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Full Length Research Paper

Development of model equations for selecting optimum parameters for dry process of shea butter extraction

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Shea butter is the fat content of the kernel of shea nut *(Vitellaria paradoxa)* which grows naturally from the wild and uncultivated state in most parts of Africa. The fat is used as edible oil and for raw material in the production of soaps, pomade, drugs and medicinal ointments. Wet extraction process is the method used in shea butter processing industry among women in African rural and urban communities. Apart from the low yield (below 20%), this wet extraction process is associated with environmental polluting effluents as by products. Experiments on dry extraction of shea butter from shea kernel were carried out using an instrumented piston-cylinder rig in conjunction with the TESTOMETRIC Universal Testing Machine (Model M500–50 KN). Shea butter was mechanically expressed when pressures of 1.5, 2.9, 5.8 and 8.8 MPa were applied at the rate of 2.50, 5.00, 7.50 and 10.00 mm/min on crushed shea kernel heated at 50, 70, 90 and 110°C. Measurements were made of oil yield, oil recovery efficiency and process loss during the mechanical expression process. The measured effects of heating temperature, applied pressure and loading speed on oil yield, oil recovery efficiency and process loss were examined using a 4^3 factorial experiment in randomized complete block design. Model equations were developed by employing multiple regression analysis using SPSS 11.0 package. Further analysis by optimization process revealed optimum heating temperature, applied pressure and loading rate of 82.24°C, 9.69 MPa and 2.50 mm min^{-1} respectively. These combinations gave 35.39% oil yield, 58.62% oil recovery efficiency and 2.83% process loss. This information provides useful data for developing a process line for dry extraction of shea butter from shea kernel.

Key words: Shea butter, dry extraction, model equations, optimization, oil yield.

INTRODUCTION

Shea nut (SN) is contained in the fruit of shea tree (ST) plant (*Vitellaria paradoxa)* which grows in the wild and uncultivated state in most parts of African savannah zones. Shea fruit (SF) contains a green epicarp, a fleshy mesocarp and a relatively hard shell (endocarp) which encloses the shea kernel (embryo). Shea kernel (SK) contains about 60% edible fat (shea butter) and the residual product, from which the butter is extracted (shea cake, SC), is an excellent ingredient for livestock feed production. Apart from this, shea butter (SB) is gaining popularity in food, soap, cosmetic, pharmaceutical, medical and engineering industries for the production of cooking oil, toilet soaps, pomade, drugs, ointments and metal cutting fluids respectively. These products, in Africa, have a lot of potential for export and as a foreign exchange earner.

According to Addaquay (2004), in the traditional wet extraction method of SB processing, SK is pounded with pestle and mortar to break it into grits. The grits are then roasted and grounded into a paste to facilitate easy extraction of the fat. The process is continued by kneading the paste in or with, water to capture the fat into an emulsion, boiling the mixture to separate the fat and skimming off the fat. The final cooling process leads to SB. This process is practiced by rural women in Mali and Burkina Faso where, according to Addaquay (2004), 80% of SB is produced traditionally. Wet extraction process is

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tedious, laborious and time consuming which is associated with low yield and environmental polluting effluents (such as waste water, slurry and shea paste) as by products. Some modified and improved methods invented in some parts of West Africa, though slightly improved the yield, are also based on wet extraction process with its associated problems.

Dry extraction process makes use of oil extraction equipment like hydraulic presses or screw expellers. This process has gained a worldwide popularity and acceptance in modern vegetable oil industry but, unfortunately, has not been widely extended to SB extraction. Relevant data like extraction temperature and pressure are needed for the operation of a dry extraction process and these are not readily available. Therefore, the objective of the work reported in this paper was to determine the optimum extraction parameters for dry process of shea butter extraction from shea kernel.

MODEL DEVELOPMENT

Ott (1977) and Babatunde (1997) described methods of multiple regression of a dependent variable Y (X_1, X_2) where X_1 and X_2 are independent variables in a 2-factor factorial design as having any of the following response equations for use in optimizing Y (X_1, X_2). The possible response equations have the following combination of X_1 and X_2:

$$Y_1 = b_o + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_2^2 + b_5X_1X_2 \qquad \ldots 1$$

$$Y_2 = b_o + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_1^2X_2 + b_5X_1X_2 \qquad \ldots 2$$

$$Y_3 = b_o + b_1X_1 + b_2X_2 + b_3X_2^2 + b_4X_2^2X_1 + b_5X_1X_2 \qquad \ldots\ldots 3$$

and

$$Y_4 = b_o + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_2^2 + b_5X_1X_2 + b_6X_2^2X_1 + b_7X_1^2X_2 + b_8X_1^2X_2^2 \qquad \ldots\ldots 4$$

where, b_o, b_1, …….., b_8 are constants; Y_1, …., Y_4 are dependent variables; and Equation 4 is a combination of Equations 1, 2 and 3.

Model equations were determined from the above response equations based on regression coefficient, coefficient of variation, standard error of estimate and F test. These parameters were determined by the statistical analysis of variance (ANOVA) and multiple regression analysis using the Statistical Package for Science and Social Sciences (SPSS 11.0 computer software package).

Extreme values of Y (X_1, X_2) were obtained by methods described by Stephenson (1975) as follows. The coordinates of the extreme values of Y (X_1, X_2), the maximum and minimum values were obtained by solving the partial differentials of Y (X_1, X_2) simultaneously as follows:

$$\frac{\partial Y(X_1, X_2)}{\partial X_1} = 0 \qquad \ldots\ldots 5$$

$$\frac{\partial Y(X_1, X_2)}{\partial X_2} = 0 \qquad \ldots\ldots 6$$

The nature of the function Y (X_1, X_2) is determined by the criteria given as:

$$\frac{\partial^2 Y(X_1, X_2)}{\partial X_1 \partial X_2} - \frac{\partial^2 Y(X_1, X_2)}{\partial X_1^{\,2}} \frac{\partial^2 Y(X_1, X_2)}{\partial X_2^{\,2}} \angle 0 \quad \ldots 7$$

$$\frac{\partial^2 Y(X_1, X^2)}{\partial X_1^{\,2}} \angle 0 \qquad \ldots\ldots 8$$

Y (X_1, X_2) is a maximum when Equation 7 is true and the right hand side of Equation 8 is less than zero; Y (X_1, X_2) is a minimum when Equation 7 is true and the right hand side of Equation 8 is greater than zero; Y (X_1, X_2) is a saddle point or give no information when the right hand side of Equation 8 is equal to, or greater than, zero at the coordinates defined by the solution to the simultaneous Equations 5 and 6. For Y (X_1) or Y (X_2), the coordinates of the extreme values of Y (X_1) or Y (X_2) are defined by the solution of the Equations 9 and 10:

$$\frac{dY(X_1)}{dX_1} = 0, for\,Y(X_1) \qquad \ldots\ldots 9$$

$$\frac{dY(X_2)}{dX_2} = 0\; for\,Y(X_2) \qquad \ldots.. 10$$

where the nature of the extreme values are given by the positive or negative signs of the second differential of Y (X_1) or Y (X_2) given by $\frac{\partial^2 Y}{\partial X_1^{\,2}}$ and $\frac{\partial^2 Y}{\partial X_2^{\,2}}$ respectively.

The function Y (X_1, X_2) is a maximum when the second differential is negative and minimum when it is positive at the values of X_1 or X_2 given by the solutions of Equations 9 or 10 for Y (X_1) or Y (X_2) respectively. Babatunde (1997) has used this same method for the development of model equation for selecting disc and tilt angles of disc plough for optimal operation.

LABORATORY EXPERIMENTS

Laboratory experiments on dry extraction of SB from crushed SK were carried out in the Engineering Materials Testing Laboratory, Technical and Scientific Services Complex, National Centre for Agricultural Mechanization (NCAM), Idofian, Ilorin, Nigeria. The average room temperature of the laboratory was about 30°C throughout the period of experimentation.

Experimental equipment

The experimental equipment consists of a piston-cylinder rig in conjunction with a Universal Testing Machine (Model M500-50 KN, TESTOMETRIC Company Ltd., England, United Kingdom). The Universal Testing Machine (UTM) is of 50 KN capacity and its vital parts includes the control console, load frame, crosshead, load cell, personal computer and the printer. The piston-cylinder rig, which was developed by Olaniyan and Oje (2007), is made up of a compression piston, a press cage cylinder, a supporting platform and an oil collecting pan. The piston serves as the pressing ram and it distributes pressure from the UTM evenly on the oilseed sample in the press cage cylinder.

A 605 W electric band heater was installed to enfold the press cage cylinder and hence serve as a heating device for extraction process. The rig was adequately instrumented with a temperature transducer to control the extraction temperature while the pressure for extraction was obtained from the UTM. The temperature transducer is a system of thermocouple connected to an Electronic Temperature Controller (Model JTC–902, Japan). The temperature range of the Electronic Temperature Controller is 0 to 400°C with the voltage of 110/220 V, frequency of 50/60 Hz and output of 840 W. In operation, the heat sensor (thermocouple probe) is inserted into the crushed SK sample through a hole drilled on the side of the press cage cylinder 70 mm height from the base. The arrangement of the equipment during extraction is as shown in Figure 1.

Experimental procedure

A sample of 200 g of crushed SK sample was weighed and transferred into the press cage cylinder. The sample was heated inside the press cage cylinder with the aid of the temperature-controlled band heater at 50°C for 30 min. Using the jug mode of the UTM, the compression piston was moved down to touch the sample in the press cage cylinder. The sample was then pre-compacted to a height of 70 mm inside the press cage cylinder by UTM loading at a constant speed of 10 mm min^{-1}. After pre-compaction, the crushed SK sample was compressed by the UTM through the compression piston at a constant loading speed of 2.50 mm min^{-1} to a pressure 1.5 MPa for 10 min. The oil expressed was collected in the oil collecting pan placed below the drainage area and weighed. All weight measurements were carried out using a Triple Beam Balance (Model M2610 g, OHAUS, New Jersey, USA). After expression, the compression piston was lifted well above the press cage cylinder by the jug mode of the UTM. The press cage cylinder (with the residual cake inside) was unscrewed and the residual cake was extruded into the cake extruding die. The experiment was repeated for the three other heating temperature levels of 70, 90 and 110°C; three other applied pressure levels of 2.9, 5.8 and 8.8 MPa; and three other loading speed levels of 5.00, 7.50 and 10.00 mm min^{-1}. Each experimental trial was replicated three times making a total of 192 treatment combinations that were carried out.

Measured parameters

Oil yield was calculated as the ratio of the weight of oil expressed to the weight of the sample before expression. It was mathematically expressed by Adeeko and Ajibola (1989) as stated in Equation 11:

$$O_Y = \frac{W_{OE}}{W_{US}} \ X \ 100\% \qquad \ldots\ldots.. \quad 11$$

Oil recovery efficiency was calculated as the ratio of the weight of oil expressed to the total weight of oil in the crushed SK sample before expression. It was mathematically expressed by Adeeko and Ajibola (1989) as shown in Equation 12:

$$O_E = \frac{W_{OE}}{xW_{US}} \ X \ 100\% \qquad \ldots\ldots..12$$

Process loss was calculated as the difference between the weight of the sample before expression and the sum total of the weights of oil expressed and residual cake after expression divided by the weight of the sample before expression. It was mathematically denoted by Olaniyan and Oje (2007) as stated in Equation 13:

$$O_L = \frac{W_{US} - (W_{RC} + W_{OE})}{W_{US}} \ X \ 100\% \qquad \ldots..13$$

where, O_Y = oil yield (%); O_E = oil recovery efficiency (%); O_L = process loss (%); W_{US} = weight of crushed SK sample before expression (g); W_{RC} = weight of residual cake after expression (g); W_{OE} = weight of oil expressed (g); and x = oil content of SK = 0.60 or 60%.

Data analysis

Based on the four response equations for an experiment with two independent variables given by Ott (1977) and shown in Equations 1 to 4, the data obtained for oil yield, oil recovery efficiency and process loss from the laboratory experiments were subjected to multiple regression analysis using SPSS 11.0 computer software package.

RESULTS AND DISCUSSION

Model equations

The data analysis was based on a two-factor (heating temperature and applied pressure) multiple regression technique. The third experimental factor (loading rate) was not included in the analysis since its best value (2.50 mm min^{-1}) has been established from the experimental results (Olaniyan and Oje, 2007). From the regression analysis, the following regression models were obtained for oil yield, oil recovery efficiency and process loss as shown in Equations 14 to 25:

$$Y_1 = -11.782 + 0.7225X_1 + 3.175X_2 - 4.319E\text{-}03X_1^2 - 0.163X_2^2 + 2.563X_1X_2 \qquad \ldots\ldots \ 14$$

$$Y_2 = -15.077 + 0.894X_1 + 2.822X_2 - 5.397E\text{-}03X_1^2 + 2.269E\text{-}04X_1^2X_2 - 3.374E\text{-}02X_1X_2 \qquad \ldots\ldots15$$

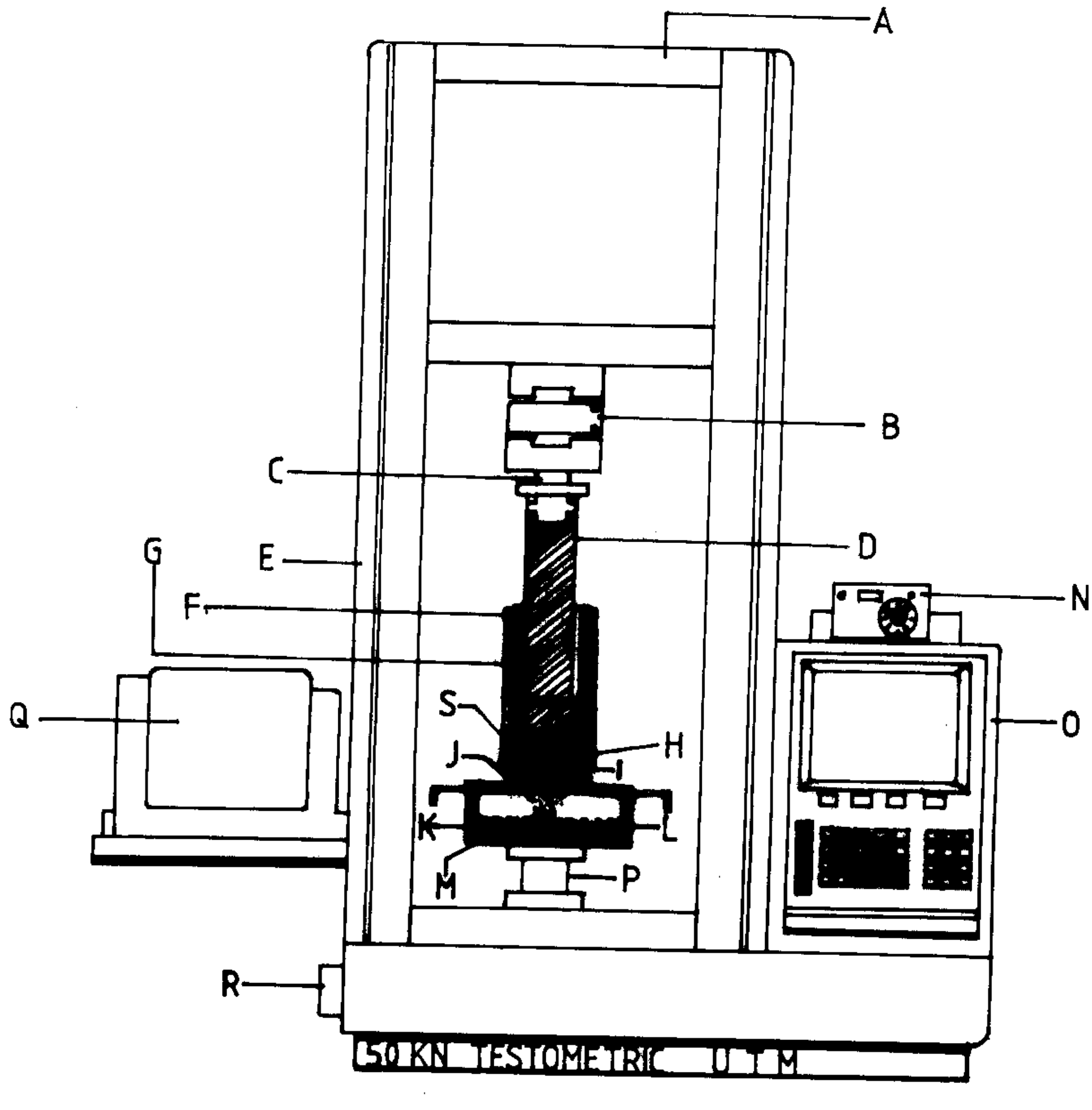

Figure 1. The mechanical expression rig: A - crosshead; B - load cell of the UTM; C - upper attachment of the UTM; D - compression piston; E - frame of the UTM; F - press cage cylinder; G - heating device; H - thermocouple probe from the electronic temperature controller; I - oilseed cake; J – drainage channel; K - supporting platform; L - oil collecting pan; M - oil expressed; N - electronic temperature controller; O - control console; P - lower attachment of the UTM; Q - computer system with printer; R - switch of the UTM; S - wire mesh.

$$Y_3 = 7.834 + 0.104X_1 + 6.415X_2 - 0.476X_2^2 + 3.911\mathrm{E}\text{-}03X_2^2X_1 - 3.794\mathrm{E}\text{-}02X_1X_2 \quad ......16$$

$$Y_4 = -19.114 + 0.834X_1 + 5.023X_2 - 4.563\mathrm{E}\text{-}03X_1^2 - 0.211X_2^2 - 3.303\mathrm{E}\text{-}03X_2^2X_1 - 2.381\mathrm{E}\text{-}04X_1^2X_2 + 4.518\mathrm{E}\text{-}05X_1^2X_2^2 \quad ....17$$

$$Y_5 = -25.999 + 1.351X_1 + 6.011X_2 - 8.012\mathrm{E}\text{-}\ 03X_1^2 - 0.296X_2^2 - 3.344\mathrm{E}\text{-}03X_1X_2 \quad ......18$$

$$Y_6 = -21.188 + 1.371X_1 + 3.101X_2 - 8.138\mathrm{E}\text{-}03X_1^2 + 2.650\mathrm{E}\text{-}05X_1^2X_2 - 7.584\mathrm{E}02X_1X_2 \quad ....19$$

$$Y_7 = 13.299 + 0169X_1 + 10.414X_2 - 0.721X_2^2 + 5.315\mathrm{E}\text{-}03X_2^2X_1 - 5.838X_1X_2 \quad ......20$$

$$Y_8 = -30.462 + 1.356X_1 + 8.253X_2 - 7.422\mathrm{E}\text{-}03X_1^2 - 0.499X_2^2\ X_2^2X_1 - 6.824\mathrm{E}\text{-}04 - 3.637\mathrm{E}\text{-}04X_1^2X_2 + 3.783\mathrm{E}\text{-}05X_1^2X_2^2 \quad ......21$$

Table 1. Analysis of variance of multiple regression of oil yield as a function of heating temperature and applied pressure.

Model	Source	Sum of squares	DF	Mean square	F	Sig.	R^2
1	Regression	1304.447	5	260.889	88.972	0.000	0.914*
	Residual	123.155	42	2.932			
	Total	1427.602	47				
2	Regression	1268.266	5	253.653	66.861	0.000	0.888*
	Residual	159.366	42	3.794			
	Total	1427.602	47				
3	Regression	1172.422	5	234.484	38.584	0.000	0.821*
	Residual	255.181	42	6.076			
	Total	1427.602	47				
4	Regression	1319.454	7	188.493	69.716	0.000	0.924*
	Residual	108.149	40	2.704			
	Total	1427.602	47				

*Significant at P ≤ 0.05.

$$Y_9 = -3.763 + 6.873E\text{-}02X_1 + 1.807X_2 + 4.219E\text{-}04 - 6.365E\text{-}02X_2^2 - 1.616E\text{-}02X_1X_2 \quad \ldots\ldots 22$$

$$Y_{10} = -8.010 + 0.216X_1 + 2.294X_2 - 5.003E\text{-}04X_1^2 + 1.941E\text{-}04X_1^2X_2 - 4.723E\text{-}02X_1X_2 \quad \ldots\ldots 23$$

$$Y_{11} = -4.431 + 0.133X_1 + 0.802X_2 + 3.345E\text{-}02X_2^2 - 1.214E\text{-}03X_2^2X_1 - 3.395E\text{-}03X_1X_2 \quad \ldots\ldots 24$$

$$Y_{12} = -3.244 + 9.176E\text{-}02X_1 + 0.468X_2 + 7.980E\text{-}05X_1^2 + 0.128X_2^2 - 3.279E\text{-}03X_2^2X + 6.760E\text{-}06X_1^2X_2 + 1.018E\text{-}05X_1^2X_2^2 \quad \ldots\ldots\ldots\ldots 25$$

where, Y_1 - Y_4 = oil yield (%); Y_5 - Y_8 = oil recovery efficiency (%); $Y_9 - Y_{12}$ = process loss (%); X_1 = heating temperature (°C); X_2 = applied pressure (MPa).

The existence and sufficiency of the regression models given in Equations 14 to 25 were examined and shown in the analysis of variance (ANOVA) of the multiple regression models shown in Tables 1 to 3. The analysis was carried out using SPSS 11.0 computer software package. The ANOVA tables indicate that the highest F-values, the highest correlation coefficient (R^2) and the lowest standard error of estimate were consistently obtained from model 4, which formed the basis of Equations 17, 21 and 25 respectively for oil yield, oil recovery efficiency and process loss. This implies that the model variables fit the data well. Also the high values of the regression sum of squares (RSS) as against the low values of error (residual) sum of squares (ESS) infer that model 4 accounted for most of the variation in the dependent variable, Y (X_1, X_2). The model is significant at 95% confidence level in each case and this is also evidence that it fits the data well and, therefore readily applicable.

Optimization process

Based on the selected model equations for oil yield Equation 17, oil recovery efficiency Equation 21 and process loss Equation 25, critical values of the process parameters were determined through partial differential equations and the results are as presented in Table 4. The table shows that, in order to maximize oil yield, a temperature of 86.62°C and a pressure of 10.42 MPa should be selected for extraction process. To maximize oil recovery efficiency, a temperature of 82.24°C and a pressure of 9.69 MPa should be used. However, in order to minimize process loss, a temperature of 55.51°C and a pressure of 7.15 MPa should be selected. Therefore, for optimal operation of a dry process of SB extraction, a temperature of 82.24°C and a pressure of 9.69 MPa should be selected for extraction.

Model validation

By substituting different values of heating temperature and applied pressure into the model equations, the expected values of oil yield, oil recovery efficiency and process loss were predicted. The predicted and measured values were subjected to a paired sample t-test using SPSS 11.0 computer software package and

Table 2. Analysis of variance of multiple regression of oil recovery efficiency as a function of heating temperature and applied pressure.

Model	Source	Sum of squares	DF	Mean square	F	Sig.	R^2
1	Regression	3399.634	5	679.927	145.794	0.000	0.946*
	Residual	195.872	42	4.664			
	Total	3595.506	47				
2	Regression	3270.643	5	654.129	84.569	0.000	0.910*
	Residual	324.863	42	7.735			
	Total	3595.506	47				
3	Regression	2927.421	5	585.484	36.807	0.000	0.814*
	Residual	668.085	42	15.907			
	Total	3595.506	47				
4	Regression	3420.635	7	488.662	111.776	0.000	0.951*
	Residual	174.871	40	4.372			
	Total	3595.506	47				

*Significant at $P \leq 0.05$.

Table 3. Analysis of variance of multiple regression of process loss as a function of heating temperature and applied pressure.

Model	Source	Sum of squares	DF	Mean square	F	Sig.	R^2
1	Regression	149.466	5	29.893	12.479	0.000	0.598*
	Residual	100.609	42	2.395			
	Total	250.075	47				
2	Regression	145.783	5	29.157	11.742	0.000	0.533*
	Residual	104.291	45	2.483			
	Total	250.075	47				
3	Regression	149.182	5	29.836	12.420	0.000	0.597*
	Residual	100.892	42	2.402			
	Total	250.05	47				
4	Regression	151.257	7	21.608	8.747	0.000	0.605*
	Residual	98.817	40	2.470			
	Total	250.075	47				

* Significant at $P \leq 0.05$.

Table 4. Critical points of heating temperature and applied pressure for optimal measured parameters.

Measured parameter (%)	Heating temperature (°C)	Applied pressure (MPa)	Nature of critical points
Oil yield	86.62	10.42	Maximum
Oil recovery efficiency	82.24	9.69	Maximum
Process loss	55.51	7.15	Minimum

the result is as shown in Table 5. From the table, the correlation coefficients were 0.956, 0.972 and 0.773 for oil yield, oil recovery efficiency and process loss respectively. These high levels of correlation show that

Table 5. Paired sample t-test of the predicted and measured values of oil yield, oil recovery efficiency and process loss.

Parameter	Paired sample	Standard deviation	Correlation coefficient	t - value
Oil yield	Predicted – measured	1.62	0.956	0.186
Oil recovery efficiency	Predicted – measured	2.04	0.972	0.039
Process loss	Predicted – measured	1.46	0.773	-0.001

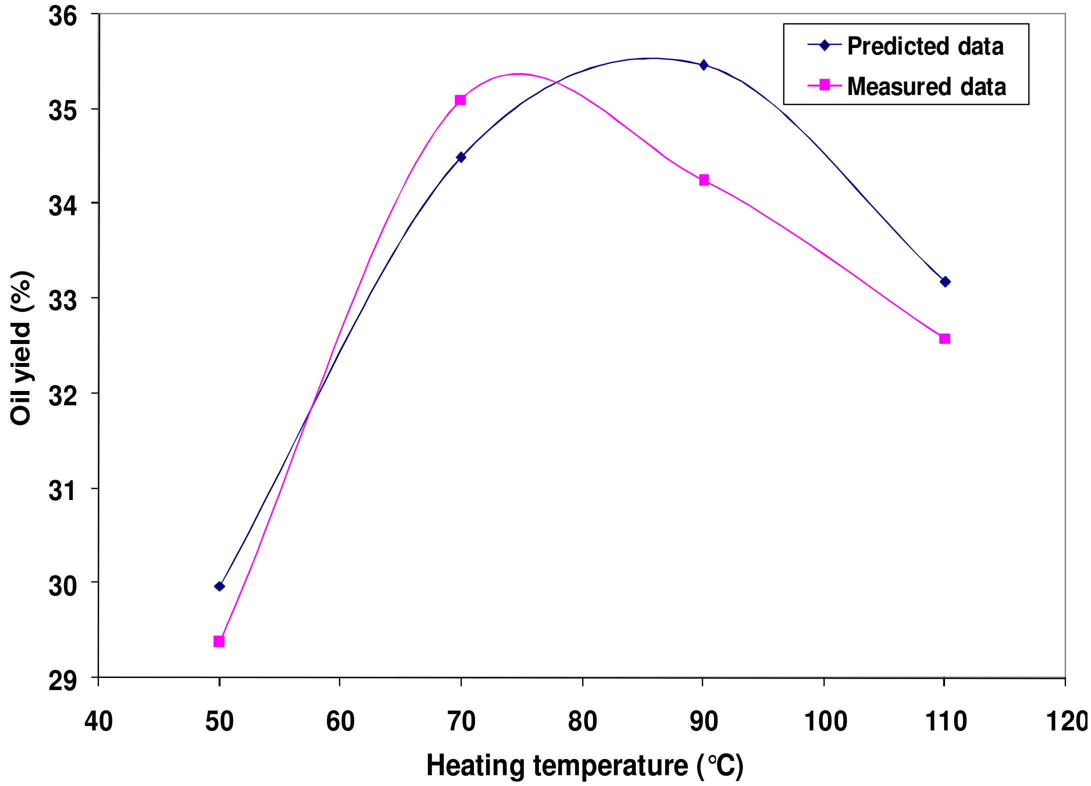

Figure 2. Effect of heating temperature on oil yield.

the predicted values compared favourably with the measured values. Therefore, the model can be used to select the best process parameters for optimal operation of a dry process of SB extraction. A careful observation of predicted and measured responses of oil yield, oil recovery efficiency and process loss to extraction temperature and pressure (Figures 2 to 7) also show high level of correlation which confirms that the model is applicable.

Practical applications

This study establishes process parameters for industrial extraction of shea butter from shea kernel. The dry process of shea butter extraction can be used for the production of good quality shea butter products for the purpose of domestic and industrial use. The quality of preosmosed carrots is much superior to the product dehydrated with the convectional method of convective dehydration. The osmotically dehydrated carrots can be used for cooking as vegetables after rehydration or can be added directly into soups, stews or casseroles before cooking. If the product is blanched before osmotic dehydration, the process can be used successfully for the preparation of carrot candy.

Conclusion

The result of the experiment and analysis showed the effect of heating temperature and applied pressure on oil yield, oil recovery efficiency and process loss. It was indicated by the result that an optimum heating

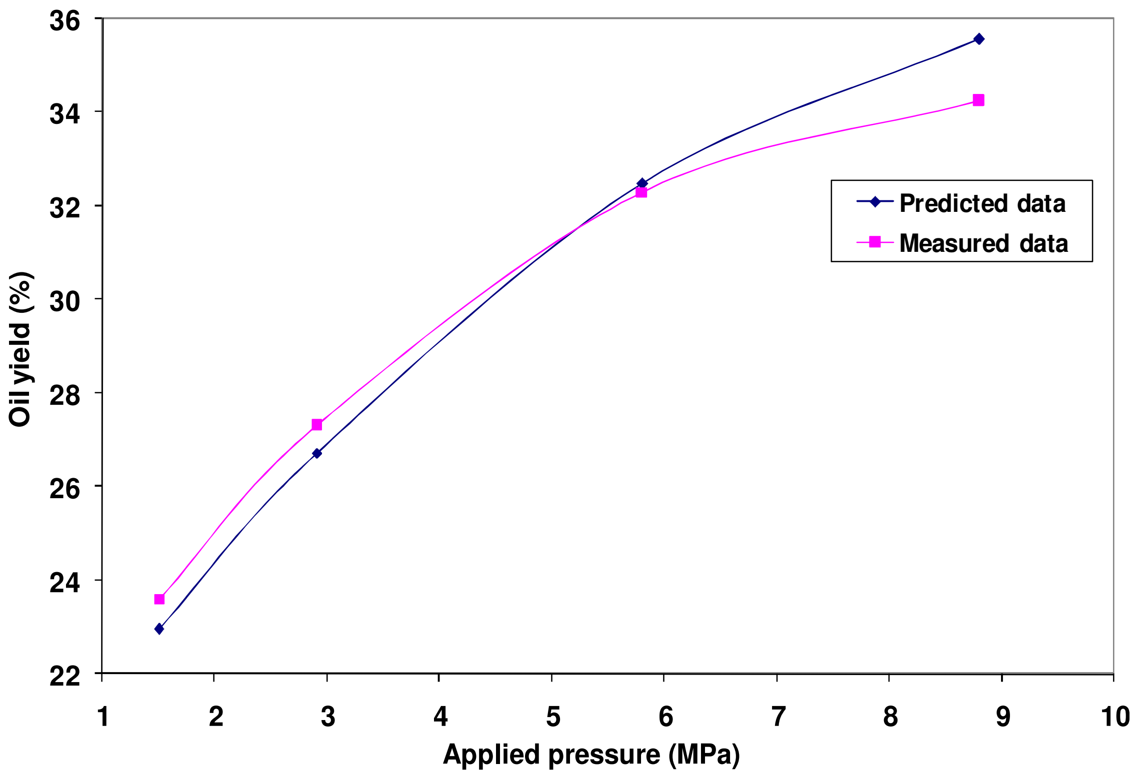

Figure 3. Effect of applied pressure on oil yield.

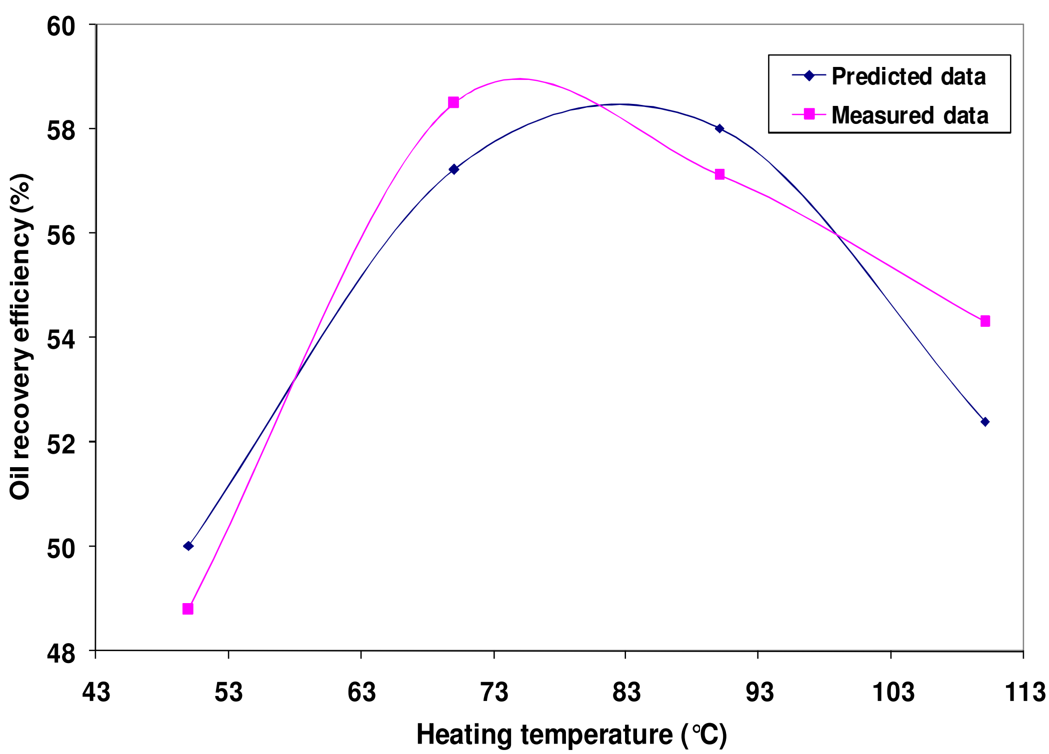

Figure 4. Effect of heating temperature on oil recovery efficiency.

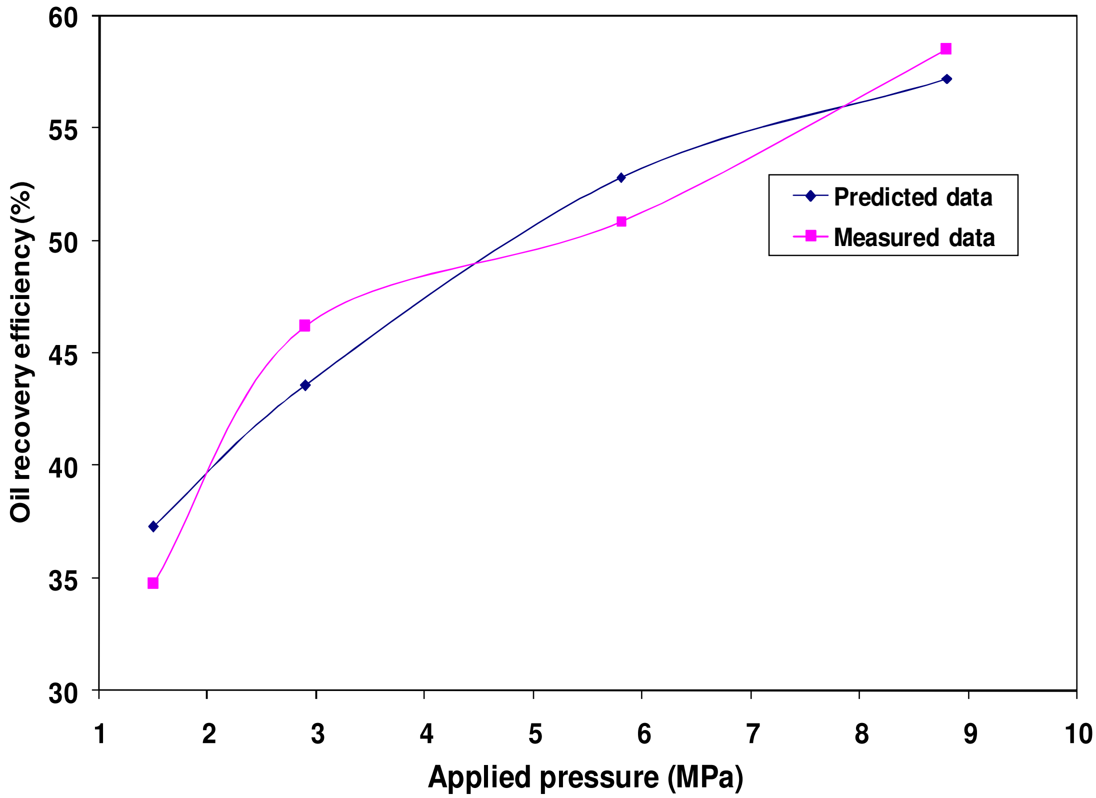

Figure 5. Effect of applied pressure on oil recovery efficiency.

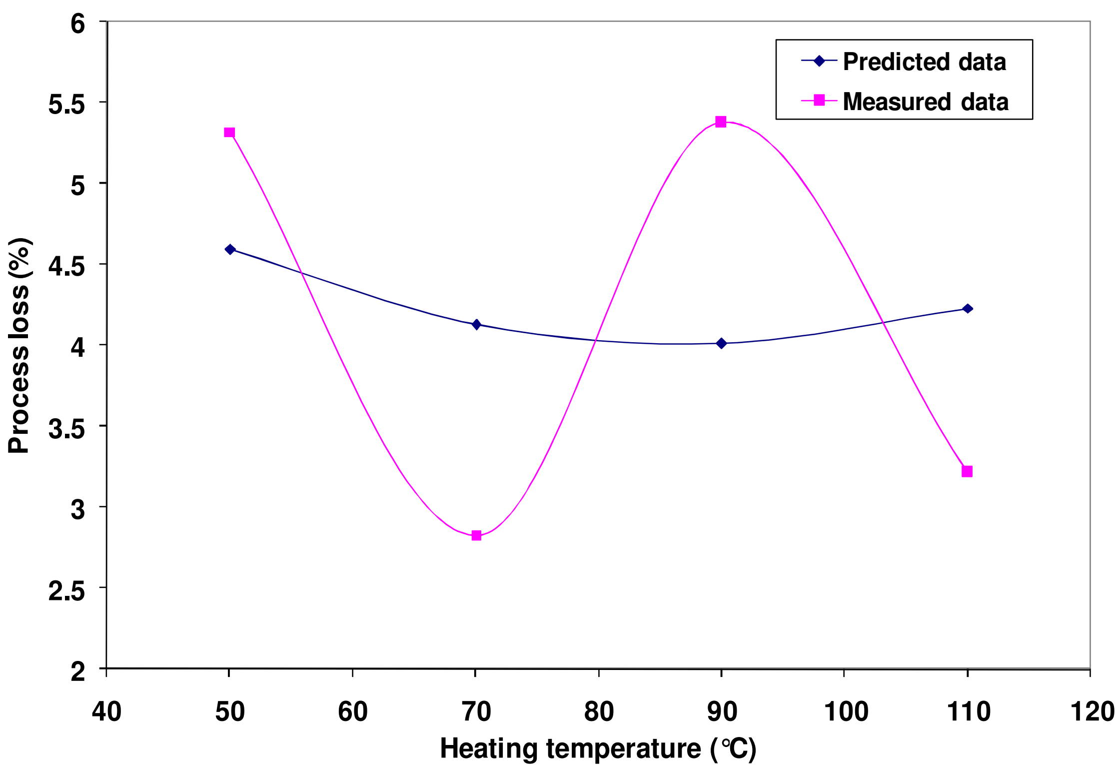

Figure 6. Effect of heating temperature on process loss.

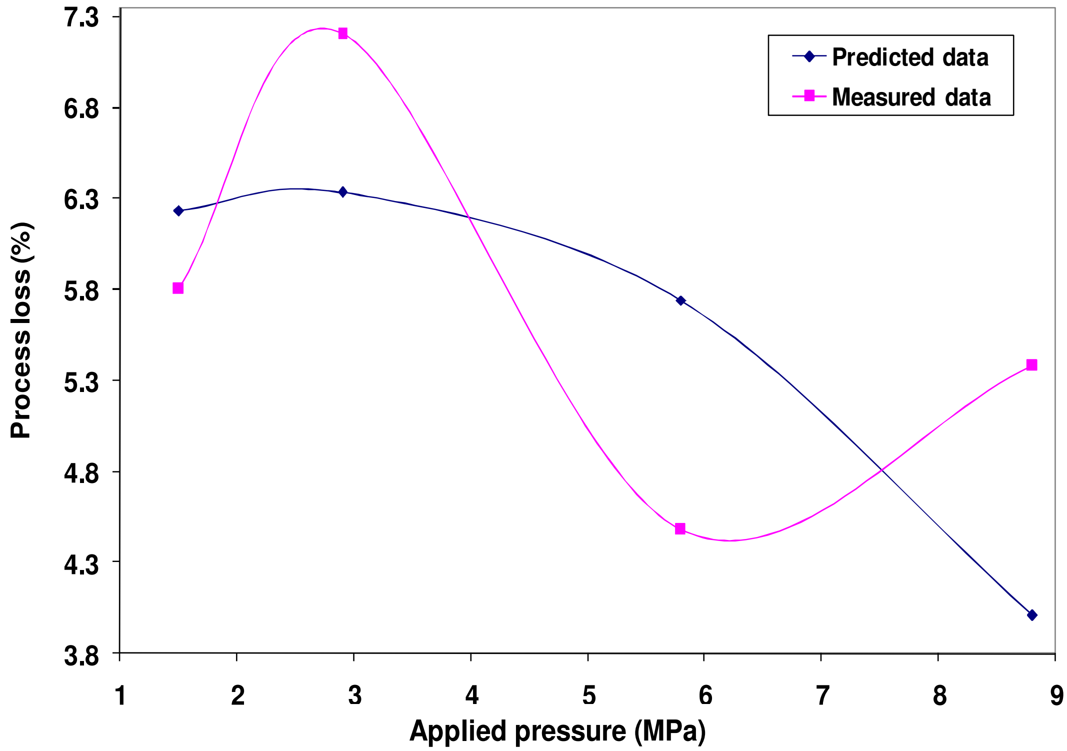

Figure 7. Effect of applied pressure on process loss.

temperature, applied pressure and loading rate of 82.24°C, 9.69 MPa and 2.50 mm min^{-1} respectively should be used for dry process of SB extraction. These combinations gave 35.39% oil yield, 58.62% oil recovery efficiency and 2.83% process loss.

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