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### DEVELOPMENT OF MODEL EQUATIONS FOR SELECTING OPTIMUM PARAMETERS FOR DRY PROCESS OF SHEA BUTTER EXTRACTION

A. M. OLANIYAN<sup>1</sup>, K. OJE<sup>1</sup>

<sup>1</sup>A. M. OLANIYAN, Department of Agricultural and Biosystems Engineering, Faculty of Engineering and Technology, University of Ilorin, P. M. B. 1515, Ilorin 240003, Kwara State, Nigeria, amolan397@hotmail.com.

<sup>1</sup>K. OJE, [kayodeoje2@yahoo.com](mailto:kayodeoje2@yahoo.com).

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**ABSTRACT** Shea butter is the fat content of the kernel of shea nut (*Vitellaria paradoxa*) which grows naturally in the wild and uncultivated 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. Traditional 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 %) and poor quality, this traditional wet extraction process has no place in the modern vegetable oil industry. 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 of oil yield, oil recovery efficiency and process loss during the mechanical expression process were taken. The measured effects of heating temperature, applied pressure and loading speed on oil yield, oil recovery efficiency and process loss were examined using a 43 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<sup>-1</sup> 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.

**Keywords:** Shea butter, Dry extraction, Model equations, Optimization, Oil yield.

**INTRODUCTION** Shea nut (SN) is contained in the fruit of shea tree (*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 roasted and grounded into a paste to facilitate easy extraction of the fat. The process is continued by kneading the paste in 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. Traditional wet extraction process is tedious, laborious and time consuming which is associated with low yield and environmental polluting effluents 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.

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 SB extraction from SK.

**MODEL DEVELOPMENT** Ott (1977) 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  $X_2$ :

$$Y_1 = b_0 + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_2^2 + b_5X_1X_2 \quad \mathbf{1}$$

$$Y_2 = b_0 + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_1^2X_2 + b_5X_1X_2 \quad \mathbf{2}$$

$$Y_3 = b_0 + b_1X_1 + b_2X_2 + b_3X_2^2 + b_4X_2^2X_1 + b_5X_1X_2 \quad \mathbf{3}$$

And 
$$Y_4 = b_0 + b_1X_1 + b_2X_2 + b_3X_1^2 + b_4X_2^2 + b_5X_1X_2 + b_6X_2^2X_1 + b_7X_1^2X_2 + b_8X_1^2X_2^2 \quad \mathbf{4}$$

where,  $b_0, b_1, \dots, b_8$  are constants, 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) and used by Babatunde (1997) 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:

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

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

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

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

$$\frac{\partial^2 Y(X_1, X_2)}{\partial X_1^2} < 0 \quad 8$$

$Y(X_1, X_2)$  is a maximum when Eq. (7) is true and the right hand side of Eq. (8) is less than zero;  $Y(X_1, X_2)$  is a minimum when Eq. (7) is true and the right hand side of Eq. (8) is greater than zero;  $Y(X_1, X_2)$  is a saddle point or give no information when the right hand side of Eq. (8) is equal to, or greater than, zero at the coordinates defined by the solution to the simultaneous Eq. (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 Eq. (9) and (10)

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

$$\frac{dY(X_2)}{dX_2} = 0 \text{ for } Y(X_2) \quad 10$$

while 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 Eq. (9) or (10) for  $Y(X_1)$  or  $Y(X_2)$  respectively.

**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. 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, UK). 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 (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). The temperature range of the Electronic Temperature Controller is 0–400 °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<sup>-1</sup>. 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<sup>-1</sup> 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<sup>-1</sup>. Each experimental trial was replicated three times making a total of 192 treatment combinations that were carried out.

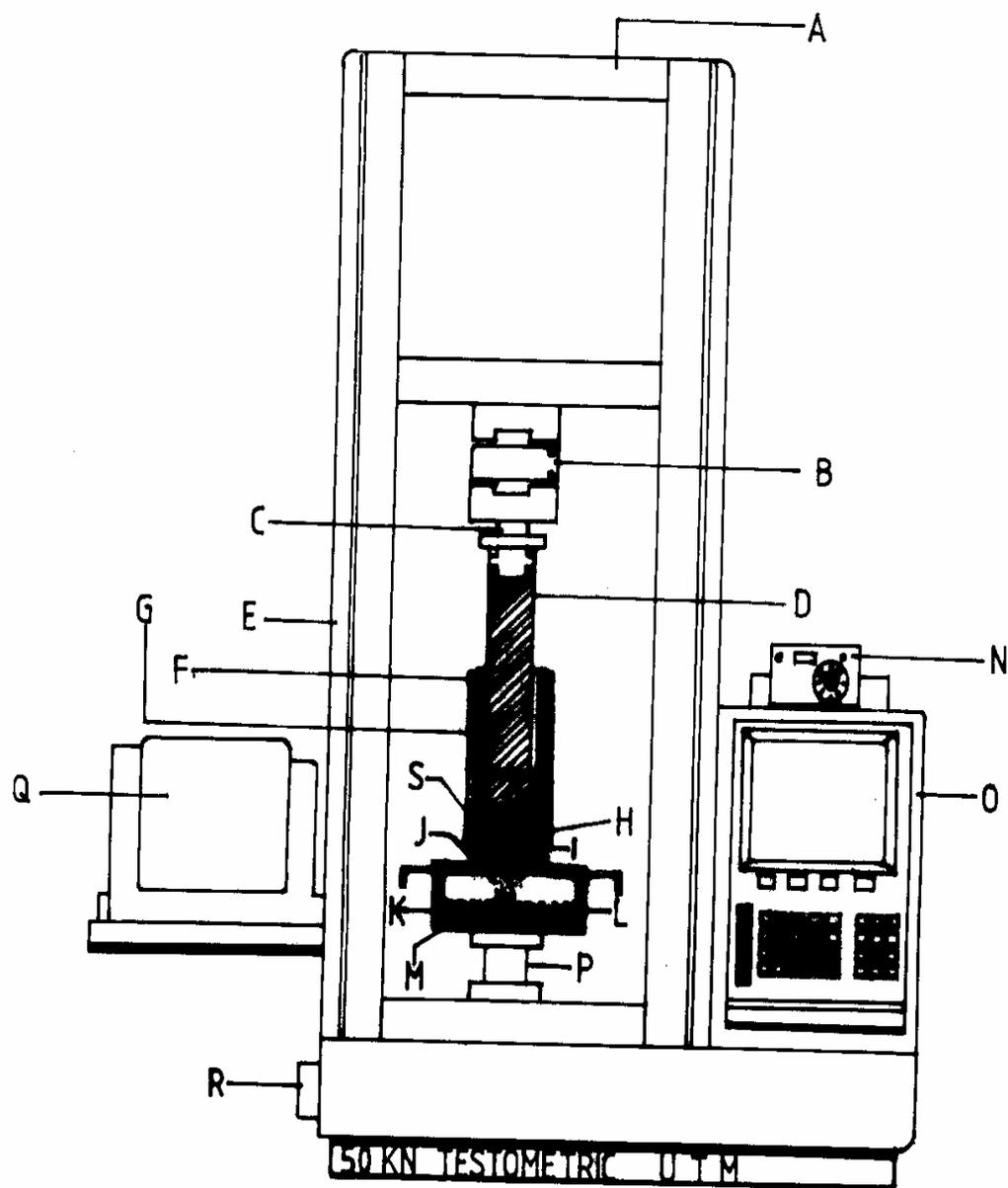


Figure 1. The mechanical expression rig: A - crosshead; B - load cell of the UTM; C - upper attachment of the UTM; D - compression piston; E - frame of the UTM; F - press cage cylinder; G - heating device; H - thermocouple probe from the electronic temperature controller; I - oilseed cake; J - drainage 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

**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 Eq. (11):

$$O_Y = \frac{W_{OE}}{W_{US}} \times 100\% \quad 11$$

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 Eq. (12):

$$O_E = \frac{W_{OE}}{xW_{US}} \times 100\% \quad 12$$

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 Eq. (13):

$$O_L = \frac{W_{US} - (W_{RC} + W_{OE})}{W_{US}} \times 100\% \quad 13$$

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 %

**RESULTS AND DISCUSSION** Based on the model in Eq. (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.

**Model equations** From the regression analysis, the following regression models were obtained for oil yield, oil recovery efficiency and process loss as shown in Eq. (14) to (16):

$$Y_1 = -19.114 + 0.834X_1 + 5.023X_2 - 4.563E-03X_1^2 - 0.211X_2^2 - 3.303E-03X_2^2X_1 - 2.381E-04X_1^2X_2 + 4.518E-05X_1^2X_2^2 \quad 14$$

$$Y_2 = -30.462 + 1.356X_1 + 8.253X_2 - 7.422E-03X_1^2 - 0.499X_2^2 - 6.824E-04 - 3.637E-04X_1^2X_2 + 3.783E-05X_1^2X_2^2 \quad 15$$

$$Y_3 = -3.244 + 9.176E-02X_1 + 0.468X_2 + 7.980E-05X_1^2 + 0.128X_2^2 - 3.279E-03X_2^2X + 6.760E-06X_1^2X_2 + 1.018E-05X_1^2X_2^2 \quad 16$$

where,  $Y_1$ ,  $Y_2$  and  $Y_3$  are oil yield, oil recovery efficiency and process loss respectively in % while  $X_1$  and  $X_2$  are heating temperature ( $^{\circ}\text{C}$ ) and applied pressure (MPa) respectively.

Analysis of Variance (ANOVA) shown in Table 1 indicates that high F-values and high Chi – square ( $R^2$ ) were obtained from model 4, which formed the basis of Eq. (14), (15) and (16) 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 the model 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 an evidence that it fits the data well and, therefore readily applicable.

Table 1. Analysis of variance of multiple regression of oil yield, oil recovery efficiency and process loss as a function of heating temperature and applied pressure

Measured	Source	Sum of	DF	Mean	F	Sig.	$R^2$
Oil yield	Regression	1319.454	7	188.493	69.716	0.000	0.924*
	Residual	108.149	40	2.704			
	Total	1427.602	47				
Oil recovery	Regression	3420.635	7	488.662	111.776	0.000	0.951*
	Residual	174.871	40	4.372			
	Total	3595.506	47				
Process loss	Regression	151.257	7	21.608	8.747	0.000	0.605*
	Residual	98.817	40	2.470			
	Total	250.075	47				

\* Significant at  $P \leq 0.05$

**Optimization process** Based on the model equations for oil yield (Eq. (14), oil recovery efficiency (Eq. (15) and process loss (Eq. (16), critical values of the process parameters were determined through partial differential equations and the results are as presented in Table 2. The table shows that, in order to maximize oil yield, a temperature of  $86.62^{\circ}\text{C}$  and a pressure of 10.42 MPa should be selected for extraction process. To maximize oil recovery efficiency, a temperature of  $82.24^{\circ}\text{C}$  and a pressure of 9.69 MPa should be used. However, in order to minimize process loss, a temperature of  $55.51^{\circ}\text{C}$  and a pressure of 7.15 MPa should be selected. Therefore, for optimal operation of dry process of SB extraction, a temperature of  $82.24^{\circ}\text{C}$  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 the result is as

shown in Table 3. 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 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 dry process of SB extraction. A careful observation of predicted and measured responses of oil yield, oil recovery efficiency and process loss to heating temperature and applied pressure (Figures 2-4) also show high level of correlation which confirms that the model is applicable.

Table 2. Critical points of heating temperature and applied pressure for optimal measured parameters

Measured parameters (%)	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

Table 3. Paired sample t-test of the predicted and measured values of oil yield, oil recovery efficiency and process loss

Parameters	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

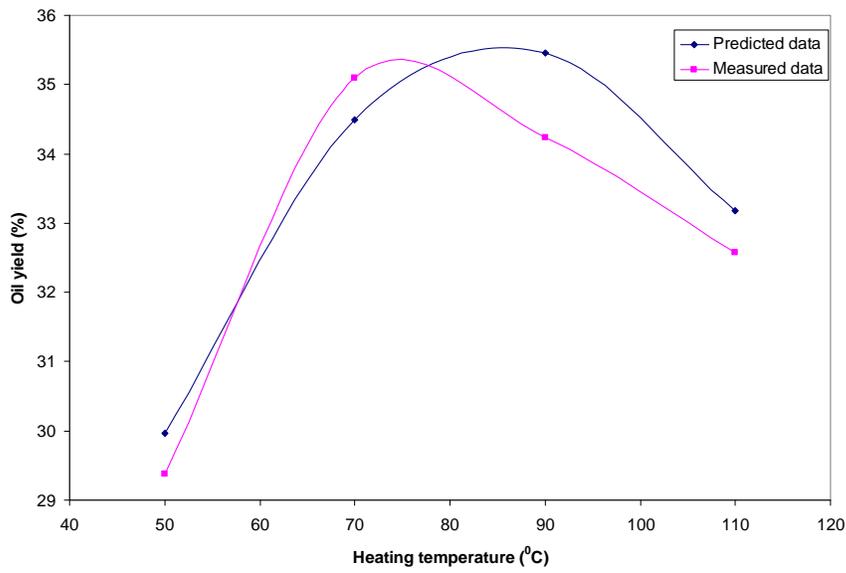


Figure 2. Effect of heating temperature on oil yield

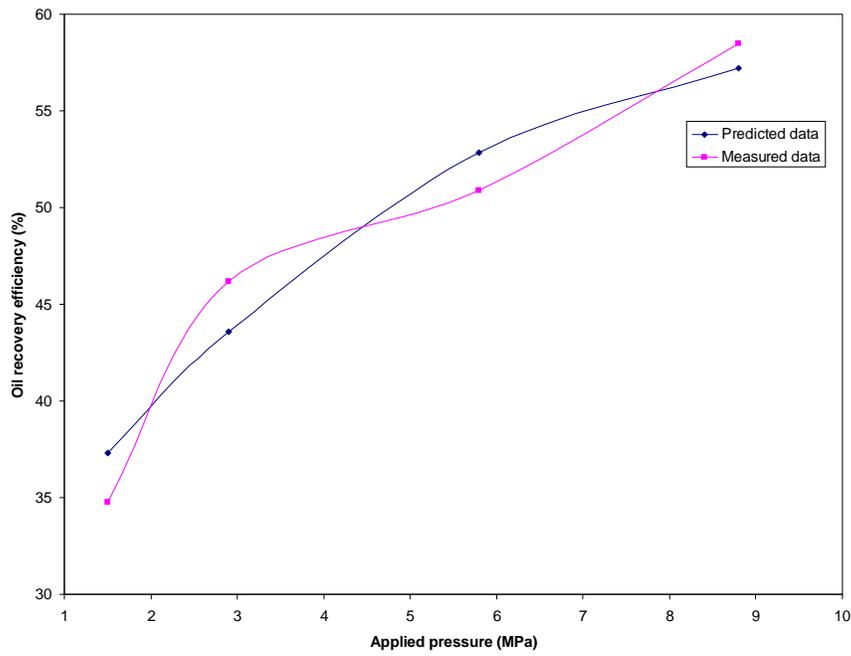


Figure 3. Effect of applied pressure on oil recovery efficiency

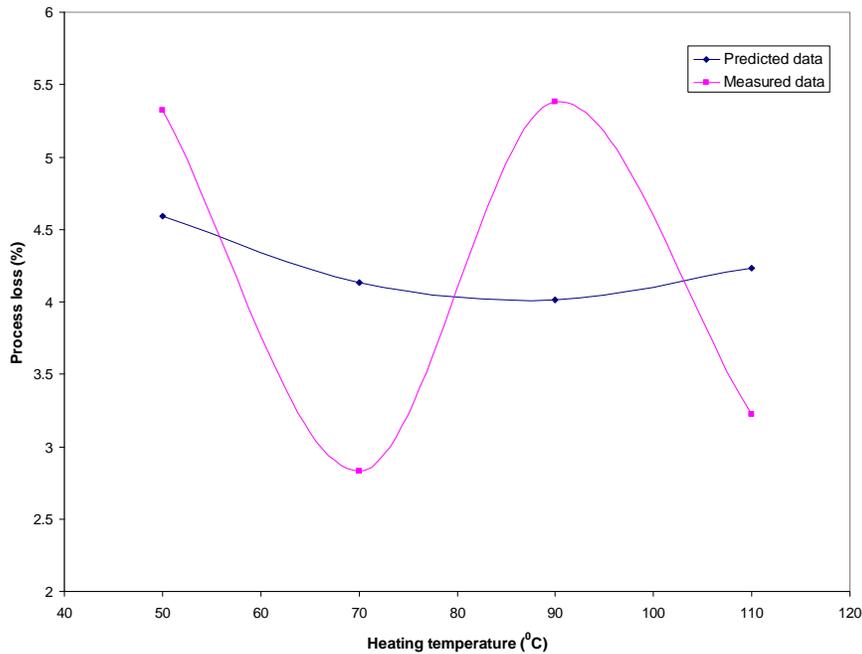


Figure 4. Effect of heating temperature on process loss

**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 temperature, applied pressure and loading rate of 82.24 °C, 9.69 MPa and 2.50 mm min<sup>-1</sup> 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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