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Full Length Research Paper

Process Optimization using Response Surface Methodology (RSM) for the Production of Modified Local Soap using Plantain Peel as a Source of Alkali

Omoruwou, F. and Olayebi, O.*

Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Delta State, Nigeria.
*Corresponding author E-mail: olayebi.oluwafemi@fupre.edu.ng

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ABSTRACT: The importance of locally produced goods to the growth of a national economy cannot be overemphasized; hence, procuring raw materials locally in industrial production is the roadmap to economic progress and development. The purpose of this study was to optimize the process parameters in high-quality local soap production using plantain peel ash as a source of alkali. Palm kernel oil (PKO) was used to make the soap, which had the following physicochemical parameters: saponification value (SV)275 mgKOH/g, acid value 7.02mgKOH/g, free fatty acid 3.51mgKOH/g, iodine value 17.68 I2/100g, peroxide value 7.784 mEq/kg, and specific gravity of 0.8818. The alkali extract from the plantain peel ash was used with the oil for soap production. A modified conventional method was adopted for better soap quality, using a response surface modelling (RSM) tool to obtain optimal production process conditions. The effect of the process variables was also analyzed. The soap produced was also analyzed by testing for foam stability, foamability, total fatty matter, moisture content and hardness of soap. The highest projected percentage soap yield of 84.17 percent was reached during a reaction time of 39.36 minutes with an ash of 17.96 g and a temperature of 55.22^0C. The model had a very high R^2 value of 0.9786, indicating that it can account for around 98 percent of the variability in the percentage soap yield. The expected R^2 of 0.9394 agrees reasonably with the corrected R^2 of 0.9594. It shows that employing a quadratic polynomial as a response surface methodology was effective in modeling the saponification process.

Keywords: Process parameters, plantain peel, palm kernel oil, soap production, saponification

INTRODUCTION

A contemporary conversation subject has been the sustainability (Ranjbari et al., 2021), environmental friendliness and cost-effectiveness of various technologies and their products as they relate to human existence and the environment (Ragheb et al., 2016). Detergents and soap production and their utilization is an example of technological issue that focuses on sustainability and environmental friendliness. The majority of soaps that are now on the shelf are produced through industrial synthesis using ingredients that come in part from fossil fuels. Sustainability is a major problem for their industrial processes since inorganic chemicals used for soap production, which are mostly made from fossil fuels, are not renewable and costly (Speight, 2011). Therefore, doing so breaches both the 2015 Paris Climate Change Agreement and the 2015 United Nations Sustainable Development Agenda (Agreement, 2018; Delbeke et al., 2019; UN, 2020). Hence, attempts are being made to employ feedstocks with an entirely agricultural background to enhance the sustainability and environmental friendliness of soap manufacturing while at the same time a reduced cost of production. Interestingly, some of the

agricultural inputs required to make soap are produced as trash. Most of these are unprocessed and unnecessary, thus they are typically disposed of by burning, dumping, or uncontrolled landfilling. This trash is generated in scattered homes, gardens, and farms, polluting the environment (Sadh et al., 2018). Among the materials utilized are palm bunches, cocoa pods, plantain peels, banana peels, maize cobs, and cassava peels. If not managed and disposed of appropriately, some of these agricultural wastes might be hazardous to both human health and the environment. They are a resource that may be used for industrial production, strengthening environmental conservation and preservation while also boosting the economy by turning waste into wealth (Babayemi and Adewuyi, 2010).

Soap, a well-known cleaning agent, is made up of metallic salts of long-chain monocarboxylic acids and is mostly used as a surfactant in washing, bathing, and cleaning (Paul et al., 2014). But it is also utilized in textile spinning and is a vital component of lubricants (Nwankwojike, 2012). It is any water-soluble salt with eight or more carbon atoms in its paraffinic fatty acid, such as caprylic acid ($C_8H_{16}O_3$), lauric acid ($C_{14}H_{32}O_2$), stearic acid ($C_{18}H_{36}O_2$), and linoleic acid ($C_{18}H_{32}O_2$) usually manufactured in bars, granules, flakes, or liquid form (Aiwize and Achebo, 2012; Warra, 2013; Zauro et al., 2016). The nature and kind of the oil used, the type of alkali, and the ratio of each key raw ingredient in the mixture all affect the quality of the soap as well as the processing. Although making soap from vegetable matter is an age-long traditional skill, the soaps made in the past were caustic to the hands, soft, black, and odorous (Hare, 2002; Waithaka and Muriuk, 2019). These qualities of soap are the aspects that influence consumers' opinions toward purchasing soap (Nalawade and Nale, 2013). Soap's physicochemical properties, such as its appearance, texture, and effect on the skin, hands, and clothes, determine the consumer's propensity to purchase it. Since soaps and detergents are used in conjunction with water, it is expected that they will be flushed down the toilet or poured down the drain as waste water. Without adequate management, they pose environmental dangers (Abulude et al., 2017).

The manipulation of the production process through engineering skills and principles helps in the production of high-quality soap from waste agricultural inputs. For instance, Ogunsuyi; et al., 2012 modified the conventional method of local soap production and compared its output with that of the conventional. Results reveal that the improved soap prepared using purified palm bunch ash-derived alkali was relatively of better quality than its traditional black soap counterpart when using several soap quality indices to evaluate the outputs. However, according to data, little to no attention has been paid to process optimization in the majority of studies on local soap manufacture in Nigeria using components supplied locally (Ogunsuyi and Akinnawo, 2012; Onyegbado et al., 2002; Zauro et al., 2016). Even if waste valorization is a step toward wealth creation, cost reduction may not be accomplished without proper resource management. Therefore, process optimization, which is the process of identifying the optimum or optimal operating conditions for a process to reduce costs and maximize efficiency, leading to a higher product yield while remaining within the bounds of the limitations, is of great importance (Kidane, 2021).

Hence, the focus of this research is to determine the optimal process conditions for making high-quality soap in Nigeria using locally generated agricultural-based waste by deploying the response surface methodology (RSM) statistical tool.

MATERIALS AND METHODS

Precursor collection and preparation

The plantain peel waste and palm kernel oil (PKO) utilized in this experiment were sourced from the Uselu market in Benin City, Edo State, Nigeria. All the reagents and chemicals used were of analytical grade and purchased from Rovet Scientific (Nig) Limited, Wire Road, Benin City, Edo State. Laboratory glassware was properly rinsed before and after use in deionized water and alcohol. For all mixing and rinsing, as well as in aqueous and alcoholic solutions, deionized water was used.

Sample preparation

The collected plantain peels were sorted, with undesirable ones being discarded. The selected peels were then washed to remove any dirt, left in the sun to dry for four days, and then placed in an air-dry oven to complete the drying process and remove any remaining moisture.

Preparation of Plantain peel ash

The dried peels were placed in the Muffle furnace at 700^{o}C for 2 hours for the peels to ash properly and then the ash was allowed to cool and was then crushed using a mortar and pestle. To get homogenous particles the crushed ash was next sieved using a 126 x 10^{-4} micron mesh analytical sieve.

Determination of alkali content in ash

The alkali content of the potash obtained from plantain peel ash was quantified using the titrimetric method (Betsy et al., 2013; Ikezu et al., 2020). A mass of 1.01g of the ash was weighed and dissolved in 100ml of distilled water. The solution was placed on a magnetic stirrer so as for the ash to dissolve properly for 30 minutes. 10ml of the clear portion was pipetted and titrated against 0.1M of HCL using two drops of phenolphthalein indicator. This was done until the pink colour becomes colourless. The alkali

content of the ash was calculated from the titre value obtained, and a percentage of the alkali present in the ash was calculated using the equation;

$$Percentage\ of\ alkali\ in\ the\ ash = \frac{mass\ of\ alkali\ in\ ash}{mass\ of\ ash} \times 100 \quad (1)$$

Determination of pH

The pH of a sample is the measure of the acidity or alkalinity of a sample. It is also a measure of the H+ or OH- of the sample. The pH of the plantain peel ash was determined using the standard method as described by US EPA, (2002). A Celsius scientific pH meter was used to measure the pH. 2g of the homogenized plantain peel ash was placed in a glass container, and 20 mL of deionized water was added, properly mixed, and allowed to stand for one (1) hour. The temperature of the suspended plantain peel ash was then measured, and the temperature on the pH meter dial was adjusted to correspond. The pH meter probe was cleaned and blot dried with deionized water. The pH meter was calibrated (with buffers of 4.00, 7.00 and 10.00) before placing the electrode in the slightly settled sample suspension whose pH was to be measured. Once the meter had stabilized, the sample pH was shown, and the reading was recorded.

Determination potash content

50g of the ash from the combusted Plantain peel was dissolved in 250ml of water and then filtered using a Whatman filter paper. The Resulting potash solution obtained was then heated with a magnetic stirrer at 80^{o}C, and to complete dryness, the residue was placed in an Air dry oven at 100^{o}C to dry to constant weight. The potash content (PCa) (% of ash) is derived as:

$$PC_a = \frac{w_2}{w_1} \times 100 \quad (2)$$

Where w_1 denotes the weight of the ash from combusted plantain peel before dissolve in water, and w_2 denotes the final weight of the residue after drying to constant weight.

Physio-chemical characterization of the plantain peel

Moisture content

The gravimetric method described by Radojevic´ and Bashkin, (2006) was used. 30g of fresh samples of the waste were weighed into a previously weighed moisture can and dried in the oven at 100^{o}C for 3 hours in the first instance. It was cooled in desiccators and weighed. It was then returned to the oven for further drying. The drying sample was weighed at an hourly interval until a constant weight was obtained. The weight of moisture loss was obtained by the difference and expressed as a percentage of the sample analyzed using the formula,

$$\%\ moisture\ content = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (3)$$

where w_1 = weight of empty moisture can; w_2 = weight of can + sample before drying and w_3 = weight of can + sample after drying

Ash content

This was carried out by furnace incineration which is referred to as dry ashing described by Nielsen, (2007); In this method, 5 g of each sample was put in a previously weighed porcelain crucible. The sample in the crucible was burnt in a muffle furnace at 700^{o}C until it became grey (ash) after 3 hours. The furnace was allowed to cool to about 100^{0}C before the sample was carefully removed and placed into the desiccators to cool. When cooled, it was reweighed, and the difference obtained the weight of the ash. This was expressed as a percentage of the weight given by the formula,

$$\%\ Ash = \frac{W_2 - W_1}{W} \times 100 \quad (4)$$

W_1 = Weight of empty crucible
W_2 = Weight of Crucible + ash
W = Weight of sample.

Physiochemical Characterization of oil

Determination of density

The density of the palm kernel oil (PKO) was determined according to the standard method. A density bottle was used in determining the density of the oil. A clean and dry stoppered bottle of 50ml volume (v) was weighed using an electronic scale to be W_1, and then it was filled with the oil and stoppered then it was reweighed as W_2. The weight of the oil was determined by:

Weight of the oil = $W_2 - W_1$

where Density is expressed as:

$$Density = \frac{W_2 - W_1}{v} \quad (5)$$

and,
v = Volume of the density bottle; W_1 = weight of density

bottle; and W_2 = weight of density bottle + oil.

Determination of specific gravity

The specific gravity of the oil was determined using the oil's density and water as a standard solution. Calculating for the specific gravity:

$$S.G = \frac{Density\ \ of\ oil}{Density\ \ of\ water} \quad (6)$$

Determination of Saponification Value

1.02g of the oil sample was weighed into a 250ml glass conical flask and 50ml of 0.5N ethanoic potassium hydroxide (alcoholic KOH) to dissolve the oil. The flask was fitted into a reflux condenser and refluxed using a heating mantle for about 60 minutes with continuous stirring. The solution was detached from the reflux condenser, allowed to cool, and three drops of phenolphthalein indicator were added. The solution was titrated with 0.5M HCL until the pink colouration disappeared (Aiwize and Achebo, 2012). The titre value was recorded, and the same procedure was carried out for blank titration (i.e. without the oil sample). The expression for saponification value (S.V) is given as:

$$S.V = \frac{(B-S0\times 0.02805\times 1000}{W} \quad (7)$$

where:
B = the volume of HCl used for the blank test:
S = the volume of solution used for the oil sample:
W = mass of the sample.

Determination of acid value

The free fatty acid was determined using the American Oil Chemists' Society (AOCS) method (1989) as described by Nielsen, (2007). Exactly 0.05 M KOH solution was prepared by dissolving 2.805g KOH (pellet) with 1000ml distilled water. 1g of the oil was weighed in a clean dried 250ml conical flask, and 10ml of benzene and 10ml of ethanol was added to the oil sample for the complete dissolution of the oil. 2 drops of phenolphthalein as indicator were added and mixed thoroughly. The mixture was then titrated against 0.05M of KOH to a faint coloured pink endpoint. The titre volume of 0.05M KOH was noted. The Acid value (AV) in mgKOH/g was calculated using the following equation:

$$AV = \frac{MW\times N\times V}{W} \quad (8)$$

where:
MW = the molecular weight of potassium hydroxide; N = the normality of Potassium hydroxide (0.05M); V = the volume of potassium hydroxide solution used in titration; and W = the weight of the oil sample.

$$\%\ FFA = \frac{AV}{2} \quad (9)$$

Ester value

The ester values of the oil blends were determined according to Pearson (1973). The ester value was determined as follows:

$$Ester\ Value = SaponificationValue\text{- Acid value} \quad (10)$$

Determination of peroxide value

Determination of peroxide value was accomplished by titration (Zauro et al., 2016). In this method, 2.54g of the oil sample was weighed into a 250ml conical flask. 30ml of acetic acid and chloroform in a ratio of 3:2 was added, then 2ml of potassium iodide (KI) solution was added and stirred to dissolution, then 2.0ml of saturated (10% in water) potassium iodide solution was then added, with constant shaking for about 60 seconds, after which the mixture turned pale yellow. 30ml of distilled water was added and then stirred adequately. 1ml of starch as an indicator was added to the mixture and was then stirred thoroughly until the mixture was purplish-black; it was then titrated against 0.1N sodium thiosulphate with constant shaking until the disappearance of the dark purple iodine colour, then the titre value was recorded. Blank titration was carried out by repeating the same procedure without the oil sample. The peroxide value was thus estimated from the formula:

$$PV = \frac{(S-B)\times N\times 1000}{W} \quad (11)$$

where:
S = Sample titre value.
B = Blank titre value.
N = Normality of sodium thiosulphate.

Determination of iodine value

The iodine value was determined by the titrimetric method (Wij's method) (British Standard 684: Section 2.13:1976) (Abdullah et al., 2013). 2.75g of the oil sample was weighed into a 250 mL conical flask and 10ml of chloroform was added then followed by 30 mL of Hanus iodine solution (i.e. iodine monochloride). The flask was securely closed, and the solution was shaken for 30 minutes in the dark. This was followed by adding 10 mL of

15% potassium iodide solution and shaking, and then 100 mL of distilled water was added. The mixture was then titrated with the iodine solution against 0.1 N sodium thiosulfate until a yellow colour formed. This was followed by adding 2 - 3 drops of starch solution, after which a blue solution formed. The titration continued until the blue colour disappeared while the volume of $Na_2S_2O_3$ at the endpoint was recorded. This process was repeated but without oil added (blank titration) (Food Safety and Standards Authority of India, 2012). The Iodine value (I.V) in I2g/100g was calculated.

$$I.V = \frac{(b-v)\times 0.01269\times c}{m} \quad (12)$$

where
c = Normality of sodium thiosulphate ($Na_2S_2O_3$) used;
b = Vol of $Na_2S_2O_3$ used for the blank;
v = Vol of $Na_2S_2O_3$ used for sample;
m = mass of the sample.

Determination of unsaponified oil

This is used to determine the weight of the substances that are not saponified by alkali hydroxides and are extractable into the ether (Dawodu et al., 2015). 5g of Oil sample was weighed in a round bottom flask; 50ml of alcoholic KOH was poured into the round bottom flask containing the oil sample and was refluxed for about 60 minutes in the reflux condenser. The solution was left to cool for about 30 minutes, after which 60ml of N-hexane, 50ml of ethanol, 50ml of distilled water and 50ml of 0.5M HCL were added before refluxing. The mass of the solution after separation was weighed at 1.84g

Optimization studies of the soap production process

Response surface methodology (RSM) modelling for optimizing the soap production. A three-variable Central Composite Design (CCD) for response surface methodology was used to develop a statistical model for optimizing the production of the local soap. The experimental design, made up of 20 runs, was developed using Design Expert® (Version 11.1.2.0, State-ease, Inc. Minneapolis, USA). The optimization process variables were considered, and their range of values is given in (Table 1).

Table 1: Experimental design using response surface methodology (RSM).

Variables	Unit	Symbol	Variables level	
			Low level	High level
Time	min^{-1}	A	15	60
Mass of Ash	g	B	5	40
Temperature	^oC	C	30	80

The low (-1) and high (+1) levels were used to choose the actual levels of the variables for CCD experiments. The process variables investigated were time (A), the mass of ash (B), and temperature (C), as stated in (Table 1). These three variables were examined at five different levels. CCD (Central - Composite Design) was used to construct twenty (20) experimental runs, as indicated in (Table 2). The experimental data were analyzed according to the response surface regression procedure to fit the second-order polynomial equation at the 5% level of significance. Based on experimental data shown in (Table 1), the regression coefficient was determined to predict the process response as a function of independent variables and their interactions were used to understand the system behaviour. The coded values of the process parameters were determined using the equation:

$$\boldsymbol{x_i} = \frac{\boldsymbol{X_i - X_0}}{\boldsymbol{\Delta X}} \quad (13)$$

Where: x_i= coded value of the ith variable; X_i = uncoded value of the ith variable; X_j = uncoded value of the ith variable at the centre point.

The mathematical correlation between the process variables and the response was determined and fitted to the quadratic polynomial expression in equation 14 below.

$$\%\ Moisture\ content = \frac{w_1}{w_2}\times 100 \quad (14)$$

Where: Y = Response; $x_i\ and\ x_j$ = Independent variable; β_0 = Constant; β_i = Linear term coefficient; β_{ii} = Quadratic term coefficient; β_{ij} = Cross-term coefficient; n = Number of process variables studied and optimized during the study (Borugadda and Goud, 2015). To evaluate the impacts of process factors and their possible interaction effects on the response variable, an analysis of variance (ANOVA) was used. R^2, a regression coefficient, assessed the model's quality and best fit. The fitted quadratic polynomial equation resulting from regression analysis was used to generate the response surface and contour plots by holding one of the independent variables constant and adjusting the other (Rao, 2011).

Experimental procedure

The local soap was made using the modified semi-boiled saponification process reported by Ogunsuyi and Akinnawo (2012). Palm kernel oil (PKO) was placed into a 500mL beaker and heated to 60 °C. Also, alkali was mixed with 100 ml of water in another 500 ml beaker and heated to 60 °C. The heated alkali solution was added to the heated PKO using a mechanical stirrer. The amount of PKO and alkali used in each of the experimental runs were

Table 2: Plan for CCD experimental design in terms of coded variables.

	Coded factors			Actual factors		
Runs	A	B	C	Time (mins)	Mass of ash (w/w %)	Temperature (℃)
1	0	-1.68	0	37.50	5.00	55.00
2	0	0	0	37.50	22.50	55.00
3	1	-1	-1	50.88	12.09	40.13
4	0	0	0	37.50	22.50	55.00
5	-1	1	1	24.12	32.91	69.87
6	1.68	0	0	60.00	22.50	55.00
7	-1	1	-1	24.12	32.91	40.13
8	-1	-1	1	24.12	12.09	69.87
9	1	1	1	50.88	32.91	69.87
10	0	0	0	37.50	22.50	55.00
11	-1.68	0	0	15.00	22.50	55.00
12	0	0	0	37.50	22.50	55.00
13	1	1	-1	50.88	32.91	40.13
14	0	0	-1.68	37.50	22.50	30.00
15	1	-1	1	50.88	12.09	69.87
16	0	0	1.68	37.50	22.50	80.00
17	-1	-1	-1	24.12	12.09	40.13
18	0	0	0	37.50	22.50	55.00
19	0	1.68	0	37.50	40.00	55.00
20	0	0	0	37.50	22.50	55.00

as specified by the experimental design in (Table 2). The heating and stirring were done continually until the liquid thickened. The soap creates a layer on the surface of the beaker, with lye beneath. A separatory funnel and a centrifuge were used to separate the lye (glycerol and borne) solution from the soap.

Physiochemical properties of the soap produced

Moisture content

Moisture content was determined by drying 10g of the sample to a constant weight at 105^{o}C according to AOAC. It was allowed to cool and then reweighed. The % moisture content was calculated from the formula below;

$$\%\ Moisture\ content = \frac{w_1}{w_2} \times 100 \quad (15)$$

Where: w_1 = weight of soap after drying; w_2 = weight of soap before dryings.

Total fatty matter

The total fatty matter (TFM) was determined by the method described by Abba et al. (2021). A 5 g of soap was weighed accurately and transferred into a 250 ml beaker. To completely dissolve the soap, 100 ml of hot water was added and 40 ml of 0.5 N HNO_3 was added to the mixture until the contents were slightly acidic. The mixture was heated over a water bath until fatty acids were floating as a layer above the solution. Then the mixture was cooled in ice water to solidify the fatty acids and separate them. 50 ml of n-Hexane was added to the remaining solution and transferred into a separating funnel. The solution was shaken and allowed to separate into two layers, and the bottom layer drained out. 50 ml of n-hexane was added to the remaining solution in the separating funnel. The fatty acid dissolved n-Hexane and again separated as in the previous case, and it was transferred and collected as fatty matter. The fatty matter was weighed in an evaporating dish and dried to constant weight in an oven. From the difference in weight, the % of the fatty matter was calculated in the given soap sample as follows:

$$\%TFM = \frac{B-A}{C} \times 100 \quad (16)$$

where: A = weight of the evaporating dish, g; B = weight of the evaporating dish + soap after drying, g; C = weight of the initial sample of soap, g.

Foam stability test

The foam stability test was done following the method described by Choudhury (2013). About 2.0 g of the soap sample was dissolved in a measuring cylinder containing 10ml of distilled water. The mixture was shaken vigorously to generate foam. Thereafter, the cylinder was allowed to stand for some time. The time taken for the foam/lather to completely disperse/disappear was recorded. The experiment was repeated, and the average time taken for the foam/lather to completely disperse/disappear was

recorded as a measure of the foam stability.

Foamability test

A method described by Belhaij and Al-Mahdy (2015) was modified and adopted to test soap foamability. About 2.0 g of the soap sample was dissolved in a measuring cylinder containing 10ml of distilled water. The mixture was shaken vigorously to generate foam. After being shaken, the cylinder was allowed to stand for some time. The height of the foam in the solution was measured and recorded. The procedure was repeated a second time, and the average height was calculated and recorded as the foamability of the soap sample.

Hardness of soap

The test method for the hardness of soap adopted was the hand-felt method, and the texture of soap was observed to be “Soft Soap”.

RESULTS AND DISCUSSION

Table 3 and 4 show the Moisture content, Dry matter content and Ash content of the precursor (plantain peel). The moisture content of the plantain peel was 85.13 percent, which is consistent with previous research (Ogidi et al., 2018). This could be because of water activities in the area where the vegetable waste was collected (Isengard, 2001). This sample's ash concentration was around 1.48 percent, which is consistent with and close to Okareh et al. (2015) estimate of 1.40 percent.

Table 3: Percentage values of plantain peel

Parameters	Plantain peels (%)
Moisture Content	85.13%
Dry Matter Content	14.87%
Ash Content	1.48%

Table 4: Plantain peel parameters.

Parameters	Plantain peel
pH of the Ash (%)	12.06
Potash content (%)	76.30
Alkali content g/dm^3	64.75

The pH obtained from a peel ash solution was 12.06, which fits into the range of pH from literature which was found to be in the range between 12.05 and 12.88. Data from this study shows the potash content of the ash from the peel to be 76.30% which is in agreement with the range from 69 to 81.9%, as reported by Babayemi et al. (2010). Potash yield depends on the nature of the soil in which the plant grows, and the efficiency of the extraction method (Babayemi et al. (2010). Fig 1 shows the FTIR of the Ash. The absorption shown in (Table 5) is the data obtained from the FTIR spectrum with wave numbers from 2985.59 cm-1 to 704.46 cm-1 and intensity ranging from 94.23 to 83.92. The IR spectrum shows a weak peak stretch at the double chain region with a wave number of 1647.5cm-^{1}, which fall between the range from 1680cm^{-1} to 1600cm^{-1}, which depicts the C=C alkenes functional group and a wave number of 1446.2cm^{-1}, which lies in the range of C=C aromatic functional group with a frequency range of 1600cm-1-1400cm-1. The IR shows a slightly broad weak peak of 1364.2 cm-1 which shows CH bending vibrations (CH_3 bend vibration) whose frequency range is 1395cm-1 – 1355cm-1. The weak peak having a frequency of 1039.9cm^{-1} in (C-O-C Acetate with a frequency range of 1040cm-1 – 1010cm^{-1}) is the frequency of esters with a range of 1300cm^{-1} – 1000cm$^{-.1}$. In the fingerprint region, IR shows a weak peak with a frequency of 704.5cm-1, which shows the –CH=CH_2 (cis) group with a range of 750cm^{-1}-650cm^{-1} (Nandiyanto et al., 2019).

Table 5: FTIR values.

Peak Number	Wave number (cm^{-1})	Intensity
1	704.46685	83.92780
2	816.28699	87.44572
3	879.65173	85.68457
4	1039.92726	86.09961
5	1364.20565	80.61109
6	1446.20708	83.75350
7	1647.48332	95.53321
8	2985.59761	94.23605

Physiochemical properties of the oil

Table 6 shows the density, specific gravity, saponification value, acid value, ester value, free fatty acid, iodine value, and peroxide value of the Palm kernel oil. The statistics in Table 6 demonstrate that the oil has a density of 0.8796kg/m^3, which is within the density range of Palm kernel oil in the literature (Amira et al., 2014; Berger, 2003; Musa, 2009). The specific gravity of the oil determined by the analysis was 0.8818, which is close to the values reported by Amira et al. (2014) and Egbuna et al. (2021), 0.904 and 0.910, respectively. The extent of adulteration can be evaluated, and this information can be utilized to accept oils as raw materials and to size pumps and pipelines in plant installations. According to research, density and specific gravity vary based on the temperature of the near surroundings. The statistics also indicate that the saponification value is 275 mg KOH/g, which is slightly higher than the findings of a study by Bart et al. (2010)

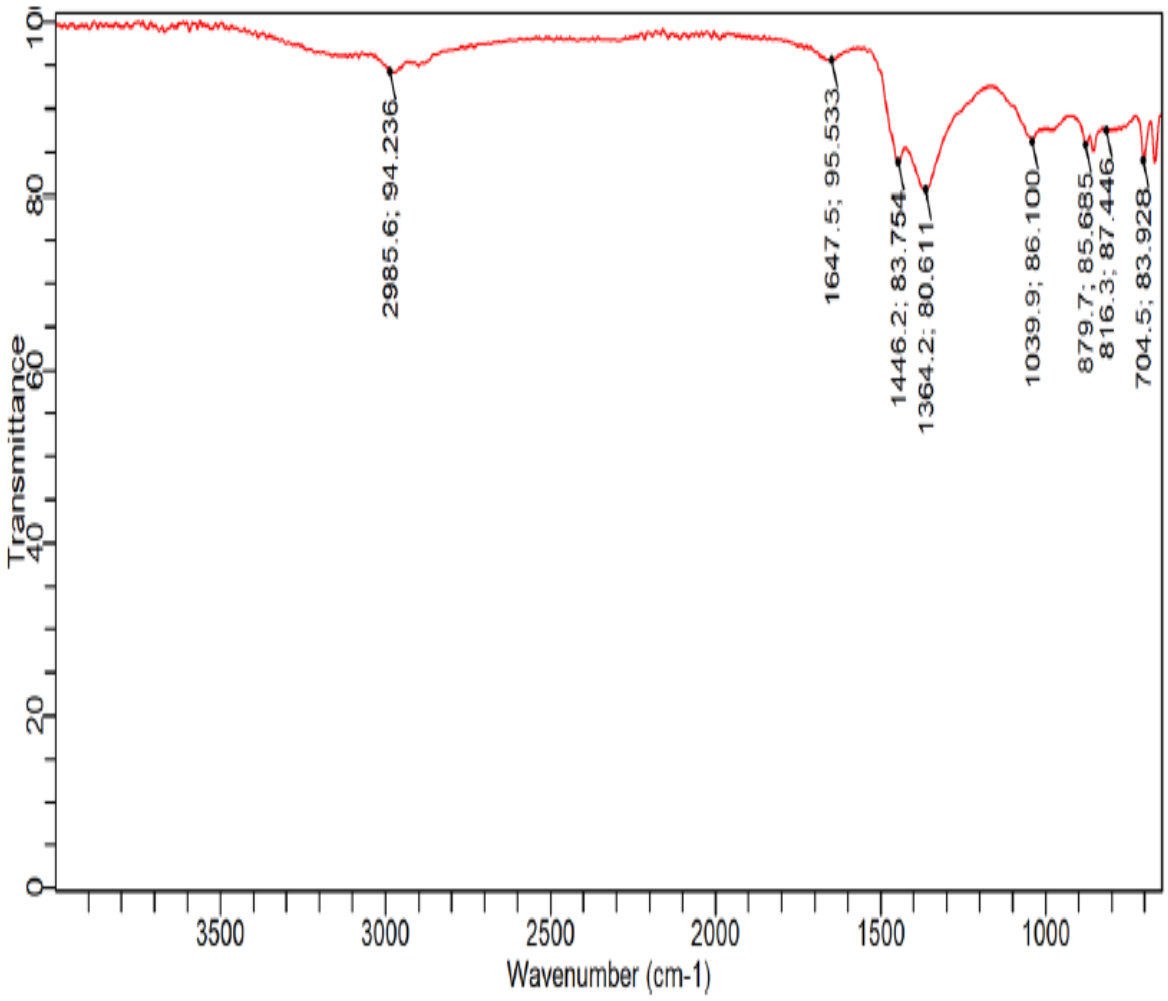


Figure 1: The FTIR of the Ash

Table 6: Palm kernel oil characterization data.

Properties	Values (Palm kernel oil)
Density(kg/m^3)	0.8796
Specific gravity	0.8818
Saponification value (mgKOH/g)	275
Acid value(mgKOH/g)	7.02
Ester Value (mgKOH/g)	267
Free Fatty Acid (mgKOH/g)	3.51
Iodine Value(I_2/100g)	17.68
Peroxide Value (*mEq/kg*)	7.874

which showed a range of 240-257 mg KOH/g and that of the Codex Committee on Fats and Oils (1999) with a range of 230 to 254 mg KOH/g. It is significant to remember that the length of the fatty chain increases as the saponification value decreases. Additionally, it can help in the detection of adulteration, which frequently results in a reduction in the fat's saponification value because, practically speaking, the higher the fat's saponification value, the more suited it is for the manufacture of soap. The suitability for soap production arises from the correlation between a high saponification number and high foamability The high saponification value of 275 mg KOH/g obtained for the

Table 7: Experimental design matrix with actual and predicted percentage soap yields.

	Coded factors			Actual factors			Soap yield (%)	
Runs	A	B	C	Time (mins)	Mass of ash (W/w %)	Temperature (℃)	Actual	Predicted
1	0	-1.68	0	37.5	5	55	68.71	68.67
2	0	0	0	37.5	22.5	55	83.68	82.14
3	1	-1	-1	50.88	12.09	40.13	65.95	65.56
4	0	0	0	37.5	22.5	55	77.99	82.14
5	-1	1	1	24.12	32.91	69.87	39.71	40.90
6	1.68	0	0	60	22.5	55	61.53	60.01
7	-1	1	-1	24.12	32.91	40.13	48.12	47.38
8	-1	-1	1	24.12	12.09	69.87	65.00	65.47
9	1	1	1	50.88	32.91	69.87	40.85	42.57
10	0	0	0	37.5	22.5	55	79.91	82.14
11	-1.68	0	0	15	22.5	55	50.75	51.13
12	0	0	0	37.5	22.5	55	81.23	82.14
13	1	1	-1	50.88	32.91	40.13	55.83	56.16
14	0	0	-1.68	37.5	22.5	30	60.99	62.40
15	1	-1	1	50.88	12.09	69.87	65.71	67.25
16	0	0	1.68	37.5	22.5	80	60.92	58.38
17	-1	-1	-1	24.12	12.09	40.13	57.58	56.67
18	0	0	0	37.5	22.5	55	81.06	82.14
19	0	1.68	0	37.5	40	55	41.19	40.10
20	0	0	0	37.5	22.5	55	88.78	82.14

PKO oil indicates that the resulting soap will have a significant potential for foam formation. The acid value (AV) of an oil is a measurement of the quality of its fatty acids. A low acid value in oil implies that the oil will be stable over time, protecting it against rancidity and making it suitable for soap manufacturing. The acid value of 7.02 mgKOH/g and a free fatty acid value of 3.51 mgKOH/g correlate with the FAO standard of 30 mgKOH/g. The considerably high acid value shows that the fat was stored improperly (Mishra, 2013). The ester value is calculated by subtracting the saponification and acid values. The degree of unsaturation in oil is measured by the iodine value. The iodine value found in this study is 17.68 I2/100g, which is substantially within the range of 14 to 21 recorded by Gunstone et al. (2007).

Optimization studies of the Saponification process

In this study, a quantitative technique was adopted, and the collected data were analyzed using the response surface methodology. The approach was used to identify the best data point. From (Table 7), it can be observed that the actual (experimental) values of soap yield ranged from 39.71 to 88.78% from the soap preparation process. The highest yield of 88.78% was obtained after a reaction time of 37.5 min, using the ash of 22.5 g at a temperature of 55^0C while the least yield of soap of 39.71% was achieved after a reaction time of 24.12 minutes using the ash of 32.91 g at a temperature of 69.87^0C. This indicates that soap yield was favoured by an increase in reaction time and a decrease in mass of ash and reaction temperature. As optimal soap production is accompanied by a reduction in the amount of ash required and the reaction temperature, it follows that the cost of production will be reduced, as the decrease in these two factors indicates a reduction in feedstock and energy consumption, respectively.

From Table 8, the F-value of the model was found to be 50.87 and with a very low p-value (< 0.0001). These values imply that the model is highly significant with respect to the percentage yield of soap from the PPA saponification of PKO. Among the main factors, reaction time (A) and mass of ash (B) with p-values of 0.0090 and <0.0001 respectively which are (< 0.05) have a significant effect on the percentage soap yield, while the effect of the temperature (C) of the saponification reaction was found to be insignificant with a p-value of 0.1745 (> 0.05). The effects of interaction factors (AB and AC) with p-values > 0.05 were found to be insignificant on the percentage soap yield, while the effect of interaction (BC) with a p-value of 0.005 (<0.05) was significant on the percentage soap yield. All quadratic factors (A^2, B^2 and C^2) were found to have very high significant effects (p-values < 0.0001) on the percentage soap yield of the saponification process. The lack of fit with a p-value of 0.8979 was insignificant, which is desirable, i.e. the model fits the soap production process using PPA and PKO.

Model summary statistics

The model summary statistics of the percentage soap yield

Table 8: ANOVA for the quadratic model of soap yield.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	4182.23	9	464.69	50.87	< 0.0001*
A - Time	95.21	1	95.21	10.42	0.0090*
B - Mass of ash	985.51	1	985.51	107.88	< 0.0001*
C - Temperature	19.52	1	19.52	2.14	0.1745
AB	0.0066	1	0.0066	0.0007	0.9791
AC	25.31	1	25.31	2.77	0.1270
BC	116.82	1	116.82	12.79	0.0050*
A^2	1271.42	1	1271.42	139.17	< 0.0001*
B^2	1387.88	1	1387.88	151.92	< 0.0001*
C^2	852.32	1	852.32	93.30	< 0.0001*
Residual	91.36	10	9.14		
Lack of Fit	20.71	5	4.14	0.2932	0.8979
Pure Error	70.65	5	14.13		
Cor. Total	4273.58	19			

*=significant

Table 9: Model summary statistics.

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	14.08	0.2575	0.1182	-0.0132	4330.07	
2FI	15.27	0.2907	-0.0367	-0.8638	7965.29	
Quadratic	**3.02**	**0.9786**	**0.9594**	**0.9394**	**259.05**	**Suggested**
Cubic	3.51	0.9827	0.9453	0.8106	809.38	Aliased

from PKO saponification is given in (Table 9). The coefficient of regression, R^2, was used to validate the fitness of the model equation. The R^2 value measures how the experimental factors and their interactions could explain variability in the observed response values. A higher R^2 value close to unity gives a better model. From Table 9, the cubic source of variability gave a higher R^2 value (0.9827). However, the cubic model is usually correlated from response surface methodology, making the quadratic source of variability with a higher R^2 value (0.9786) the best model to evaluate the fitness of the saponification process than the two-factor interaction (2FI) and linear sources of variation with lesser R^2 values.

This was further expressed by the high and close values of the adjusted and predicted R^2 values of 0.9594 and 0.9394 respectively (difference lesser than 0.2) in the model summary statistics of (Table 10 and Figure 2) show the predicted versus actual plot of the saponification process.

Fig 2: Predicted vs. actual plot of soap production from PKO

From the plot, it can be observed that a linear and close correlation exists between the actual (experimental) and predicted values of the soap production process. This indicated that there is reasonable agreement between the predicted and actual values of the soap yields of the saponification process and showed that response surface methodology is efficient in analyzing the saponification reaction of PKO with PPA using a quadratic polynomial.

A very high R^2 value of 0.9786 was observed, which indicates that the model can account for about 98% of the variability in the percentage soap yield. The predicted R^2 of 0.9394 is in reasonable agreement with the adjusted R^2 of 0.9594; i.e. the difference is less than 0.2. This indicates that the response surface methodology was efficient in modelling the saponification process using a quadratic polynomial.

CV is a measure of expressing standard deviation as a percentage of the mean. Smaller values of CV give better reproducibility. In general, a high CV indicates that variation in the mean value is high and does not satisfactorily develop an adequate response model. The CV of 4.74% is within the acceptable range. Adequate precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. The ratio of 19.6719 indicates an adequate signal. This model can therefore be used to navigate the design space.

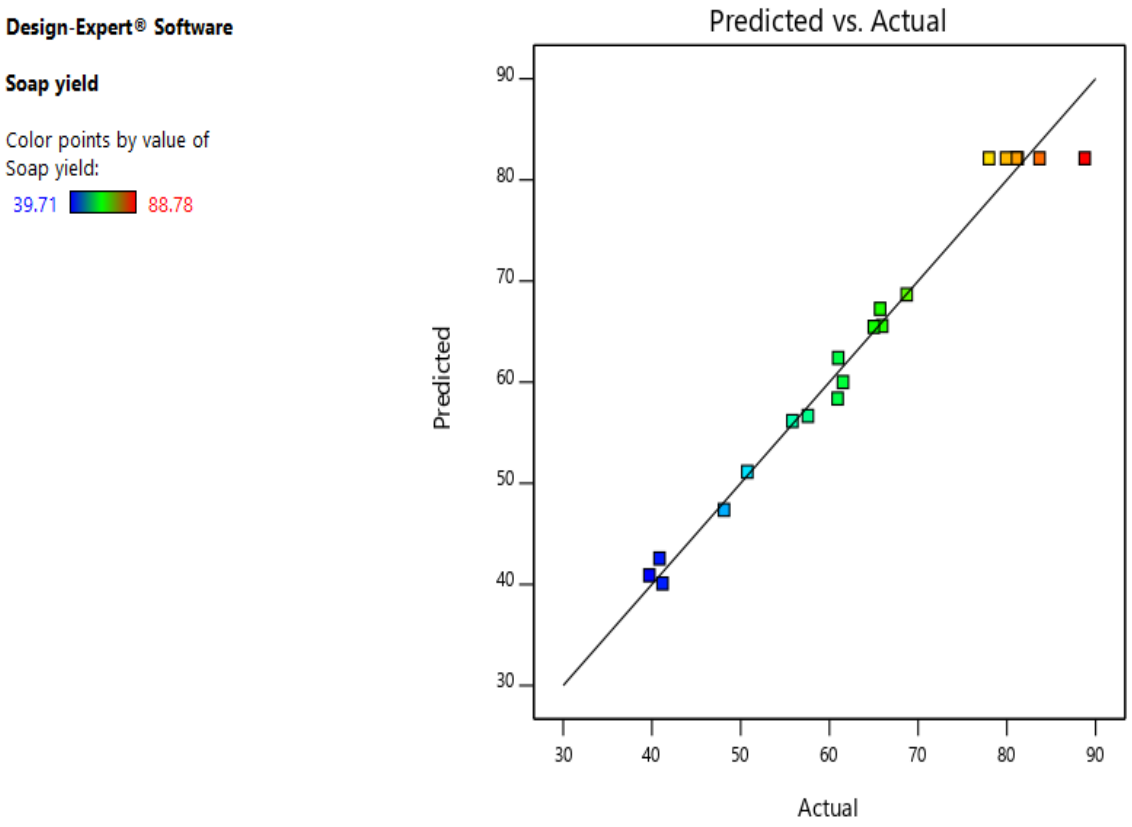


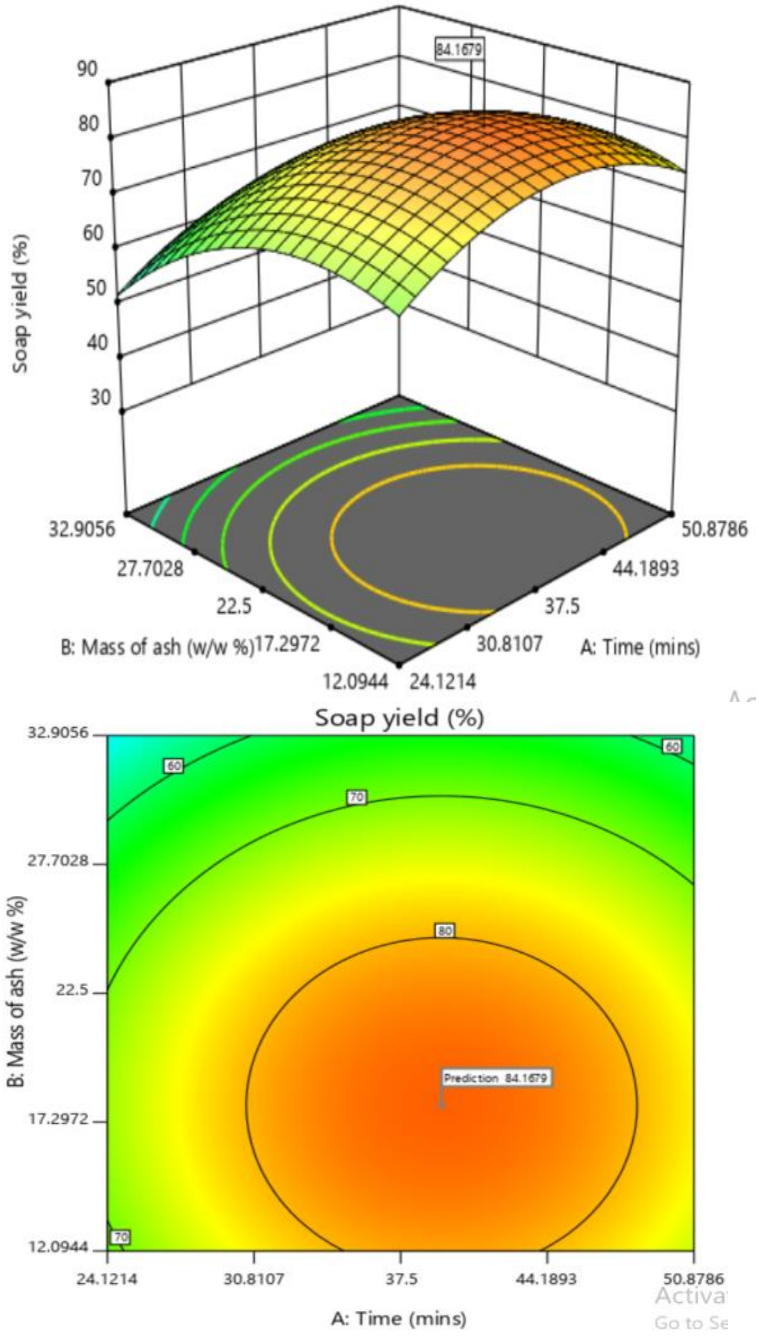
Figure 2: Predicted vs. actual plot of soap production from PKO

Table 10: Fit statistics of regression model.

Parameter	Value
R^2	0.9786
Adjusted R^2	0.9594
Predicted R^2	0.9394
Mean	75.71
Standard Deviation	3.02
CV%	4.74
Adequate Precision	19.6719

Effect of the interaction of variables on percentage soap yield

The effect of the saponification process factors considered on the percentage yield of soap from PKO and PPA reaction is shown by the response surface and contour plots in Figures 8 (a) to (c). Figures 3- 8: Response surface and contour plots showing the effect of the interaction of (a) reaction time and mass of ash (b) reaction time and temperature, and (c) mass of ash and temperature on the percentage soap yield. The effect of the interaction of reaction time and mass of ash (AB) on the percentage soap yield is shown in Figure 8 (a). It can be observed from the plot that the concurrent increase in the reaction time and mass of ash lead to a decrease in the percentage soap yield at a constant temperature. As seen from the plot, the ash mass had a more dominant effect than the reaction time on the percentage soap yield. The interaction of reaction time and temperature (AC) on the percentage soap yield is shown in Figure 8 (b) at the constant mass of ash. It can be observed from the plot that the concurrent increase in the reaction time and temperature lead to a decrease in the percentage soap yield. Also, from the plot, it can be seen that the effect of reaction time was more dominant on the percentage soap yield than the effect of temperature. The effect of the interaction of mass of ash and temperature (BC) on the percentage soap yield is shown in Figure 8 (c) at a constant reaction time. It can be observed from the plot that the concurrent increase in the mass of ash and temperature lead to a decrease in the



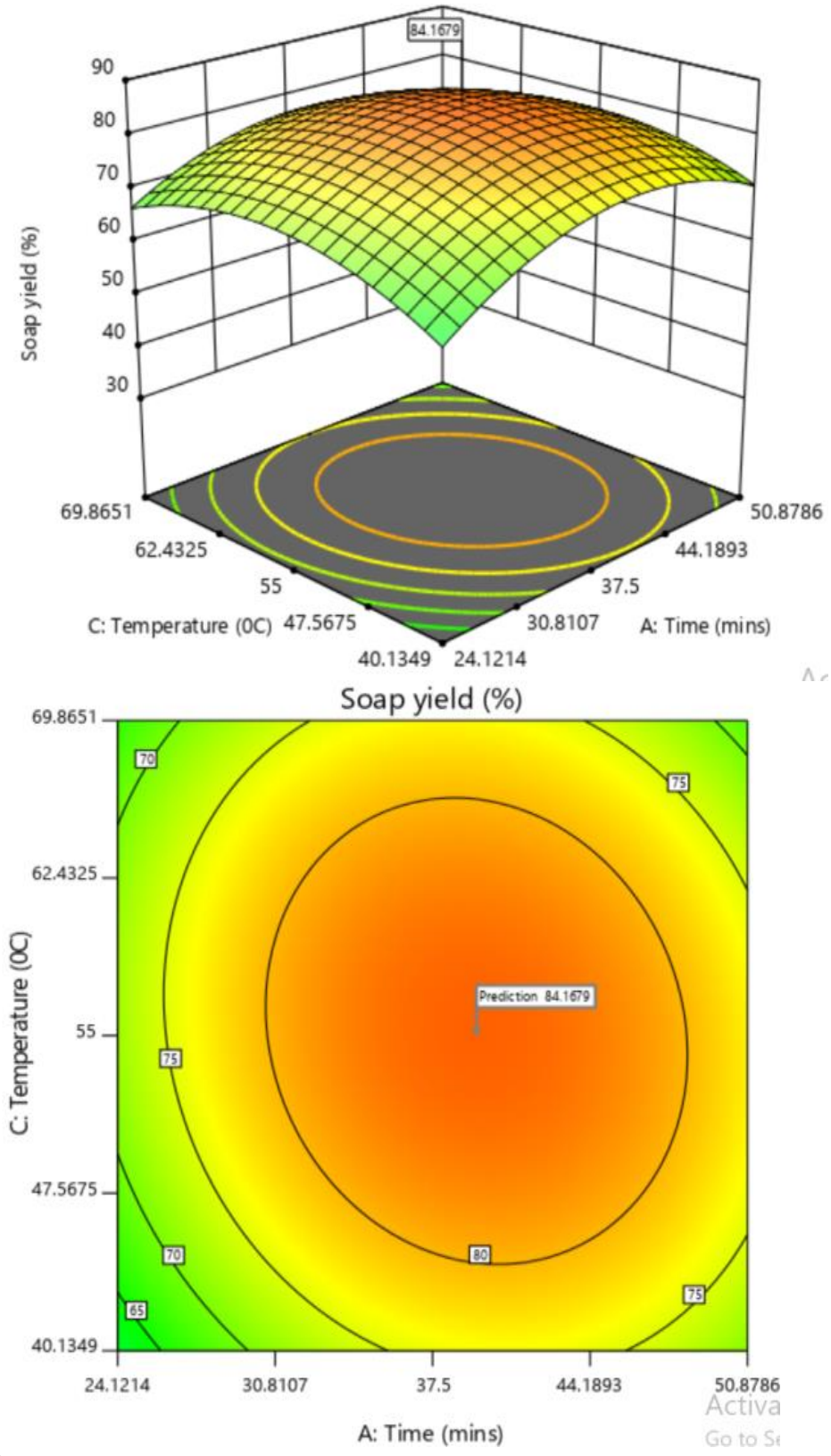
(a)

Figure 8a: Response surface and contour plots showing the effect of the interaction of (a) reaction time and mss of ash

percentage soap yield. The plot shows that ash mass had a more dominant effect on the percentage soap yield tham temperature.

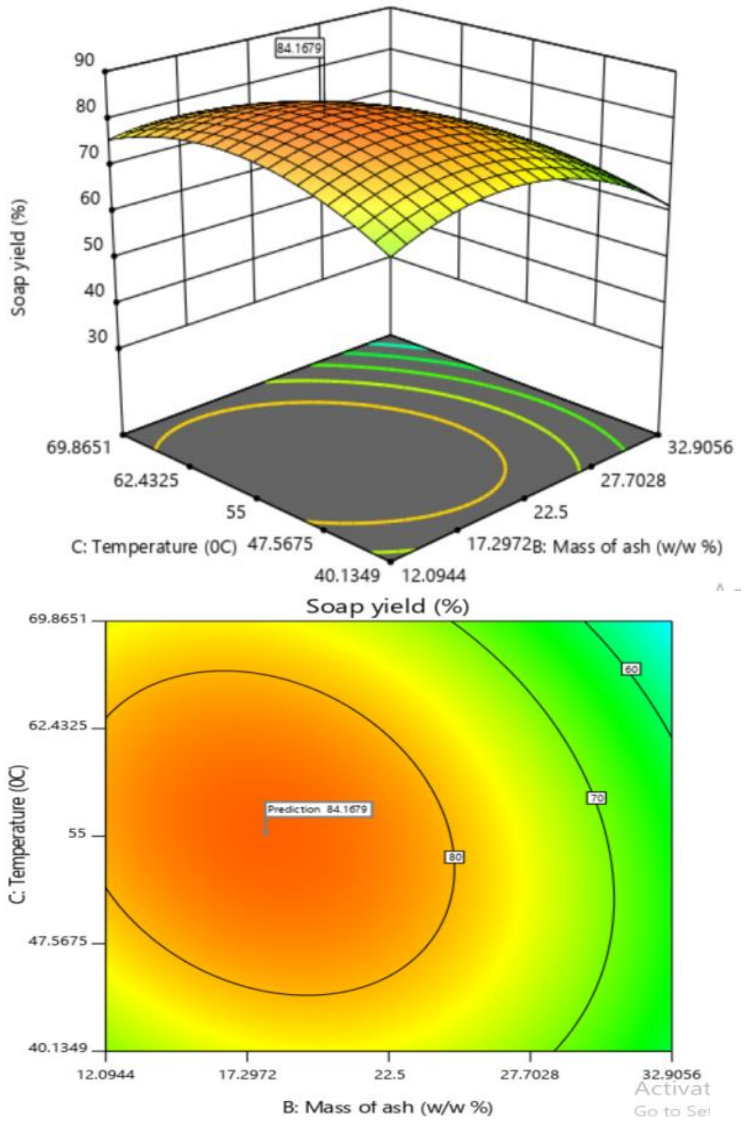
Optimization

From the numerical optimization studies of the saponification process, the achieved maximum desirability of 0.906 from the optimization of soap yield indicates that it is possible to reach about 91% of maximum soap yield. The maximum percentage soap yield of 84.17% predicted was obtained after a reaction time of 39.36 min using the ash of 17.96 g and a temperature of 55.22^{0}C. The desirability ramp shows the optimization condition of the



(b)

Figure 8b: Response surface and contour plots showing the effect of the interaction of (b) reaction time and temperature,



(c)

Figure 8c: Response surface and contour plots showing the effect of the interaction of (mass of ash and temperature on the percentage soap yield.

process in (Figure 8).

Physiochemical properties of the soap

Table 11 shows the physicochemical properties of the soap.

Table 11: Properties of Soap Produced.

Properties of Soap	Palm Kernel oil
Foam Stability (min $^{-1}$)	7.85
Foamabiltiy (mm)	45
Moisture Content (%)	10.7
Total Fatty Matter (g)	3.5
Hardness of Soap	Soft

Foam-stability

The foam stability was determined by measuring the time for the lather formed by the soap with pure water to collapse—the result obtained from the analysis of the soap samples for foam stability was 6 min 40 seconds.

Foamability

Foamability is a test of satisfiable foam ability determined by measuring the height of the lather formed in the soap. The result obtained from this test showed the height of the foam to be 4.5cm (19ml) in a measuring cylinder of 50ml(12.5cm). This value correlates with the findings of

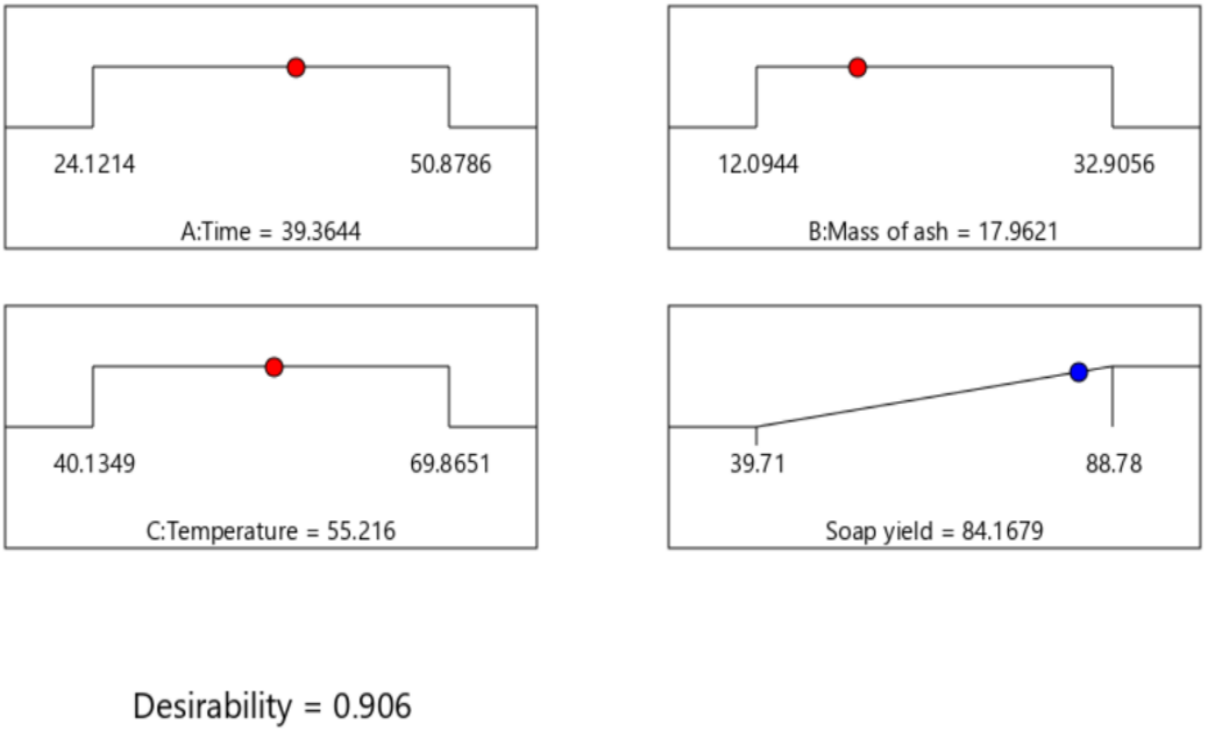


Figure 9: Desirability ramp of the optimization process.

Ogunsuyi and Akinnawo (2012) with a 15 ml foamability.

Moisture content

This is the level of water in the soap. The moisture content obtained from the soap sample was 10.7%. This result is consistent with Vivian et al. (2014) research on some commercial soap in Kenya, where the moisture content ranges from 10.91% to 22.69%. However, a substantial difference was noticed between the moisture content of our research and that of Ogunsuyi and Akinnawo (2012), who operated on identical feedstock with a 29.05% moisture level. This large variance in the measured moisture content can be attributable to variations in process conditions for both works.

Total fatty matter (TFM)

The result obtained from the analysis of the soap samples for TFM showed 70.6% for the soap sample. Total Fatty Matter (TFM) is one of the most essential criteria that define the quality of soap. The minimal TFM for commercial, high-quality, moisture-finished soap is 78 percent, but soap with 50 percent TFM is typically dry 1. powder, harsher, and irritating on the skin (Hare, 2002; Kuntom, 1998). Therefore, the 70.6% TFM obtained in this study indicates that it is a soft, mild soap that may be used on the skin without causing any problems.

Hardness of soap

From the texture of soap obtained by the hand felt method it was concluded that this was soft soap. This is consistent with the TFM finding, which indicates that the soap is soft. Additionally, it corresponds to the iodine value of the PKO. The higher the iodine value of the oil used the softer the resulting soap (Christopher, 2015).

Conclusion

In this study, soap production using alkali derived from agricultural waste ash (plantain peel) and Palm kernel oil was carried out. Both high saponification number and iodine number indicate this oil to be highly suitable for soap making. The acid value was found to be acceptable in line with results from the literature. The unsaponifiable matter was also within acceptable limits, and the oil can be used without being refined. From the optimization studies, it can be established that;

Reaction time and mass of ash have significant effects on the percentage soap yield from the saponification reaction, while the effect of the temperature on the saponification reaction was insignificant.

2. The maximum percentage soap yield of 88.78% was obtained after a reaction time of 37.5 min using the ash of 22.5g and a temperature of 55^0C. The relevance of this conclusion lies in the fact that the knowledge is applicable to the design of the soap production reactor especially, when it is required to scale up from laboratory to industrial level.
3. A very high R^2 value of 0.9786 was observed, indicating that the model can account for about 98% of the variability in the percentage soap yield. The predicted R^2 of 0.9394 is in reasonable agreement with the adjusted R^2 of 0.9594. It indicates that the response surface methodology was efficient in modelling the saponification process using a quadratic polynomial.
4. The heavy dependence on synthetic chemicals for soap production would drastically be reduced if a concerted effort is made to improve this local source of raw material for soap making. The use of raw materials from agricultural waste, such as plantain peel will serve as a better alternative source to caustic soda for soap production because of its environmental friendliness. It will also reduce the heavy dependence on synthetic chemicals usually imported for soap production.

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