

## Optimisation of Kernels Preparation Conditions Involved in the Press Extraction of Shea (*Vitellaria paradoxa* Gaertner F.) Butter

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**Abstract** In this study, different conditions of press extraction of shea butter were optimised. Response Surface Methodology (RSM) using the Doehlert experimental design has been employed in the optimisation. The independent variables considered were roasting time (0-60 min), roasting temperature (50-300°C), cooking time (0-300 min) and cooling time (0 -60 min). Four-second order polynomial models were generated. The models were well described the responses: extraction yield, acid value, peroxide value and Lovibond red colour of the process with satisfactory fits in terms of absolute average deviation and coefficient of determination. The optimum conditions were found to be 10-15 min roasting time, 160-225°C roasting temperature, 140-275 min cooking time and 30-40 min of cooling time. The optimum responses were 45.7% extraction yield, 19.3 acid value, 5.4 peroxide value and 1.6 Lovibond red colour. The statistical relation between the four independent variables and the process responses was well described.

**Keywords:** optimisation, RSM- Doehlert experimental design, shea, kernels, butter, pressing, physicochemical characteristics

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### **1. Introduction**

Shea butter obtained from kernels of shea tree (Vitellaria paradoxa Gaertner F.) fruit, is highly appreciated for its multi uses. It is used as; cooking and frying fat, Cocoa Butter Substitute (CBS) in chocolate and confectionary, useful base for preparing ointments to treat inflammations, rashes on children, ulcers, for wrinkles and dry skin, as cosmetic moisturizing cream, an antiseptic for the treatment of wounds, as hair oil and rub for rheumatic pains [1]. Shea butter deteriorates when extracted inadequately, with the principal decomposition reaction being oxidation [2]. In the traditional aqueous extraction method of shea butter which is very predominant in the Central African region and is the main source of supply of local markets with shea butter, the seed is ground, boiled in water and the oil is scooped off the top of the mixture [3]. This procedure is tedious, labour-intensive, time consuming and gives relatively low extraction yields of about 20% [4]. Moreover, the produced butter is of an inferior quality; yellow, brown and dark coloured, in poor hygienic condition and with high acid and peroxide values [5]. Womeni et al. (2006) studied the effect of cooking

(boiling water) and drying of shea nuts on the quality of solvent extracted shea butter. They reported that temperature and time of thermal treatment has a significant influence on the yield and the quality of shea butter, but miss control of optimal parameters could lead to dangerous chemical modifications (colour, odour, **nutritional values**, etc) of the shea butter [6]. Oil content of shea kernel is about 35 - 60 % [4].

Press extraction is influenced by a number of variables including the type of oil seeds, the roasting and cooking conditions (temperature, time), the cooling time, the pressing force and the volume of oilseed to be pressed. Operating at elevated temperature enhances the oil yield [7]. However, high temperature usually leads to the deterioration of oil, with the principal decomposition reaction being oxidation [8]. The optimum conditions of a manual press for extracting oil from oilseeds have been studied [9,10] as an alternative for the traditional aqueous methods. Such studies could help in introducing many changes in its design and thus could reduce the hard efforts exercised by women during the processing of shea butter.

Optimisation studies using response surface methodology (RSM) were deeply investigated [11,12]. Furthermore, the optimum conditions could assist designers to manufacture simple unit operations that could limit or eliminate the tedious practice of shea butter extraction (roasting, grinding and cooking pre-treatments). Then absolute average deviation (AAD) and coefficient of determination ( $\mathbb{R}^2$ ) could draw to investigate the adequacy of the proposed models.

The present study involves optimisation of some parameters that are likely to affect extraction yield. The general practice of determining these optima is by onevariable-at-time approach. One of the disadvantages of this approach is that it does not include interaction effects among the variables and is unable to determine the true optimum conditions. In order to overcome this problem, optimization studies were done using response surface methodology RSM [13,14]. RMS is a collection of mathematical and statistical technique that is useful for modeling and analyzing situations in which a response of interest is influenced by several variables, especially if there is a need to optimize the responses of a process [15]. Doehlert matrix as an experimental design represents a uniform distribution of experimental points in space of coded variables. It is used particularly when there is a need to cover an experimental domain of any form of uniformly distributed points in order to explore the total domain (margins and interiors). Moreover, it permits to follow in a sequential manner in studying a response surface of second degree [16]. Polynomial equations with and without interaction could be proposed as models for the mentioned processes. A few studies have been reported on the thermal pre-treatment of shea kernels prior press extraction and since, shea butter is a promising multifunctional edible fat. It seemed to be important to study roasting and steam cooking processes of shea kernel. The objective of this study was to evaluate the influence of roasting time, roasting temperature, cooking time and cooling time on the extraction yield of shea butter using manual vertical screw press described by Kapseu *et al.* (2002) [17]. The second preoccupation was to assess the quality of the shea butter in terms of acid value, peroxide value and Lovibond red colour.

### 2. Materials and Methods

### 2.1. Sun Dried Shea Kernels

Sun dried shea kernels were purchased from Penie village in Chad in the second week of September, 2005. The kernels were winnowed manually to remove husk, fine peel, infected kernels and foreign matter and then the sample was pooled and packed in polythene bag and transported to the laboratory of applied chemistry in faculty of pure and applied science, university of N'djamena. After one week the kernels were characterised for moisture and the total lipid content using the methods described by AFNOR (1981) [18] and IUPAC (1979) [19], respectively.

All the chemicals used were of reagent grade.

Table 1. Doehlert experimental design of four independent variables employed for press extraction of shea butter.

Run No	Roasting time	Roasting temperature	Cooking time (min)	Cooling time (min)
	$X_1(X_1)$	$(^{\circ}C) X_{2} (x_{2})$	X <sub>3</sub> (X <sub>3</sub> )	X <sub>4</sub> (X <sub>4</sub> )
1	30.5 (0.000)	175 (0.000)	150 (0.000)	30 (0.000)
2	60 (1.000)	175 (0.000)	150 (0.000)	30 (0.000)
3	0 (-1.000)	175 (0.000)	150 (0.000)	30 (0.000)
4	45.3 (0.500)	300 (0.866)	150 (0.000)	30 (0.000)
5	15.8 (-0.500)	50 (-0.866)	150 (0.000)	30 (0.000)
6	45.3 (0.500)	50 (-0.866)	150 (0.000)	30 (0.000)
7	15.8 (-0.500)	300 (0.866)	150 (0.000)	30 (0.000)
8	45.3 (0.500)	216.7 (0.289)	300 (0.816)	30 (0.000)
9	15.8 (-0.500)	133.3 (-0.289)	0.00 (-0.816)	30 (0.000)
10	45.3 (0.500)	133.3 (-0.289)	0.00 (-0.816)	30 (0.000)
11	30.5 (0.000)	258.3 (0.577)	0.00 (-0.816)	30 (0.000)
12	15.8 (-0.500)	216.7 (0.289)	300 (0.816)	30 (0.000)
13	30.5 (0.000)	91.8 (-0.577)	300 (0.816)	30 (0.000)
14	45.3 (0.500)	216.7 (0.289)	187.5 (0.204)	60 (0.791)
15	15.8 (-0.500)	133.3 (-0.289)	112.5 (-0.204)	0 (-0.791)
16	45.3 (0.500)	133.3 (-0.289)	112.5 (-0.204)	0 (-0.791)
17	30.5 (0.000)	258.3 (0.577)	112.5 (-0.204)	0 (-0.791)
18	30.5 (0.000)	175 (0.000)	262.5 (0.612)	0 (-0.791)
19	15.8 (-0.500)	216.7 (0.289)	187.5 (0.204)	60 (0.791)
20	30.5 (0.000)	91.8 (-0.577)	187.5 (0.204)	60 (0.791)
21	30.5 (0.000)	175 (0.000)	37.5 (-0.612)	60 (0.791)
v th	e coded value of variables			

X the coded value of variables

**X** the real value of variables.

# **2.2. Optimisation of Extraction Conditions and Statistical Analysis**

The response surface methodology using Doehlert experimental matrix was used to optimise the extraction

process. SigmaPlot version 9.01 and Mathcad softwares were used for statistical analysis (ANOVA), regression models and graphical optimisation. Besides, the fit of models was verified by the coefficient of determination ( $R^2$ ) and the absolute average deviation (AAD). Four

independent variables namely roasting time (X<sub>1</sub>: 0 - 60 min), roasting temperature (X<sub>2</sub>: 50 - 300°C), cooking time (X<sub>3</sub>: 0 - 300 min) and cooling time (X<sub>4</sub>: 0 - 60 min) were chosen. The ranges of independent parameters were selected based on literature review and preliminary studies. Twenty-one different experiments were presented in Table 1 according to the experimental design for the four parameters. The experiments were figured in coded (x) and real (X) values. The response functions (Y<sub>i</sub>) measured were extraction yield (E<sub>ield</sub>), acid value (Y<sub>Av</sub>), peroxide value (Y<sub>Pv</sub>) and Lovibond red colour (Y<sub>Colour</sub>) of shea butter. The responses were related to the coded values (x<sub>i</sub>) by the second order polynomial that shown in equation 1.

$$Y_{i} = b_{0} + \sum_{i=1}^{k} b_{i}X_{i} + \sum_{i=1}^{k} b_{ii}X_{i}^{2} + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} b_{ij}X_{i}X_{j}$$
(1)

The coefficients of the polynomial were represented by  $b_0$  (constant term),  $b_i$  (linear effects),  $b_{ii}$  (quadratic effects) and  $b_{ij}$  (interaction effects).  $X_i$  and  $X_j$  are the independent variables.

The analyses of variance (ANOVA) were generated and the effect and regression coefficients of individual, quadratic and interaction terms were determined. The significances of all terms in the polynomial were judged statistically at a probability (P) of 0.001, 0.01 and 0.05. The regression coefficients were then used to make statistical calculation to generate contour map and response surface graphs from the regression models.

### 2.3. Determination of Extraction Yield

The yield of the oil recovered from the ground shea kernels ( $Y_{ield}$ ) was calculated as a ratio of mass of the extracted oil to the mass of dry sample (equation 2), while the extraction yield ( $E_{ield}$ ) was calculated from the amount of extracted oil to the total amount of oil available ( $L_c$ ) in the sample (equation 3).

$$Y_{ield} = \frac{M_0 (g)}{M_d (g)} *100$$
 (2)

$$E_{ield} = \frac{Y_{ield}}{L_c} *100$$
(3)

Where  $M_o$  is the mass of oil recovered in gram,  $M_d$  is the mass of dry sample;  $L_c$  is the total lipid content.

### 2.4. Characterization of the Extracted Butter

The colour of shea butter was measured using Lovibond method described by Sun *et al*, 2001[20] and ISO 15305, 1998 [21], while acid and peroxide values were evaluated using the method described in AFNOR (1981) [18]. Each experiment in the mentioned analysis was replicated twice and the average value was used.

### 2.5. Roasting and Cooking of the Kernels

Figure 1, shows the steps involved in the press extraction process. In roasting process, previously cleaned, washed and dried sand was put in a steel dish, and then the dish put in an electrical oven. The oven was adjusted to the desirable temperature for one hour. In each experiment of the roasting process, the sample of sun dried shea kernel was mixed with the heated sand and stirred every two minutes for a length of time called roasting time. After roasting the sample was pounded in a wooden mortar until it became flourlike, and then sieved to 2 - 3mm squared mesh, using standard sieves [22]. In the cooking process, the sieved sample was put in a Pyrex flask (100 ml capacity), firmly covered with aluminum sheet and then placed in a thermostatic water bath containing boiling water. The beaker and the boiling water were separated by a 4 - 5 cm distance such that only steam from the bath was used in cooking. After cooking, the sample was put in a crucible to cool at ambient condition  $(28 \pm 2^{\circ}C)$  for a length of time called cooling time.



Figure 1. Flow sheet for the press extraction process of shea butter

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### **2.6. Extraction Procedure**

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In each experiment of extraction process, 30 g of the cooked sample was introduced into small cotton bag of a known weight and then pressed. Pressing force was applied slowly up to the maximum degree according to the methods described by Kapseu et al., 2002 [17]. After extraction, the oil was allowed to cool in a crucible for 30 minutes, then weighed and kept for further analysis.

Table 2. In	Table 2. Influences of cooking time, roasting time, roasting temperature and cooling time on the responses of extraction process			
Run No	Extraction yield (%)	Acid value	Peroxide value	Lovibond red colour
1	$43.84\pm0.47$	$19.76\pm0.29$	$6.06\pm0.21$	$2.17 \pm 0.12$
2	$36.54\pm0.29$	$19.66\pm0.33$	$6.01\pm0.37$	$9.87\pm0.21$
3	$41.65\pm0.47$	$19.67\pm0.45$	$6.00\pm0.17$	$0.33 \pm 0.06$
4	$33.22\pm0.37$	$14.32\pm0.62$	$3.04\pm0.56$	$70.00\pm0.05$
5	$42.71\pm0.24$	$19.64\pm0.29$	$6.27\pm0.03$	$0.38\pm0.10$
6	$37.9\pm0.34$	$19.81\pm0.03$	$6.05\pm0.27$	$0.23\pm0.08$
7	$35.94\pm0.15$	$19.82\pm0.06$	$6.05\pm0.07$	$11.45\pm0.13$
8	$38.07\pm0.08$	$19.76\pm0.26$	$6.02\pm0.30$	$4.23\pm0.06$
9	$31.43\pm0.05$	$19.90\pm0.04$	$14.09\pm0.35$	$3.22\pm0.16$
10	$32.86\pm0.53$	$17.12\pm0.32$	$8.65\pm0.78$	$1.38\pm0.03$
11	$22.04\pm0.20$	$14.45\pm0.39$	$7.01\pm0.47$	$70.00\pm0.05$
12	$42.45\pm0.42$	$19.74\pm0.09$	$4.02\pm0.14$	$2.17\pm0.15$
13	$39.23\pm0.07$	$19.73\pm0.13$	$6.09\pm0.52$	$0.22\pm0.07$
14	$37.67\pm0.48$	$19.62\pm0.21$	$4.04\pm0.28$	$4.45\pm0.13$
15	$36.18\pm0.03$	$19.72\pm0.23$	$8.09\pm0.33$	$0.28 \pm 0.08$
16	$38.96 \pm 0.35$	$19.88\pm0.32$	$8.04\pm0.31$	$0.30 \pm 0.10$
17	$29.77\pm0.09$	$14.45\pm0.41$	$6.10\pm0.07$	$70.00\pm0.05$
18	$35.65\pm0.31$	$19.74\pm0.65$	$6.05\pm0.06$	$2.62\pm0.13$
19	$38.53\pm0.28$	$19.83\pm0.06$	$6.03\pm0.20$	$3.25\pm0.09$
20	$40.96\pm0.16$	$19.79\pm0.15$	$9.06\pm0.26$	$0.23\pm0.03$
21	$34.35\pm0.18$	$19.79\pm0.16$	$8.01\pm0.55$	$2.20\pm0.10$

### 3. Results and Discussion

The experimental results for extraction yield, acid value, peroxide value and Lovibond red colour under different treatment conditions are shown in Table 2.

The results of the analysis of variance, goodness of fit and the adequacy of models are summarised in Table 3. The data showed a good fit with the equation 1, which were statistically acceptable at P < 0.05 level. The values of coefficient of determination for the extraction yield, acid value, peroxide value and the colour are 0.94, 0.90, 0.90 and 0.92 respectively. These values of  $R^2$  showed

that the proposed models are adequate. Mendenhall, 1975 [23] reported that the closer the value of  $R^2$  to the unity, the better the empirical models the actual data. In fact, Joglekar and May 1987 [24] suggested that, for a good fit of a model,  $R^2$  should be at least 80.0%. On the other hand, the low absolute average deviation (AAD) values for the extraction yield, acid value, peroxide value and Lovibond red colour (0.06, 0.15, 1.5 and 4.83 respectively) confirmed the adequacy of the models. Therefore, the models could be used to generate surface response curves to explain the influence of the independent factors on the responses studied.

Table 3. Regression coefficients, coefficient of determination ( $R^2$ ) and absolute average deviation (AAD) for the four responses of the press

Coefficient $E_{ied}(\%)$ Acid valuePeroxide valueColour $b_0$ $43.84^{**}$ $19.800^{***}$ $6.00^{***}$ $2.2$ $b_1$ $-1.878$ $-0.800$ $0.875$ $8.01$ $b_2$ $4.262^{**}$ $-1.986^{**}$ $-1.862^{**}$ $30.595^{**}$ $b_3$ $6.122^{***}$ $1.525^{**}$ $-2.573^{**}$ $-10.123$ $b_4$ $1.732$ $0.838$ $-0.157$ $-10.123$ $b_12$ $1.207$ $-3.060$ $-1.588$ $33.776$ $b_13$ $3.987$ $2.738$ $5.158^{**}$ $-9.634$ $b_14$ $-1.713$ $0.412$ $-2.014$ $-8.465$ $b_23$ $7.105^{**}$ $2.675$ $-2.303$ $-31.19$ $b_14$ $0.975$ $2.665$ $-2.303$ $-31.19$ $b_14$ $0.289$ $-1.719$ $0.200$ $2.9$ $b_22$ $4.949$ $-1.767$ $-0.917$ $23.468$ $b_33$ $-11.333^{**}$ $-1.561$ $2.607$ $10.437$ $b_44$ $-7.121^{*}$ $0.395$ $1.062$ $5.609$ $R^2$ $0.94$ $0.90$ $0.90$ $0.92$	CALL ACTION PLOCESS					
b_043.84***19.800***6.000***2.2b_1-1.878-0.800-0.8758.01b_2-4.262**-1.986**-1.862*30.595**b_36.122***1.525*-2.573**-13.673*b_41.7320.838-0.157-10.123b_121.207-3.060-1.58833.776b_133.9872.7385.158*-9.634b_14-1.7130.412-2.014-8.465b_237.105*2.1711.976-38.522b_240.9752.665-2.303-31.119b_340.289-1.7191.32414.911b_11-4.7450.0000.0002.9b_22-4.949-1.767-0.91723.468b_3-11.333**-1.5612.60710.437b_4-7.121*-0.3951.0625.609R20.940.900.900.92AAD0.060.151.504.83	Coefficient	E <sub>ield</sub> (%)	Acid value	Peroxide value	Colour	
b1-1.878-0.800-0.8758.01b2-4.262**-1.986**-1.862*30.595**b36.122***1.525*-2.573**-13.673*b41.7320.838-0.157-10.123b121.207-3.060-1.58833.776b133.9872.7385.158*-9.634b14-1.7130.412-2.014-8.465b237.105*2.1711.976-38.522b240.9752.665-2.303-31.119b340.289-1.7191.32414.911b11-4.7450.0000.0002.9b22-4.949-1.5612.60710.437b33-11.333**-1.5612.60710.437b44-7.121*0.900.900.900.92AAD0.060.151.504.83	b <sub>0</sub>	43.84***	19.800***	6.000***	2.2	
b2-4.262**-1.986**-1.862*30.595**b36.122***1.525*-2.573**-13.673*b41.7320.838-0.157-10.123b121.207-3.060-1.58833.776b133.9872.7385.158*-9.634b14-1.7130.412-2.014-8.465b237.105*2.1711.976-38.522b240.9752.665-2.303-31.119b340.289-1.7191.32414.911b11-4.7450.0000.0002.9b22-4.949-1.7670.91723.468b33-11.33**-1.5612.60710.437b44-7.121*-0.3951.0625.609R20.940.900.900.92AAD0.060.151.504.83	<b>b</b> <sub>1</sub>	-1.878	-0.800	-0.875	8.01	
b36.122***1.525*-2.573**-13.673*b41.7320.838-0.157-10.123b121.207-3.060-1.58833.776b133.9872.7385.158*-9.634b14-1.7130.412-2.014-8.465b237.105*2.1711.976-38.522b240.9752.665-2.303-31.119b340.289-1.7191.32414.911b11-4.7450.0000.0002.9b224.949-1.7670.91723.468b33-11.33**-1.5612.60710.437b44-7.121*0.3951.0625.609R20.940.900.900.92AAD0.660.151.504.83	<b>b</b> <sub>2</sub>	-4.262**	-1.986**	-1.862*	30.595**	
b41.7320.838-0.157-10.123b121.207-3.060-1.58833.776b133.9872.7385.158*-9.634b14-1.7130.412-2.014-8.465b237.105*2.1711.976-38.522b240.9752.665-2.303-31.119b340.289-1.7191.32414.911b11-4.7450.0000.0002.9b22-4.949-1.767-0.91723.468b33-11.333**-1.5612.60710.437b44-7.121*0.3951.0625.609R <sup>2</sup> 0.940.900.900.92AAD0.060.151.504.83	<b>b</b> <sub>3</sub>	6.122***	1.525*	-2.573**	-13.673*	
$b_{12}$ 1.207-3.060-1.58833.776 $b_{13}$ 3.9872.7385.158*-9.634 $b_{14}$ -1.7130.412-2.014-8.465 $b_{23}$ 7.105*2.1711.976-38.522 $b_{24}$ 0.9752.665-2.303-31.119 $b_{34}$ 0.289-1.7191.32414.911 $b_{11}$ -4.7450.0000.0002.9 $b_{22}$ -4.949-1.767-0.91723.468 $b_{33}$ -11.333**-1.5612.60710.437 $b_{44}$ -7.121*-0.3951.0625.609 $R^2$ 0.940.900.900.900.92AAD0.060.151.504.83	$b_4$	1.732	0.838	-0.157	-10.123	
$b_{13}$ $3.987$ $2.738$ $5.158^*$ $-9.634$ $b_{14}$ $-1.713$ $0.412$ $-2.014$ $-8.465$ $b_{23}$ $7.105^*$ $2.171$ $1.976$ $-38.522$ $b_{24}$ $0.975$ $2.665$ $-2.303$ $-31.119$ $b_{34}$ $0.289$ $-1.719$ $1.324$ $14.911$ $b_{11}$ $-4.745$ $0.000$ $0.000$ $2.9$ $b_{22}$ $-4.949$ $-1.767$ $-0.917$ $23.468$ $b_{33}$ $-11.333^{**}$ $-1.561$ $2.607$ $10.437$ $b_{44}$ $-7.121^*$ $-0.395$ $1.062$ $5.609$ $R^2$ $0.94$ $0.90$ $0.90$ $0.92$ AAD $0.06$ $0.15$ $1.50$ $4.83$	b <sub>12</sub>	1.207	-3.060	-1.588	33.776	
$b_{14}$ -1.7130.412-2.014-8.465 $b_{23}$ 7.105*2.1711.976-38.522 $b_{24}$ 0.9752.665-2.303-31.119 $b_{34}$ 0.289-1.7191.32414.911 $b_{11}$ -4.7450.0000.0002.9 $b_{22}$ -4.949-1.767-0.91723.468 $b_{33}$ -11.333**-1.5612.60710.437 $b_{44}$ -7.121*-0.3951.0625.609 $R^2$ 0.940.900.900.92AAD0.060.151.504.83	b <sub>13</sub>	3.987	2.738	5.158*	-9.634	
$b_{23}$ $7.105^*$ $2.171$ $1.976$ $-38.522$ $b_{24}$ $0.975$ $2.665$ $-2.303$ $-31.119$ $b_{34}$ $0.289$ $-1.719$ $1.324$ $14.911$ $b_{11}$ $-4.745$ $0.000$ $0.000$ $2.9$ $b_{22}$ $-4.949$ $-1.767$ $-0.917$ $23.468$ $b_{33}$ $-11.33^{**}$ $-1.561$ $2.607$ $10.437$ $b_{44}$ $-7.121^*$ $-0.395$ $1.062$ $5.609$ $R^2$ $0.94$ $0.90$ $0.90$ $0.92$	b <sub>14</sub>	-1.713	0.412	-2.014	-8.465	
$b_{24}$ 0.9752.665-2.303-31.119 $b_{34}$ 0.289-1.7191.32414.911 $b_{11}$ -4.7450.0000.0002.9 $b_{22}$ -4.949-1.767-0.91723.468 $b_{33}$ -11.333**-1.5612.60710.437 $b_{44}$ -7.121*-0.3951.0625.609 $R^2$ 0.940.900.900.92AAD0.060.151.504.83	b <sub>23</sub>	7.105*	2.171	1.976	-38.522	
b340.289-1.7191.32414.911b11-4.7450.0000.0002.9b22-4.949-1.767-0.91723.468b33-11.333**-1.5612.60710.437b44-7.121*-0.3951.0625.609R <sup>2</sup> 0.940.900.900.92AAD0.060.151.504.83	b <sub>24</sub>	0.975	2.665	-2.303	-31.119	
$b_{11}$ -4.7450.0000.0002.9 $b_{22}$ -4.949-1.767-0.91723.468 $b_{33}$ -11.333**-1.5612.60710.437 $b_{44}$ -7.121*-0.3951.0625.609 $R^2$ 0.940.900.900.92AAD0.060.151.504.83	b <sub>34</sub>	0.289	-1.719	1.324	14.911	
b22-4.949-1.767-0.91723.468b33-11.333**-1.5612.60710.437b44-7.121*-0.3951.0625.609R <sup>2</sup> 0.940.900.900.92AAD0.060.151.504.83	b <sub>11</sub>	-4.745	0.000	0.000	2.9	
b33-11.333**-1.5612.60710.437b44-7.121*-0.3951.0625.609R²0.940.900.900.92AAD0.060.151.504.83	b <sub>22</sub>	-4.949	-1.767	-0.917	23.468	
b44-7.121*-0.3951.0625.609R20.940.900.900.92AAD0.060.151.504.83	b <sub>33</sub>	-11.333**	-1.561	2.607	10.437	
R <sup>2</sup> 0.94     0.90     0.90     0.92       AAD     0.06     0.15     1.50     4.83	b <sub>44</sub>	-7.121*	-0.395	1.062	5.609	
AAD 0.06 0.15 1.50 4.83	$\mathbb{R}^2$	0.94	0.90	0.90	0.92	
	AAD	0.06	0.15	1.50	4.83	

\*\*\* *P* < 0.001, \*\* *P* < 0.01, \* *P* < 0.05.

From Table 3 it is observed that roasting and cooling times had no significant influence (P < 0.05) on the responses. However, longer roasting and cooling times reduce the extraction yield and increase the colour of shea butter. There is neither an interaction nor quadratic significant (P < 0.05) influence of the independent parameters on the acid value and the colour of shea butter, but the interaction of roasting and cooking time increased the peroxide value of the shea butter at P < 0.05 level. The extraction yield was significantly reduced by the quadratic effect of cooking (P < 0.01) and cooling (P < 0.05) times, whereas the interaction of roasting temperature and cooking time significantly (P < 0.05) increased the extraction yield.

To aid visualisation, the effect of roasting time  $(X_1)$ , roasting temperature  $(X_2)$ , cooking time  $(X_3)$  and cooling time  $(X_4)$  on the extraction yield, acid value, peroxide value and the colour of shea butter are presented in Figure 2 - Figure 6 using response surface graphs.

3.1. Extraction Yield

The influence of roasting time, roasting temperature, cooking time and cooling time on the extraction yield of shea butter during the press extraction process is presented in Figure 2 (a, b, c). When cooking and cooling times were kept constant at central values; 150 and 30 min respectively, the response surface (Figure 2a) indicated that the extraction yield increased with an increase in roasting time up to 43% at 20 min and then dramatically decreased to 34.5% at 60 min. In the same vein, the yield was increased slightly from 40.5 to 41.5% at roasting temperature 148°C after which it sharply decreased to 31% at 300°C roasting temperature. The increase of extraction yield with an increase in roasting temperature was probably due to the denature of proteins and similar substances by heating. Heating decreases the affinity of oil for solid surfaces of seeds. Thereby increasing the rate at which oil leaves the surface into the extraction medium. On the other hand, the decrease in extraction yield after 20 min of roasting time could be attributed to thermal polymerisation and decarboxylation of the oil as a result of high temperature.



Figure 2. Response surface graph showing the variation of extraction yield of shea butter from sun dried shea kernels as a function of roasting temperature and roasting time (a), roasting temperature and cooking time (b), roasting temperature and cooking time (c)

### 3.2. Acid Value

Response surface plots and the model constants for acid value of press extracted shea butter are presented in Figure 3

and table III. The acid value was negatively related to the linear effects of roasting temperature and positively to the linear influence of cooking time at P < 0.05 confidence level. The interaction of roasting temperature and cooking

time was positively related to the model constant ( $b_{23} =$ 2.2), but showed a negative effect on the acid value of shea butter. When the roasting and cooling times were maintained constant at the central points (Figure 3), it was observed that the acid value decreased rapidly as roasting temperature increased. The slight increase in acid value with the increase in cooking time could be attributed to chemical and enzymatic hydrolysis of oil in presence of heat and moisture, while the decrease in acid value with the increase of cooking time after 148 min could be related to deactivation of lipase by the vapour. This argument could be supported by the fact that the diffusion of heat from the heating medium (vapour) to the interior of the bulk of oilseed particle increases with time and so does the deactivation of the enzyme. The decrease in acid value with the increase in roasting temperature is probably due to polymerisation and decarboxylation as already mentioned before.



Figure 3. Variation of acid value of press extracted shea butter as a function of roasting temperature and cooking time



Figure 4. Variation of peroxide value of press extracted shea butter from sun dried shea kernels as a function of roasting temperature and cooking time

### 3.3. Peroxide Value

From Table 3, the model constants showed that, peroxide value was negatively related to the linear effects of roasting ( $b_2 = -1.9$ ) and cooking ( $b_3 = -2.6$ ) times at P <

0.05 confidence level, while the interactions of roasting temperature and cooking time ( $b_{23} = 2.0$ ) and roasting time and cooking time ( $b_{13} = 5.2$ ) were positively related to the peroxide value of shea butter. It should be noted that, negative value of model constant signifies a lowering effect of the peroxide value of shea butter. The decrease in peroxide value with an increase in roasting temperature and cooking time (Figure 4) could be due to decomposition of peroxides, which are unstable at high temperature.

### 3.4. Lovibond Red Colour

In table III, it is observed that, the linear terms of: roasting time, roasting temperature, the interactions of roasting time and roasting temperature; cooking time and cooling time and the quadratic terms of the four independent parameters studied had a negative influence on the colour of press extracted shea butter. On the other hand, the linear terms of cooking time, cooling time and the interaction effects of (roasting time and cooking time), (roasting time and cooling time), (roasting temperature and cooking time) and (roasting temperature and cooling time) had a positive effect on the colour of shea butter. It should be noted that the negative value of model constant indicates lowering of colour intensity of shea butter. The increase in colour index with an increase in roasting temperature (Figure 5) could be attributed to colour formation by both non-enzymatic browning reaction and phospholipids degradation during the roasting process or to the fact that colour reversion occurred. Sonntag, 1982 [25] reported that deep oxidation products (chromophore and 6-quinone), known as tocored were isolated from soybean oil. These products were responsible for colour reversion. It was seen with the necked eye that, when roasting temperature increased over 220°C without intervention of cooking process, a dark browning colour was formed.



Figure 5. Variation of Lovibond red colour of press extracted shea butter as a function of roasting temperature and cooking time

# **3.5.** The Optimum Condition of Extraction Yield

With the aim to point out the optimal conditions of press extraction process, a graphical optimisation was conducted using Sigmaplot software (SigmaPlot ®, 2004) [26]. Such a methodology consists of overlaying the

curves of the four contour plots obtained from the Doehlert experimental design according to the specific criteria imposed. The optimum conditions were defined in order to get high extraction yield (> 45%) and minimum acid value, peroxide value (< 7%) and colour (0 - 10 Lovibond red colour) to meet nutritional and industrial economic requirements. Figure 6 shows the overlaying plot in which the purple shaded area represents the optimum domain satisfying the imposed criteria.



**Figure 6.** The optimum region identified by overlaying contour plots of the four responses ( $E_{ield}$ ,  $Y_{Av}$ ,  $Y_{Pv}$  and  $Y_{colour}$ ) of press extraction process as a function of roasting temperature and cooking time

The optimum domain under these conditions were 10 - 15 min roasting time, 160 - 225°C roasting temperature, 140 - 275 min cooking time and 30 - 40 min cooling time. The corresponding responses were 45.7 % extraction yield, 19.3 (mg KOH/g oil) acid value, 5.4 (meq O<sub>2</sub>/kg oil) peroxide value and 1.6 Lovibond red colour. These results confirmed that these optimum conditions had not affected the acid value of shea butter extracted from raw sun dried shea kernels (19.8). To confirm these results, additional laboratory studies permitted us to take the following as optimum points: roasting time (10 ± 3 min), cooling time (30 ± 5 min), roasting temperature (190 ± 2°C) and the steam-cooking time (200 ± 5 min).

The shea butter obtained in these conditions, could belong to category 1 reported by Mégnanou1 *et al*, (2013) [27] which used in cosmetics and pharmaceutical industries, if the optimum cooking conditions stated by Bup *et al.*, (2009) [28] are used to cook the shea nut before processing, in order to improve the acid value.

### 4. Conclusion

Response surface methodology using Doehlert experimental design was successfully employed in the optimisation of press extraction process. Four second order polynomial models with satisfactory validation in terms of coefficient of determination ( $R^2$ ) and absolute average deviation (AAD), were generated to describe the press extraction process. The optimum press extraction conditions were found to be 10 - 15 min roasting time, 160 - 225°C roasting temperature, 140 - 275 min cooking time and 30 - 40 minute cooling time. The corresponding responses of these optimum conditions were: 45.7% extraction yield, 19.3 (mg KOH/g oil) acid value, 5.4 (meq  $O_2$ /kg oil) peroxide value and 1.6 Lovibond red colour.

### Nomenclature

AAD	Absolute average deviation
E <sub>ield</sub>	Extraction yield (%)
Av	Acid value (mg KOH/g)
L <sub>c</sub>	Total lipid content (%)
m	Mass (g)
Mc	Moisture content (%)
M	Mass of oil recovered (g)
$\mathbf{M}_{\mathbf{d}}$	Mass of dry sample (g)
<b>b</b> <sub>0</sub>	Constant term
bi	Linear constant effect
bii	Quadratic constant effect
bij	Interaction constant effect
X <sub>i</sub> , Xj	Constant independent parameters
Xi	Coded value of the independent
	parameter
Pv	Peroxide value (meq O <sub>2</sub> /kg)
meq	milliequivalent
Y <sub>Av</sub>	Measured acid value
Yi	Response function
Y <sub>ield</sub>	Yield of the oil recovered $(g/g)$
Y <sub>Colour</sub>	Measured Lovibond colour scale
Yloss	Measured oil loss (%)
Y <sub>Pv</sub>	Measured peroxide value
$\mathbf{R}^2$	Coefficient of determination
Р	Probability

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