for all sensory attributes.

The significant effects of the ingredients variables and their interactions on the sensory responses were demonstrated by the response surface plots in *Figure 3* and *Figure 4*. The diagrams showed that the maximum scores for pungency, creaminess and overall acceptance exist within the testing range of ingredient variables used in this study. The acceptability score was high at the lowest waterbase level, hence the diagrams in *Figure 3* were plotted with the waterbase level fixed at 50%.

Superimposing the optimum acceptable areas of the contour plots revealed that pungency acceptability, overall acceptability, and acceptance of creaminess were the limiting factors in attaining the optimum. The shaded region where contours within limits of acceptance overlapped is considered as predicted optimum region *(Figure 5)*. The mean sensory scores for this region are >3.7 for all attributes. The ingredient level in this region is in the range of 50–52% water base, 32–33% coconut milk, 14–15.7% onion and 2.8–3.9% chilli.

Verification experimentation

ANOVA and DMRT showed that scores for optimum products made either from coconut milk powder (OPT) or fresh coconut milk (OPT 20) were significantly higher (p <0.05) for colour, pungency, creaminess and overall acceptability *(Table 6)*. OPT was observed to be significantly higher in creaminess intensity as compared to OPT 20 with the value of 3.94 and 3.40 respectively. As for pungency intensity, there seemed to be no significant difference among the treatments (p <0.05). The sample OPT 10 made from the same optimum formulation but with 50% less fat content *(Table 7)* was less preferred than OPT by the panelists.

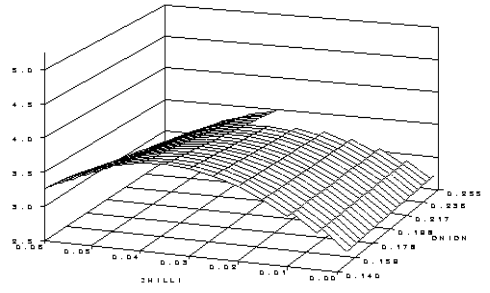
Process optimization

Table 8 shows the mean scores for the sensory attributes as affected by the grilling temperatures and times. ANOVA and DMRT of the 12 samples from the temperature/time combinations showed significant difference for all responses (p <0.05) except for colour and coating thickness. Regression equations were computed for the response *(Table 9)* which had shown significant differences in the ANOVA. Each regression equation showed no significant lack of fit (p <0.05).

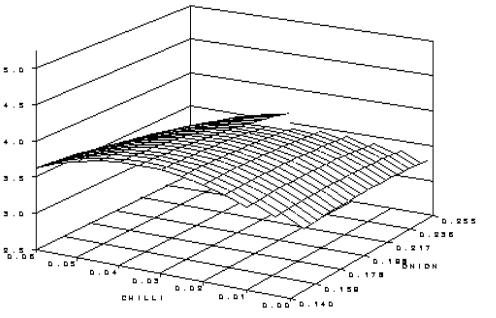
The response surface plots in *Figure 6* showed that samples processed at the highest temperatures/times combinations received higher scores for both degree of doneness and overall acceptability.

Chemical analysis

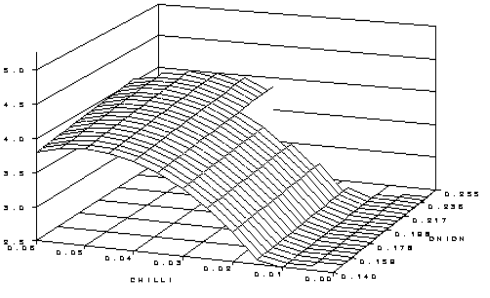
The overall mean values of fat contents in 'percik' sauce samples from the 5 treatments, namely: OPT, OPT 20, OPT 10, FORM and TRAD were 19.8, 20.6, 11.3, 11.6 and 14.8% respectively *(Table 7)*. The changes in moisture and fat contents during grilling are shown in *Figure 7* and *Figure 8* respectively. The moisture, protein and fat contents in raw black pomfret were 74.85, 20.16 and 3.12%, respectively. The moisture content decreased to 64.3% and fat content increased to 10.1% in the product prepared from the optimum sauce formulation and grilled at 240 °C for 25 min *(Table 10)*. The protein content remained constant. The calcium, phosphorous, sodium, potassium and vitamin C contents in the product were



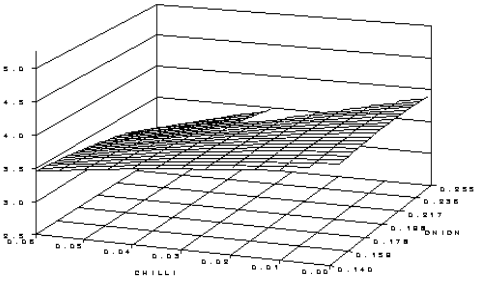
a) Pungency acceptability



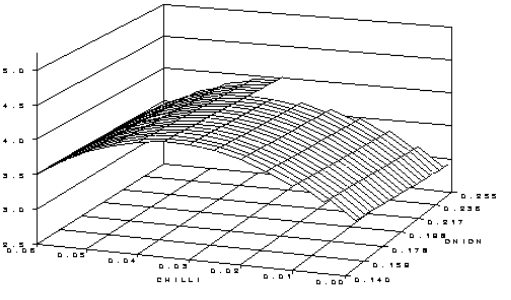
b) Creaminess acceptability



c) Perceived intensity of pungency



d) Perceived intensity of creaminess



e) Colour acceptability

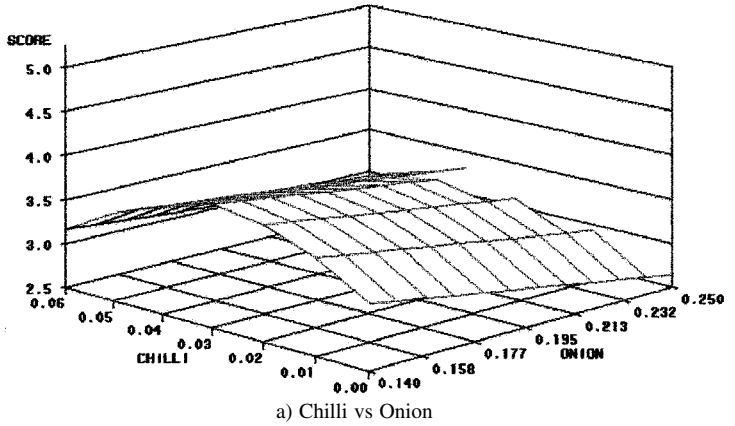
Figure 3. Response surface plots of sensory responses with respect to chilli and onion proportions in the formulations

Table 6. Average scores for the sensory attributes of products in the verification experimentation

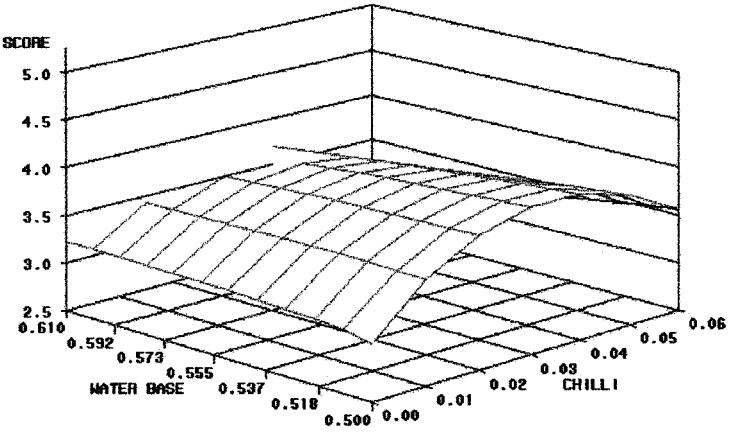
Sample* code	Pungency intensity	Creaminess intensity	Colour liking	Pungency liking	Creaminess liking	Overall liking
OPT	3.13a	3.94a	3.54a	3.55a	3.42a	3.44a
OPT 20	3.28a	3.40b	3.72a	3.59a	3.58a	3.56a
OPT 10	3.66a	2.82c	3.02b	2.96b	2.92b	2.77b
FORM 2	3.49a	2.90c	2.70c	3.07b	2.84b	2.71b
TRAD	3.17a	2.90c	2.76c	3.00b	2.91b	2.74b

Means are calculated from panelist responses (n = 75) for each attribute. Means in a column followed by the same letter are not significantly different ($p < 0.05$) according to DMRT

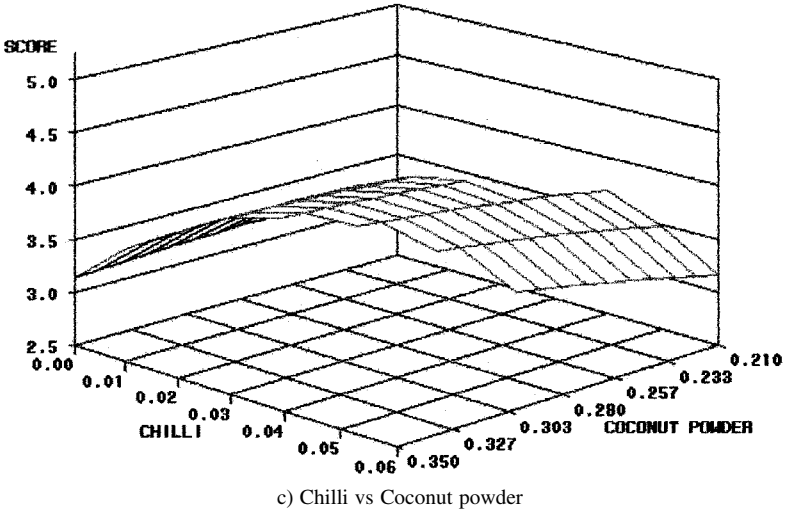
*Samples codes used correspond to description in *Table 2* i.e. OPT (optimum formulation using coconut milk powder), OPT20 (optimum formulation using fresh coconut milk), OPT10 (as OPT 20 with lower fat content), FORM 2 (second best formulation), TRAD (traditional recipe)



a) Chilli vs Onion



b) Water base vs Chilli



c) Chilli vs Coconut powder

Figure 4. Response surface plots of overall acceptability with respect to ingredient proportions in the formulations

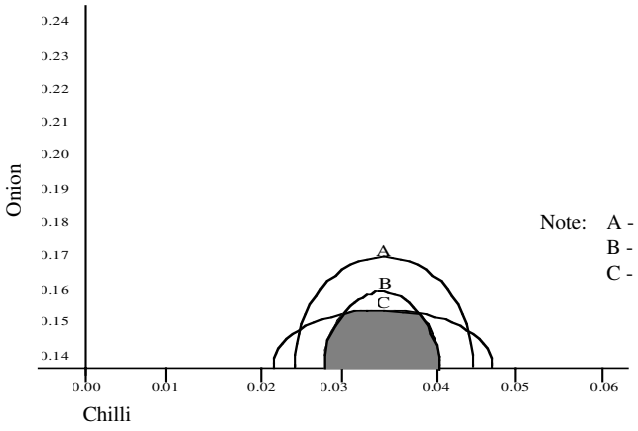


Figure 5. Superimposed contour plots with shaded area showing the optimum region

Table 7. The moisture and fat contents in the 'percik' sauce prepared for the verification experimentation

Composition (%)*	Sample code[1]				
	OPT	OPT 20	OPT 10	FORM 2	TRAD
Moisture	55.6 (±0.58)	64.8 (±0.15)	75.5 (±0.54)	75.2 (±0.45)	71.2 (±2.3)
Fat	19.75 (±0.72)	20.6 (±1.4)	11.25 (±0.78)	11.6 (±0.82)	14.8 (±1.1)

*Average of three replicates
[1]Sample codes referred to *Table 2* and *Table 6*

Table 8. Central composite rotatable design arrangement of 2-factor process variables and DMRT for the sensory attributes

Run no.	Variable level		Mean response					
	Y1	Y2	Juiciness	OA	Thickness	Colour	Doneness	Hardness
1	1	1	3.66d	4.64a	4.50a	4.40a	4.77ab	4.26ab
2	1	–1	4.37ab	4.32abcd	4.14a	4.33a	4.36d	4.48ab
3	–1	1	3.82cd	4.28bcd	4.30a	4.33a	4.61bc	4.29ab
4	0	0	4.25abc	4.44abcd	4.30a	4.34a	4.52bcd	4.53ab
5	–1	–1	4.34ab	4.24cd	4.23a	4.41a	4.85a	4.65ab
6	2	0	3.94bcd	4.56abc	4.73a	4.67a	4.62abc	4.36ab
7	–2	0	4.44a	4.20d	4.32a	4.27a	4.48cd	4.62a
8	0	0	4.25abc	4.41abcd	4.34a	4.29a	4.62abc	4.41ab
9	0	2	3.92bcd	4.39abcd	4.33a	4.41a	4.74ab	4.19ab
10	0	–2	4.59a	4.35abcd	4.34a	4.32a	4.57bcd	4.58ab
11	0	0	4.43a	4.47abcd	4.68a	4.63a	4.69abc	4.62a
12	0	0	4.29abc	4.61ab	4.51a	4.40a	4.68abc	4.50ab
Significant level			**	*	n.s.	n.s.	**	*

Scale : 0 = very unacceptable, 5 = very acceptable except for hardness (0 = dry, 5 = soft) and juiciness (0 = dry, 5 = moist)
Y1 = temperature in Celcius (–2 = 150 °C, –1 = 172.5 °C, 0 = 195 °C, 1 = 217.5 °C, 2 = 240 °C)
Y2 = time in minutes (–2 = 20 min, –1 = 22.5 min, 0 = 25 min, 1 = 27.5 min, 2 = 30 min)
** = significant at 1% level
* = significant at 5% level
n.s. = not significant
Means in a column followed by the same letter are not significantly different from each other

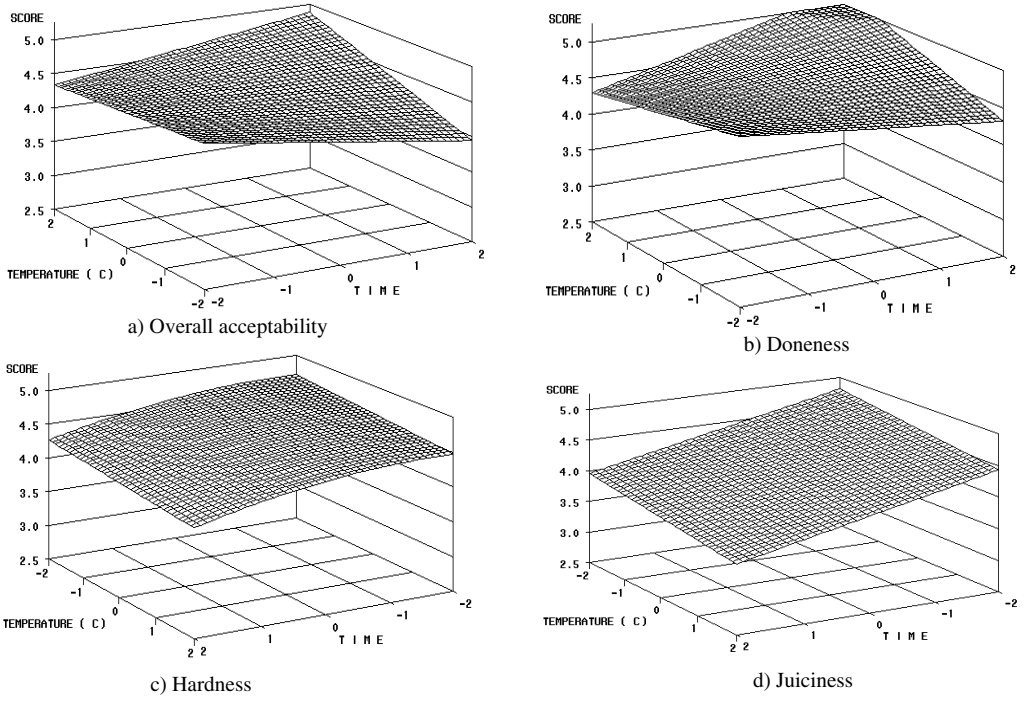


Figure 6. Changes in sensory attributes as affected by grilling temperature and time of 'ikan percik'

Table 9. Reduced models with process variables as independent variables

Attribute	Predictive equation	R^2
Juiciness	4.1933 – 0.2138*TIME – 0.0954*TEMP	0.70
Overall acceptability	4.4125 + 0.0963*TIME + 0.0688*TEMP*TIME	0.60
Doneness	4.6296 + 0.0621 + TEMP + 0.0688*TIME + 0.0813*TEMP*TIME	0.65
Hardness	4.4896 – 0.0592 + TEMP – 0.1125*TIME – 0.0304*TIME*TIME	0.83

All variables with a P-value < 0.05 were kept in the regression model. The models were reduced using stepwise procedure of SAS (SAS institute Inc. 1985)

0.037, 0.169, 0.537, 0.369 and 0.003%, respectively.

Discussion

Formulation optimization

Our preliminary survey showed that 6–15 types of ingredients were used in the recipes for the preparation of 'ikan percik'. All recipes contained fresh coconut milk, onion and dried chilli as the main ingredients except for the recipe from the state of Kelantan which contained white pepper instead of dried chilli. Small amounts of lemon grass, turmeric, salt, sugar, tamarind and shrimp paste were included in most recipes. Screening trials were conducted to study the effect of these ingredients on the acceptability of the product. The screening indicated that the flavour was affected mainly by the proportion of the main ingredients. Other minor ingredients can be kept at a certain constant level to produce a well-balanced flavour impact on the final product. Therefore, the ingredients were grouped into four components as discussed by Hare (1974) so that mixture design can be employed to derive the optimum formulation with minimum number of experimentation runs.

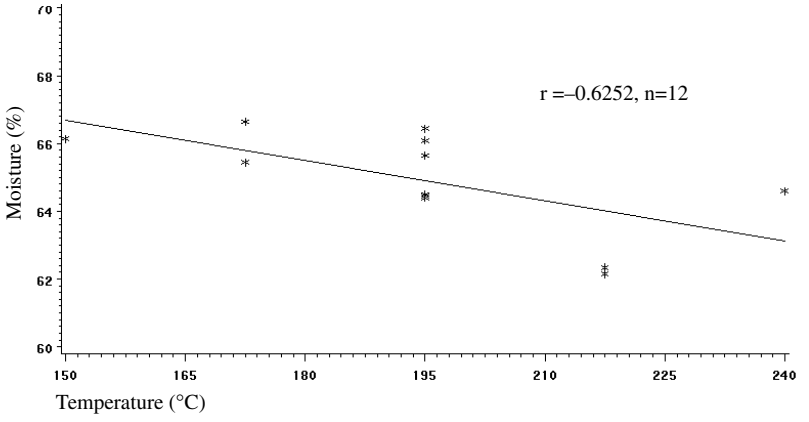


Figure 7. The effect of grilling temperature on moisture content in savoury grilled fish

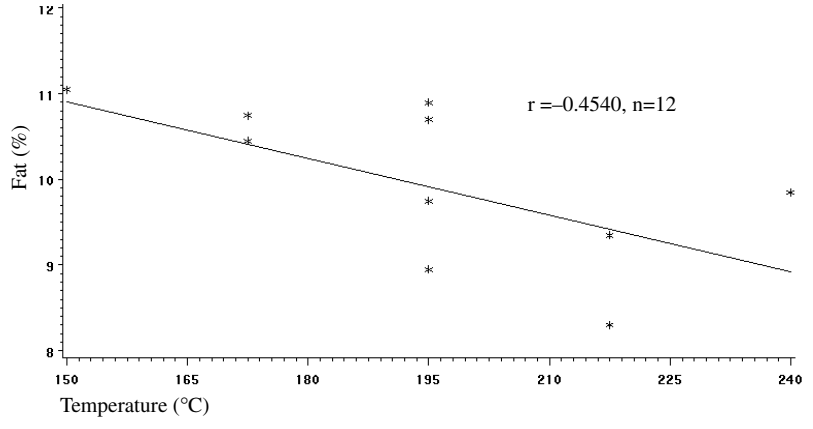


Figure 8. The effect of grilling temperature on fat content in savoury grilled fish

Table 10. Proximate and nutrient compositions in raw and processed black pomfret

Proximate composition	Composition in 100 g	
	Raw fish	Savoury grilled fish
Moisture	74.85 ± 2.1	64.3 ± 2.3
Protein (g)	20.16 ± 1.9	20.45 ± 0.07
Fat (g)	3.12 ± 2.01	10.05 ± 1.18
Carbohydrates (g)	N.A.	1.75 ±0.35
Crude fiber (g)	N.A.	0.4 ± 0.15
Ash (g)	N.A.	2.6 ± 0.28
Calcium (mg)	N.A.	37.45 ± 4.35
Phosphorous (mg)	N.A.	169.1 ± 5.3
Sodium (mg)	N.A.	537.7 ± 25.2
Potassium (mg)	N.A.	369 ± 4.0
Ascorbic acid (mg)	N.A.	2.8 ± 0.70

N.A. = not analysed

The results from the formulation experiment revealed a significant interaction between the four groups of ingredients namely water, coconut milk powder, onion and chilli, on all of the sensory attributes. Chilli seemed to be the most critical factor affecting the overall acceptability of 'ikan percik'. Formulations containing too low or too high chilli proportion were not acceptable. It is important to incorporate the right proportion of chilli in the formulation in order to maximize the product acceptance**.** The acceptability for pungency was optimal around 3–4% chilli level and decreased with further increased or decreased in the amount of chilli. Apart from flavour, chilli also influenced the colour acceptability of the product. The significance of ingredient interactions in protein mixtures was observed by Artega et al. (1993).

Optimization is a procedure for developing the best possible product with maximum overall acceptability (Sidel and Stone 1983). Overall acceptability was high at the lowest range of waterbase and highest range of coconut milk proportions *(Figure 4b* and *Figure 4c)*. Higher water proportion will dilute the impact of other ingredients on the sensory attributes and this was found to be unacceptable for a product like 'ikan percik'.

Contrary to the popular believe that 'ikan percik' tastes better with a high proportion of onion, results in *Figure 4a* showed that the overall acceptability decreased with the increase in onion proportion. Onions have a distinctive sweet sulphur-like flavour with a slight bitter aftertaste (Shankaranarayana et al. 1974). The decrease in overall acceptability with the increase in onion may be due to the bitter aftertaste.

Verification experimentation

Confirmatory experiment was carried out to validate the acceptability of the product made using the established optimum formulation as suggested by Shewfelt et al. (1997) and Hare (1974). Three treatments representing the optimum formulation (OPT), the second best formulation (FORM 2) and the best traditional formulation (TRAD) were used. Two other treatments OPT 20 and OPT 10 were included to explore the effect of fresh coconut milk of different fat levels on the product acceptability.

The results in *Table 6* confirm that products made using optimum formulation (OPT and OPT 20) were significantly superior to the others including the traditional formulation (TRAD) for colour, creaminess, pungency and overall taste. Both coconut milk powder and freshly pressed coconut milk may be used in the preparation of 'ikan percik' without affecting its acceptability. This finding has a practical importance for mass production of frozen 'ikan percik' since coconut milk powder is easier to handle compared to fresh coconut milk.

There was no significant difference (p <0.05) on perceived pungency intensity among samples. This was as expected since all treatments contained essentially the same level of chilli *(Table 2)*. Govindarajan et al. (1987) reported that the perceived pungency of chilli is linearly related to the total capsaicinoids content in product. However, the acceptability for pungency differed significantly (p <0.05) between samples of different fat contents *(Table 6)*. The fat content in the sauces for OPT and OPT 20, which received higher acceptability, was 50% higher than those in OPT 10 and FORM 2 *(Table 6* and *Table 7)*. Fat seemed to modify the effect of pungent flavour on panelists taste buds. Food of different fat contents is known to cause a generation of a totally different taste sensations by the same flavour compound (deRoos 1997). The fact that capsaicin is very soluble in lipids but sparingly soluble in water (Govindarajan 1986) may explain this effect. Similar observation was obtained in the formulation experimentation where samples with high chilli and high coconut milk were more

acceptable than those with high chilli and low coconut milk *(Figure 4c)*. Higher fat content in OPT and OPT 20 also seemed to have a positive effect on colour acceptability of the product. The red colour of 'ikan percik' is due to the present of fat soluble carotenoids, capsanthin in chilli (Francis 1985). The positive influence of fat on the perceived pungent flavour and colour of the product, as shown by this study may explain why traditional chilli-spiced foods like 'ikan percik' taste better with higher fat levels.

The results obtained in this study clearly show that constrained vertex mixture design is very useful in establishing the optimum formulation for traditional products which contain many types of ingredients. It helps to minimize the number of experiments to a manageable level.

Process optimization

A good 'ikan percik' is expected to have a well-grilled appearance and flavour, be well-coated with the sauce, reddish in colour and have a rich creamy savoury flavour. These sensory attributes were used to evaluate and determine the optimal grilling conditions. The grilling temperature/time combinations used in this study gave no significant effect on the coating thickness and colour of the product *(Table 8)*. The ANOVA showed that there was no significant difference in the 12 experimental runs for these variables. ln this experiment the viscosity of the sauce and the concentration of the red pigment in the sauce were the same for all treatment levels since the same sauce formulation was used for all experimental runs. The thickness of the sauce that adhered to the fish after grilling seemed to be influenced most by the viscosity of the sauce rather than the grilling conditions. The red carotenoids in chilli are fat soluble and heat stable, therefore the colour was not affected much by the grilling process.

Apart from appearance and flavour, texture was found to be an important attribute that affect the acceptability of a food product including fish (Sawyer et al. 1984; Prinyawiwathul et al. 1997). In this study textural attributes, juiciness and hardness, were used to elucidate the effect of grilling temperatures and times on the texture of 'ikan percik'. ANOVA for both variables showed significant difference among the samples with relatively high R^2 - value of 0.70 and 0.83 respectively in the fitted models *(Table 9)*.

Juiciness and hardness were defined as the perceived degree of oil and/or water in the sample and the perceived force required to break the sample with the molar teeth, respectively (Sawyer et al. 1984). Both textural attributes were significantly affected by the process conditions. The juiciness decreased and hardness increased as the grilling temperature and time increased. These textural changes occurred as a result of moisture loss during grilling *(Figure 7)*. The grilling conditions used for the preparation of 'ikan percik' has resulted in a moisture loss of about 14 *% (Table 10)*. The process has resulted in an increase of fat content in the final product.

Other researchers had reported a similar moisture loss from cooked fish muscle that resulted in the increase in fat content (Agren and Hanninen 1993; Bhattacharya et al. 1993). However, the increase in fat content in 'ikan percik' was mainly due to the outside coating rather than as a direct effect of moisture loss from the fish muscle. The initial fat content in the raw fish was lower than in the grilled product *(Table 10)*. Therefore, the higher fat content in the product at any temperature of grilling certainly come from the sauce adhering to the fish surface. The amount of fat in the product seemed to be related to the grilling temperature *(Figure 8).* Higher temperatures will result in lower fat content in the product.

The degree of doneness and the overall acceptability of the product had R^2 - value of 0.65 and 0.60 respectively in the fitted model. Degree of doneness and overall acceptability responses were higher for products grilled at higher temperatures

(>217 °C) and times (>25 min) even though products were acceptable at all time/ temperature combinations tested in this study. Panelists preferred the product to be slightly dry and hard as dictated by the higher overall acceptability score for products grilled at maximum temperatures (above 217 °C) and times (25 min or longer) used in this study. Therefore, based on the above observations the optimum range of processing conditions for 'ikan percik' was established to be at 220–240 °C for 25–30 min.

Conclusion

The results obtained in this study showed that constrained vertex mixture design was very useful in determining the optimum formulation for a traditional product which contained multi-ingredients like 'ikan percik'. Careful selection of variables affecting the product quality is essential to ensure meaningful results.

The sample made from the optimum formulation had been proven to be significantly superior to other samples. The grilling process was improved using an electric griller instead of traditional method to produce uniform product. The process was optimized at 220–240 °C for 25–30 min. The optimum product grilled at 240 °C for 25 min contained 64.3% moisture, 20.4% protein and 10% fat.

This study showed that frozen 'ikan percik' was well-accepted by the taste panelists. Therefore, with the available standard formulation and mechanized grilling process, mass production of 'ikan percik' is possible.

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