Use of Factorial Design of Experiment and Response Surface Methodology for the Study of the Effects of Different Types of Oil on the Yield of Soap.

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ABSTRACT

The work was carried out to study the yield of soap using different oils and oil blends. The experimental design, randomization, optimization and statistical analysis were carried out using Box Benken Design Expert 10.0. The design of experiment was done using four independent factors (variables), at three categorical levels. The optimum oil yield for palm kernel oil (P.K.O), coconut oil, P.K.O and olive oil, P.K.O and coconut oil blends and olive oil and coconut oil blend are 179.90 g, 150.22 g, 118.90 g, 130.99 g and, 105.00 g, respectively. The yield of soap at these optimized values for P.K.O, coconut oil, P.K.O and olive oil, P.K.O and coconut oil blends and olive oil and coconut oil blend are respectively, 123 g, 12.09 g, 79 g, 88.6 g and59.99 g. This showed that P.K.O is the best oil for soap production for it guarantees very good yield of soap, and other oils could be blended with PKO when special soaps are required. The coefficient of determination (R^2) for P.K.O, coconut oil, PKO and olive oil, PKO and coconut oil blend, olive and coconut oil blend are 0.8007, 0.6649, 0.9739, 0.9266 and 0.9131, respectively. Mathematical models developed were found significant ($p<0.05$) for predicting the response variable. The various contour and 3-D surface graphs plotted can be used to determine the response variable in lieu of the mathematical models.

Key words: Soap yield, oil yield, oil blends, oils and palm kernel oil (PKO)

INTRODUCTION

Soaps are widely used in our society. Soap is a product of the reaction between a fat/oil and caustic soda (sodium hydroxide):

Fat + 3NaOH → glycerin + 3soap

Soap is produced industrially in four basic steps. (Colgate Palmolive, 2012, Schuman, *et al*, 2005)

Step 1 – Saponification - where a mixture of tallow (animal fat) and coconut oil is mixed with sodium hydroxide and heated. The soap produced is the salt of a long chain carboxylic acid.

Step 2 - Glycerin removal

Salt is added to the wet soap causing it to separate out into soap and glycerin in salt water.

Step 3 - Soap purification

Any remaining sodium hydroxide is neutralized with a weak acid such as citric acid and two thirds of the remaining water removed.

Step 4 - Finishing

Additives such as preservatives, colour and perfume are added and mixed with the soap and it is shaped into bars for sale.

Soft and hard soap

Depending on the nature of the alkali used in their production, soaps have distinct properties. Sodium hydroxide (NaOH) gives "hard soap", whereas, when potassium hydroxide (KOH) is used, a soft soap is formed (Thorsten *et al.*, 2005).

Soaps are useful surfactants for washing, bathing; and cleaning, they are also used in textile spinning and are important components of lubricants. Soaps for cleansing are obtained by treating vegetable or animal oils and fats with a strong alkaline solution. The production of soap therefore helps to boost the economy of the country and provide jobs for many people through entrepreneurship.

RESPONSE SURFACE

Here a response of interest is influenced by several variables and the objective is to optimise this response (Giunt, *et al.*, 1996; Montgomery, 2005). It can be expressed as:

$$y = f(x_1, x_2) + e \qquad (1)$$

The variables x_1 and x_2 are independent variables and the response y depends on them. The dependent variable y is a function of x_1, x_2; the error term, is denoted as e. The error term e, represents any measurement error on the response, as well as other types of variations not counted for in f. It is a statistical error that is assumed to distribute normally with zero mean and variance (Toropov *et al,* 1996). In most *RSM* problems, the true response function f is unknown. In order to develop a proper approximation for f, we usually start with a low-order polynomial in some small region. If the response can be defined by a linear function of independent variables, then the approximating function is a **first-order model**. A first-order model with 2 independent variables can be expressed as:

$$y = \beta_o + \beta_1 x_1 + \beta_2 x_2 + \varepsilon \qquad (2)$$

If there is a curvature in the response surface, then a higher degree polynomial should be used (Myers *et al*, 1995, McNeil, 1990). The approximating function with 2 variables is called a second order model. This is given in Eq. 3.

$$y = b_o + b_1 x_1 + b_2 x_2 + b_{11} x_{11}^2 + b_{22} x_{22}^2 + b_{12} x_1 x_2 + e. \qquad (3)$$

In general, all *RSM* problems use either one or the mixture of the both of these models. In each model, the levels of each factor are independent of the levels of other factors. In order to get the most efficient result in the approximation of polynomials the proper experimental design must be used to collect data. Once the data are collected, the Method of Least Square is used to estimate the parameters in the polynomials (Anderson *et al*,2007, Cavitch,1994, Douglas, 2005)

This work was aimed at optimisation of yield of soap using a combination of different types of oils by the method of response surface and design of experiment. The variables that make up the soap were also optimised.

The multiple-regression model can be written in a matrix form as

$$y = X\beta + e \qquad (4)$$

Where:

$$\mathbf{y} = \begin{bmatrix} y_1 \\ y_2 \\ \vdots \\ y_n \end{bmatrix}_{(nX1)} \quad \mathbf{X} = \begin{bmatrix} 1 & x_{11} & x_{12} \cdots & x_{1q} \\ 1 & x_{21} & x_{22} \cdots & x_{2q} \\ \vdots & \vdots & \vdots & \vdots \\ 1 & x_{n1} & x_{n2} \cdots & x_{nq} \end{bmatrix}_{(nXk)} \quad \mathbf{ß} = \begin{bmatrix} \beta_0 \\ \beta_1 \\ \vdots \\ \beta_q \end{bmatrix}_{(kX1)} \quad \mathbf{e} = \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \vdots \\ \varepsilon_n \end{bmatrix}_{(nX1)}$$

$\boldsymbol{y}$ is an (n x 1) vector of observations, $\boldsymbol{X}$ is an (n x k) matrix of levels of independent variables, $\boldsymbol{\beta}$ is a (k x 1) vector of regression coefficients, and $\boldsymbol{e}$ is an (n x 1) vector of random errors (Montgomery, 2005). If X is a (k x k) matrix, then the linear system $\boldsymbol{y} = \boldsymbol{X\beta} + \boldsymbol{e}$ has a unique least squares solution given by**:**

$$\beta = (X^t X)^{-1} X^t y \tag{5}$$

The estimated regression equation is

$$\hat{y} = X\hat{\beta} \tag{6}$$

Orthogonal First-Order Design

In order to design a model that is efficient it is necessary to take the estimation of variances into consideration. The orthogonal first-order design minimizes the variance of the regression coefficients.B_j. A first-order design is orthogonal if the off-diagonal elements of the $(X^t X)$matrix are all zero (Montgomery, 2005). Consequently, the cross-products of the columns of the X matrix sum to zero, the inverse matrix of $(X^t X)$ can be obtained easily, and all of the regression coefficients are uncorrelated. When the columns of the $\boldsymbol{X}$ matrix are mutually orthogonal then the levels of the corresponding variables are linearly independent

METHODOLOGY

The permutation and optimization were carried out using Response Surface Methodology (Box-Benken Design) in order to reduce the number of experimental runs and to develop a second order regression model from the Analysis of Variance (ANOVA). This was used in order to reduce cost and determine the oil optimum yields among the four different types of oil used in this work. The yield (response) in this work is the mass of the soap produced. The results will help to determine optimum constituents of soap which yield a very good response (product mass, g).

The constituents considered are quantity of palm kernel oil, (P.K.O) (g), quantity of olive oil (g), quantity of coconut oil (g), quantity of sodium hydroxide solution (ml), quantity of soda ash solution (ml), and quantity of sodium sulphate solution (ml)

THE EXPERIMENTAL DESIGN

Main constituents of soap such as P.K.O (g), olive oil (g), and coconut oil (g), and others such as sodium hydroxide solution (ml), soda ash solution (ml), and sodium sulphate solution (ml) were

investigated in order to optimise these constituents for achieving maximum mass of soap. Three main factors were considered: quantity of Oil, quantity of Sodium hydroxide, and quantity of sodium carbonate. Actual process variable (x_i) was related to the coded process variables as shown in Equ.6 below:

$$X_i = \frac{x_i - x_o}{\Delta x} \quad (6)$$

Where;

X_i is the dimensionless coded value of the independent variables,

x_i is the actual value of the independent variables at the design center point,

Δx is the variable increments about the center point.

The center point chosen for the design was 50% soap constituents at a concentration of 28 % w/v. The study was divided into two major parts. First, the preliminary experiments and second, the optimisation of soap mass. The study was carried out using a second order polynomial model. The experimental values of the variables used in the coded and actual form in this study are given in Tables 3.1, 4.2, 4.4, 4.6, 4.8, 4.10 and 4.12. below

Table 1 Constituents Employed in a Box-Benken Design for Optimisation.

Range of constituents	**-1**	**0**	**1**
Mass of Oil (g)	100	125	150
NaOH solution (cm^3)	55	70	85
Na_2CO_3 (cm^3)	45	60	75
Na_2SO_4(cm^3)	22	37	52

Statistical analysis

The data obtained from the experiments were analyzed statistically using test of significance and response surface methodology, so as to fit the second-order mathematical model generated by the Design-Expert Software version 9.0.3 (Stat-Ease Inc., Minneapolis, USA). To correlate the response variable to the independent variables, multiple regressions was used to fit the coefficient of the polynomial model of the response. The quality of the fit of the model was evaluated using Analysis of Variance (ANOVA). The fitted second-order mathematical model is described in Eq. 7 below:

$$y = b_o + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{i=1}^{k} b_{ij} x_i x_j + e \qquad 7$$

where, y is response variable (mass of soap produced). It was also used in the individual optimisation of variables such as P.K.O., b_o is the intercept value, b_i (i = 1, 2, ..., k) is the first order model coefficient, b_{ij} (i=1...k, j=1...k) is the interaction effect, and b_{ii} represents the quadratic coefficients of x_i, and e is the random error.(Aguwa, et al 2012)

RESULTS

The results of the ANOVA for PKO is shown in Table 4.1. The model F-value of 11.95 with low P-value (P<0.0001) implied a high significance for the regression model. The goodness of fit of the model was checked by calculating the coefficient of determination (R^2), which must not be less than 0.80 (80%) for the good fit of a model. Here, the R^2 value of 0.8007 obtained indicated that the sample variation of 80.07% for the soap production could be attributed to the independent variables (quantity of NaOH, quantity of Na_2CO_3, mass of Oil, quantity of Na_2SO_4) mole ratio of catalyst to oil and separation time) and only 19.03 % of the total variations are not explained by the model. Hence, the model could be used in theoretical prediction of the yield (%) of the soap mass. The developed regression model is shown in Equ.8.

$Y = 161.07 + 6.87A + 8.87B – 2.35C + 2.67D – 1.31AB + 0.62AC – 0.035AD + 7.08BC – 8.5BD – 0.36CD + 9.71A^2 – 7.04B^2 – 0.28C^2 – 0.71D^2$. …… 8

Where;

A is quantity of oil

B is quantity of sodium hydroxide (Na_2OH)

C is quantity of sodium carbonate (Na_2CO_3)

D is quantity of sodium sulphate (Na_2SO_4)

Table 2. Experimental Table for P.K.O Optimisation

Run	Quantity of P.K.O (g)	Quantity of NaOH (ml)	Quantity of Na_2CO_3 (ml)	Quantity of $NaSO_4$ (ml)	Response (g)
1	125	85	60	52	160.50
2	125	70	60	37	161.17
3	125	70	75	52	165.47
4	150	55	60	37	170.33
5	125	85	45	37	168.50
6	150	70	75	37	175.69
7	125	55	45	37	159.20
8	125	70	60	37	161.17
9	100	70	60	37	164.00
10	125	70	60	22	160.49
11	125	55	75	37	123.38
12	100	70	45	37	163.09
13	125	70	45	52	159.50
14	125	55	60	52	159.90
15	150	70	60	22	175.75
16	125	70	60	37	161.17
17	125	85	75	37	160.99
18	100	70	75	37	162.70
19	150	70	60	52	174.60
20	125	55	60	22	125.70
21	100	85	60	37	164.70
22	150	85	60	37	179.90
23	100	70	60	52	162.99
24	100	55	60	37	149.90
25	125	85	60	22	160.30
26	125	70	45	22	158.90
27	150	70	45	37	173.60
28	125	70	60	37	161.17
29	125	60	75	22	165.30

Table 3. ANOVA for P.K.O Optimisation.

Source	Sum of squares	Df	Mean square	F Value (Fisher values)	P Value (Probability values
Model	3313.9914	1	236.71	4.02	0.0069
A	567.05	1	567.05	9.62	0.0078
B	944.83	1	944.83	16.03	0.0013
C	66.55	1	66.55	1.13	0.3059
D	85.39	1	85.39	1.45	0.2487
AB	6.84	1	6.84	0.12	0.7384
AC	1.54	1	1.54	0.026	0.8740
AD	4.9×10^{-3}	1	4.9×10^{-3}	8.31×10^{-5}	0.9929
BC	200.36	1	200.36	3.40	0.0865
BD	289.00	1	289.00	4.90	0.0439
CD	0.51	1	0.51	8.67×10^{-3}	0.9271
A2	611.22	1	611.22	10.37	0.0062
B2	321.17	1	321.17	5.45	0.0350
C2	0.52	1	0.52	8.882×10^{-3}	0.9263
D2	3.27	1	3.27	0.056	0.8171
Residual	8.27	14	58.94		
Lack of Fit	824.93	10	82.49	1790.22	< 0.0001
Pure Error	0.18	4	0.046		
Cor.Total	4139.11	28			
$R^2 = 0.8007$	Adj. $R^2 = 0.6013$	Pred. $R^2 = 0.7481$	Std. Dev. = 7.68	Mean 161.77	

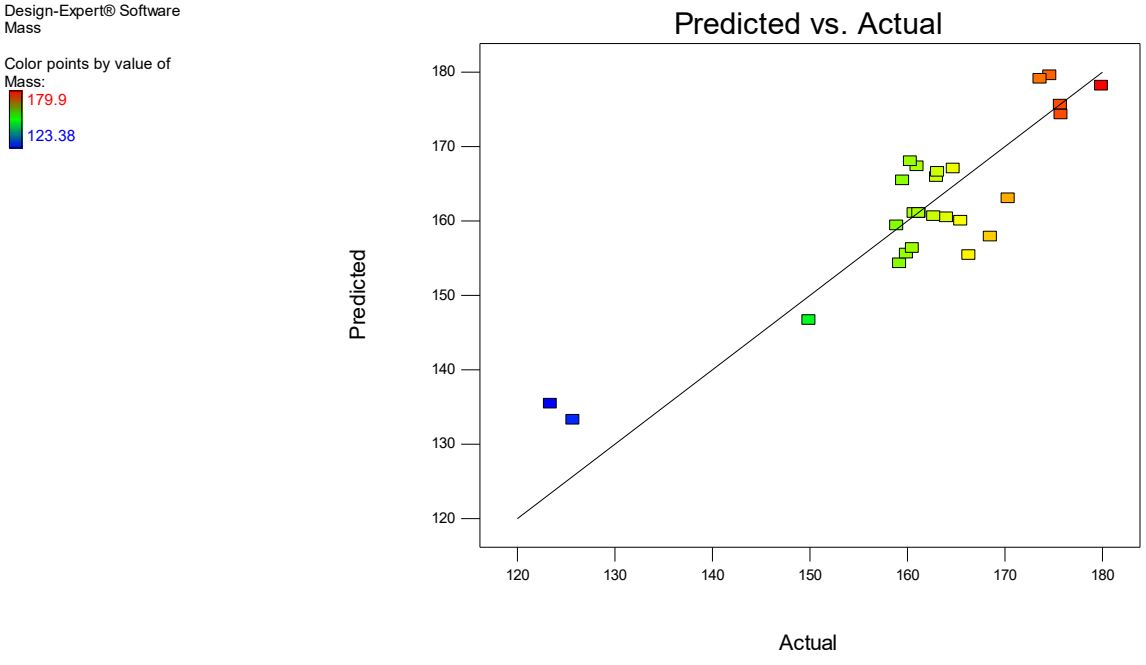


Figure 1: Predicted Vs Actual for P.K.O

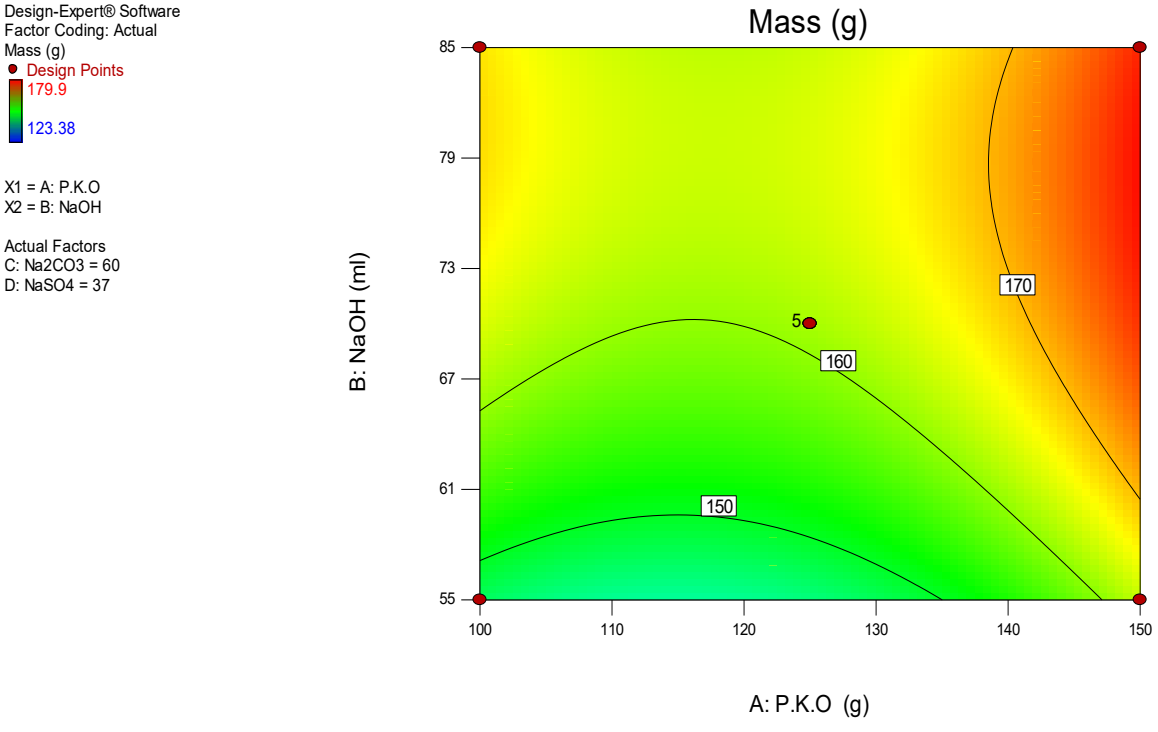


Figure 2: Contour Plot for P.K.O

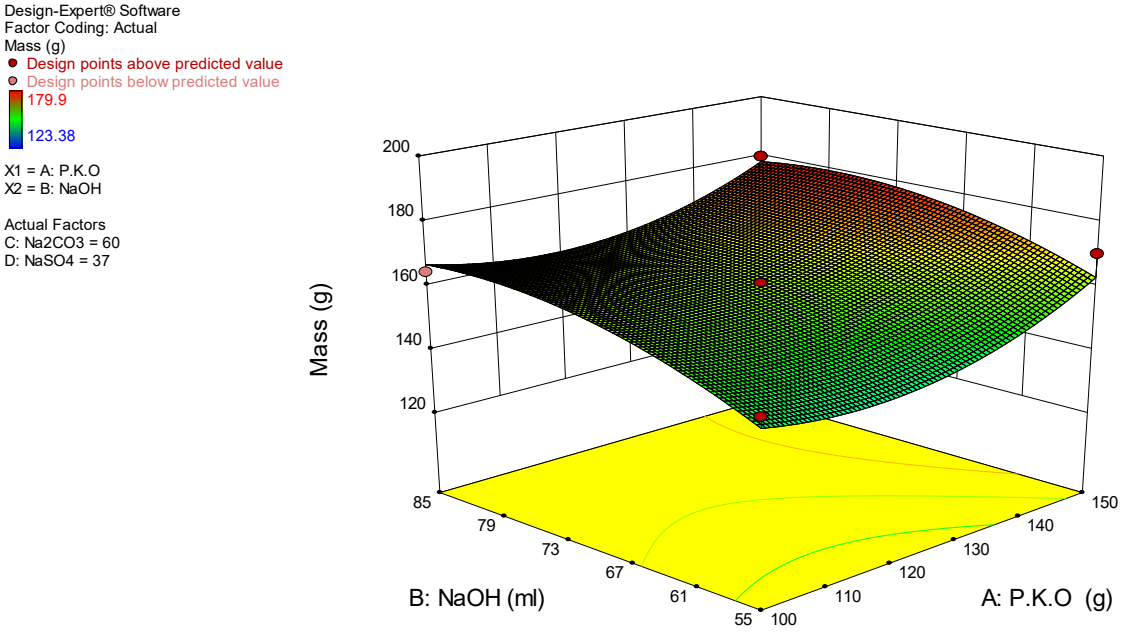


Figure 3. 3D surface plot for P.K.O

Table 4. Experimental Table for Coconut Oil

Run	Quantity of Coconut Oil (g)	Quantity of NaOH (ml)	Quantity of Na_2CO_3 (ml)	Quantity of $NaSO_4$ (ml)	Response (g)
1	125	85	60	52	133.42
2	125	70	60	37	130.39
3	125	70	75	52	121.51
4	150	55	60	37	139.48
5	125	85	45	37	130.97
6	150	70	75	37	144.69
7	125	55	45	37	120.09
8	125	70	60	37	130.39
9	100	70	60	37	120.09
10	125	70	60	22	121.99
11	125	55	75	37	123.09
12	100	70	45	37	121.32
13	125	70	45	52	129.81
14	125	55	60	52	123.53
15	150	70	60	22	142.91
16	125	70	60	37	130.39
17	125	85	75	37	130.56
18	100	70	75	37	120.81
19	150	70	60	52	143.00
20	125	55	60	22	121.23
21	100	85	60	37	120.61
22	150	85	60	37	150.22
23	100	70	60	52	125.70
24	100	55	60	37	121.51
25	125	85	60	22	128.70
26	125	70	45	22	125.56
27	150	70	45	37	140.34
28	125	70	60	37	130.39
29	125	60	75	22	122.59

Table 5. ANOVA for Coconut Oil

Source	Sum of squares	df	Mean square	F Value	P Value
Model	10164.81	1	726.06	1.98	0.1061
A	1189.22	1	1189.22	3.25	0.0939
B	1964.80	1	5.37	0.0361	
C	886.83	1	2.42	0.1418	
D	1.88	1	5.139×10^{-3}	0.9439	
AB	33.87	1	0.093	0.7654	
AC	5.90	1	0.016	0.9007	
AD	7.90	1	0.022	0.8853	
BC	3103.05	1	8.48	0.0114	
BD	1.46	1	4.002×10^{-3}	0.9505	
CD	7.10	1	0.019	0.8912	
A2	626.68	1	1.71	0.2117	
B2	780.89	1	2.13	0.1661	
C2	1010.41	1	2.76	0.1188	
D2	68.53	1	0.19	0.6718	
Residual	5122.13	14			
Lack of Fit	5122.13	10			
Pure Error	0.000	4			
Cor.Total	15286.95	28			
$R^2 = 0.6649$	Adj. $R^2 = 0.3299$	Pred. $R^2 = -0.9300$	Std. Dev. = 19.13	Mean = 126.10	

The results of ANOVA calculation for coconut oil optimisation are shown in Table 4.3. The model's F-value of 1.98 with low P-value ($P<0.0001$) implied a high significance for the regression model. The goodness of fit of the model was checked by the value of coefficient of determination. In this case, the R^2 value of 0.6649 indicated that the sample variation of 66.49 % for the soap production was attributed to the independent variables (quantity of NaOH, quantity of Na_2CO_3, mass of Oil, quantity of Na_2SO_4) mole ratio of catalyst to oil and separation time) and only 33.51 % of the total variations are not explained by the model. The developed regression model is shown

in Equ.9. describing the relationship between the oil response (Y) and the coded values of independent factors and their respective interactions.

$$Y = 130.39 + 9.95A + 12.80B + 8.60C + 0.40D + 2.91AB + 1.21AC + 1.40AD - 27.85BC + 0.61BD - 1.33CD + 9.83A^2 - 10.97B^2 - 012.48C^2 + 3.25D^2. \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 9$$

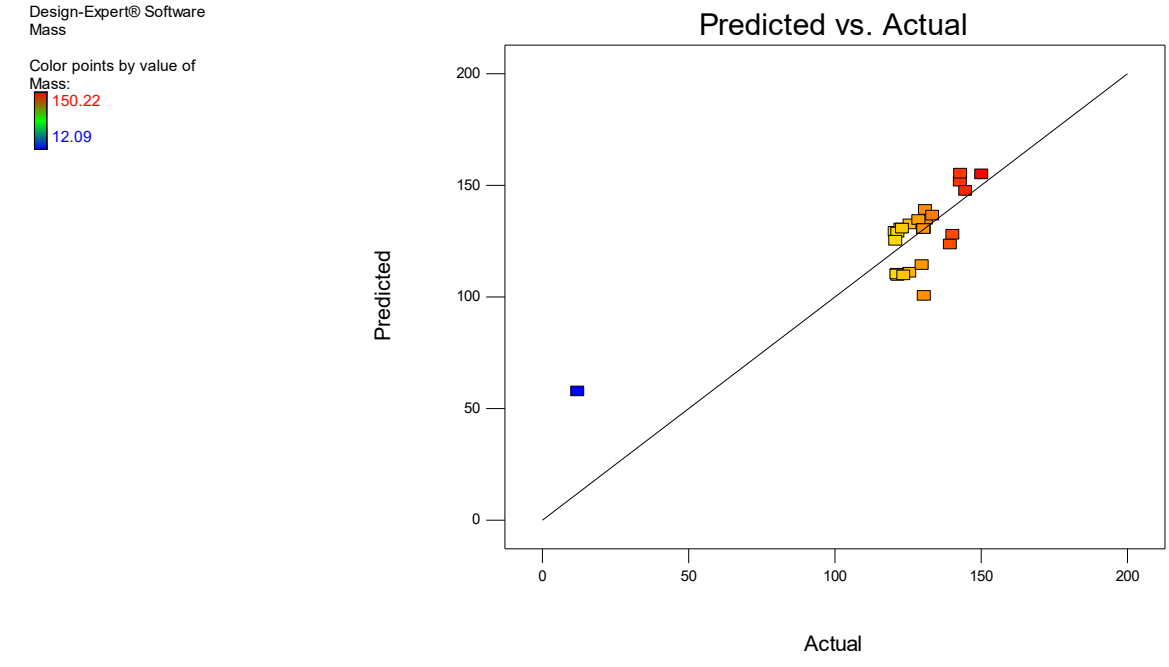


Figure 4. Plot of Predicted Vs Actual for Coconut Oil

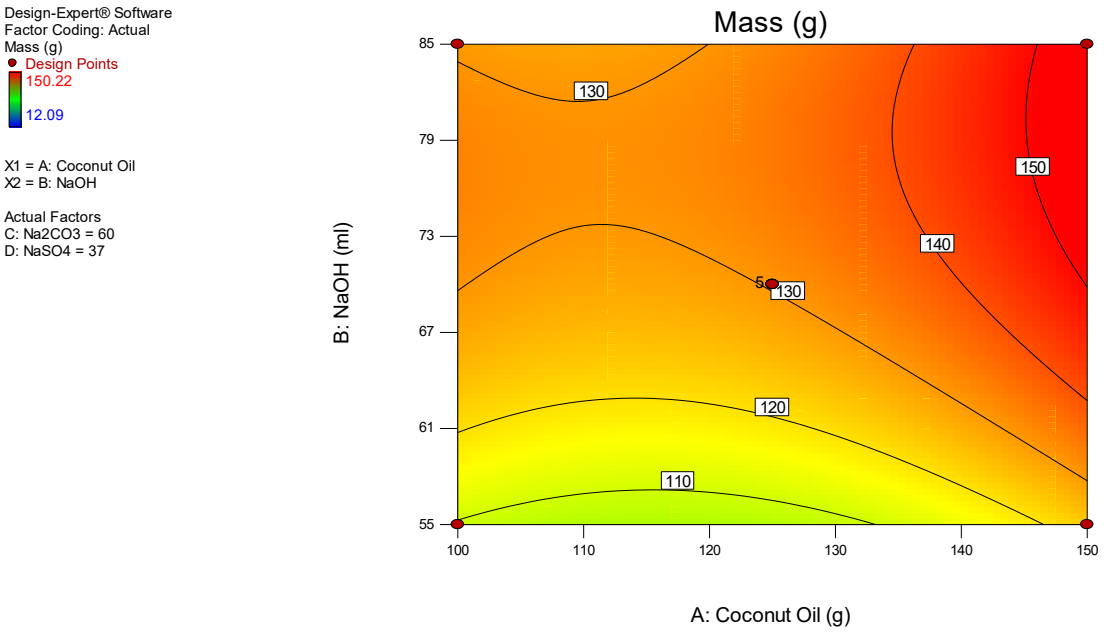


Figure 5: Contour plot for Coconut Oil

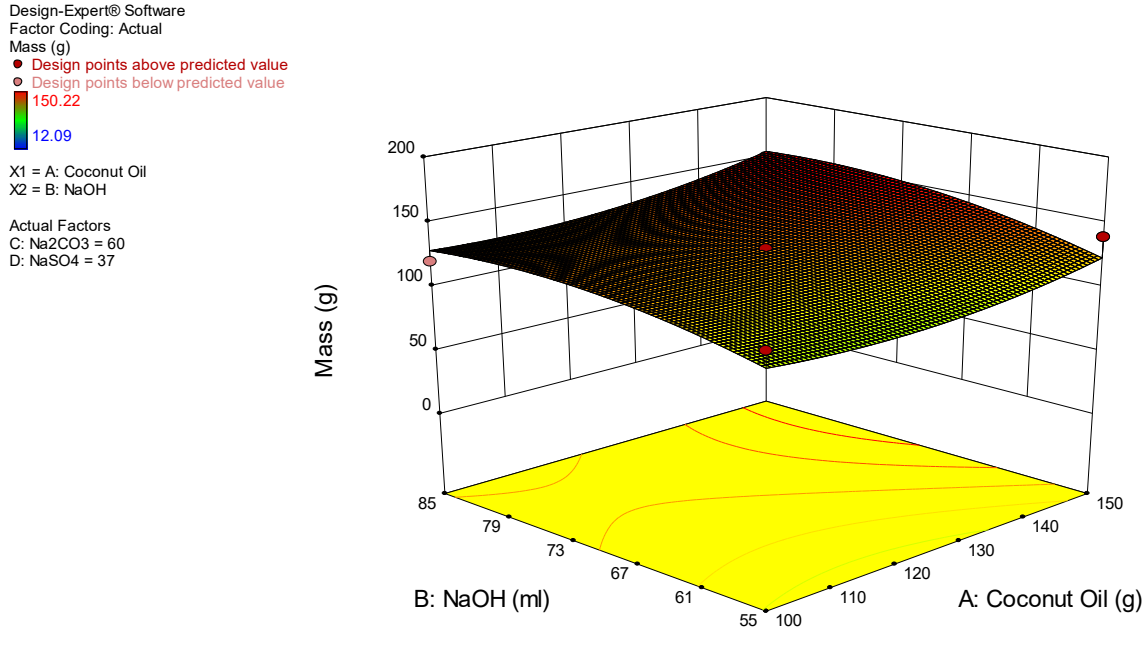


Figure 6. 3D Surface plot for Coconut oil.

Table 6. Experimental Table for PKO and Olive Oil

Run	Quantity of PKO & Olive Oil blend (g)	Quantity of NaOH (ml)	Quantity of Na_2CO_3 (ml)	Quantity of $NaSO_4$ (ml)	Response (g)
1	125	85	60	52	95.89
2	125	70	60	37	82.71
3	125	70	75	52	83.35
4	150	55	60	37	105.85
5	125	85	45	37	89.80
6	150	70	75	37	116.26
7	125	55	45	37	81.99
8	125	70	60	37	82.79
9	100	70	60	37	80.70
10	125	70	60	22	81.90
11	125	55	75	37	82.70
12	100	70	45	37	80.00
13	125	70	45	52	88.90
14	125	55	60	52	82.80
15	150	70	60	22	115.90
16	125	70	60	37	82.71
17	125	85	75	37	99.89
18	100	70	75	37	81.99
19	150	70	60	52	116.40
20	125	55	60	22	81.90
21	100	85	60	37	82.70
22	150	85	60	37	118.90
23	100	70	60	52	81.80
24	100	55	60	37	79.00
25	125	85	60	22	94.00
26	125	70	45	22	88.59
27	150	70	45	37	115.90
28	125	70	60	37	82.71
29	125	60	75	22	83.60

Table 7. ANOVA for PKO and Olive Oil

Source	Sum of squares	Df	Mean square	F Value	P Value
Model	4902.79	14	350.20	37.24	< 0.0001
A	3397.30	1	3397.30	361.30	<0.0001
B	266.40	1	266.40	28.33	0.0001
C	0.53	1	0.53	0.056	0.8159
D	15.53	1	15.53	1.65	0.2196
AB	21.86	1	21.86	2.32	0.1496
AC	0.74	1	0.74	0.079	0.7832
AD	0.090	1	22.00	2.34	0.1484
BC	22.00	1	22.00	2.34	0.1484
BD	32.43	1	32.43	3.45	0.0844
CD	0.078	1	0.078	8.33×10^{-3}	0.9285
A^2	1137.71	1	1137.71	120.99	< 0.0001
B^2	20.67	1	20.67	2.20	0.1604
C^2	50.49	1	50.49	5.37	0.0361
D^2	19.55	1	19.55	2.08	0.1713
Residual	131.64	14	9.40		
Lack of Fit	131.64	10	13.61		
Pure Error	0.000	4	0.000		
Cor.Total	5034.44	28			

$R^2 = 0.9739$ Adj. $R^2 = 0.9477$ Pred. $R^2 = 0.8494$ Std. Dev. = 3.07 Mean = 90.80

The results of ANOVA calculations for the blend of coconut oil and olive oil are presented in Table 4.5. The model F-value of 37.24 with low P-value (P<0.0001) implied a high significance for the regression model. The goodness of fit of the model was checked by the value of coefficient of determination (R^2). In this case, the R^2 value of 0.9739 indicated that the sample variation of 97.39 % for the soap production is attributed to the independent variables (quantity of NaOH, quantity of Na_2CO_3, mass of Oil, quantity of Na_2SO_4) mole ratio of catalyst to oil and separation time) and only 2.61 % of the total variations are not explained by the model. The developed regression model is shown in Equ.10.

$$Y = 82.71 + 16.83A + 4.71B + 0.21C + 1.41D + 2.34AB - 0.43AC + 0.15AD + 2.35BC + 2.85BD - 0.14CD + 13.24A^2 + 1.78B^2 - 2.79C^2 + 1.74D^2. \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 10.$$

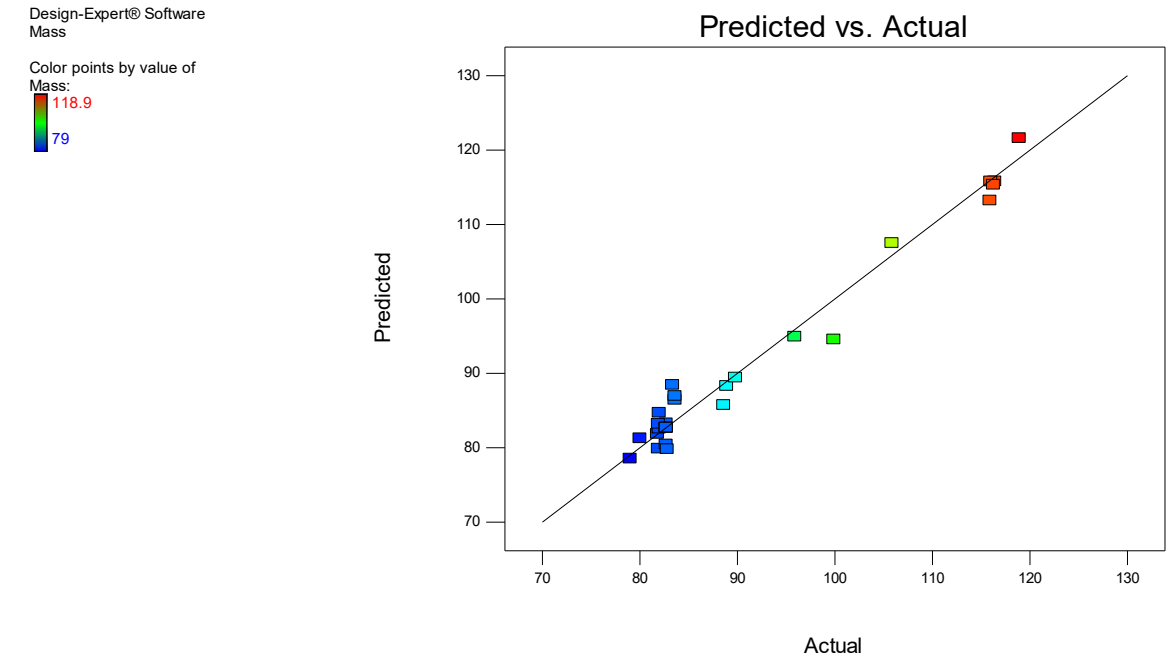


Figure 7. Predicted Vs Actual plot for P.K.O and Olive Oil

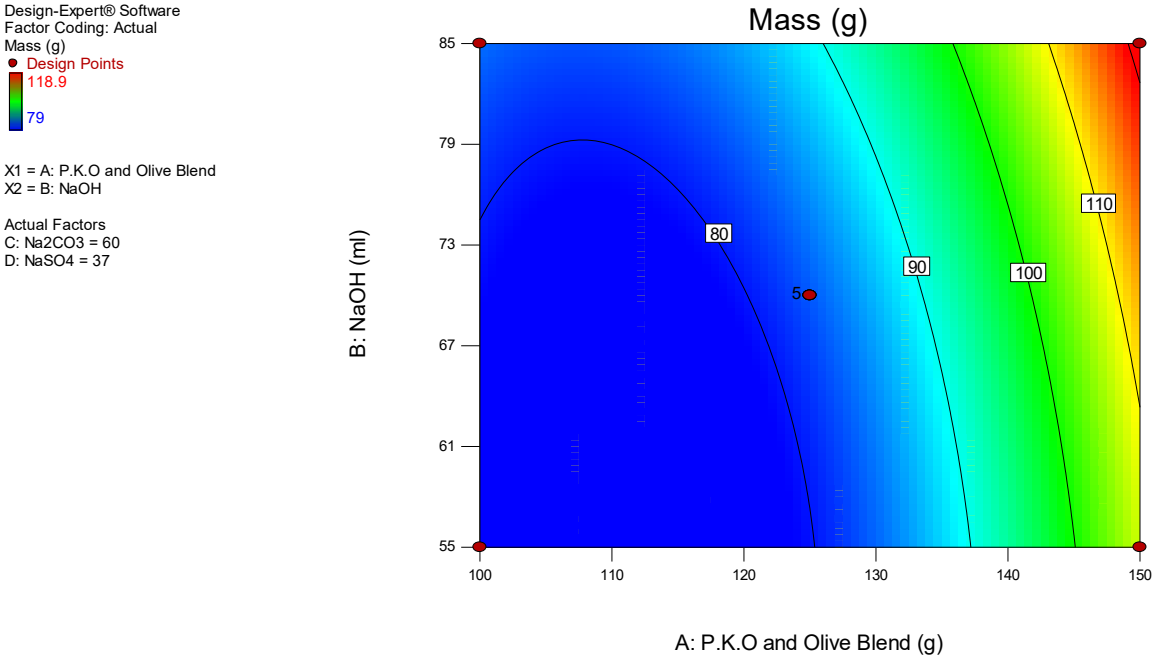


Figure 8. Contour plot for P.K.O and Olive Oil

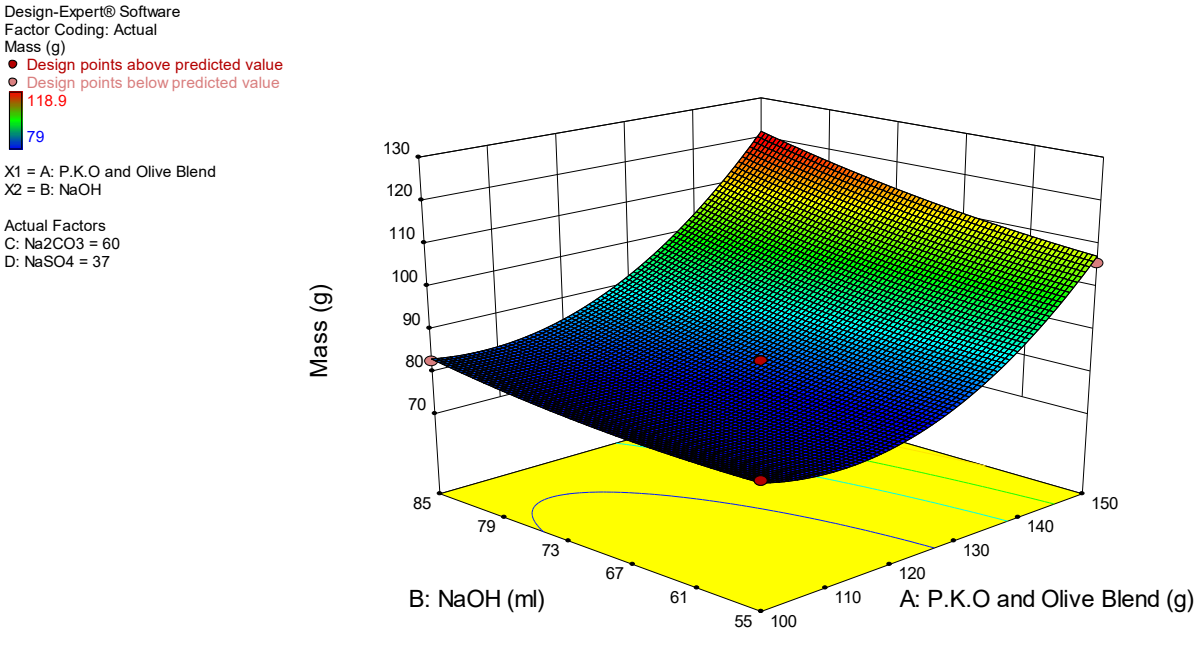


Figure 9. 3D Surface plot for P.K.O and Olive Oil.

Table 8. Experimental Table for PKO and Coconut Oil blend

Run	Quantity of PKO and Coconut Oil blend (g)	Quantity of NaOH (ml)	Quantity of Na_2CO_3 (ml)	Quantity of $NaSO_4$ (ml)	Response (g)
1	125	85	60	52	93.52
2	125	70	60	37	89.79
3	125	70	75	52	89.67
4	150	55	60	37	123.40
5	125	85	45	37	90.10
6	150	70	75	37	128.22
7	125	55	45	37	126.12
8	125	70	60	37	89.79
9	100	70	60	37	57.85
10	125	70	60	22	87.45
11	125	55	75	37	126.50
12	100	70	45	37	88.60
13	125	70	45	52	89.50
14	125	55	60	52	124.20
15	150	70	60	22	127.60
16	125	70	60	37	89.79
17	125	85	75	37	92.59
18	100	70	75	37	88.70
19	150	70	60	52	128.90
20	125	55	60	22	125.50
21	100	85	60	37	90.90
22	150	85	60	37	130.99
23	100	70	60	52	89.90
24	100	55	60	37	125.91
25	125	85	60	22	93.90
26	125	70	45	22	88.59
27	150	70	45	37	127.90
28	125	70	60	37	88.99
29	125	60	75	22	89.85

Table 9. ANOVA for PKO and Coconut Oil blend

Source	Sum of squares	df	Mean square	F Value	P Value
Model	8543.68	14	610.26	12.63	< 0.0001
A	3107.30	1	3107.30	64.30	< 0.0001
B	2123.21	1	2123.21	43.94	< 0.0001
C	1.77	1	1.77	0.037	0.8509
D	5.2×10^{-3}	1	5.2×10^{-3}	1.078×10^{-3}	0.9919
AB	453.69	1	1.77	0.037	0.0084
AC	0.012	1	0.012	2.5×10^{-4}	0.9876
AD	0.42	1	0.42	8.743×10^{-3}	0.9268
BC	1.10	1	1.10	0.023	0.8821
BD	0.21	1	0.21	4.379×10^{-3}	0.9482
CD	0.25	1	0.25	5.07×10^{-3}	0.9422
A^2	1576.95	1	1576.95	32.63	< 0.0001
B^2	1646.47	1	1646.47	34.07	< 0.0001
C^2	10.68	1	10.68	0.22	0.6455
D^2	22.64	1	22.64	0.47	0.5048
Residual	676.53	14	48.32		
Lack of Fit	676.53	10	67.65		
Pure Error	0.000	4	0.000		
Cor. Total	9220.21	28			
$R^2 = 0.9266$ Adj. $R^2 = 0.8532$ Pred. $R^2 = 0.5774$ Std. Dev. = 6.95 Mean = 104.14					

The results of ANOVA calculated for the blend of PKO and coconut oil is shown in Table 4.7 The model F-value of 37.24 with low P-value ($P<0.0001$) implied a high significance for the regression model. The goodness of fit of the model was checked by the value of coefficient of determination (R^2), which should be at least 0.70 (70%) for the good fit of a model. In this case, the R^2 value of 0.9266 indicated that the sample variation of 92.66 % for the soap production is attributed to the independent variables (quantity of NaOH, quantity of Na_2CO_3, mass of Oil, quantity of Na_2SO_4) mole ratio of catalyst to oil and separation time) and only 2.61 % of the total variations are not explained by the model. The developed regression model is shown in Equ.11

$$Y = 89.79 + 16.09A - 13.30B + 0.38C + 0.021D + 10.65AB + 0.055AC + 0.33AD + 0.53BC + 0.23BD - 0.25CD + 15.59A^2 + 15.93B^2 + 1.28C^2 + 1.874D^2. \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 11$$

Model and Diagnostic Graphs

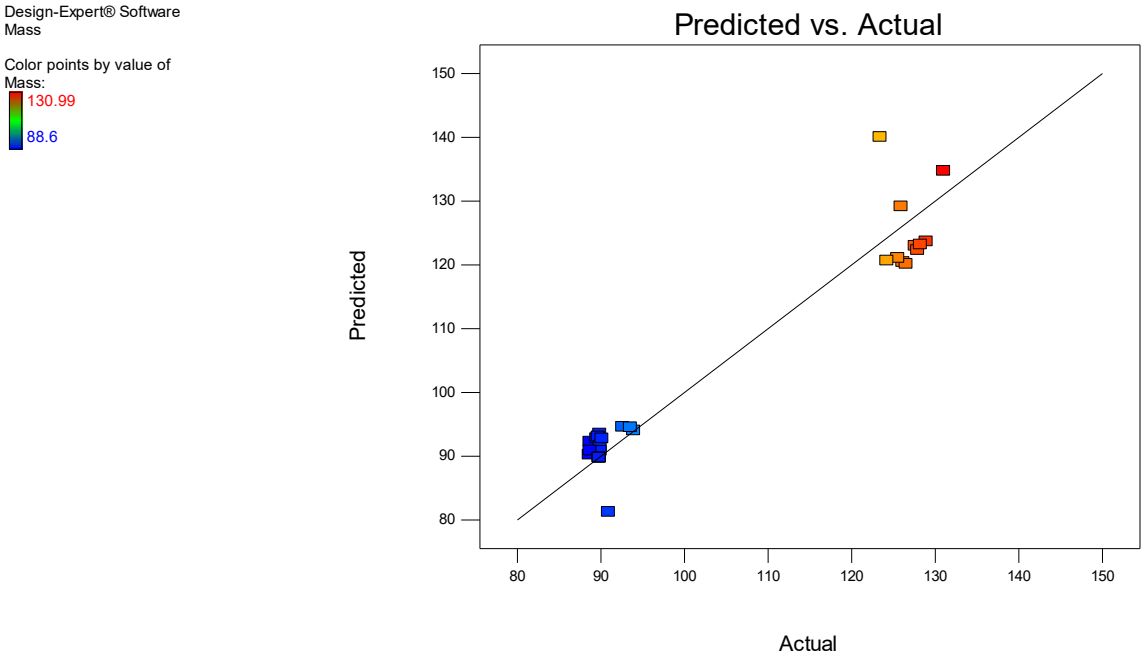


Figure 4.0d: Predicted Vs Actual plot for P.K.O and Coconut Oil

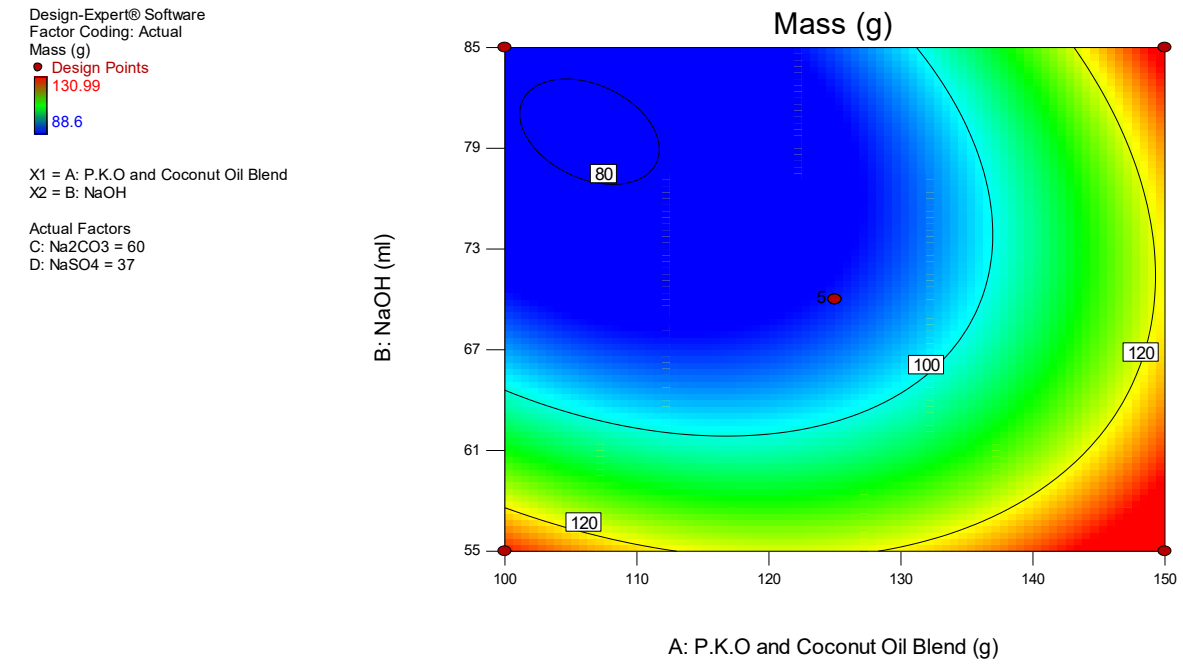


Figure 4.1d: Contour Plot for P.K.O and Coconut Oil

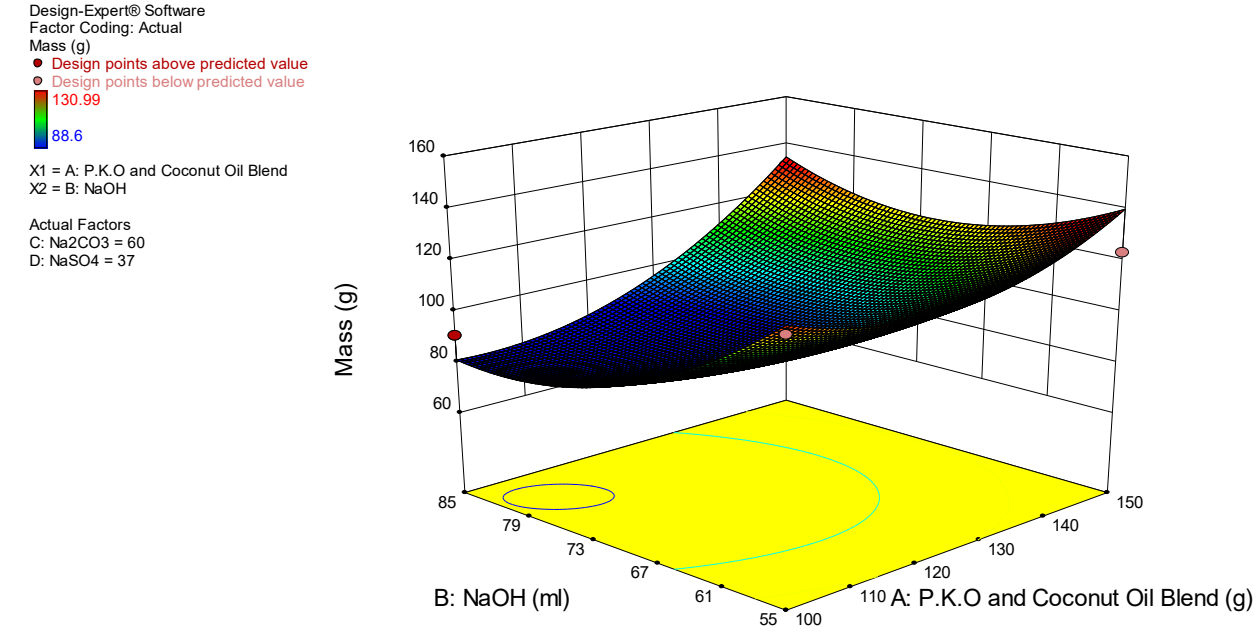


Figure 4.2d: 3D Surface plot for P.K.O and Coconut Oil blend

Table 4.10: Experimental Table for Olive and Coconut Oil blend

Run	Quantity of Olive and Coconut Oil (g)	Quantity of NaOH (ml)	Quantity of Na_2CO_3 (ml)	Quantity of $NaSO_4$ (ml)	Response (g)
1	125	85	60	52	73.85
2	125	70	60	37	60.09
3	125	70	75	52	66.09
4	150	55	60	37	96.03
5	125	85	45	37	72.89
6	150	70	75	37	99.61
7	125	55	45	37	63.99
8	125	70	60	37	60.09
9	100	70	60	37	60.60
10	125	70	60	22	67.99
11	125	55	75	37	63.89
12	100	70	45	37	60.00
13	125	70	45	52	70.99
14	125	55	60	52	63.09
15	150	70	60	22	99.89
16	125	70	60	37	60.09
17	125	85	75	37	74.80
18	100	70	75	37	60.71
19	150	70	60	52	99.99
20	125	55	60	22	64.91
21	100	85	60	37	61.90
22	150	85	60	37	105.00
23	100	70	60	52	60.90
24	100	55	60	37	59.99
25	125	85	60	22	73.20
26	125	70	45	22	60.08
27	150	70	45	37	98.90
28	125	70	60	37	60.09

29	125	60	75	22	66.00

Table 4.11: ANOVA for Olive and Coconut Oil blend

Source	Sum of squares	Df	Mean square	F Value	P Value
Model	6426.26	14	459.02	10.51	< 0.0001
A	3380.83	1	3380.83	77.38	<0.0001
B	206.17	1	206.17	4.72	0.0475
C	1.51	1	1.51	0.034	0.8554
D	46.69	1	46.69	1.07	0.3188
AB	12.46	1	12.46	0.29	0.6017
AC	9.095E-013	1	9.095E-013	2.082E-014	1.0000
AD	283.92	1	283.92	6.50	0.0232
BC	1.01	1	1.01	0.023	0.8813
BD	1.53	1	1.53	0.035	0.8545
CD	29.27	1	29.27	0.67	0.4268
A^2	2382.68	1	2382.68	54.54	<0.0001
B^2	87.88	1	87.88	2.01	0.1780
C^2	19.46	1	19.46	0.45	0.5154
D^2	247.30	1	247.30	5.66	0.0321
Residual	611.64	14	43.69		
Lack of Fit	611.64	10	61.16		
Pure Error	0.000	4	0.000		
Cor.Total	7037.90	28			
R^2 = 0.9131 Adj. R^2 = 0.8262 Pred. R^2 = 0.4994 Std. Dev. = 6.61 Mean = 72.82					

The results of ANOVA calculation for the blend of coconut oil and olive oil is presented in Table 4.11. The model F-value of 10.51 with low P-value (P<0.0001) implied a high significance for the regression model. The goodness of fit of the model was checked by the coefficient of determination (R^2), which should be at least 0.70 (70%) for the good fit of a model. In this case, the R^2 value of 0.9131 indicated that the sample variation of 91.31 % for the soap production is attributed to the independent variables (quantity of NaOH, quantity of Na_2CO_3, mass of Oil, quantity of Na_2SO_4)

mole ratio of catalyst to oil and separation time) and only 8.69 % of the total variations are not explained by the model. The developed regression model is shown in Equ.12.

$$Y = 60.09 + 16.79A + 4.14B + 0.35C - 1.97D + 1.77AB - 3.319 \times 10^{-15}AC + 8.42AD + 0.50BC + 0.62BD - 2.70CD + 19.174A^2 + 3.68B^2 - 1.73C^2 + 6.17D^2. \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad 12$$

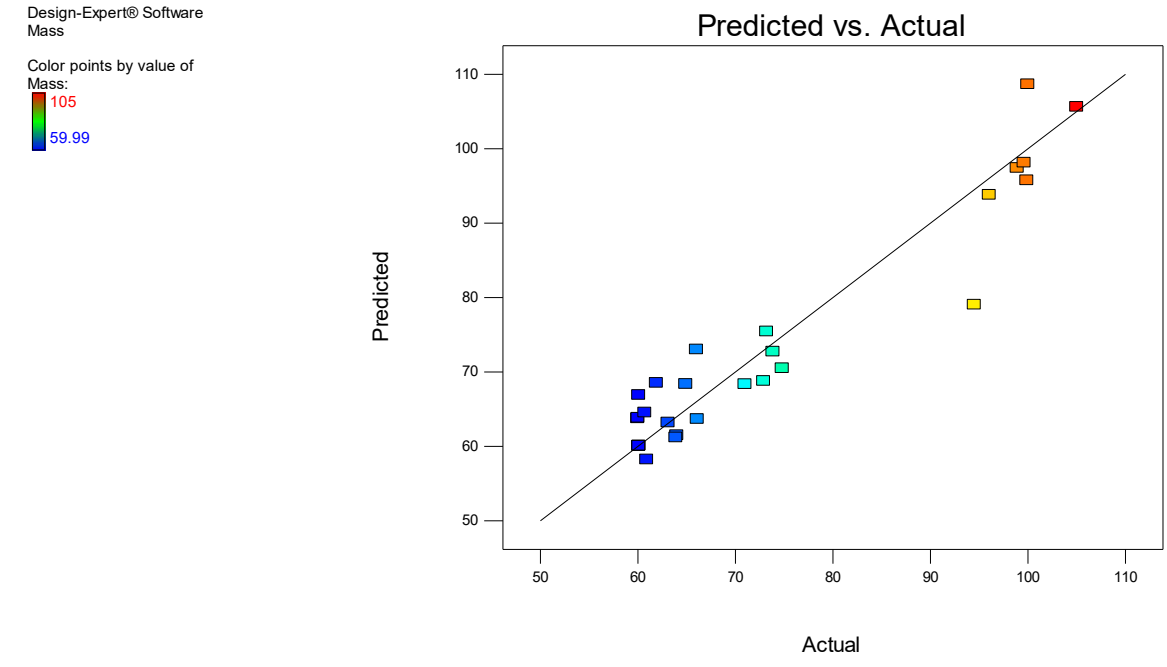


Figure 4.0f: Predicted Vs Actual plot for Olive and Coconut Oil blend

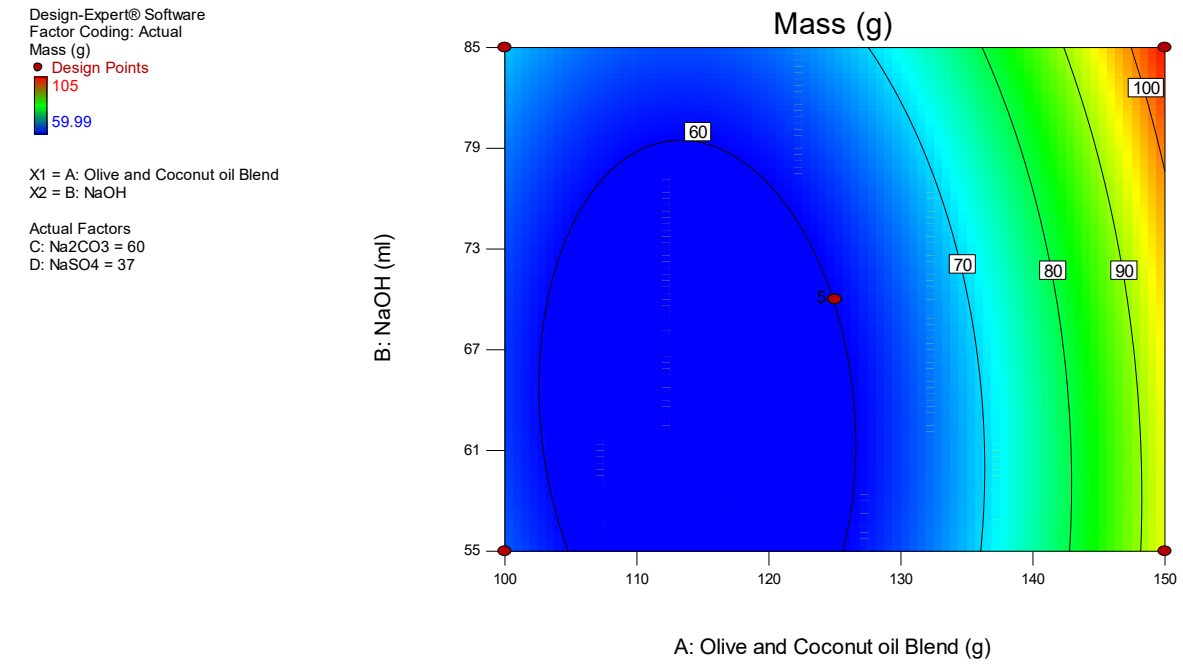


Figure 4.1f: Contour Plot for Olive and Coconut Oil blend

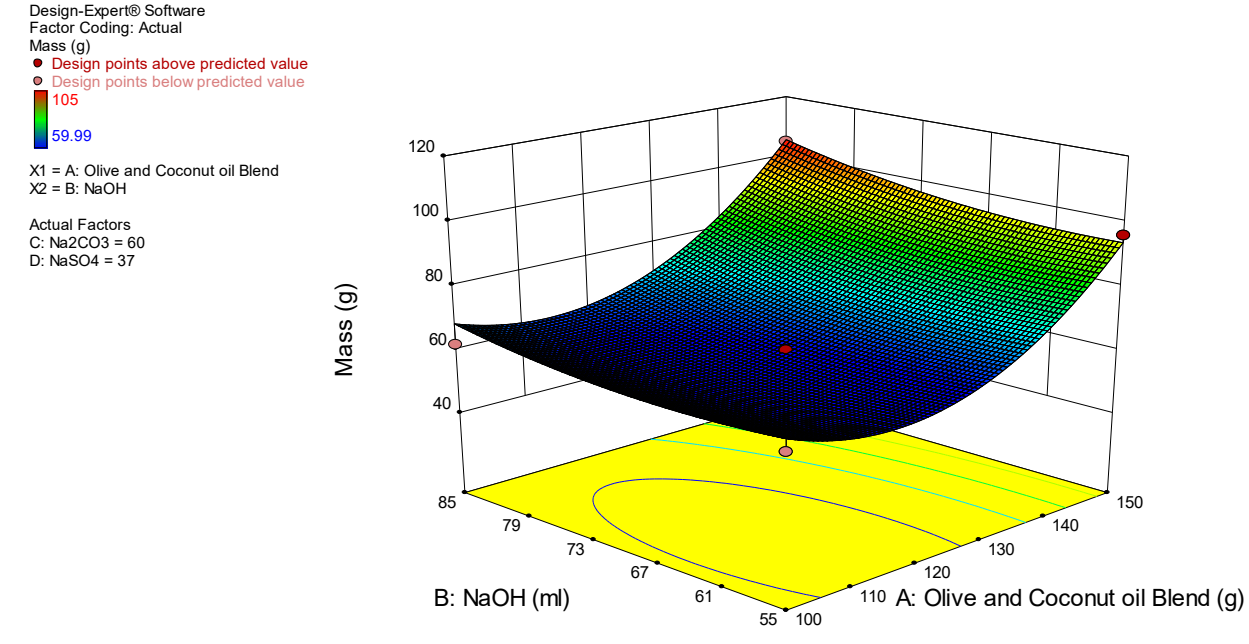


Figure 4.2f: 3D Surface plot for Olive and Coconut Oil blend

4.2 DISCUSSION

This work was aimed at comparative study of the production of soap using different oils and oil blends. The experimental design, randomization, optimization and statistical analysis were carried out using Box Benken Design Expert 10.0. The optimum mass (g) for P.K.O, coconut oil, P.K.O and olive oil, P.K.O and coconut oil blends, olive oil and coconut oil blend, were 179.90 g, 150.22 g, 118.90 g, 130.99 g, and 105.00 g respectively. The coefficient of determination (R^2) for P.K.O, coconut oil, PKO and olive oil, PKO and coconut oil blends, olive oil and coconut oil blend, were 0.8007, 0.6649, 0.9739, 0.9266, and, 0.9131, respectively. The closer the value of R^2 to 1.0, the better the prediction of the response variable from the independent variables. Tables 4.0, 4.2, 4.4, 4.6, and 4.10 showed the experimental results from the design of experiment, Tables 4.1, 4.3, 4.5, 4.7, and 4.11 showed the statistical analysis of the experimental results; these tables include the residual errors, coefficient of determination (R^2), predicted R^2, adjusted R^2, the Fischer value, and the Probability value (P). Figures 4.0a, 4.0b, 4.0c, 4.0d, 4.0f, are the plots of predicted versus actual. These showed the significance of the model; from the plot, the closer the points of the actual response to the predicted (calculated from the model), the more significance the model. Figures 4.1a, 4.1b, 4.1c, 4.1d, and, 4.1f, are the contour plots which were used to explore the potential relationship between these variables. The Contour plots display the relationship in two dimensions, with x- and y-factors (quantity of Oil and quantity of NaOH) plotted on the x- and y-scales and response values (mass of soap produced) represented by contours. The contour plots show how the quantity of NaOH (y) and mass of Oil (or oil blend) (x) affect the mass (contours) of the produced soap. Figures 4.2a, 4.2b, 4.2c, 4.2d, and, 4.2f, are the 3D surface plots. These were used to explore the potential relationship between the variables. 3D surface plots display the relationship in three dimensions, with predictor variables on the x- and y-scales, and the response (z) variable represented by a smooth surface (surface plot). The plots showed how quantity of NaOH (y) and mass of oil (or oil blend) (x) affect the mass (surface) of the produced soap. The coefficient of determination (R^2) also showed how the independent variables had affected the experimental results (response variable). The closer the value of R^2 to 1.0, the better the prediction of the response variable from the independent variables.

CONCLUSION

The optimum mass (g) of P.K.O, coconut oil, P.K.O and olive oil blend, P.K.O and coconut oil blend, and olive and coconut oil blend, were 179.90 g, 150.22 g, 118.90 g, 130.99 g, and, 105.00 g respectively. The yield of soap at optimum values were, PKO alone, 123.38 g, coconut oil alone 12.09 g, blend of olive oil and PKO ,79g, blend of coconut oil and PKO is 88.6 g, blend of olive oil and coconut 59.99 g. These results indicate that PKO is still the best oil for soap production. Other oils could be blended to have a significant yield of soap. For example, using coconut oil alone, the yield is just 12.09 g for an optimum of 150.22 g of oil input whereas when blended with PKO the yield improved to 88.6 g of soap. Such type of oil must be blended to get a good result.

Authors Contributions: Conceptualization, C.Madu, Methodology, C. Madu, Experimentation, C. Madu,, B.S. Adesina, Investigation and analysis, C.Madu, B.S. Adesina, Original drafting, C. Madu, B.S. Adesina, Editing, B.S. Adesina.

Conflict of Interest: The Authors declare no conflict of interest. They have read and agreed to publish the work.

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