Predicting the Nutritional and Rancidity Properties of Dehydrated Catfish (*Clarias gariepinus*) Using Response Surface Methodology

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ABSTRACT: Catfish (*Clarias gariepinus*), the most popular fish species cultivated in Nigeria is rich in nutrients but highly perishable thus, it requires processing for preservation. In order to determine the optimal dehydration parameters, the combined effects of brine concentration [3.0, 6.0, and 9.0% (w/v)], brining time (30, 60, and 90 min), and drying temperature (90, 110, and 130°C) were investigated to predict the nutritional and rancidity properties of the dehydrated catfish using response surface methodology (RSM). The study showed that brine concentration, drying temperature, and the interaction of brine concentration and brining time significantly ($P < 0.05$) influenced the nutritional and rancidity properties of dehydrated catfish. However, the optimal process parameters: 7.83% brine, 90 min, and 110.38°C produced dehydrated catfish of high protein content (60%), low moisture (6.0%), free fatty acid (1.2%), thiobarbituric acid (0.10 mg malondialdehyde/kg), and total volatile nitrogen (10.0 mg nitrogen/100 g) with no detectable levels of peroxide value, indicating good nutritional quality and lower lipid oxidation for shelf stability. RSM models with a high range of predictive R^2 (77 ~ 88%) were obtained at the set conditions showing the RSM potential as a feasible tool in this regard. The dehydration technique employed in this study is effective for high nutrient retention, especially the protein content, which could ameliorate the problem of malnutrition especially where fresh fish is not accessible, simple in operation and economical to encourage commercial applications with a potential for food security.

Keywords: catfish, dehydration, response surface methodology, nutritional properties, rancidity indices

INTRODUCTION

Fish is a nutrient-rich food considered to be a cheaper and healthier source of animal protein than red meat, especially in developing countries (1,2). Fish is also free from religious prejudice, which is common for beef and pork and of a wider consumer acceptance (3). The most popular species of fish cultivated in Nigeria is African catfish (*Clarias gariepinus*), which is a freshwater fish containing protein of higher biological value compared with other protein sources (4,5) and high in mineral content, especially calcium (6).

However, fresh catfish is highly susceptible to deterioration due to its high moisture content and requires refrigeration, which is difficult in Nigeria due to the erratic supply of electricity (7). Also, the supply of fresh catfish to low access areas is associated with a high mortality rate of live-catfish on transit as a factor of stress during the distribution (8). All these factors limit the nutritive

and economic potential of catfish in Nigeria. Therefore, processing by dehydration could be an appropriate method to remove most of the water present in fish muscle to preserve the nutrient content and extend the shelf-life (9).

Accordingly, fish dehydration is considered cheaper than refrigeration/freezing and canning (10,11) and when the processing is effectively done, dehydrated catfish has a high level of sensory acceptability (6), which is necessary to derive the nutrients contained. Moreover, when assessing the quality of dehydrated catfish, many studies regarding the effect of processing/dehydration method on proximate composition exists, but the results varied considerably (12-14). The rancidity indices [pH, free fatty acid (FFA), thiobarbituric acid (TBA), total volatile nitrogen (TVN), and peroxide value (PV)] are used to assess the shelf-stability of the processed fish based on the recommendations set by the United States Food and Drug Administration (2,15).

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Consequently, using simple mathematical expressions (models) to describe the data obtained from the analysis of chemical properties of dehydrated catfish processed under optimal parameters, when sources of variations are minimized, could serve as a tool for quick predictive purposes. Furthermore, an economical way of finding the best set of parameters among a specified set of alternatives (optimisation) is through modern statistically derived experimental designs such as response surface methodology (RSM) (16). RSM is an experimental design with a collection of mathematical and statistical modelling techniques which have been used effectively in the optimization and monitoring of food processes. This combines product treatment to the outputs and establishes a regression equation (model) to describe inter-relations between input parameters and product properties (17).

Despite the vast literature on the effects of processing methods on the quality of processed fish, scarce information exists on modelling efforts on the nutritional and rancidity properties of dehydrated catfish as influenced by the dehydration parameters. Therefore, this study aimed at using RSM to develop models, which can be employed for predicting and optimising the proximate composition and rancidity indices of the dehydrated catfish.

MATERIALS AND METHODS

Materials

Freshly harvested live catfish of about 6 months old, averagely weighing 524 ± 20 g with an average length of 42 ± 2 cm each, were obtained from the Fisheries Research and Teaching Farm of the Federal University of Technology, Akure (FUTA), Nigeria. Common salt used for brining was purchased from a local market in Akure, Nigeria. Other chemicals used for analyses were of analytical grade.

Experimental design for catfish processing

A face-centred full-factorial central composite design of the RSM (version 10.0.1, Stat-Ease, Inc., Minneapolis, MN, USA) was used to develop different combinations of process parameters (runs) and to evaluate the combined effect, at three levels each, of brine concentration (3.0, 6.0, and 9.0%; g NaCl/100 mL water), brining time (30, 60, and 90 min), and drying temperature (90, 110, and 130°C) on the dependent variables (responses). The responses were: proximate composition (moisture, crude protein, crude fat, and ash contents) and rancidity indices (pH, FFA, TBA, TVN, and PV). Seventeen experimental runs were conducted with three of the replicates at the centre point, eight factorial points, and six axial points.

Brining operation

The fish were slaughtered, beheaded, degutted, thoroughly washed with clean water, and drained. Brine solutions of the required concentrations [3.0, 6.0, and 9.0% (w/v)] were prepared by dissolving the salt in water at ambient temperature ($26 \pm 2^\circ\text{C}$). Multiple ratios of brine volume to fish were made to minimise the dilution effect of moisture diffusion from the fish muscle (6). Headless catfish of an average weight of 254 g was randomly selected and dipped into brine solutions for the appropriate time (30, 60, or 90 min). Brined catfish arranged on mesh trays were left for at least 1 h to allow brine equilibration and draining of the excess solution.

Drying operation

The dryer was a cylindrical, double walled insulated stainless steel of 2 cm thickness consisting of a drying chamber having 8 electrical heating coils (1.5 kW each) enclosed in glass tubes, which were equidistantly fixed around the wall of the chamber with connections to a temperature regulator. The drying chamber contained a set of three mesh trays on which the brined catfish were arranged. The dryer was preheated to the desired temperature while the samples were being prepared to ensure temperature stability when the fish was introduced. A dry bulb thermometer was placed inside the dryer through the vent at the top to monitor the temperature. The fish were occasionally checked to ensure no case hardening occurred during drying. The drying of catfish at 90, 110, and 130°C was done for 27 h, 24 h: 40 min and 12 h: 49 min, respectively. Samples with a similar temperature factor were dried as a batch. Dehydrated catfish were cooled and muscle parts milled with the aid of a blender (MARLEX Electrolite, MARLEX, Dabhel, India), sealed in polyethylene bags and labelled prior to analyses. The method described by Eyo (18) was employed in the production of dehydrated catfish.

Determination of proximate composition

The proximate composition of the fresh and dehydrated catfish was determined to assess the percentages of moisture, crude protein, crude fat, and ash contents using the Association of Official Analytical Chemists methods (method number: 925.10, 960.52, 2003.05, and 923.03, respectively) (19).

Determination of rancidity indices

The pH of the homogenate samples of the fresh and dehydrated catfish was measured using a digital pH meter (HI98107, Hanna Instruments, Inc., Woonsocket, RI, USA). The FFA, TBA, TVN, and PV were determined using the method described by Pearson (20); the absorbance for TBA was read using a spectrophotometer (AJ-1C03, Anjue Medical Equipment Co., Ltd., Anhui, China).

Statistical analysis

Analysis of variance (ANOVA) was used to establish differences among treatments and Duncan's multiple range tests were used to separate treatment means using SPSS (21). Significance was accepted at $P < 0.05$. The data was fitted into RSM models [quadratic, two-factor interaction (2FI), and linear]. To correlate the response with the independent variables, multiple regressions were used to fit the coefficient of the polynomial model, which was further subjected to backward regression or transformation analysis to improve the fit. The lack-of-fit, coefficient of determination (R^2), adjusted R^2 , predicted R^2 , and adequate precision were used to evaluate the quality of the fitted model (22). The response surface plots were prepared to represent a function of two independent variables while fixing the other variable at the optimum value. The fitted quadratic response model is as described in Eq. 1 (17).

$$Y = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ij} X_i^2 + \sum_{i < j}^k \sum_j b_{ij} X_i X_j + e \quad (1)$$

where Y is the modelled response to be predicted, i and j denote linear and quadratic coefficients, respectively, while b_0 is intercept, b_i is a first-order model coefficient, k is the number of factors, and e is a random number.

Optimization procedures

The numerical multi-response optimization procedure was done using the desirability concept (with value close to 1) to determine the optimum level of the processing

parameters by setting goals for the parameters to be in range (22), nutrient compositions were maximised for nutritional quality, pH in range, moisture content, and other rancidity indices were minimised for shelf stability.

RESULTS AND DISCUSSION

Predicting the nutritional and rancidity properties of dehydrated catfish

The nutritional (proximate compositions) and rancidity indices of dehydrated catfish as obtained from the experiment (Table 1 and 2, respectively) were significantly ($P < 0.05$) influenced by the dehydration parameters and were adequately predicted by the RSM models. All the obtained models were significant ($P < 0.05$). Backward regression and transformation analysis improved the fit of the models as shown in Table 3. The variance inflation factors of the describing models were quite low (ranged 1.00~1.36), most were of ideal nature (value=1.00), which implied correct predictions. Also, the response surface 3D plots in Fig. 1 and 2 indicated significant effects of processing parameters on the proximate compositions and rancidity indices, respectively, of the dehydrated catfish.

Predicting the proximate composition of dehydrated catfish

Moisture: The moisture contents ranged from 5.47~8.41% (Table 1) from the initial value of 76.12% and were generally lower than the value (15.62%) obtained

Table 1. Proximate compositions of fresh raw and the dehydrated catfish samples (%)

RSM run	A	B	C	Moisture	Crude protein	Crude fat	Ash
1	6	60	110	5.81±0.01 ^{ghi}	64.06±0.01 ^d	22.33±0.29 ^{fg}	11.00±0.00 ^{cde}
2	6	90	110	5.47±0.01 ^h	61.15±0.00 ^f	22.60±0.52 ^{fg}	12.29±0.61 ^{bc}
3	3	60	110	7.80±0.24 ^{bc}	55.74±0.01 ^o	26.60±0.90 ^{cd}	8.85±0.15 ^{gh}
4	3	30	90	7.59±0.02 ^c	59.86±0.01 ^k	26.20±1.53 ^{bcd}	10.00±1.00 ^{efg}
5	6	30	110	6.41±0.02 ^{de}	59.93±0.01 ^j	24.70±0.30 ^{de}	10.77±0.23 ^{def}
6	9	90	130	6.64±0.01 ^d	55.10±0.01 ^p	27.10±1.01 ^b	12.05±0.95 ^{bcd}
7	9	60	110	6.18±0.00 ^{efg}	60.04±0.01 ⁱ	23.50±0.50 ^{ef}	11.00±0.00 ^{cde}
8	6	60	110	5.75±0.00 ^{gh}	64.33±0.01 ^c	19.17±0.29 ^h	11.00±0.00 ^{cde}
9	9	90	90	5.50±0.10 ^h	61.90±0.01 ^e	19.30±0.32 ^h	14.00±1.00 ^a
10	6	60	130	6.06±0.00 ^{efgh}	55.79±0.01 ⁿ	26.40±0.68 ^{bc}	9.32±0.05 ^{gh}
11	3	90	130	6.60±0.03 ^d	60.10±0.01 ^h	21.64±1.73 ^g	9.57±0.54 ^{fgh}
12	6	60	110	6.13±0.01 ^{efgh}	61.09±0.01 ^g	19.67±0.64 ^h	10.77±0.23 ^{def}
13	6	60	90	6.30±0.10 ^{def}	65.26±0.01 ^b	17.00±0.50 ⁱ	11.00±1.00 ^{cde}
14	3	90	90	8.41±0.01 ^a	59.84±0.02 ^j	26.50±0.00 ^{bc}	9.11±0.84 ^{gh}
15	3	30	130	6.02±0.81 ^{fgh}	53.34±0.03 ^q	30.80±0.52 ^a	8.33±0.93 ^h
16	9	30	130	7.97±0.00 ^b	57.65±0.00 ^m	21.30±1.30 ^g	11.46±1.48 ^{bcd}
17	9	30	90	8.40±0.18 ^a	68.85±0.01 ^a	10.70±1.44 ^j	12.50±0.50 ^b
FRF	—	—	—	76.12	17.29	2.93	1.68

Values are means of triplicate determinations±standard deviations.

Different letters (a-q) along the columns are significantly different at $P < 0.05$.

A, brine concentration (%); B, brining time (min); C, drying temperature (°C); FRF, fresh raw fish.

Table 2. Rancidity indices of fresh raw and the dehydrated catfish samples

RSM run	A	B	C	pH	FFA	TBA	TVN	PV
1	6	60	110	6.10±0.09 ^f	1.28±0.05 ^{bcd}	0.03±0.00 ^{kl}	9.60±0.10 ^h	ND
2	6	90	110	6.10±0.00 ^f	1.19±0.29 ^{de}	0.04±0.00 ^{jk}	9.80±0.10 ^h	ND
3	3	60	110	6.15±0.05 ^{ef}	1.49±0.02 ^a	0.13±0.00 ^{ab}	19.60±0.21 ^b	ND
4	3	30	90	6.30±0.00 ^c	0.91±0.06 ^f	0.14±0.00 ^a	14.00±0.10 ^f	ND
5	6	30	110	6.15±0.05 ^{ef}	1.34±0.04 ^{bc}	0.06±0.00 ^{hij}	5.60±0.00 ⁱ	ND
6	9	90	130	6.30±0.00 ^c	0.73±0.04 ^g	0.11±0.00 ^{bc}	14.00±0.50 ^f	ND
7	9	60	110	6.10±0.00 ^f	0.88±0.00 ^f	0.10±0.00 ^{cd}	18.20±0.00 ^c	ND
8	6	60	110	6.20±0.00 ^{de}	1.28±0.03 ^{bcd}	0.05±0.00 ^{ijk}	14.00±0.00 ^f	ND
9	9	90	90	6.20±0.00 ^{de}	1.20±0.02 ^{cde}	0.09±0.00 ^{cdef}	14.00±0.00 ^f	ND
10	6	60	130	6.20±0.00 ^{de}	1.18±0.03 ^{de}	0.07±0.00 ^{ghi}	12.60±0.20 ^g	ND
11	3	90	130	6.50±0.00 ^a	1.21±0.00 ^{cde}	0.02±0.00 ^j	15.40±0.10 ^e	ND
12	6	60	110	6.00±0.00 ^g	1.20±0.02 ^{cde}	0.03±0.00 ^{kl}	14.00±0.00 ^f	ND
13	6	60	90	6.00±0.00 ^g	1.33±0.01 ^{bc}	0.08±0.00 ^{defg}	14.00±0.00 ^f	ND
14	3	90	90	6.25±0.05 ^{cd}	1.17±0.04 ^{de}	0.10±0.00 ^{cde}	14.00±0.00 ^f	ND
15	3	30	130	6.45±0.05 ^{ab}	1.10±0.03 ^e	0.09±0.00 ^{cdef}	21.00±0.09 ^a	ND
16	9	30	130	6.40±0.00 ^b	0.78±0.03 ^{fg}	0.11±0.00 ^{bc}	14.00±0.00 ^f	ND
17	9	30	90	6.10±0.00 ^f	0.47±0.00 ^h	0.13±0.00 ^{ab}	16.80±0.00 ^d	ND
FRF	—	—	—	6.6	nd	0.086	11.2	ND

Values are means of duplicate determinations±standard deviations.

Different letters (a-l) along the columns are significantly different at $P<0.05$.

A, brine concentration (%); B, brining time (min); C, drying temperature (°C); FFA, free fatty acid (%); TBA, thiobarbituric acid (mg malondialdehyde/kg); TVN, total volatile nitrogen (mg nitrogen/100 g); PV, peroxide value (milliequivalents of oxygen/kg); FRF, fresh raw fish.

nd, not determined; ND, not detected.

Table 3. ANOVA and regression coefficients for models' predictions of the proximate composition and rancidity indices of dehydrated catfish

Response	Moisture	Crude protein	Crude fat	Ash	pH	FFA	TBA	TVN
Model (Reduc/Transf.)	RQuadratic	2FI	2FI/Sqrt (Fat)	Linear	RQuadratic	RQuadratic	RQuadratic	RQuadratic
Model (P -value)	0.0005	0.0064	0.0013	<0.0001	0.0004	0.0063	0.0032	0.0031
A	ns	ns	0.0026	<0.0001	0.0321	0.0048	ns	ns
B	0.0227	ns	ns	ns	ns	ns	0.0311	ns
C	ns	0.0011	0.0037	0.0217	0.001	ns	ns	ns
AB	0.0012	0.0342	0.0057	na	na	na	na	na
AC	0.0085	ns	0.0131	na	na	na	ns	ns
BC	na	ns	ns	na	na	ns	na	na
A ²	0.0004	na	na	na	0.0162	0.0075	0.0008	0.0002
B ²	na	na	na	na	0.0162	na	na	0.007
C ²	na	na	na	na	na	na	na	na
LOF (P -value)	0.1595	0.3942	0.3494	0.029	0.8933	0.0638	0.1446	0.683
R ²	0.8754	0.7858	0.8483	0.8088	0.843	0.7332	0.7662	0.7101
Adjusted R ²	0.8007	0.6572	0.7573	0.7646	0.7717	0.6119	0.6599	0.6135
Predicted R ²	0.4448	0.4545	0.6025	0.6596	0.6441	0.2541	0.4039	0.3812
Adeq. precision	9.848	10.997	13.68	14.444	11.659	8.003	8.252	9.924

FFA, free fatty acid; TBA, thiobarbituric acid; TVN, total volatile nitrogen; Reduct/Transf., reduction or transformation; RQuadratic, reduced quadratic model; 2FI, two-factor interaction model; 2FI/Sqrt, transformation square root two-factor interaction model; A, brine concentration (%); B, brining time (min); C, drying temperature (°C); LOF, lack of fit; R², coefficient of determination; Adeq. precision, adequate precision.

ns, not significant; na, not applicable.

by Chukwu and Shaba (12) in an electric oven with dried catfish at 120°C without prior brining. This shows that the brining operation in the present study enhanced dehydration and lower moisture content as a result of an osmotic effect of the brine on moisture drip from the fish

muscle, similar to the findings of Sobukola et al. (23). This is evidence in the significance ($P<0.05$) of the brining parameters (brining time, interaction of brine concentration and brining time, and quadratic term of brine concentration) on the moisture content (Table 3). The

Fig. 1. Response surface 3D plots describing the effect of brining time and brine concentration at a fixed drying temperature (110°C) on the proximate composition [moisture (A), crude protein (B), crude fat (C), and ash content (D)] of dehydrated catfish.

contribution of the interactions between brine concentration and drying temperature was also significant. Moreover, observations during the experiment show that the temperature effect during drying contributed to a greater percentage of moisture loss through evaporation. Low moisture content is an index of shelf stability, which suggests a reduced rate of spoilage of dehydrated catfish either by rancidity or microbial activities during storage, similar to the report of Kumolu-Johnson et al. (24). Fig. 1A shows the quadratic effect of brine concentration and brining time at a fixed drying temperature (110°C) on moisture content. There was an initial decrease in moisture content as the brine concentration increased, but the significance was aided by the increase in brining time. The modified quadratic model could predict 88% of the variations in the moisture content observed (Table 3). The regression model developed for predicting the moisture content is shown in Eq. (2).

$$\text{Moisture} = 5.99 - 0.17A - 0.38B - 0.29C - 0.70AB + 0.51AC + 1.12A \quad (2)$$

where A is brine concentration, B is brining time, and C is drying temperature.

Crude protein: The crude protein content of dehydrated

catfish ranged from 53.34 to 68.85% (Table 1). A protein content of 55% and above is said to be an indicator of good nutritive quality in dried catfish (13). High protein retention (except the lower limit in the range from run 15) indicated a good nutritional quality of the dehydrated catfish, similar to the report of Aberoumand and Karimi reza abad (25). Table 3 shows that the description by the 2FI model of the effects of drying temperature, and interaction between brine concentration and brining time on protein content were significant ($P < 0.05$). According to Okpala and Okoli, a predictive R^2 value of 79% (0.7858) is adequate (26). The crude protein content of dehydrated catfish increased with the increase in brine concentration but decreased with a decrease in brining time (Fig. 1B). However, it was observed that a high range of minimum to maximum values (56.7~66.5%) was expressed for the effects of interacting brine concentration and drying temperature within the ranges used at a fixed brining time (60 min), indicating a positive effect for higher protein content. The developed model of coded factors is described in Eq. (3).

$$\text{Crude protein} = 60.24 + 1.47A - 0.15B - 3.37C - 2.03AB - 1.47AC + 1.40BC \quad (3)$$

Crude fat: The fat contents ranged from 10.70~30.80%. Low-fat contents were obtained in most of the samples dried at 90°C (Table 1). This could mean more fat accompanied moisture exudation for a longer period of time during drying at the lower temperature. A similar observation was made by Chukwu and Shaba (12) for kiln- (60~70°C) and electric-oven-dried (120°C) catfish with a comparable range of values of 21.20 and 29.60% lipid content, respectively. The brine concentration, drying temperature, brine concentration-brining time interaction, and brine concentration-drying temperature interaction significantly ($P<0.05$) influenced the crude fat content (Table 3). A high R^2 (0.8483) implied a good correlation between the predicted and experimental values. Crude fat content decreased with an increase in brine concentration but increased with a reduction in brining time (Fig. 1C). Nevertheless, the observation during the drying process and the analysis showed that higher drying temperatures significantly favoured higher values of crude fat, indicating the reversed case of the above exudation discussion. The transformation regression model developed for predicting crude fat of dehydrated catfish is described in Eq. (4).

$$\text{Sqrt (crude fat)} = 4.73 - 0.33A + 0.063B + 0.31C + 0.32AB + 0.28AC - 0.18BC \quad (4)$$

Ash content: The ash content values ranged from 8.33~14.00% (Table 1). The range is relatively higher than the range of values (4.14 to 5.33%) reported by Omodara and Olaniyan (13), which may be due to the higher brine concentrations used and the lower moisture contents of the dehydrated catfish in the present work. Similarly, Jittinandana et al. (27) related increased ash contents of the cooked product of brined and smoked rainbow trout fish fillets for water loss and increased brine concentration. Table 3 shows that brine concentration (A) and drying temperature were the significant ($P<0.05$) process parameters with brine concentration showing a more significant effect on the ash content. However, the P -value for lack of fit (0.0290) was significant, implying that the predictive linear model did not have a good fit. The adjusted R^2 (0.7646) is in reasonable agreement with the predicted R^2 (0.6596) indicating a correct prediction. Fig. 1D shows the effect of brining time and brine concentration at a fixed drying temperature (110°C) on the ash content. There is a significant increase in ash content with increased brine concentration, while the effect of brining time was not significant. The model for predicting ash content of the dehydrated catfish is presented in Eq. (5).

$$\text{Ash} = 10.77 + 1.52A + 0.40B - 0.59C \quad (5)$$

The above analyses indicated that the dehydrated catfish samples showed high retention of nutrient compositions, which implies a higher nutrient intake per unit quantity of the product consumed which could ameliorate malnutrition especially in areas where fresh catfish is less accessible. In addition to being effective in preserving the nutrient contents, the dehydration technique employed is simple in operation and economical to encourage commercial applications for a wider distribution of catfish within the national and foreign markets.

Predicting the rancidity indices of dehydrated catfish

pH: The pH is suggested as a guideline to assess the quality of fish and fish products (28). Generally, a pH of 6.5 or less is acceptable for dehydrated fish (29). The range of pH values (6.00~6.50) in this work is within the suggested limits (Table 2). The brine concentration, drying temperature, quadratic effects of brine concentration, and brining time were significant ($P<0.05$) in describing the pH of fish (Table 3). The adjusted and predicted R^2 values, 0.7717 and 0.6441, respectively were in agreement showing a good correlation between the predicted and experimental values. Fig. 2A shows the quadratic effects of brine concentration and brining time at a fixed drying temperature (110°C) on the pH of dehydrated catfish. The developed model for predicting the pH is presented in Eq. (6).

$$\text{pH} = 6.07 - 0.055A - 5.000E - 003B + 0.10C + 0.12A^2 + 0.12B^2 \quad (6)$$

FFA: The determination of FFA is an index of the quality of fat and subsequently of the food in which the fat is contained (30). A maximum FFA of $\leq 1.38\%$ has been set as the standard for fish grading (20). The obtained range, (Table 2) in all the runs [except the highest value (1.49) in run 3] were found within the standard limits in the samples and are comparable with the values reported by Seifzadeh et al. (31) for Kilka fish preserved in sodium alginate ($1.00 \pm 0.20\%$) and whey protein ($1.10 \pm 0.15\%$). Low values of FFA in the present experiment are attributed to inactivation of lipolytic enzymes by the brining and thermal processing employed, similar to the report of Al-Saghir et al. (32). Also, Chukwu and Shaba (12) noted that oven drying could retard lipid oxidation in catfish. These factors indicate that the dehydration technique used in this study could preserve the dehydrated catfish against oxidative rancidity for shelf stability. The brine concentration and its quadratic effect significantly ($P<0.05$) affected the FFA index (Table 3). The R^2 (0.7332) less than 0.75 implied that the developed model may not be adequate for predicting FFA of dehydrated catfish at the set conditions; adequate predictive R^2 value should be ≥ 0.75 (26). Fig. 2B shows that low

Fig. 2. Response surface 3D plots describing the effect of processing parameters on the rancidity indices [pH (A), free fatty acid (FFA) (B), thiobarbituric acid (TBA) (C), and total volatile nitrogen (TVN) (D)] of dehydrated catfish at a fixed drying temperature (110°C), brine concentration, brining time, and drying temperature (110°C), respectively.

drying temperature (about 90°C) and low brining time (about 30 min) favored a low value (1.1%) of FFA at a fixed brine concentration (6%). However, observations during data analysis showed that the interaction of brine concentration and brining time at a constant temperature (in another response surface 3D graph not presented) better favors lower values of FFA (0.60~1.25%), with brine concentration showing a more significant decrease. The regression model for predicting FFA is presented in Eq. (7).

$$\text{FFA} = 1.26 - 0.18A + 0.090B - 8.000E - 003C - 0.12BC - 0.26A^2 \quad (7)$$

TBA: The TBA value is widely used as an indicator of the degree of lipid oxidation (33). The TBA values ranged from 0.02~0.14 mg malonaldehyde (M)/kg (Table 2) and were much lower than the maximum recommended limit (1~2 mg M/kg) for fish samples (34). This stresses the preservative effect of the combinations of the process parameters employed for dehydration of catfish in this study. From Table 3, the adequate predictive R^2 (77%) was obtained; $R^2 \geq 75\%$ is adequate for predictive pur-

poses (26). Fig. 2C shows that at a fixed brining time (60 min), a non-linear decrease occurred in TBA with increasing brine concentration (below 6%) and drying temperature reaching the minimum (0.04 mg M/kg) at the highest temperature (130°C) but increased with a further increase in brine concentration, indicating a quadratic effect. The regression model for predicting TBA is presented in Eq. (8).

$$\text{TBA} = 0.053 + 6.300E - 003A - 0.018B - 0.015C + 0.016AC + 0.051A^2 \quad (8)$$

TVN: The TVN involves the denaturation of protein by autolytic de-amination of amino acids (35). It is measured as an index of the freshness of fish (29). The rejection limit for the freshness of fish products is set at 25 mg nitrogen (N)/100 g (36), while staleness is considered at a value in excess of 30 mg N/100 g (20). The obtained range of values, 5.60 to 21.00 mg N/100 g TVN, (Table 2) was within the acceptable limits for the freshness of dried fish stressing the effectiveness of the dehydration technique used. However, the R^2 value (0.71), which is less than 0.75 (Table 3) may not be adequate for

Fig. 3. Response surface 3D plot showing multi-response optimisation conditions for the dehydration of catfish.

prediction purposes as indicated by Okpala and Okoli (26). Interacting brine concentration and brining time at a fixed drying temperature (110°C) produced an initial low value (8.7 mg N/100 g) TVN at the brine concentration of 6.1% and the lowest brining time of 30 min, while an additional increase in these parameters led to a rise in the value of TVN of dehydrated catfish showing a quadratic effect (Fig. 2D). The developed model for predicting TVN value of dehydrated catfish is presented in Eq. (9).

$$\text{TVN} = 12.59 - 0.70A - 0.42B + 6.93A^2 - 4.27B^2 \quad (9)$$

PV: The PV [milliequivalents of oxygen (mEq O₂)/kg] was below the detectable levels in all the samples which corroborates the findings of Bragadóttir et al. (15) that reported 0.0 mEq O₂/kg PV for salted and dried capelin fish and confirms the preservative effect of the dehydration technique used. This, coupled with the low values obtained for all the other rancidity indices parameters determined, indicate a lower susceptibility to oxidative deterioration and shelf stability of dehydrated catfish.

Additionally, the developed models were adequate for quick prediction purposes for the proximate composition and rancidity indices of dehydrated catfish produced under similar conditions, showing the RSM potential as a feasible tool.

Process optimization

The result of numerical multi-response process optimization based on the desirability concept with the set goal that maximized the nutrient contents, and minimized moisture content and rancidity indices, predicted the optimal process parameters at a 7.83% brine concentration, 90 min brining time, and 110.38°C drying temperature with the highest and good desirability value of 0.598 (approximately 60% level) as indicated in Fig. 3. The pre-

Table 4. Experimental values for responses under multi-response optimisation conditions

Response	Pre-dicted	95% CI low	95% CI high	Validation
Moisture (%)	5.5	4.99486	5.99077	6.0±0.1
Crude protein (%)	59.7	56.8505	62.0019	60.0±0.0
Crude fat (%)	23.1	20.6721	26.2079	23.0±0.3
Ash (%)	12.1	11.3245	12.6897	12.0±0.5
pH	6.2	6.11175	6.28297	6.0±0.1
FFA (%)	1.14	0.96405	1.29369	1.2±0.04
TBA (mg MDA/kg)	0.058	0.03371	0.07934	0.100±0.000
TVN (mg N/100 g)	10.06	7.25005	12.7107	10.0±0.05

n=1; a=0.05.

CI, confidence interval; FFA, free fatty acid; TBA, thiobarbituric acid; MDA, malondialdehyde; TVN, total volatile nitrogen.

dicted conditions were adjusted to 8.0% brine, 90 min, and 110°C for experimental convenience (37). The experimental values obtained at the optimised conditions produced dehydrated catfish that had the following: moisture content (6.0%), protein (60.0%), fat (23.0%), ash (12.0%), pH (6.0), FFA (1.2%), TBA (0.10 mg M/kg), and TVN (10.0 mg N/100 g), which were close to the predicted values. These values were also found within the predicted confidence interval levels (Table 4), which give an indication of the expected process average. Hence, the adjusted predicted dehydration parameters: 8% brine concentration (w/v), 90 min brining time, and 110°C drying temperature optimised the chemical properties of dehydrated catfish at the set goal with high retention of nutrients (especially the protein content), low moisture, and rancidity index values. These imply good nutritional quality and less lipid oxidation for shelf stability.

Consequently, brine concentration, brining time, and drying temperature at the levels used had a positive significant influence on the nutritional and rancidity properties of the dehydrated catfish. However, the quadratic term of brine concentration had the highest regression coefficient in all of the models obtained in this study, showing brine concentration as the most significant factor.

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AUTHOR DISCLOSURE STATEMENT

The authors declare no conflict of interest.

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