Thesis for the Degree of Master of Fisheries Science

Process Optimization and Quality Evaluation of Gwamegi from Pacific Saury (*Cololabis saira*) using Response Surface Methodology

by

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KOICA-PKNU International Graduate Program of Fisheries Science

Graduate School of Global Fisheries

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February 2017

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반응표면법을 이용한 과메기 제조의 최적화와 품질평가

Advisor: Prof. Yang Bong Lee

by

Neema Idda Respickius

A thesis submitted in partial fulfilment of the requirements for the degree of

Master of Fisheries Science

in KOICA-PKNU International Graduate Program of Fisheries Science,
Graduate School of Global Fisheries
Pukyong National University

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February 24, 2017

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Abstract

Drying- freezing process and quality evaluation of Gwamegi from Pacific Saury was optimized by using response surface methodology. A fractional 3-level-3-factor experimental design which consisted of 15 experimental treatments and three replicates at the center points, was adopted to see the effect of such independent variables as drying cycles (4-12 numbers), drying temperature (5-15 °C) and drying time (3-5 hours) on the responses of moisture content (%), weight loss (%), volatile basic nitrogen (mg/100g) and trimethyl amine. Data were analyzed using SAS statistical software and a second-order polynomial models were obtained for predicting the response. Also, the 3-dimensional graphs were drawn with Maple 8 software. From regression analyses, the coefficient of regressions (R^2) for moisture content, weight loss, VBN and TMA were 0.9519, 0.8578, 0.9691 and 0.8230 respectively, showing that moisture content and VBN fits better in a model. The minimum and maximum values of moisture content, weight loss, VBN and TMA ranged from 27.73 ± 0.39% to 50.45 ± 0.64%, 22.98 ± 0.94% to 52.49±0.58%, 6.3 ± 0 to 16.8 ± 1.98 mg/100g and 40 ± 3.67 to 117.85± 3.34 respectively. The optimal drying conditions are temperature of 15.6 °C, time of 5.1 hours and drying cycles of number 12. Drying temperature has the most significant effect followed by drying cycle and drying time. These results provide us with the basic information required to control qualities of Gwamegi and other dried fish and fishery products.

Keywords: Gwamegi, VBN, TMA, GC-FID, response surface methodology

1 . Introduction

Pacific saury (*Cololabis saira*) is the small pelagic fat-rich fish. It is consumed when it is semidried, dried or fried in Korea. Most of Korean people prefer to consume when it is semi dried. Semi-dried product from Pacific saury is called Gwamegi. Gwamegi is produced by naturally drying fish in the shade using sea winds during winter period for about 15 days to reach a moisture content of 30-45% (Kang et al, 2015). The process was performed by repetitive freezing during night time and defrosting during daytime. The production sites of commercial Gwamegi are located in the east coastal area of Korea, including Pohang City. There are two types of Gwamegi which are produced recently skinned and unskinned Gwamegi.

Gwamegi has higher nutritional value and exceptional flavor, and its consumption is steadily increasing. Not only producing semi dried fish “principally Gwamegi” is to get a shelf stable product but also to promote important sensory changes. While the product is being dried, the tissues of Gwamegi mature to produce an exceptional flavor with a high level of unsaturated fatty acids. The product has been shown to be effective in preventing adult diseases like arteriosclerosis and hypertension since it contains a lot of Eicosapentaenoic acid (EPA) and Docosahexaenoic acid (DHA), which is effective in expanding blood vessels, restraining the cohesion of a blood platelet, and lowering blood pressure as reported by (Kang et al, 2011). It is currently being used as a good tool for food therapy and source of therapeutic substances for the treatment of coronary diseases, auto-immune diseases, and anaemia and protein energy malnutrition (Nahid et al., 2014). It is also an important source of iron, calcium, iodine, potassium, vitamin A, vitamin B2, vitamin B6, poly-unsaturated fatty acids, other minerals, vitamins, micronutrients (Holma and Maalekuu, 2013; Mohanty et al., 2013).

The quality of Gwamegi product in different drying method is different. The natural drying method, though important for preservation have various effects on the sensory and nutritional quality of Gwamegi because it has been observed that different processing and drying methods have different effects on the nutritional compositions of fish (Oparaku and Mgbenka, 2012). The fish can be dried to various degrees with moisture levels in the final product ranging from about 10% to 60%. Processing temperatures may range from less than 5°C to up to 120°C and processing times from half an hour to several months. The fish may be dried only or smoked only or there may be a combination of smoking and drying (Chukwu and Shaba, 2009). Oven drying is effective as preservative method since it reduces the water activity of fish muscle; consequently inhibit bacterial growth hence prolongs the shelflife of the fish. It also needs low initial investment cost, protects the fish from dust, microorganisms and does not depend on environmental condition. On the other hand, oven drying method is safer and faster than the natural drying method (Aberoumand and Karim, 2015).

The natural drying method, in which Gwamegi is dried during winter time to reach that moisture content has a lot of problems to the final product. It is time consuming since the products dried for a long period due to the drying speed to be very slow as a result change the quality of the product like imparting bad smell (fishy or rancid) and colour change. Due to the fact that the process is done in an open air it contaminates the product by microbes. Not only deteriorate the quality of the product by accelerates the multiplication of pathogenic microorganism inflow during manufacturing processes, but also facilitates the growth of microorganisms during rainy days since this method is weather dependent. Kang et al, 2011 reported that many food safety problems may arise since biogenic amine is excessively generated due to poor processing and distribution conditions of that product. Also other researchers did studies on Gwamegi focus on effects of drying conditions on biogenic amines and lipid oxidation (Shim et al., 2011); fatty acid composition, total amino acid and mineral content (Yoon et al., 2010), the nutrient composition of commercial Gwamegi admixed with functional ingredients (Jang et al., 2010), general composition, rheometric and microbiological changes on the storage temperature and duration (Lee at al., 2008) and processing and characteristics of canned (Park et al., 2012).

By application of response surface methodology (RSM), which shows the relationship between dependent variables and independent variables, has been used to determine the optimum extraction conditions for bioactive components (Edwards and Jutan, 1997). Using RSM, Cho et al. (2004, 2005) studied the optimization of gelatin extraction from shark (Isurus oxyrinchus) cartilage and yellowfin tuna skin. These studies demonstrated that RSM is an effective tool for investigating the optimum extraction conditions for producing gelatin. The main advantage of RSM is its ability to reduce the number of experimental trials needed to assess multiple parameters and their interactions to provide sufficient information for statistical acceptable result (Lee et al., 2009). Therefore, the objective of this study was to optimize the drying-freezing processing conditions of Gwamegi product using response surface methodology and to determine the effect of these conditions on the quality of Gwamegi product

2 . Materials and Methods

2.1. Fish collection and preparation

Pacific sauries (*Cololabis saira*) were purchased from a local Fish market in Busan, South Korea. The average weight of fish ranges from 85.5g to 132.7g. Fish samples were de-scaled, de-gutted, cut into halve to remove the backbone (jaws) in order to remain with the fish fillet and the head were removed. The prepared fishes were washed, drained and stored in the freezer under -20°C for 25 days ready for drying process.

2.2. Experimental design

Response surface methodology (RSM) was employed for optimizing the processing conditions on Gwamegi product. A fractional 3-level-3-factor experimental design which consisted of 15 experimental treatments and three replicates at the center points was adopted to see the effect of independent variables on the responses. The drying-freezing cycle (4, 8, and 12 numbers), drying time (3, 4, and 5 hours) and drying temperature (5, 10, and 15°C) were used as independent variables. The selected responses were weight loss (%, Y_1), moisture content (%, Y_2), volatile basic nitrogen (VBN) content (mg%, Y_3) and trimethyl amine (TMA) content (%, Y_4).

Table 1. Independent variables of the process and their corresponding levels

Independent variables	**Symbol**		**Level**		
	Uncoded	**Coded**	**-1**	**0**	**1**
Drying Cycle (no.)	X_1	x_1	4	8	12
Drying Time (hrs)	X_2	x_2	3	4	5
Temperature (°C)	X_3	x_3	5	10	15

Table 2. Experimental coded and uncoded values utilized for the full-factorial design in our experiment

Run no.	Coded			Uncoded		
	x_1	x_2	x_3	X_1	X_2	X_3
1	1	1	0	12	5	10
2	1	-1	0	12	3	10
3	-1	1	0	4	5	10
4	-1	-1	0	4	3	10
5	1	0	1	12	4	15
6	1	0	-1	12	4	5
7	-1	0	1	4	4	15
8	-1	0	-1	4	4	5
9	0	1	1	8	5	15
10	0	1	-1	8	5	5
11	0	-1	1	8	3	15
12	0	-1	-1	8	3	5
13	0	0	0	8	4	10
14	0	0	0	8	4	10
15	0	0	0	8	4	10

2.3. Determination of moisture content

To determine moisture content, 5g of the sample was dried in an oven at a temperature of 105°C for 10 hours to reach constant weight. The difference weight before and after is the water evaporated during drying time, and percentage of moisture content was calculated with this formula:

$$\%\text{Moisture content} = ((W_b - W_a) / W_a) \times 100)$$

Where

Wb is the weight of sample before drying operation

Wa is the weight of sample after drying operation.

2.4. Determination of weight loss

To determine the weight loss, the sample was measured before and after dried for a specified condition in a specified cycle. The difference weight was then calculated as percentage weight loss with this formula:

$$\%\text{Weight loss} = ((W_b - W_a) / W_a) \times 100)$$

Where

Wb is the weight of sample before drying operation

Wa is the weight of sample after drying operation

2.5. Determination of trimethylamine

Sample was grinded to make a test liquid solution. 50ml of water and 40g of NaOH was prepared by mixing it together. 4ml of the test liquid from sample and 3ml of NaOH were weighed and mix into vial bottle. The mixed liquid was then incubated into oven for 7min at 10°C. The 200μL of the gas was taken on the headspace by syringe and injected to the Gas Chromatography Flame Ionized Detector (GC-FID). The peak area for TMA was drawn after 40min by the computer.

2.6. Determination of volatile basic nitrogen

This determination involves making test liquid, diffusion and titration process. 10g of sample was respectively added in a beaker with 50ml of distilled water then extracted for 30min. The mixture was filtrated and makes it to 100ml with the distilled water. The diffusion and titration reaction was carried out where by 1ml of test liquid was poured in the bottom of outer well (B) and 1ml of 0.01N-H_2SO_4 in inner well (C).of the conway unit. Vaseline gel was applied at contact part of cover plate and outer well. The cover plate was closed to about 2/3, and 1ml of K_2CO_3 saturated solution added in inner well (A). Then rapidly close the cover plate, and fix it by clip. The conway unit was shake to mix outer well's solution equally and leave it at 25℃ for

60min. The cover plate was opened and 1 drop of Brunswik indicator added in the inner well (C). Titrate by 0.01N-NaOH using micro burette.until the colour change to green. The titration was repeated 2 times. Then VBN was calculated by using the formula:

VBN (mg/100g) = 0.14 × (b-a) × F/W × 100 × D

Where:

W is sample's quantity

F is Factor of 0.01N-NaOH

D is Dilution rate

0.14 is Amount of VBN (mg) to 0.01N-H_2SO_4 1ml

a is blank amount

b is the titration amount

2.7. Statistical analysis

All experiments were carried out in duplicate. Regression analysis and analysis of variance (ANOVA) were performed using statistical analysis SAS software program (version 9.4) to observe the statistical significance at 95% confidence interval i.e. P≤0.05. ANOVA tables were obtained and the effects of regression coefficients of individual linear and quadratic were determined. Data obtained were fitted to the second-order polynomial equation Then, the three dimensional figures were drawn with Maple 8 software program.

3 . Results and Discussion

The experimental values and analysis of variance for response variables on three independent variables from 15 experimental treatments of Box-Behnken design for response surface methodology were presented in table 2. The data were analysed statistically to examine the optimum condition for processing Gwamegi and the results of each variable are presented here under.

3.1. Moisture content of drying processing of Gwamegi

From table 4, it was observed that the mean moisture content was 38.22% with the coefficient of regression (R^2) in the value of 0.9519.This results indicates the model fitted better to that equation The highest value of moisture content was 50.45±0.64% found in the sample dried for 4 hours at a temperature of 5°C for 8 drying cycles. The lowest value of moisture content was 27.73±0.39% found in the sample dried for 4 hours at a temperature of 15°C for 12 drying cycles (table 3).The moisture content value in this study was in the range of that reported by Shah et al, 2013 which is 20.05% to 65.08%.on herring fillet. Moisture content in the herring fillet decreased significantly throughout the drying period. According to that study decrease in moisture content can be explained by increased dehydration of herring fillet during the drying period.

The analysis of variance (ANOVA) results of moisture content confirms the satisfactoriness of the statistical model on drying temperature and drying cycles since their probability > F values are less than 0.05 and statistically significant at 95% confidence interval. The result shows that the drying temperature has the most significant effect followed by drying cycle and drying time.

The multiple polynomial regression analysis result was used to fit the data into the following second-order equation of moisture content as follows:

$$MC\ (\%) = 68.69-0.57438*X_1-0.6875*X_2-2.5725*X_3+0.00875*X_1^2-0.12875*X_1*X_2-0.055*X_2^2-0.00125*X_1*X_3-.0178*X_2*X_3+0.0686*X_3^2$$

Independent variable $\mathbf{X_1}$, $\mathbf{X_2}$ and $\mathbf{X_3}$ denotes drying cycles (numbers), time (hrs) and temperature (°C) respectively.

From the formula obtained in Table 5, the three dimensional figures were drawn using Maple 8 which shows the relationship between two factors and one factor fixed at the centre value.

Fig. 1 is three dimensional figure which shows the effect of drying temperature and drying time at the fixed value of drying cycle number 8 on moisture content. As shown in fig 1, there is strong inversely relationship between moisture content on both time and temperature. As temperature and time increases, moisture content decreases. This decrease in moisture content during drying by temperature is due to removal of water by evaporation from the fish sample. It means that the lower value of moisture content is appeared in the higher value of temperature and time. This present study revealed that, the moisture content was more affected by temperature compared to the drying time. The drying temperature was in the value of 15.2°C when the moisture content is 35%.and time was in the value of 5.1 hour when the moisture content is 44 %. Since the temperature has the more effect on the drying of Gwamegi, to control it is very important. It means the temperature is not supposed to go beyond 15.2°C when drying Gwamegi in order to obtain the semi dried product. If the temperature will go beyond that amount you will produce a Gwamegi which is very hard. Similar studies were explained by Oladele and Odedeji, 2008, Idah and Nwamkwo, 2013 on moisture content of catfish steaks The effect of moisture content on drying rate was also studied by Modibbo et al, 2014 and revealed that as drying time increases, moisture content decreases and drying will therefore proceed more rapidly with temperature increase. This shows that the heat radiation penetration required to drying the fish sample. Similar observation has been reported in the effect of smoke-drying temperature and time done by Idah and Nwamkwo, 2013 that, moisture content decreases as temperature and time increases.

Fig. 2 shows the relationship between temperature and drying cycle on moisture content at a fixed drying time of 4 hours. According to fig 1, as moisture content decreases both drying cycle and temperature increases. At the temperature value of

15.2°C the moisture content was 38% and at drying cycle of number 12 the moisture content was 44%. This trend of result was similar to that observed from the effect of time and temperature on moisture content found in fig 1. This might be due to dehydration of that Gwamegi by evaporation as number of drying cycle's increases as a result the moisture content decreases. The findings of this study are also in agreement with Mohamed et al. (2011) who reported that, as moisture content decreases the number of drying cycles increases. From that study the moisture content reduced from74.99% to 18.511% of fish fillet from Tilapia. In other study Rowland et al. (2009), reported that, the higher the temperature, the lower the moisture content.

Fig 3 presents the effect of time and drying cycles on moisture content at the fixed drying temperature of 10° C. As depicted in Fig 3 there is negative relationship between the time and drying cycles on moisture content. It means that as moisture content decreases both time and drying cycles increases. The effect of drying cycle on moisture content is more compared to the effect of drying cycle as observed in this study. The moisture content was observed to the value of 39% when the drying time was 5.1 hours and drying cycle was number 12 when the moisture content is 38%. In drying process of Gwamegi, the drying cycle is very important to indicate that the product is ready for consumption and it attain the required moisture content. This means that the more the number of drying cycles the better the Gwamegi. The same phenomenon of drying time also observed from fig. 1.

Table 3. Results of moisture content of the drying process of Gwamegi

Treatment	Variation Levels			Response function
	X_1	**X_2**	**X_3**	**Moisture content (%)**
1	12	5	10	32.07±0.10
2	12	3	10	38.09±0.13
3	4	5	10	37.63±0.53
4	4	3	10	41.59±0.58
5	12	4	15	27.73±0.39
6	12	4	5	39.64±0.51
7	4	4	15	38.64±0.91
8	4	4	5	50.45±0.64
9	8	5	15	29.82±0.26
10	8	5	5	43.70±0.43
11	8	3	15	34.31±0.44
12	8	3	5	47.85±0.22
13	8	4	10	38.23±0.33
14	8	4	10	35.80±0.29
15	8	4	10	37.75±0.35

Values are mean ± SD of duplicate analyses

X_1, **X_2** and **X_3** denotes drying cycles (no.), time (hrs) and temperature (°C) respectively.

Table 4. Result of statistical analyses for moisture content

Response surface for variable of moisture content	
Response mean	38.220000
Root MSE	2.248677
R-square	0.9519
Coefficient of variation	5.8835

Table 5. Coefficient regression model estimated by multiple regression analysis for moisture content

Parameter	**DF**	**Estimate**	**Standard error**	**t Value**	**Pr > |t|**	**Parameter estimate from coded data**
Intercept	1	68.69	24.1493	2.84	0.0361	37.26
C	1	-0.57438	1.728918	-0.33	0.7532	-3.8475
Tm	1	-0.6875	9.919276	-0.07	0.9474	-2.3275
T	1	-2.5725	1.383135	-1.86	0.122	-6.3925
C*C	1	0.00875	0.073141	0.12	0.9094	0.14
Tm*C	1	-0.12875	0.281085	-0.46	0.6661	-0.515
Tm*Tm	1	-0.055	1.170249	-0.05	0.9643	-0.055
T*C	1	-0.00125	0.056217	-0.02	0.9831	-0.025
T*Tm	1	-0.017	0.224887	-0.08	0.9427	-0.085
T*T	1	0.0686	0.04681	1.47	0.2027	1.715

DF, C, Tm, and T denote; degree of freedom, drying cycles, drying time and drying temperature, respectively.

The values are the estimated value of the statistical result of SAS

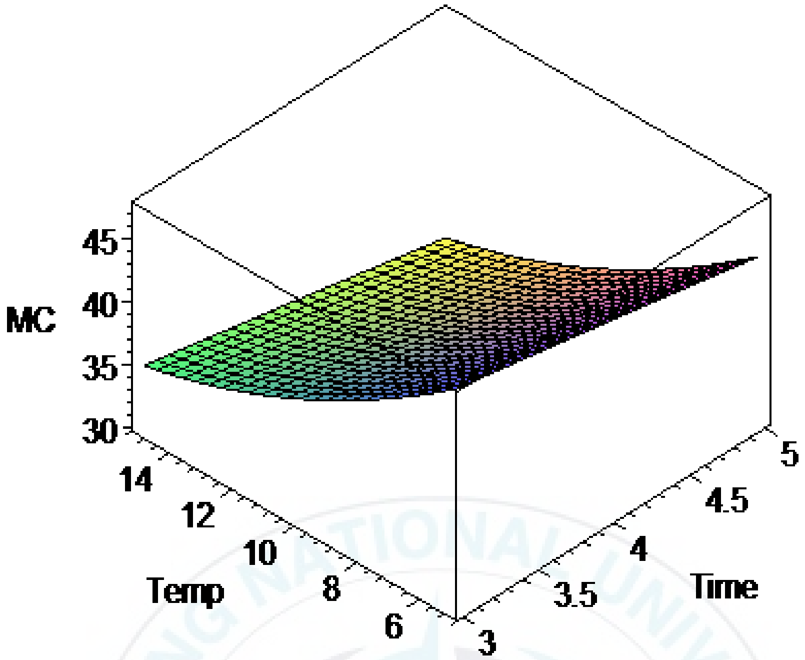

Figure 1. Relationship between temperature (°C) and time (hrs) on moisture content (%) of Gwamegi product.

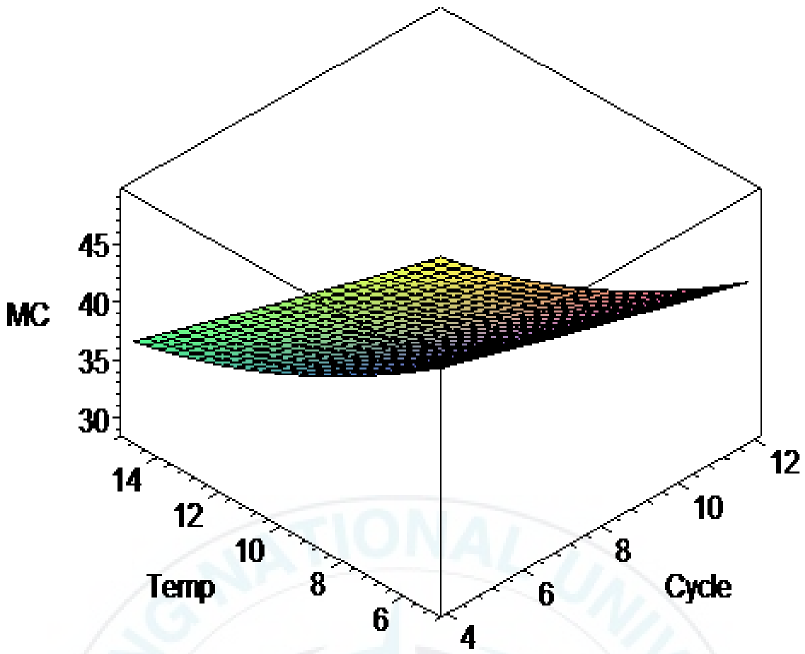

Figure 2. Relationship between temperature (°C) and cycle (no.) on moisture content (%) of Gwamegi product.

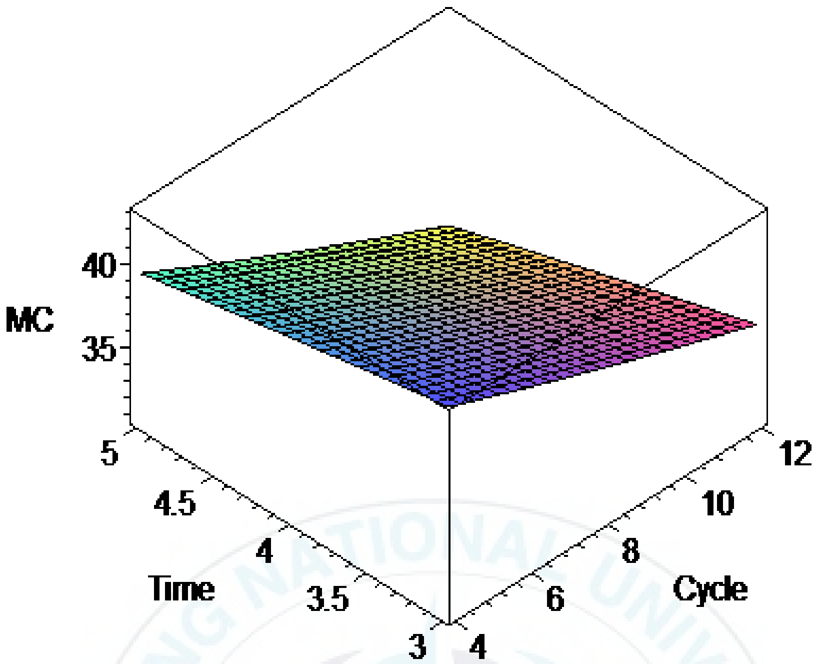

Figure 3. Relationship between time (hrs) and cycle (no.) on moisture content (%) of Gwamegi product.

3.2. Weight loss of drying processing of Gwamegi

The mean weight loss was 35.01% with the coefficient of regression (R^2) in the value of 0.8578 as shown in table 7. The highest value of weight loss was 52.49±0.58% found in the sample dried for 4 hours at a temperature of 15°C for 12 drying cycles. The lowest value of weight loss is 22.98±0.94% found in the sample dried for 4 hours at a temperature of 5°C for 4 drying cycles (Table 6). From table 8, the formula was made as:

$$WL(\%)=57.888333+3.108542*X_1-10.466667*X_2-7.092667*X_3-0.241042*X_1^2+0.582500*X_1*X_2+1.003333*X_2^2+0.0595*X_1*X_3+0.123*X_2*X_3+0.331933*X_3^2$$

Where;

X_1, **X_2**, and **X_3** denotes drying cycles (number), time (hrs) and temperature (°C) respectively.

The above formula was used to show the effect of two different factors with one factor fixed at the central value on weight loss then three dimensional figure drawn on Maple 8. The analysis of variance (ANOVA) on weight loss has no significant effect on both factors i.e drying cycle, drying time and temperature since their probability > F values are greater than 0.05 at 95% confidence interval. This means that the interaction effects of both factors were not significant.

Fig. 4 presents the effect of temperature and time on weight loss at a fixed value of drying cycle number 8.As shown from fig. 4, the relationship of weight loss on those factors is a minimum shape. There is direct relationship between time and temperature on weight loss. As weight loss increases the time and temperature increases too. The weight loss increases gradually at start then reaches minimum point at temperature of 10°C. From that point it increases rapidly at increasing rate. It means the higher the value of weight loss the higher the value of temperature and vice versa. Same trend also was observed from time, as weight loss increases time increases too. The results of this study agree with Mustapha et al. (2014) who demonstrated that weight loss is the result of the moisture reduction with time in a sample of *C.gariepinus*. A progressive decrease in weight of *C gariepinus* was recorded from the first day of drying until a constant weight loss was recorded. The rate of weight loss was faster in the first 4 days of drying, but slowed down after wards till a weight was recorded as observed from *C gariepinus.*

Fig. 5 shows the effect of temperature and drying cycle on weight loss at the fixed value of drying time of 4 hours. As depicted in fig.5, the weight loss and drying cycle show the direct relationship means that as weight loss increases the drying cycle and temperature increases. At the same time the drying cycle show the more effect on weight loss compared to temperature. Same trend was observed too in fig. 4 on the temperature side. But there is a limited study done on weight loss and drying cycle during drying process of fish. Many authors Kaymak-Ertekin and Sultanogla, 2000 reported that there is an increase of water loss with increase in temperature but not the weight loss.

Fig. 6 presents the effect of time and drying cycle on weight loss at the fixed value of drying temperature at 10°C. As shown in this Fig. 6, the weight loss has the positive relationship with time and drying cycle. It means as weight loss increases both drying cycle and time tend to increase too and vice versa. The weight loss was observed to the value of 22% when the drying time was 5.1 hours and drying cycle was number 12 when the moisture content is 40%. In this relationship drying cycle also show similar phenomenon as in fig. 5.

This study revealed that, drying cycle is also an important factor to consider in the drying process since it can act as an indicator for the product example in the case of Gwamegi product.

Table 6. Results f weight loss of the drying process of Gwamegi

Treatment	Variation levels			Response function
	X_1	**X_2**	**X_3**	**Weight loss (%)**
1	12	5	10	37.75±2.36
2	12	3	10	29.29±0.19
3	4	5	10	24.55±1.72
4	4	3	10	25.41±0.36
5	12	4	15	52.49±0.58
6	12	4	5	46.89±1.98
7	4	4	15	23.82±0.04
8	4	4	5	22.98±0.94
9	8	5	15	50.55±0.61
10	8	5	5	42.26±1.91
11	8	3	15	39.32±0.18
12	8	3	5	33.49±2.81
13	8	4	10	32.03±1.41
14	8	4	10	32.68±0.62
15	8	4	10	31.60±0.68

Values are mean ± SD of duplicate analyses

X_1, **X_2** and **X_3** denotes drying cycles (numbers), time (hrs) and temperature (°C) respectively.

Table 7. Result of statistical analysis for weight loss

Response surface for variable of weight loss	
Response mean	35.007333
Root MSE	6.078081
R-square	0.8578
Coefficient of variation	17.3623

Table 8. Coefficient regression model estimated by multiple regression analysis for weight loss

Parameter	**DF**	**Estimate**	**Standard Error**		**Pr>| t|**	**Parameter Estimate from Coded Data**
Intercept	1	57.888333	65.27448	0.89	0.4158	32.103333
C	1	3.108542	4.673194	0.67	0.5354	8.7075
Tm	1	-10.466667	26.811386	-0.39	0.7123	3.45
T	1	-7.092667	3.738555	-1.90	0.1163	2.57
C*C	1	-0.241042	0.197696	-1.22	0.2771	-3.856667
Tm*C	1	0.5825	0.75976	0.77	0.4779	2.33
Tm*Tm	1	1.003333	3.163133	0.32	0.7639	1.003333
T*C	1	0.0595	0.151952	0.39	0.7115	1.19
T*Tm	1	0.123	0.607808	0.20	0.8476	0.615
T*T	1	0.331933	0.126525	2.62	0.0469	8.298333

DF, C, Tm, and T mean degree of freedom, drying cycles, drying time and drying temperature, respectively.

The values are the estimated value of the statistical result of SAS

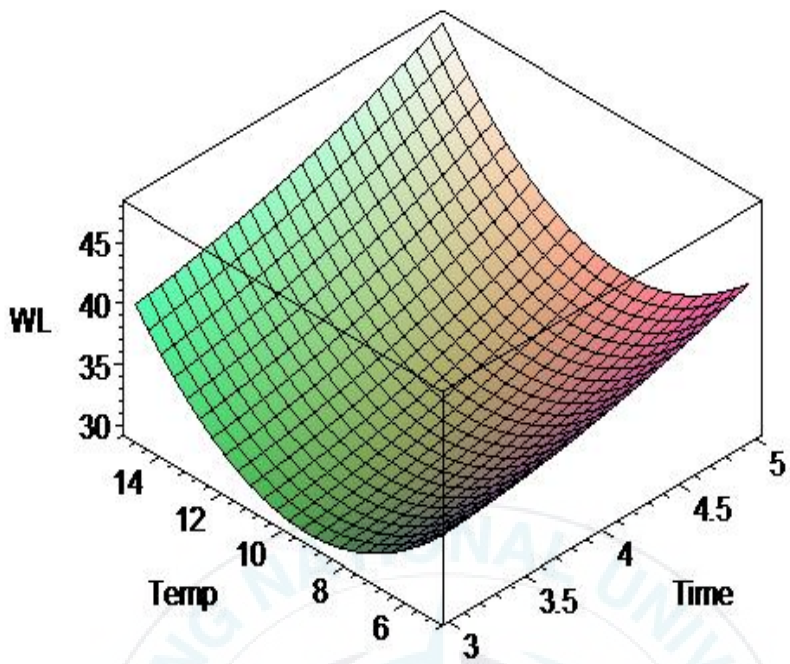

Figure 4. Relationship between temperature (°C) and time (hrs) on weight loss (%) of Gwamegi product.

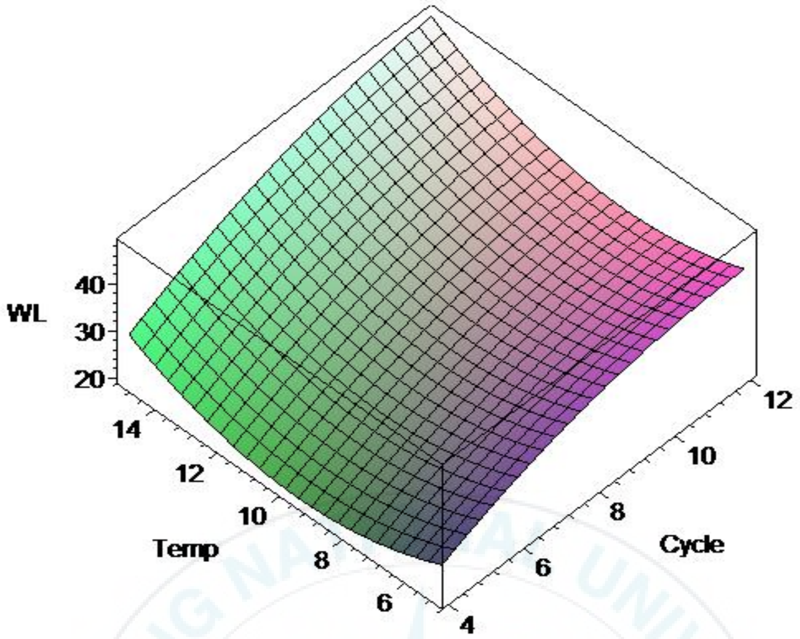

Figure 5. Relationship between temperature (°C) and cycle (no.) on weight loss (%) of Gwamegi product.

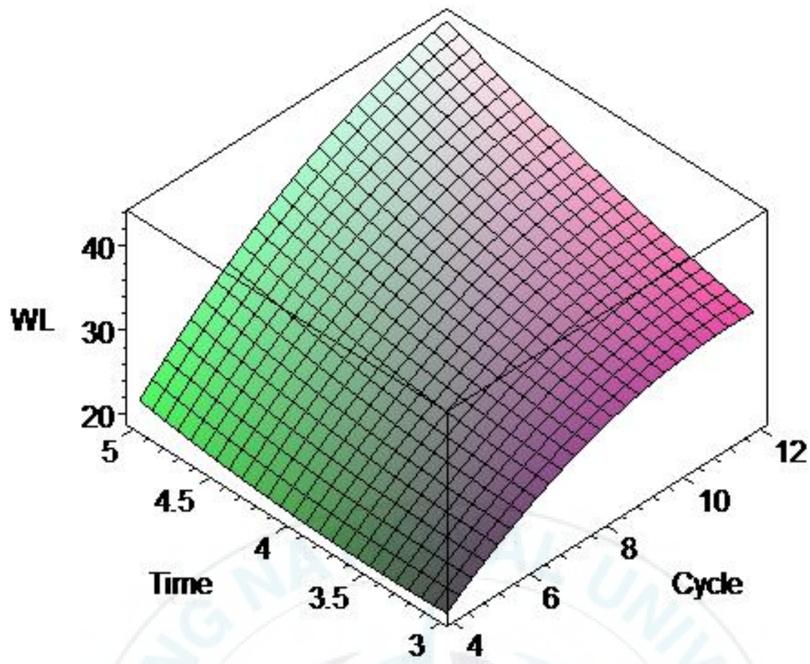

Figure 6. Relationship between time (hours) and cycle (no.) on weight loss (%) of Gwamegi product.

3.3. Volatile basic nitrogen of drying processing of Gwamegi

Table 9 shows the VBN values calculated from the data obtained on titration process. The VBN concentration ranges from 6.30±0 to 16.80±1.98 mg/100g. The range of VBN concentration obtained from this study is in agreement with the one reported by Muhammet and Sevim, 2007 of freshly caught fish which varied between 5 and 20 mg/100 g. It gives us an indication that Gwamegi product is fresh and can be consumed without any problem. The data was subjected to SAS and the results are as follows. From ANOVA the coefficient of determination (R^2) was 0.9691 and coefficient of variation was 8.5516.as shown in table 10. These results signify good precision and consistency of the experiment. The significance of each coefficient is determined by F value and probability > F value. The estimates data from table 11 was used to fit the second-order polynomial equation model of multiple regression analysis as shown below:

$$VBN(mg/100g):=57.166667-2.522917*X_1-8.983333*X_2-0.748333*X_3+0.133073*X_1^2-0.13125*X_1*X_2+0.729167*X_2^2+0.13125*X_1*X_3+0.385*X_2*X3+0.113167*X_3^2$$

Where by independent variables $\mathbf{X_1}$, $\mathbf{X_2}$ and $\mathbf{X_3}$ denotes drying cycle (number), time (hrs) and temperature (°C) respectively.

Fig. 7 is three dimension figure which show the relationship between temperature and time on VBN at the fixed value of drying cycle number 8. As shown in fig. 7, there was direct relationship between temperature and time on VBN with a minimum phenomenon. The drying temperature was in the value of 15.2°C when the VBN concentration is 9.2 mg/100g.and time was in the value of 5.1 hour when the VBN concentration is 11 mg/100g. Temperature and time increases rapidly as VBN increases. Similar observations were explained by Gwak and Eun, (2010) on Gulbi a traditional Korean food as the temperature and time increases VBN values also increases. Also Gulsun et al., 2009 reported that, VBN values increased according to time of storage. The temperature has the more effect on VBN compared to time. This might be due to the biochemical decomposition of the nitrogenous compounds with the effect of temperature increase.

Fig. 8 shows the effect of time and drying cycle on VBN at a fixed temperature of 10°C. There was a positive relationship between time and drying cycle on VBN. This observation agree with the findings of Mohamed et al, (2011) who studied the Coral trout (*Plectropomusspp)* fish and found that the VBN increases from 7.7mg/100g to 29.4 mg/100g after the time interval of 25 days. The highest value of VBN was observed when the interval time was increased at 40 days (37.8 mg/100g). But from this study the VBN concentration was observed to the value of 10 mg/100g when the drying time was 5.1 hours and drying cycle was number 12.

Fig. 9 shows the relationship between temperature and drying cycle on VBN at a fixed time of 4 hours. There was a positive relationship between temperature and drying cycle on VBN. It means as the temperature and drying cycles increases the

VBN also increases. These results coincide with the study done by Mohamed et al., 2011 who found that the VBN values were higher in the dried samples of Tilapia fish fillets. According to him the difference is due to partial dehydration of dried samples and subsequent concentration of VBN constituents.

Table 9. Results of volatile basic nitrogen for drying process of Gwamegi

	Variation Levels			**Response function**
Treatment	X_1	X_2	X_3	VBN (mg /100g)
1	12	5	10	10.50±0.99
2	12	3	10	11.20±0.99
3	4	5	10	9.10±0.99
4	4	3	10	7.70±0.99
5	12	4	15	16.80±1.98
6	12	4	5	10.50±0.99
7	4	4	15	7.70±1.98
8	4	4	5	11.90±0.99
9	8	5	15	11.90±0.99
10	8	5	5	7.00±0
11	8	3	15	9.80±1.99
12	8	3	5	12.60±0.99
13	8	4	10	7.00±0.99
14	8	4	10	7.00±1.98
15	8	4	10	6.30±0

Values are mean ± SD of duplicate analyses

X_1, **X_2** and **X_3** denotes drying cycles (no.), time (hrs) and temperature (°C) respectively.

Table 10. Result of statistical analysis for volatile basic nitrogen

Response surface for variable of volatile basic nitrogen	
Response mean	9.800000
Root MSE	0.838053
R-square	0.9691
Coefficient of variation	8.5516

Table 11. Coefficient regression model estimated by multiple regression analysis for volatile basic nitrogen

Parameter	DF	Estimate	Standard Error	t value	Pr>| t|	Parameter Estimate from Coded Data
Intercept	1	57.166667	9.000136	6.35	0.0014	6.766667
C	1	-2.522917	0.644346	-3.92	0.0112	1.575000
Tm	1	-8.983333	3.696787	-2.43	0.0594	-0.350000
T	1	-4.748333	0.515477	-9.21	0.0003	0.525000
C*C	1	0.133073	0.027259	4.88	0.0045	-2.129167
Tm*C	1	-0.131250	0.104757	-1.25	0.2656	-0.525000
Tm*Tm	1	0.729167	0.436137	1.67	0.1554	0.729167
T*C	1	0.131250	0.020951	6.26	0.0015	2.625000
T*Tm	1	0.385000	0.083805	4.59	0.0059	1.925000
T*T	1	0.113167	0.017445	6.49	0.0013	2.829167

DF, C, Tm, and T mean degree of freedom, drying cycles, drying time and drying temperature, respectively.

The values are the estimated value of the statistical result of SAS

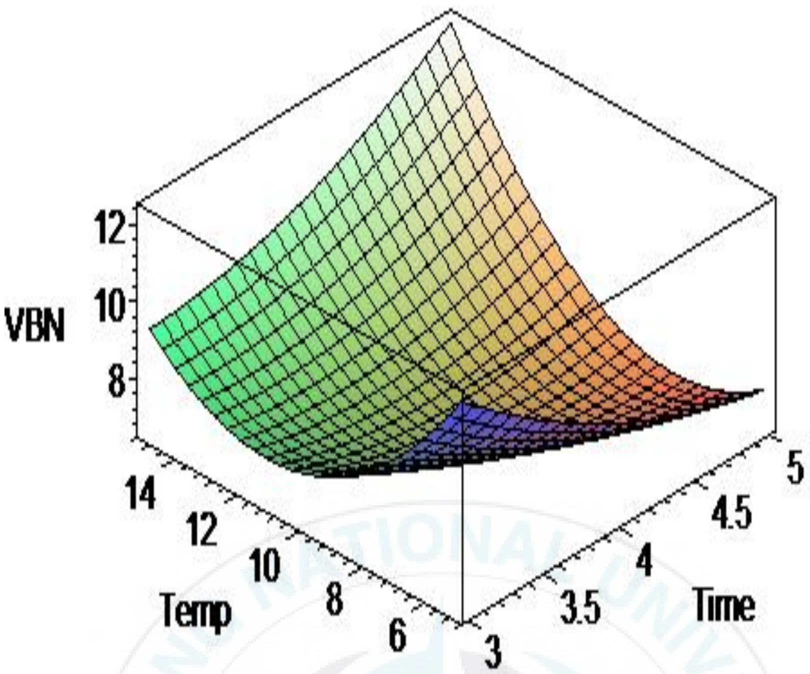

Figure 7. Relationship between temperature (°C) and time (min.) on VBN of Gwamegi product.

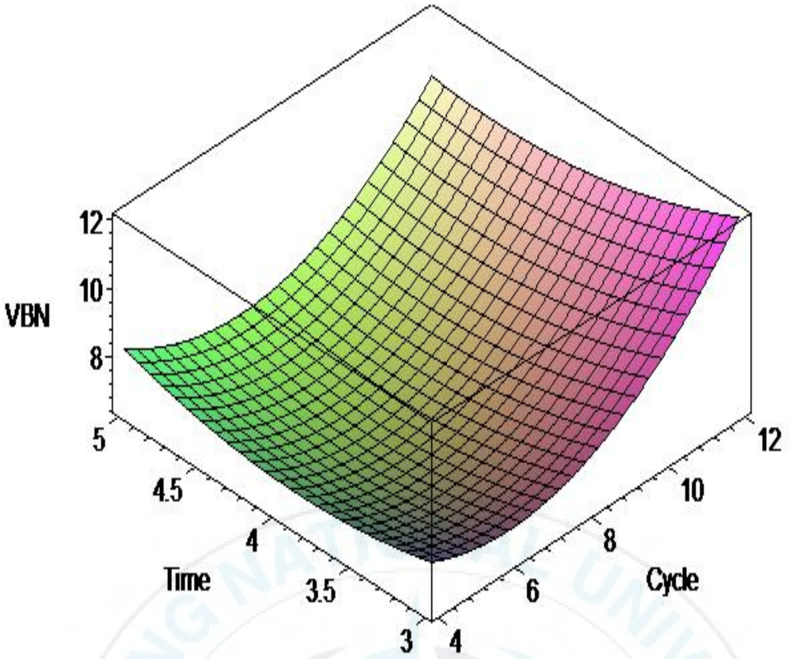

Figure 8. Relationship between time (min.) and drying cycle (no.) on VBN of Gwamegi product.

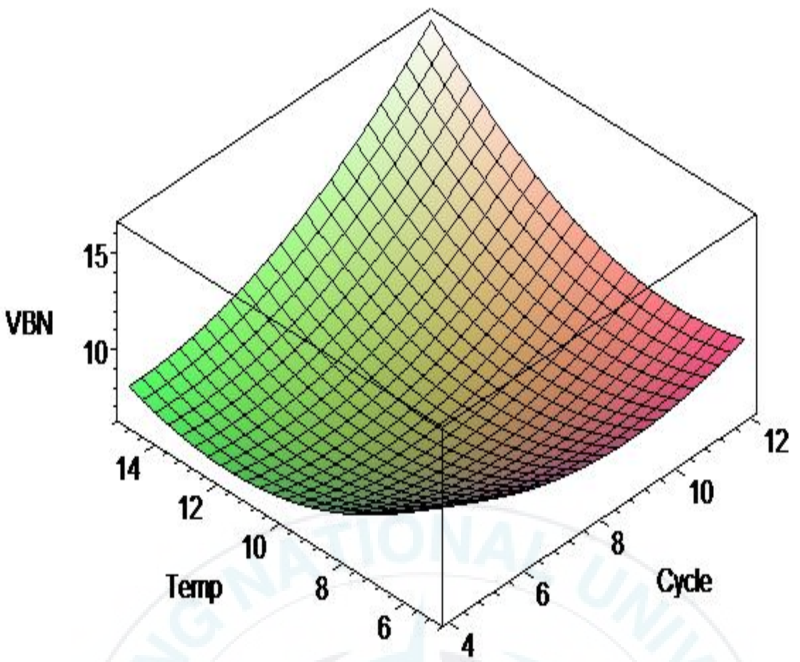

Figure 9. Relationship between temperature (°C) and drying cycle (no.) on VBN of Gwamegi product.

3.4. Response surface modeling for TMA standard on Gwamegi

The activity of TMA standard was assessed using response surface methodology. The concentration of Gwamegi (ml), concentration of NaOH (%), temperature (°C) and time (min) were used as independent variables. The TMA pick-up area was the only dependent variable in this experiment. The values of 33 experimental treatments on four independent variables and one dependent variable are presented in the table 12. The values of TMA peak up areas range from 11.40 ± 3.67 to 117.85 ± 3.34 respectively (table 12). The data were subjected to statistical analysis SAS software for RSREG procedure and second-order polynomial equation was fitted to the experimental data to obtain the response surface for the variables, regression models, independent variables and regression estimates as shown in table 13, 14, 15 and 16 respectively. The ANOVA results for the TMA peak up area on standard Gwamegi indicate that all regression model equations i.e linear, quadratic, cross product.were significant ($P<0.05$) as. indicated in table 14. The coefficient of determination (R^2) is 0.8230 and coefficient of variation (CV) is 25.4531 as shown in table 13. The variables NaOH, temperature and time were significant ($P<0.05$) but concentration of Gwamegi was not significant. The temperature has more effect among others on this analysis.as shown in table 15. The multiple polynomial regression analysis result was used to fit the data obtained from table 16 into the following second order equation model as follows:

$$TMA\_area=374.626931-41.902665*X_1-7.812572*X_2-2.820057*X_3+2.343327*X_4+1.319126*X_1^2+0.247762*X_1*X_2+0.189230*X_2^2+0.095930*X_1*X_3+0.346409*X_2*X_3+0.259009*X_3^2+3.691280*X_1*X4-0.583056*X_2*X_4+0.798611*X_3*X_4-1.195439*X_4^2$$

Where $\mathbf{X_1}$, $\mathbf{X_2}$, $\mathbf{X_3}$ and $\mathbf{X_4}$ denotes the independent variables of Gwamegi concentration (ml), NaOH concentration (%), temperature (°C) and time (min) respectively.

Fig.10 shows the relationship between temperature and time on TMA-area at the fixed points of 3ml concentration of Gwamegi and 15% NaOH concentration. According to the fig. 10, as time and temperature increases the TMA area decreases in a minimum phenomenon. However temperature has more effect on TMA area compared to time.

Fig.11 presents the effect of time and Gwamegi concentration on TMA area at the fixed points of 15% NaOH concentration and 18°C temperature. As depicted from fig. 11, TMA area was decreasing as time and concentration of Gwamegi was increasing in a maximum phenomenon.

As shown in fig. 12, the relationship between Gwamegi concentration and NaOH concentration were fixed at a time of 7min. and temperature of 18°C. The TMA area and NaOH shows the direct relationship while TMA area and Gwamegi concentration shows the inversely relationship. Similar trend of relationship between TMA area and Gwamegi concentration was observed in figure 11 too.

Fig. 13 describes the effect of temperature and NaOH on the TMA area at a given time of 7 min and Gwamegi concentration of 3mL. As shown in fig. 13, temperature increases rapidly as TMA area decreases. On the other hand, NaOH increases as TMA area increases.

Table 12. Response surface results for TMA peak-area of Gwamegi

Treatment	Variation levels				Response function
	X_1	X_2	X_3	X_4	TMA Peak area
1	4	20	11	9	54.60± 0.78
2	4	10	11	5	91.15±1.09
3	2	20	11	5	68.35±1.41
4	4	20	25	5	30.50± 1.41
5	2	10	25	5	11.40± 3.67
6	4	10	25	9	56.10± 2.83
7	2	20	25	9	37.70± 2.63
8	2	10	11	9	73.15± 1.91
9	3	15	18	7	45.30± 2.40
10	3	15	18	7	44.70± 6.29
11	3	15	18	7	46.80± 0.85
12	4	20	11	5	86.35± 1.41
13	4	10	25	5	46.65± 4.96
14	2	20	11	9	63.30± 1.05
15	2	10	11	5	93.50± 0.71
16	2	10	25	9	46.50± 1.27
17	4	10	11	9	96.00± 1.41
18	2	20	25	5	65.2± 0.21
19	4	20	25	9	85.30± 0.98
20	3	15	18	7	46.70± 4.33
21	3	15	18	7	46.50± 0.30
22	3	15	18	7	42.43± 0.61
23	1	15	18	7	72.30± 0.84
24	5	15	18	7	16.10± 4.94
25	3	5	18	7	44.85± 0.01
26	3	25	18	7	74.75± 0.73
27	3	15	4	7	117.85± 3.34
28	3	15	32	7	91.55± 0
29	3	15	18	3	12.60± 3.39
30	3	15	18	11	30.90± 3.68
31	3	15	18	7	45.30± 1.42
32	3	15	18	7	41.48± 0
33	3	15	18	7	43.70± 7.60

$\mathbf{X_1}$, $\mathbf{X_2}$, $\mathbf{X_3}$ and $\mathbf{X_4}$ denotes the independent variables of Gwamegi concentration (ml), NaOH concentration (%), temperature (°C) and time (min) respectively.

Table 13. Result of statistical analysis for TMA peak-area

Response surface for variable of TMA peak area	
Response mean	56.457879
Root MSE	14.370287
R-square	0.8230
Coefficient of variation	25.4531

Table 14. Results showing the secondary model from table 12

Regression	DF	Type 1 Sum of Squares	R-Square	F Value	Pr>F
Linear	4	4744.4597	340.961	5.74	0.0037
Quadratic	4	8458.018	640.155	10.24	0.0002
Cross product	6	4082.746	2917.579	3.3	0.0229
Total model	14	17285	661.492	5.98	0.0003

1) DF means degree of freedom.

2) The probabilities for rejecting the null hypothesis for each parameter are shown in their values.

Table 15. Results of statistical analysis of the independent variables obtained from table 12

Factor	**DF**	**Sum of Squares**	**Mean of Square**	**F value**	**Pr>F**
Concentration	5	1704.806	340.961	1.65	0.1975
NaOH	5	3200.777	640.155	3.1	0.0343
Temperature	5	14588	2917.579	14.13	<.0001
Time	5	3307.461	661.492	3.2	0.0305

1) DF means degree of freedom.

2) The probabilities for rejecting the null hypothesis for each parameter are shown in their values.

Table 16. Coefficient regression model estimated by multiple regression analysis for TMA peak-area

Parameter	**DF**	**Estimate**	**Pr > | t|**
Intercept	1	374.6269	0.0008
Conc	1	-41.90267	0.071
NaOH	1	-7.812572	0.1351
Temp	1	-22.82006	<.0001
Time	1	2.343327	0.8588
Conc*Conc	1	1.319126	0.095
NaOH*Conc	1	0.247762	0.7474
NaOH*NaOH	1	0.18923	0.089
Temp*Conc	1	0.09593	0.7864
Temp*NaOH	1	0.346409	0.0055
Temp*Temp	1	0.259009	0.0011
Time*Conc	1	3.69128	0.0671
Time*NaOH	1	-0.583056	0.1464
Time*Temp	1	0.798611	0.0093
Time*Time	1	-1.195439	0.0859

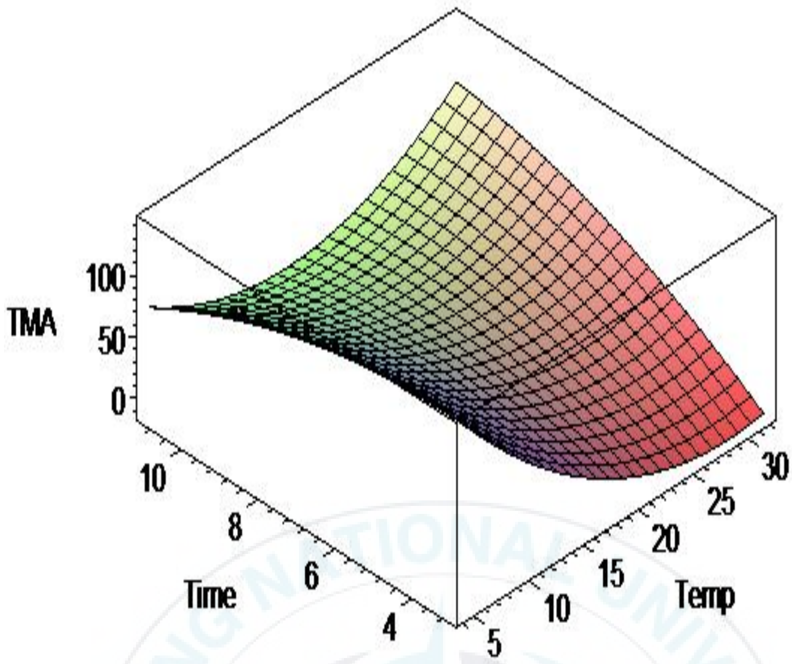

Figure 10. Relationship between temperature (°C) and time (min) on TMA of Gwamegi product.

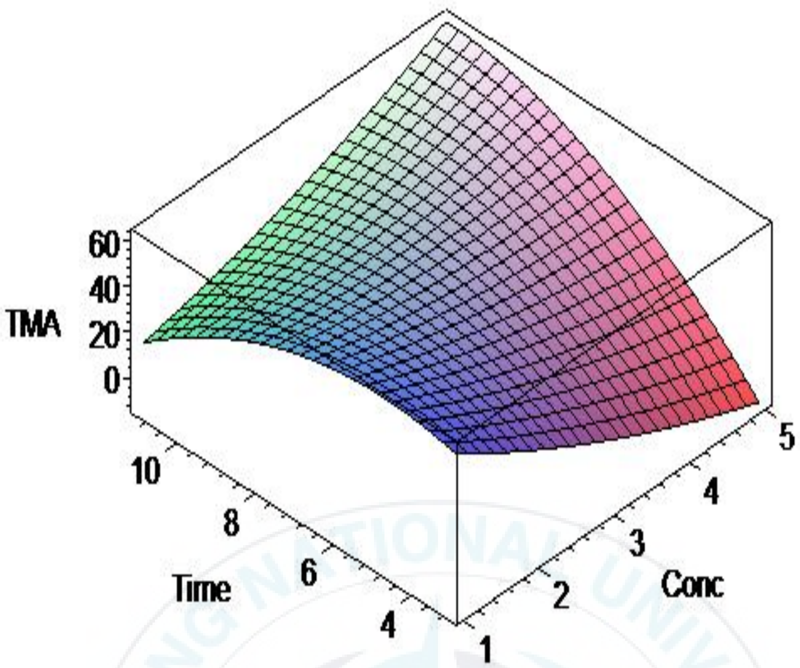

Figure 11. Relationship between time (min) and concentration (mL) on TMA of Gwamegi product.

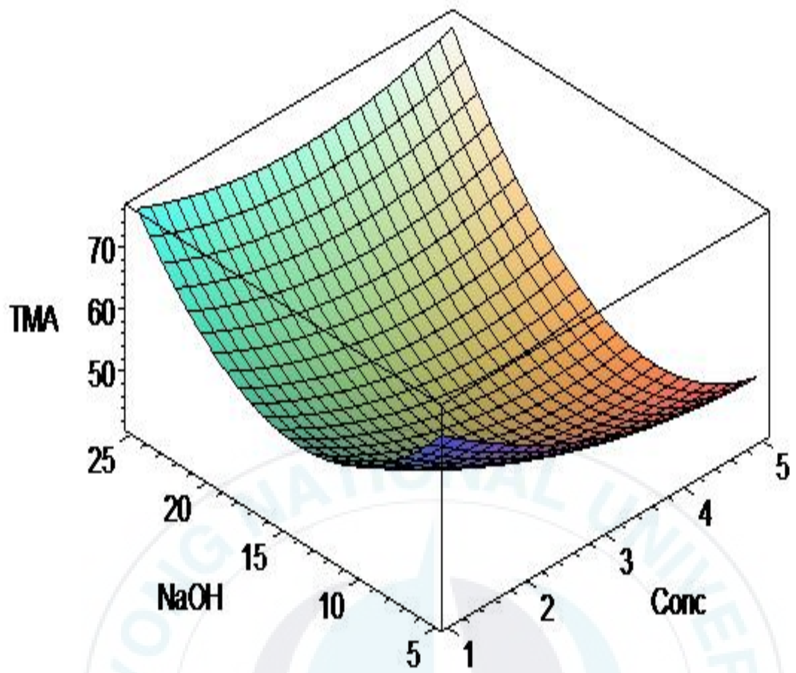

Figure 12. Relationship between concentration (mL) and NaOH (%) on TMA of Gwamegi product.

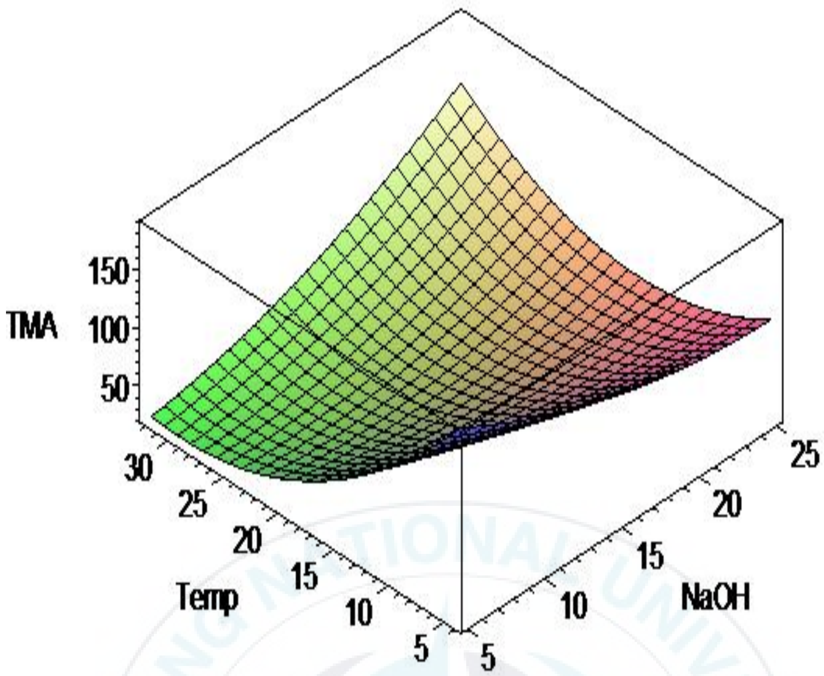

Figure 13. Relationship between temperature (°C) and NaOH (%) on TMA of Gwamegi product.

4 . Conclusion and Recommendations

Response surface methodology was employed to optimize the processing conditions of Gwamegi. The drying freezing conditions are significantly influenced by drying temperature, time and cycles. The optimal drying condition is temperature of 15.6 °C, time of 5.1 hours and drying cycles of 12 numbers. Drying temperature has shown the most significant effect on Gwamegi processing followed by drying cycles.and time. These results provide us with the basic information required to control qualities of Gwamegi and other dried fish and fishery products.

It is recommended that, further studies be conducted on the sensory evaluation aspects to observe people's perception on the product dried by oven using the above mentioned optimal drying conditions found from this study.

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Acknowledgement

I would like to express my sincere thanks to my advisor, Prof Yang-Bong Lee for his willingness in supervising my Masters studies and research work, for his kindness, tolerance and words of encouragement in the course of my studies. His technical guidance and supervision inspired me to complete my thesis work successfully and timely. I am grateful to my thesis committee members Prof. Sungchul C. Bai and Prof. Young-Mog Kim for their valuable remarks and constructive critique on this work. I would like to offer my gratitude to South Korea Government and KOICA scholarship program for financial support in pursuing this Masters Degree.

Special thanks to Dr. Kyoungmi Kang, the KOICA Program Coordinator and other staff members for their assistance and cooperation throughout the stay in South Korea. I thank my lab mates in the Food Biochemistry laboratory for being with me and assisting in the laboratory work experiments and my fellow students for their support. My deepest appreciation goes to Dr. Respickius Casmir for his untiring assistance in proof reading my thesis work and make it wonderful one. Finally, I am particularly grateful to almighty God for his tremendous love throughout the course of my studies in South Korea.

Dedication

This work is dedicated to my lovely husband Respickius Casmir and my lovely sons Victor, Frank and Adon.