Effect of extraction variables on the proximate composition of coconut milk: a response surface approach

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ABSTRACT

To our knowledge, there seem to be no report on the effect of extraction variables on the proximate composition of coconut milk. In this study, response surface methodology (RSM) with a central composite design (CCD), consisting three factors (extraction time, extraction temperature and coconut meat particle size) was used to study the effect of process conditions on the proximate compositions of coconut milk. Results revealed that process variables significantly (p<0.05) affected the proximate compositions of coconut milk. The R² values of ash, fibre and carbohydrate were 0.9244, 0.8822 and 0.8876 respectively, while that of fat and protein were 0.6048 and 0.6866, respectively. Results also indicated that moisture, ash, fat, protein, fibre and carbohydrate contents of coconut milk ranged from 75.40 to 81.97%, 1.03 to 4.3%, 62.70 to 78.39%, 6.33 to 32.16%, 0.05 to 0.15% and 2.25 to 60.0% respectively. The optimum conditions obtained for extraction of coconut milk with a desirability index of 75.30% were 16.27 min extraction time, 40°C extraction temperature and < 1617 μm particle size of coconut meat. The estimated amount of moisture, ash, fat, protein, fibre and carbohydrate were 79.03, 3.48, 73.82, 25.45, 0.16 and 29.34% respectively.

Keywords: Coconut milk extraction optimization central composite design response surface methodology proximate compositions

Introduction

Coconut palm (Cocos nucifera), family Palmacea, is a large palm growing up to 30 m (98 ft) tall, with pinnate leaves 4-6 m (13-20 ft) long (Pradeepkumar et al., 2008; Alonge and Adetunji, 2011). Coconut palm is one of the most important cultivated trees which grow extensively in Nigeria. It is referred to as the “tree of life” because of its different uses (Alonge and Adetunji, 2011). The fruit may be eaten as a snack or used for various food and industrial applications.

Coconut water serves as a source of sugar. It is also used as a delicious, non-alcoholic beverage and often substituted for blood plasma in emergency surgical operation (Alonge and Adetunji, 2011). Coconut oil is used for cooking and as cream. Coconut meat (analogue) is highly nutritious and rich in vitamins and minerals. It is classified as a “functional food” because it provides many health benefits beyond its nutritional content (Edem, 2016).

Coconut milk is the natural oil-in-water emulsion extracted from the endosperm of mature coconut (Cocos nucifera) (Tangsuphoom and Coupland, 2009; Raghavendra and Raghavarao, 2010) and it plays an important role in many traditional foods of Asian and Pacific regions (Chiewchan and Phungamngoen, 2006), including Nigeria. Coconut milk is rich in oils and various nutrients. It is used for sauces, prepared foods (Edem, 2016) as well as serving as an important raw material in the production of other dairy like products such as yoghurt (Edem and Elijah, 2016a). The colour and rich taste of the milk can be attributed to the high oil content and sugars. Coconut milk is obtained from finely grated coconut meat that is steeped in hot water and then filtered (Edem and Elijah, 2016b). Coconut milk is rich in phosphorus. The milk supplies the body with nearly a quarter of daily value of iron thereby resulting in the

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prevention of anaemia (Belewu et al., 2013). The milk helps to decrease the risk of joint inflammation due to its high selenium content, and could promote the health of prostrate gland when consumed (Belewu et al., 2005). In Nigeria, the extraction of coconut milk is usually done manually and thus it is time and energy consuming. Extraction is carried out under diverse process conditions resulting in inconsistent quality products. However, an optimum extraction condition of coconut milk has been established by Edem and Elijah (2016b) to be: extraction time, extraction temperature and coconut meat particle size of 15 min, 40 °C and ≤ 1617 μm, respectively.

It is believed that factors such as extraction time, extraction temperature and particle size of coconut meat could have great influence on the quality of coconut milk (Edem and Elijah, 2016c). This is evident in a recent study conducted by Elijah et al. (2018) on the effect of extraction variables on the mineral composition of coconut milk. The study found that extraction variables stated above had significant effect on the mineral composition of coconut milk.

To the best of our knowledge, the effect of extraction time, extraction temperature and coconut meat particle size on the proximate composition of coconut milk has not been documented. Thus, this research was undertaken to determine the effect of extraction variables on the proximate composition of coconut milk using response surface methodology. Response surface methodology has been successfully applied in optimization processes in food and found to be an effective tool.

Materials and methods

Sample collection

Matured coconuts (7-8 months old) of the dwarf variety were collected directly from the coconut trees in EmVic farm in Ibesikpo Asutan Local Government Area, Akwa Ibom State, Nigeria and transported to the Processing Laboratory of the Department of Food Science and Technology, University of Uyo, Uyo, Akwa Ibom State, Nigeria, for analysis.

Extraction of coconut milk

Coconut milk was extracted following the method reported by Edem and Elijah (2016b) with slight modification. The coconut was dehusked and cracked to separate the meat from the shell. The coconut water obtained was transferred into a container and stored for further use. The brown skin of the coconut meat was carefully removed and the meat thoroughly washed and then grated using a manual grater fabricated by the Department of Food Engineering, University of Uyo, Nigeria, with different particle size numbers. The grated coconut meat was mixed in a ratio of 1:1 with a solution containing 75% distilled water and 25% coconut water, and allowed to stand in a water bath at temperatures (33.18 – 66.81 °C) and time (6.59 – 23.4 min) specified by the experimental design. The resulting slurry was pressed and filtered through cheese cloth to separate the milk. Coconut milk obtained was pasteurized at 90 °C for 30 min and allowed to assume room temperature (37 °C) before analyzing for proximate composition. Based on the experimental design, a total of 20 coconut milk samples, of varied extraction temperature, extraction time and coconut meat particle sizes (as shown in Table 2), were obtained.

Proximate composition of coconut milk

The proximate composition of coconut milk was determined according to the AOAC (2005) methods.

Experimental design and statistical analysis

The experimental variables (range and levels) for the effects of extraction conditions on proximate composition of coconut milk are presented in Table 1. Effect of extraction variable on proximate composition of coconut milk was determined using Design Expert software version 10.0 (Stat-Ease Inc., Minneapolis, MN, USA). Response Surface Methodology (RSM) with a Central Composite Design (CCD) was used to study linear, interaction and quadratic effects of extraction variables on proximate composition of coconut milk. Three (3) independent variables; extraction time (X1), extraction temperature (X2) and coconut meat particle size (X3), each having 5 different coded levels from low (-1), to medium (0) and high (+1) as well as star points (+α), were used for efficient determination of curvature and quadratic term. The dependent variables, moisture, ash, fat, protein, fibre and carbohydrate contents of the extracted milk, were evaluated as responses. The design matrix of the CCD and experimental runs are presented in Table 2. The effect of independent variables on the dependent variables was assessed using multiple regression analysis (Elijah et al., 2018). The full quadratic equation of the response variables for coconut milk extraction was derived using RSM as follows:

\[ Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \]  

(1)

where Y is the response; \( \beta_0 \) is the constant; \( \beta_1, \beta_2, \beta_3 \) are the coefficients of linear regression; \( \beta_{12}, \beta_{13}, \beta_{23} \) are the coefficients of interaction regression; \( \beta_{11}, \beta_{22} \) and \( \beta_{33} \) are the coefficients of quadratic regression and \( X_1, X_2, X_3 \) are the independent variables.
Table 1. Experimental range and levels of the independent variables

| Independent Variables | Factor | Unit   | -α   | -1   | 0     | +1   | +α   |
|-----------------------|--------|--------|------|------|-------|------|------|
| Time                  | X₁     | Min    | 6.591| 10   | 15    | 20   | 23.409|
| Temperature           | X₂     | °C     | 33.182| 40   | 50    | 60   | 66.8179|
| Particle size         | X₃     | µm     | 1114.86| 1617 | 2353.5| 3090| 3592.14|

Results and discussion

Fitting the model and response surface analysis

Following the experimental design, results of RSM experimental runs on the effect of extraction time (X₁), extraction temperature (X₂) and coconut meat particle size (X₃) on the proximate composition of coconut milk are presented in Table 2. Results show that moisture, ash, fat, protein, fibre and carbohydrate contents of coconut milk ranged from 75.40 to 81.97%, 0.13 to 4.33%, 62.70 to 78.39%, 6.33 to 32.16%, 0.05 to 0.15% and 2.25 to 60.0%, respectively. These experimental data were used to generate the best predicted model and its statistical analysis using RSM software.

The fitness of the final predicted polynomial model was estimated using the results of analysis of variance (ANOVA) presented in Table 3. The regression model which had the highest R², including other parameters were shown to be significant (p<0.05) and effective in describing the effect of independent variables on the dependent variables. For this reason, moisture, ash and fibre were fitted to quadratic model. Fat and protein were fitted to reduced quadratic model, because there were a number of insignificant terms in the regression model, while carbohydrate was fitted to reduced linear model. In order to develop a statistically significant regression model, the insignificant terms were removed from the model thus resulting in a model’s efficiency in describing response (Sudamalla et al., 2012; Edem and Elijah, 2016 b). However, the model’s result for moisture content was not significant, which indicated that this parameter could not be explained by regression model and the independent variables had little or no effect to the response (moisture content). Hence, further action was inconsequential as the mean value is a better predictor of the response.

Table 3 Analysis of variance (ANOVA) for the fitted polynomial model of the dependent variables

| Response     | Source   | Df | Sum of sq. | Mean sq. | F-value | p-value |
|--------------|----------|----|------------|----------|---------|---------|
| Moisture     | Model    | 1  | 19.97      | 6.66     | 2.28    | 0.1188  |
|              | Residual | 16 | 46.78      | 2.92     |         |         |
|              | Lack-of-fit | 11 | 35.06      | 3.19     |         |         |
|              | Pure error | 5  | 11.72      | 2.34     |         |         |
|              | Total    | 19 | 66.74      |          |         |         |
| Ash          | Model    | 9  | 26.38      | 2.93     | 13.58   | 0.0002*|
|              | Residual | 10 | 2.16       | 0.22     |         |         |
|              | Lack-of-fit | 5  | 2.11       | 0.42     |         |         |
|              | Pure error | 5  | 0.044      | 8.817E-003|         |         |
|              | Total    | 19 | 28.54      |          |         |         |
| Fat          | Model    | 2  | 1156.28    | 578.14   | 8.67    | 0.00025*|
|              | Residual | 17 | 1134.14    | 66.71    |         |         |
|              | Lack-of-fit | 12 | 1130.97    | 94.25    |         |         |
|              | Pure error | 5  | 3.17       | 0.63     |         |         |
|              | Total    | 19 | 2290.42    |          |         |         |
| Protein      | Model    | 6  | 421.80     | 70.30    | 3.07    | 0.0424*|
|              | Residual | 13 | 297.27     | 22.87    |         |         |
|              | Lack-of-fit | 8  | 286.95     | 35.87    |         |         |
|              | Pure error | 5  | 10.33      | 2.07     |         |         |
|              | Total    | 19 | 719.08     |          |         |         |
| Fibre        | Model    | 9  | 0.023      | 2.585E-003| 8.32    | 0.0014*|
|              | Residual | 10 | 3.107E-003 | 3.107E-004|         |         |
|              | Lack-of-fit | 5  | 3.107E-003 | 6.214E-004|         |         |
|              | Pure error | 5  | 0.000      | 0.000    |         |         |
|              | Total    | 19 | 0.026      |          |         |         |
| Carbohydrate | Model    | 1  | 2045.05    | 2045.05  | 9.17    | 0.0072*|
|              | Residual | 18 | 4012.51    | 222.92   |         |         |
|              | Lack-of-fit | 13 | 2797.11    | 215.16   |         |         |
|              | Pure error | 5  | 1215.40    | 243.08   |         |         |
|              | Total    | 19 | 6057.56    |          |         |         |

*Values are significant at p = 0.05
The model’s goodness-of-fit was ascertained by the coefficient of determination ($R^2$) presented in Table 4. Jusoh et al. (2013) noted that the best $R^2$ value for a good model fitting is between 0.8 and 1.0. The $R^2$ values of ash, fat, protein, fibre and carbohydrate were 0.9244, 0.6048, 0.6866, 0.8822 and 0.8876, respectively. This indicated that over 80% variations in the observed values of ash, fibre and carbohydrate were explained by respective regression models. However, $R^2$ less than 0.8 had been reported previously (Gupta et al., 2014; Edem and Elijah, 2016b), signifying a fair fit of the model, yet reliable in making predictions. Thus, $R^2$ of 0.6048 and 0.6866 for fat and protein respectively, indicated that the models were fairly fitted. This showed that only 60.48% variations in fat and 68.66% variations in protein were explained by respective models.

**Effect of extraction time, extraction temperature and coconut meat particle size on ash content of coconut milk**

The ash content of coconut milk as affected by extraction time ($X_1$), extraction temperature ($X_2$) and coconut meat particle size ($X_3$) is presented in Table 4. Regression model showing the effect of process variables on ash content of coconut milk is given in equation 2.

$$\text{Ash} = 4.30 + 0.31X_1 + 0.46X_1X_3 + 0.88X_2X_3 - 0.77X_1^2 - 0.42X_2^2 - 0.78X_3^2 \quad (2)$$

Result shows that extraction temperature, interaction of extraction time and temperature, interaction of extraction temperature and coconut meat particle size and quadratic effects of extraction time, extraction temperature and coconut meat particle size had significant ($p<0.05$) effect on the ash content of coconut milk (Table 4). In addition, the coefficients of estimate (Table 4) represent the relative impact of process variables on the ash content of coconut milk. Positive and negative coefficients in the model indicate synergistic and antagonistic effects. The main effect of extraction temperature (Fig. 1a) showed that increase in extraction temperature resulted in increase in ash content of the coconut milk. Similarly, the interaction effect of extraction time and coconut meat particle size led to a gradual increase in the ash content of the coconut milk (Fig. 1b). This may be the result of increase in time which could increase the diffusion rate of soluble components of coconut and consequently increase ash content of the coconut milk. The effect of extraction temperature and coconut meat particle size resulted in increased ash content of coconut milk (Fig. 1c). Similar result was reported by Elijah et al. (2018) who found out that the interaction between extraction temperature and coconut meat particle size had a synergistic effect which resulted in increased mineral elements of coconut milk studied.

**Effect of extraction time, extraction temperature and coconut meat particle size on the fat content of coconut milk**

The estimated regression (Table 4) showing the effect of extraction time ($X_1$), extraction temperature ($X_2$) and coconut meat particle size ($X_3$) on the fat content of coconut milk is expressed by the equation (3).

$$\text{Fat} = 73.68 - 6.46X_1 - 6.32X_3^2 \quad (3)$$

### Table 4. Estimated regression coefficients, coefficients of determination of the fitted polynomial model for the proximate composition of coconut milk at the design response surface

| Source Model | Moisture Coef | Ash Coef | Fat Coef | Prot. Coef | Fibre Coef | CHO Coef |
|--------------|---------------|----------|----------|------------|------------|----------|
| Constant     | 79.23         | 4.30     | 73.68    | 19.62      | 0.15       | 17.10    |
| Linear       | -             | -        | -        | -          | -          | -        |
| Interaction  | -             | -        | -        | -          | -          | -        |
| Quadratic    | -             | -        | -        | -          | -          | -        |
| $\beta_0$    | 3.06          | 0.0995   | -        | NS         | -          | NS       |
| $\beta_1$    | 0.31          | 1.21     | 0.042    | 0.0067     | 0.0003     | -        |
| $\beta_2$    | 0.46          | 0.0193   | 0.46     | 0.77       | 0.46       | 0.46     |
| $\beta_3$    | 0.88          | 0.0003   | 0.0001   | 0.88       | 0.46       | 0.46     |
| $\beta_1^2$  | -             | -        | -        | -          | -          | -        |
| $\beta_2^2$  | -             | -        | -        | -          | -          | -        |
| $\beta_3^2$  | -             | -        | -        | -          | -          | -        |
| $R^2$        | 0.924         | 0.6048   | 0.6866   | 0.8822     | 0.8876     | 0.8876   |

Coef = coefficient; p.v=p-value; NS=not significant values; * = Values are significant at $p =0.05$
Fig 1. Effect of extraction time and extraction temperature on ash content of coconut milk; Effect of extraction time and particle size of coconut meat on the ash content of coconut milk; Effect of extraction temperature and particle size of coconut meat on the ash content of coconut milk

Result shows that coconut meat particle size and quadratic effect of coconut meat particle size had significant (p<0.05) effect on the fat content of coconut milk. However, extraction temperature, extraction time and interaction of time and temperature were not significant. The negative main effect of coconut meat particle size indicates that increase in coconut meat particle size (Fig. 2) resulted in decreased fat content of coconut milk. This could be due to the fact that larger particle size has a smaller contact surface area and is more resistant to solvent entrance and subsequent diffusion of the soluble components of coconut milk (Sayyar et al., 2009).

Similar results were reported by Edem and Elijah (2016b); the larger the particle size of coconut meat, the lower the yield and by and Elijah et al. (2018), who claimed that increased coconut milk particle size resulted in decreased amount of the mineral elements of the milk.

Effect of extraction time, extraction temperature and coconut meat particle size on the protein content of coconut milk

The protein content of coconut milk was found to be a function of the quadratic effect of extraction time only. The estimated regression showing this effect is expressed by the equation (4).

\[ \text{Protein} = 19.62 + 3.06X_1^2 \quad (4) \]

Result indicates that quadratic term of time was the only variable that had significant (p<0.05) effect on the protein content of coconut milk. A negative coefficient of quadratic time indicates an antagonistic effect of extraction time on protein. Figure (3) revealed that increase in extraction time resulted in increased protein of coconut milk.

Effect of extraction time, extraction temperature and coconut meat particle size on the fibre content of coconut milk

The fibre content of coconut milk ranged from 0.05 to 0.15%, indicating that the variation between the maximum and minimum value of fibre content was 0.1% during extraction at different process variables. The interaction effect of extraction temperature and coconut meat particle size, as well as the quadratic terms of time and particle size of coconut meat, had significant (p<0.05) effects on the fibre content of coconut milk. The estimated regression showing this effect is expressed by the following equation (5).

\[ \text{Fibre} = 0.15 + 0.038X_1^2 X_3 - 0.019X_1^2 - 0.019X_3^2 \quad (5) \]

The positive coefficient of interaction of extraction temperature and coconut meat particle size indicated a synergistic effect on fibre content of coconut milk (Figure 4a). Figure 4a showed that increase in extraction temperature and particle size of coconut meat resulted in an increase in the fibre content of coconut milk. Also, the negative coefficients of quadratic time and quadratic particle size of coconut meat showed that there is a turning point (15 min for time and 2353.5 µm for particle size of coconut meat). Everything beyond this limit time and particle size of coconut meat had a negative effect on fibre content of coconut milk (Fig. 4b). This is evident in Figure 4b having a concave shape.
Fig 2. Effect of extraction time and particle size of coconut meat on the fat content of coconut milk

Fig 3. Effect of extraction time and extraction temperature on protein content of coconut milk

Fig 4. Effect of extraction temperature and particle size of coconut meat on fibre content of coconut milk; Effect of extraction time and particle size of coconut meat on fibre content of coconut milk

Effect of extraction time, extraction temperature and coconut meat particle size on the carbohydrate content of coconut milk

Result shows that the main effect of coconut meat particle size was the only significant term influencing the carbohydrate content of coconut milk. However, extraction time, temperature, interaction effects of process variables and quadratic terms of extraction time, extraction temperature and coconut meat particle size showed no significant effect on carbohydrate content of coconut milk. The relationship showing this effect is expressed by the following equation (6).

\[
\text{Carbohydrate} = 17.10 - 12.24X_3 \\
\]

The 3-D surface model graph (Fig. 5) showed that, irrespective of time, coconut meat particle size is a major factor influencing the carbohydrate content of coconut milk. The increase in coconut meat particle size led to decreased carbohydrate content of coconut milk.

Process Optimization

Considering the proximate composition of coconut milk, the optimum extraction time (X_1), extraction temperature (X_2) and coconut meat particle size (X_3), for extraction of coconut milk with the optimal conditions at the highest desirability index of 0.753 (75.30%) to obtain the highest quantity of moisture, ash, fat, protein, fibre and carbohydrate were 16.27 min extraction time, 4! °C extraction temperature and <1617 μm particle size of coconut meat. The estimated amount of moisture, ash, fat, protein, fibre and carbohydrate were 79.03, 3.48, 73.82, 25.45, 0.16 and 29.34%, respectively.
Conclusions

This study has clearly shown the application of RSM in the study of effect of extraction time, extraction temperature and particle size of coconut meat on proximate composition of coconut milk. Results indicated that extraction time, extraction temperature and particle size of coconut meat had a significant effect on proximate composition of coconut milk. The optimum extraction time, extraction temperature and particle size of coconut meat were estimated at 16.27 min, 40 °C and <1617 µm, respectively. Thus, within the experimental range and conditions used in this study, the respective regression models can be used to make predictions on the effect of independent variables on proximate composition of coconut milk.

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