Predicting the Nutritional and Rancidity Properties of Dehydrated Catfish (C*larias gariepinus*) using Response Surface Methodology

By

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Introduction

- Fishes are aquatic animals, providing a significant protein intake in the diets of a large proportion of people in the developing countries (Adeyeye *et al*., 2016).

- Catfish is of good economic value (which must be exploited) and widely sought in many parts of the world including Nigeria because of its palatability (Adeparusi *et al*., 2010).

- Fish is an extremely perishable food item due to high water content thus, required preservation for future use (Balachandra, 2011).

- The removal of most of the water present in fish muscles by dehydration, extends the shelf-life without the need for refrigeration (Olayemi, 2012).

- The quality of dehydrated catfish can be assessed using a range of physical, physicochemical, biological and organoleptic tests (Adam and Sidahmed, 2012).

Introduction Contd.

- Contributing to the understanding of the influence of processing parameters on product properties are the mathematical expressions (models) which can adequately describe the data obtained from the analyses. The use of Response Surface Methodology (RSM) aided this.

- RSM is an experimental design with a collection of mathematical and statistical modelling technique which has been used effectively in the optimisation and monitoring of food processes. It relates product treatment to the outputs and establishes a regression equation (model) to describe inter-relations between input parameters and product properties (Awolu and Layokun 2013).

Statements of Problem

Limitation of values

- Despite the numerous food and economic values imbedded in catfish (*Clarias gariepinus*), accessing these values is limited.

•Inefficient and prevailing traditional methods of fish processing and preservation in Nigeria (Eyo, 2001, Oladele and Oladeji, 2008; Olayemi, 2012).

•Adverse colour and aroma changes, as well as health risk associated with smoked catfish (Fronthea, 2003; Adeyeye *et al*., 2016).

• Limited affordable modern dehydration techniques / expensive and sophisticated equipment/operations in some other fish preservation methods like freezing, freeze drying, canning, etc (George, 2010).

•Product instability during storage leading to dried fish losses (Aworh *et al*., 2002; Ward, 2003).

Justification for the research

Simple dehydration technique with effective process parameters for catfish preservation could serve as an improvement over the traditional and alternative to the sophisticated modern methods.

Using simple mathematical expressions (models) to describe the data obtained from the analysis of chemical properties of dehydrated catfish processed under optimal parameters, could serve as a tool for quick predictive purposes.

Objectives

The specific objectives of this work are to:

- develop the process parameters interactions using RSM,
- determine the chemical properties of dehydrated catfish,
- assess the potential of RSM for predicting the proximate composition and rancidity indices of dehydrated catfish,
- establish the optimum conditions for simple processing method (dehydration) for catfish.

Materials and Methods

Materials

- Freshly harvested headless and eviscerated catfish (*Clarias gariepinus*) of average weight 254 g were used in the study,

- common salt (Mr Chef brand) was used to make brine solution,

- convective electric dryer was used for the drying of catfish and

- the chemicals used for the analyses were of analytical grade.

Response Surface Methodology (RSM) design

Design

- A three-factor face centered central composite design (CCD) was used.

Independent variables / factors

- Brine concentration (3.0, 6.0, 9.0%), brining time (30, 60, 90 min) and drying temperature (90, 110, 130 ^{o}C).

Table 1: Response surface Methodology (RSM) design runs

Run	A: Brine Concentration (%)	B: Brining Time (min)	C: Drying Temperature (^oC)
1	6	60	110
2	6	90	110
3	3	60	110
4	3	30	90
5	6	30	110
6	9	90	130
7	9	60	110
8	6	60	110
9	9	90	90
10	6	60	130
11	3	90	130
12	6	60	110
13	6	60	90
14	3	90	90
15	3	30	130
16	9	30	130
17	9	30	90

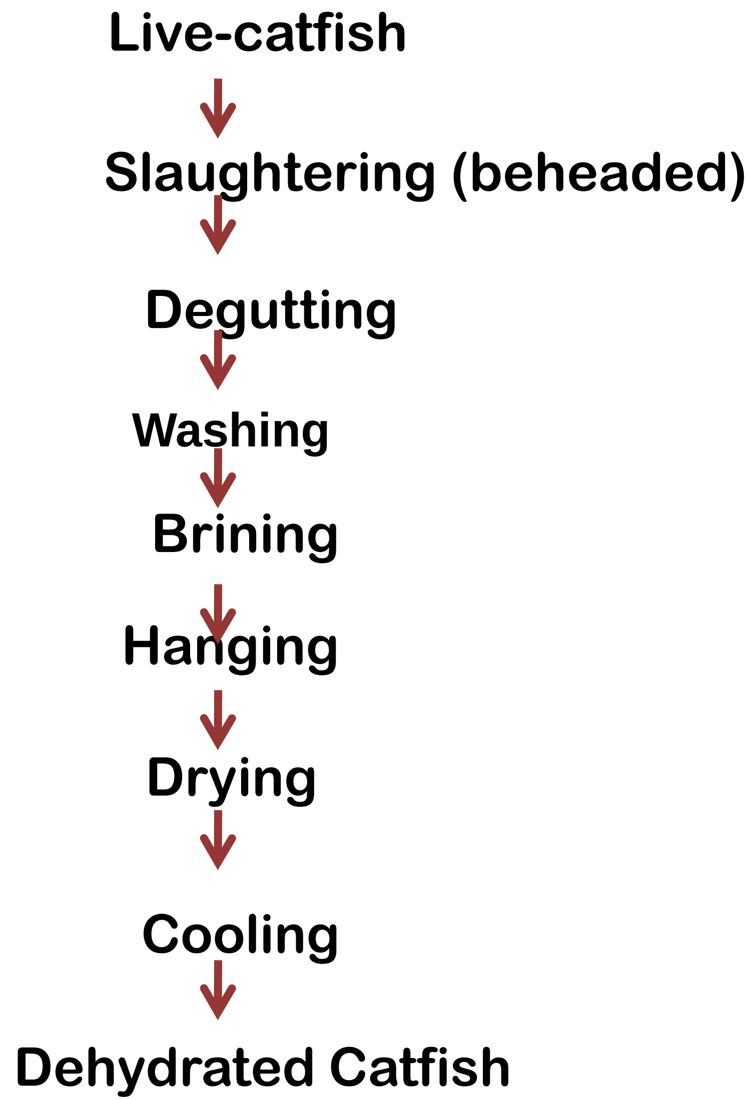

Figure 1: Processing of dehydrated catfish (Eyo, 2001).

Chemical analyses

Proximate composition (moisture, crude protein, fat and ash contents)

- Proximate composition (AOAC, 2005).

pH, FFA, TBA, TVN and PV

- pH - digital pH meter (98107).
- Free fatty acid, thiobarbituri acid, total volatile nitrogen and peroxide value were determined by Pearson (1976) method.

Modelling / process optimisation

- The proximate composition and rancidity indices' data was modelled via the RSM analysis. High coefficient of determination (R^2), Adjusted R^2, Predicted R^2 and lack of fit p- values indicates the fit of model. Transformation and backward regression were used to improve the fit.

The fitted quadratic response model is as described in Eq. 1 (Awolu and Layokun 2013) .

$$Y = b_o + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ij} X_i^2 + \sum_{i1<j}^{k} \sum_{j}^{k} b_i X_i X_j + e \quad (1)$$

- Numerical optimisation based on desirability concept was done to predicted the optimal dehydration conditions.

Data analysis/Software

- Analysis of variance (ANOVA) was used to establish differences among treatments and Duncan's multiple range tests were used to separate the means. Significance difference was accepted at $p < 0.05$. SPSS version 17 was used.

- Design expert (DX 10.0.1) was used for the RSM statistical analysis and modelling of data.

RESULTS

Table 2: Proximate compositions of dehydrated catfish

RSM run	A	B	C	Moisture content (%)	Crude Protein (%)	Crude fat (%)	Ash (%)
1	6	60	110	$5.81^{ghi}\pm0.01$	$64.06^{d}\pm0.01$	$22.33^{fg}\pm0.29$	$11.00^{cde}\pm0.00$
2	6	90	110	$5.47^{h}\pm0.01$	$61.15^{f}\pm0.00$	$22.60^{fg}\pm0.52$	$12.29^{bc}\pm0.61$
3	3	60	110	$7.80^{bc}\pm0.24$	$55.74^{o}\pm0.01$	$26.60^{cd}\pm0.90$	$8.85^{gh}\pm0.15$
4	3	30	90	$7.59^{c}\pm0.02$	$59.86^{k}\pm0.01$	$26.20^{bcd}\pm1.53$	$10.00^{efg}\pm1.00$
5	6	30	110	$6.41^{de}\pm0.02$	$59.93^{j}\pm0.01$	$24.70^{de}\pm0.30$	$10.77^{def}\pm0.23$
6	9	90	130	$6.64^{d}\pm0.01$	$55.10^{p}\pm0.01$	$27.10^{b}\pm1.01$	$12.05^{bcd}\pm0.95$
7	9	60	110	$6.18^{efg}\pm0.00$	$60.04^{i}\pm0.01$	$23.50^{ef}\pm0.50$	$11.00^{cde}\pm0.00$
8	6	60	110	$5.75^{gh}\pm0.00$	$64.33^{c}\pm0.01$	$19.17^{h}\pm0.29$	$11.00^{cde}\pm0.00$
9	9	90	90	$5.50^{h}\pm0.10$	$61.90^{e}\pm0.01$	$19.30^{h}\pm0.32$	$14.00^{a}\pm1.00$
10	6	60	130	$6.06^{efgh}\pm0.00$	$55.79^{n}\pm0.01$	$26.40^{bc}\pm0.68$	$9.32^{gh}\pm0.05$
11	3	90	130	$6.60^{d}\pm0.03$	$60.10^{h}\pm0.01$	$21.64^{g}\pm1.73$	$9.57^{fgh}\pm0.54$
12	6	60	110	$6.13^{efgh}\pm0.01$	$61.09^{g}\pm0.01$	$19.67^{h}\pm0.64$	$10.77^{def}\pm0.23$
13	6	60	90	$6.30^{def}\pm0.10$	$65.26^{b}\pm0.01$	$17.00^{i}\pm0.50$	$11.00^{cde}\pm1.00$
14	3	90	90	$8.41^{a}\pm0.01$	$59.84^{l}\pm0.02$	$26.50^{bc}\pm0.00$	$9.11^{gh}\pm0.84$
15	3	30	130	$6.02^{fgh}\pm0.81$	$53.34^{q}\pm0.03$	$30.80^{a}\pm0.52$	$8.33^{h}\pm0.93$
16	9	30	130	$7.97^{fgh}\pm0.00$	$57.65^{m}\pm0.00$	$21.30^{g}\pm1.30$	$11.46^{bcd}\pm1.48$
17	9	30	90	$8.40^{a}\pm0.18$	$68.85^{a}\pm0.01$	$10.70^{j}\pm1.44$	$12.50^{b}\pm0.50$
FRF				76.12	17.29	2.93	1.68

Values are means of triplicate determinations ± standard deviations. Means with different superscript along the columns are significantly different ($p<0.05$). A = Brine concentration (%); B = Brining time (min); C = Drying temperature (^{o}C); FRF = fresh raw fish.

Table 3: Rancidity indices of dehydrated catfish

RSM run	A (%)	B (min)	C (°C)	pH	FFA (%)	TBA (mg M/kg)	TVN (mg N/100g)
1	6	60	110	6.10^{f}±0.09	1.28^{bcd}±0.05	0.031^{kl} ±0.000	9.60^{h}±0.10
2	6	90	110	6.10^{f}±0.00	1.19^{ab}±0.29	0.043^{jk} ±0.000	9.80^{h}±0.10
3	3	60	110	6.15^{ef}±0.05	1.49^{a}±0.02	0.133^{ab} ±0.000	19.60^{b}±0.21
4	3	30	90	6.30^{c}±0.00	0.91^{f}±0.06	0.141^{a} ±0.000	14.00^{f}±0.10
5	6	30	110	6.15^{ef}±0.05	1.34^{bc}±0.04	0.062^{hij} ±0.000	5.60^{i}±0.00
6	9	90	130	6.30^{c}±0.00	0.73^{g}±0.04	0.110^{fgh} ±0.060	14.00^{f}±0.50
7	9	60	110	6.10^{f}±0.00	0.88^{f}±0.00	0.104^{cd}±0.000	18.20^{c}±0.00
8	6	60	110	6.20^{de}±0.00	1.28^{bcd}±0.03	0.048^{ijk}±0.000	14.00^{f}±0.00
9	9	90	90	6.20^{de}±0.00	1.20^{cde}±0.02	0.092^{cdef}±0.000	14.00^{f}±0.00
10	6	60	130	6.20^{de}±0.00	1.18^{de}±0.03	0.070^{ghi}±0.000	12.60^{g}±0.20
11	3	90	130	6.50^{a}±0.00	1.21^{cde}±0.00	0.016^{l}±0.000	15.40^{e}±0.10
12	6	60	110	6.00^{g}±0.00	1.20^{cde}±0.02	0.031^{kl}±0.000	14.00^{f}±0.00
13	6	60	90	6.00^{g}±0.00	1.33^{bc}±0.01	0.084^{defg}±0.000	14.00^{f}±0.00
14	3	90	90	6.25^{cd}±0.05	1.17^{de}±0.04	0.102^{cde}±0.000	14.00^{f}±0.00
15	3	30	130	6.45^{ab}±0.05	1.10^{e}±0.03	0.094^{cdef}±0.000	21.00^{a}±0.09
16	9	30	130	6.40^{b}±0.00	0.78^{fg}±0.03	0.110^{bc}±0.000	14.00^{f}±0.00
17	9	30	90	6.10^{f}±0.00	0.47^{h}±0.00	0.133^{ab}±0.000	16.80^{d}±0.00
STD	-	-	-	≤6.5	1.38	1-2	≤25

Values are means of duplicate determinations ± standard deviations. Means with different superscript along the columns are significantly different ($p < 0.05$).

Key: RSM = Response surface methodology; A = Brine concentration, B = Brining time, C = Drying temperature; FFA = Free fatty acid; TBA = Thiobarbituric acid; TVN = Total volatile nitrogen; STD = Standard. **NB:** PV was below detectable level in all the runs-(Bragadotirr, *et al*., 1998-0.0 meq O_2/kg in salted and dried capelin fish).

Table 4: ANOVA and regression coefficients of the RSM models describing the effects of processing parameters on the chemical properties of dehydrated catfish

Response	Moisture content	Crude protein	Crude fat	Ash	pH	FFA	TBA	TVN
Model	RQuad.	2FI	2FI/Sqrt (Fat)	Linear	RQuad.	RQuad.	RQuad.	RQuad.
Model (p-value)	0.0005	0.0064	0.0013	<0.0001	0.0004	0.0063	0.0032	0.0058
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	0.0010	ns	ns	ns
AB	0.0012	0.0342	0.0057	ns	ns	ns	ns	ns
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	0.0070
C^2	na	na	na	na	na	na	na	na
LOF p-value	0.1595	0.3942	0.3494	0.0290	0.8933	0.0638	0.1446	0.6830
R^2	0.8754	0.7858	0.8483	0.8088	0.8430	0.7332	0.7662	0.7101
Adj. R^2	0.8007	0.6572	0.7573	0.7646	0.7717	0.6119	0.6599	0.6135
Pred. R^2	0.4448	0.4545	0.6025	0.6596	0.6441	0.2541	0.4039	0.3812
Adeq. Precision	9.848	10.997	13.680	14.444	11.659	8.003	8.252	9.924

*$p < 0.05$

Key: FFA = Free fatty acid; TBA = Thiobarbituric acid; TVN = Total volatile nitrogen; RQuad. = Reduced Quadratic; Sqrt = Square root; A = Brine concentration; B = Brining time; C = Drying temperature; LOF = Lack of fit; R^2 = Coefficient of determination; Adj = Adjusted; Pred. = Predicted; Adeq = Adequate.

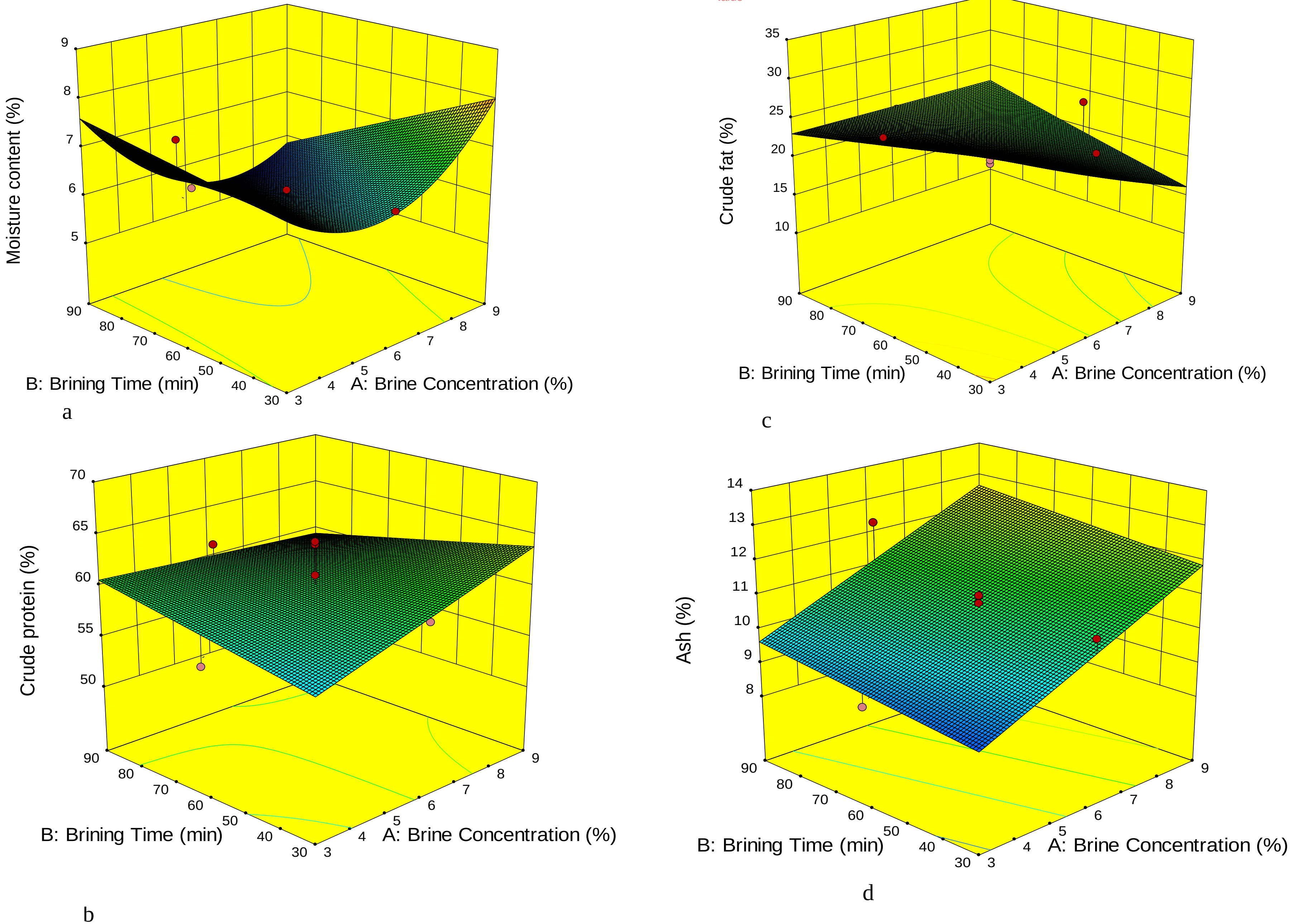

Figure 2 (a-d): Response surface 3D plots describing the effect of brining time and brine concentration at a fixed drying temperature (110°C) on the proximate composition of dehydrated catfish.

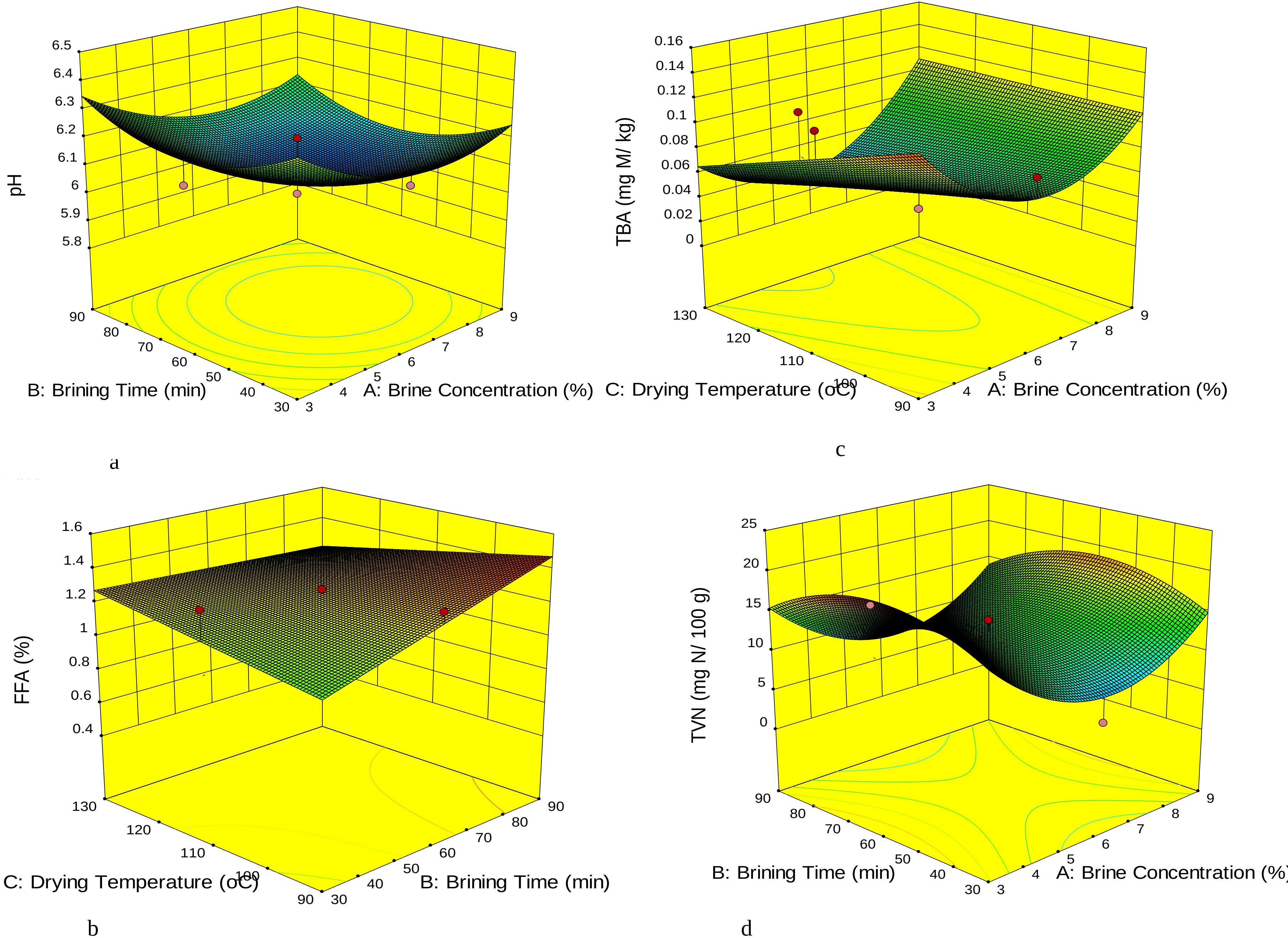

Figure 3 (a-d): Response surface 3D plots describing the effect of processing parameters on rancidity indices of dehydrated catfish

Final equations in terms of coded factors (the developed models) for the proximate composition and rancidity indices of dehydrated catfish

Moisture content = +5.99-0.17*A -0.38*B-0.29*C-0.70*A*B + 0.51*A*C + 1.12*A^2

Crude protein = +60.24+1.47*A-0.15*B -3.37*C-2.03*A *B-1.47*A *C +1.40*B*C

Sqrt (Crude fat) = +4.73-0.33*A+0.063*B+0.31*C+0.32*A*B+0.28*A*C-0.18*B*C

Ash = +10.77+1.52*A+0.40*B-0.59*C

pH = +6.07-0.055*A-5.000E-003*B+0.10*C+0.12*A^2+0.12*B^2

FFA = +1.26-0.18*A+0.090*B-8.000E-003*C-0.12*B *C-0.26*A^2

TBA = +0.053+6.300E-003*A-0.018*B-0.015*C+0.016*A*C +0.051*A^2

TVN = +12.59-0.70*A-0.42*B+0.42*C-1.40*A *C+6.93*A^2-4.27*B^2

where, A = Brine concentration; B = Brining time; C = Drying temperature; Sqrt = Square root; FFA = Free fatty acid; TBA = Thiobarbituric acid; TVN = Total volatile nitrogen.

Process optimisation Result

The optimal dehydration conditions based on desirability concept were predicted at 7.83% brine concentration, 90 min brining time, and 110.38^{o}C drying temperature with the highest and good desirability value of 0.598 (approximately 60% level) as indicated in (Fig. 4). The predicted conditions were adjusted to 8.0% brine, 90 min and 110^{o}C for experimental conveniences, bearing in mind the setting of the temperature regulator on the dryer used.

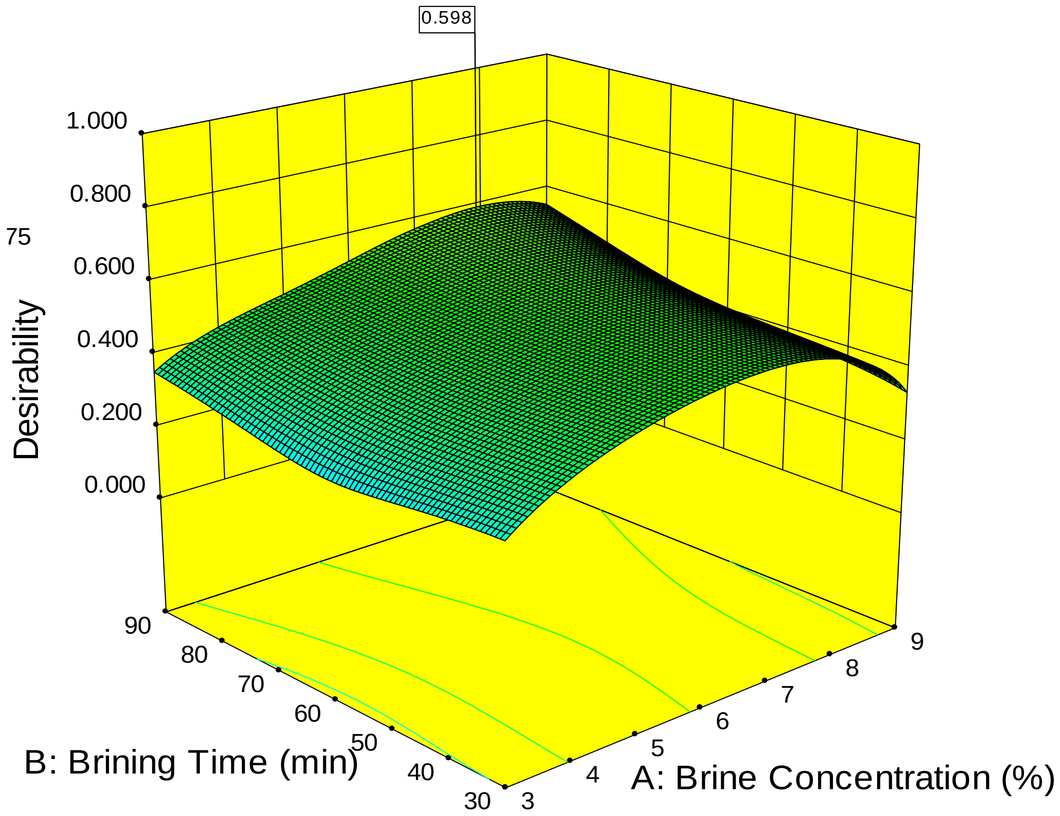

Figure 4: Response surface 3D plot showing multi-response optimisation conditions for the dehydration of catfish

Table 5: Experimental values for responses under multi-response optimization conditions

Response	Predicted	95% CI low	95% CI high	Validation
Moisture content (%)	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 M/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

CI = Confidence Interval; n = 1; a = 0.05

Conclusions

❖Processing parameters significantly improved the chemical quality of dehydrated catfish.

❖Adequate predictions of the tried models shows good agreement with the experimental and adequate for predictive purposes showing the RSM potential as a feasible tool in this regard.

❖ Optimal dehydration conditions (8% brine, 90 min, 110 ^{o}C) for catfish has been established for high retention of nutrients and good rancidity properties for storage stability. These could ameliorate the problem of malnutrition especially where fresh fish is less accessible.

❖Finally, the dehydration technique employed in this investigation is effective, simple in operation and economical to encourage commercial application with a potential for food security.

Recommendation

Suggested further work;

- amino acids profile of the product may be necessary to further ascertain the established nutritional quality of the dehydrated catfish (*Clarias garipinus*).

Contribution to knowledge

This work has been able to:

•establish a simple dehydration technique that could preserved the quality of dehydrated catfish on shelf and during storage,

•provide innovative approach of using a statistical tool (like RSM) for modelling and optimising the chemical properties of dehydrated catfish.

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