



## Application of Response Surface Methodology in Palm Kernel Seed Oil Extraction Using n-Hexane

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### ABSTRACT

In this work the extraction of palm kernel seed oil using n-Hexane was studied through the application of Response Surface Methodology (RSM). Two factors, time and solute-solvent ratio were considered, while the response was the percentage of yield achieved. Central Composite Design (CCD) in the Design Experiment v11 package was employed which gave 13 experiments that lead to linear model relating the response and the variables. Analysis of variance (ANOVA) indicated that the model was significant as indicated in the model F-value of 7.30 or P-value = 0.0124., while B was also a significant model parameter with P-value = 0.0039

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### INTRODUCTION

In solvent extraction, oil in material is leached with a suitable solvent under suitable conditions. In the process, oil (the soluble material) is dissolved by the solvent while the (the insoluble material) is retained unaffected by the solvent.

The extent of extraction is affected by the following factors; the kind of solvent, the temperature of solvent, the ratio of solvent to the solute, the number of extraction stages, the shape of the particles, the porosity of the material, and the contact time (Aletor, 1990). The most common solvent used is n-hexane, because of its low price, low toxicity, suitable boiling point for recovery and handling, and its availability. Solubility of oil in hexane increases with temperature. The extraction of oil of either vegetable or animal origins fine pharmaceuticals from plants materials, and perfumes from flowers are process of modern development (ASA, 2003).

As a result of physicochemical research, great achievements were made by developing useful products from an abundant and locally available raw material. Unlimited quantities of raw materials were manufactured for the

pharmaceuticals, foods and other allied industries, allowing for making products available to the various needs of the people and world over. Also, as a result of current advances due to the most recent research in the extraction methods and to the newest analytical techniques and procedures such as column chromatography, mass spectrometer, paper and thin layer chromatography, infra-red spectrometry and nuclear magnetic resonance spectroscopy. The precise composition of the naturally occurring seeds can be known. Kernel oil has both food and industrial uses. It is processed into cooking oil and edible oil. Edible oil is used as "butter fat" substitute in the manufacture of "filled milk". Hydrogenated fat in the manufacture of "vegetable lard" and baker's margarine (Aletor, 1999).

In industry, kernel oil is used in the manufacture of pure kernel oil soap. It is largely used in the manufacture of toilet soaps. In its modified or original form, it is used for painting and varnish in industry. Kernel oil is also processed into a methyl esters, fatty acids, and fatty alcohols. Its intermediate products are the raw materials in

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the manufacture of detergents, plasticizers surfactants, emulsifiers and other organic chemical products. Kernel cake or meal is used as an ingredient in the blending of animal feeds (Knothe, 1997).

The aim of this research project is to carry out solvent extraction of palm kernel oil via n-hexane using a response surface methodology.

## MATERIAL AND METHOD

### Materials and Equipment

Reagent used include n-hexane absolute, Phenolphthalein indicator, 0.14M NaOH solution, 0.1N KOH, 0.5M ethanol potassium hydroxide solution, 0.5M HCl solution, 30ml Hanus solution and 0.1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution.

Equipment used include 250ml soxhlet extractor, Chiller, Heating mantle, Stop watch, Analytical balance, Beaker 100ml, 250ml volumetric flask, Palm kernel flour, 250ml conical flask, Starch indicator, Cylindrical test tube, Thermometer and Gas chromatography mass spectrometry (GC-MS) machine (GC-MS-QP2010shimadzu Japan).

### Palm Kernel Oil Extraction

The N-hexane was measured by the use of a measuring cylinder (250ml) and poured into the round bottom flask, while the palm kernel flour was weighed using an analytical balance, the sample was placed inside the thimble and placed in the soxhlet extractor. The chiller the source of heat was filled with cold water and the condenser was connected to it. The water bath was also filled with cold water and set on while the parts of the soxhlet extractor was connected i.e. the round bottom flask, the soxhlet extractor and the condenser and placed inside the water bath

The stop watch was used to monitor the time required for the first run of extraction of palm kernel oil. After which all the other runs were performed in the procedure as the first at different time intervals. The result is as shown in Table 3.1.

## PHYSIOCHEMICAL ANALYSIS OF THE PALM KERNEL OIL EXTRACTED

The following physiochemical analyses of the palm kernel oil were carried out in this order;

### Free Fatty Acid (FFA) Test

The method used for the determination was that of British standards institute no. 684. 1g of the palm kernel oil was placed in a 250ml conical flask and warmed. 25ml of methanol was added with thorough stirring, followed by 2 drops of phenolphthalein indicator and a drop of 0.14M NaOH solution. The contents were then titrated with 0.14M NaOH solution until a light pink color which persisted for one minute was seen. The end point was recorded and used to calculate the FFA

$$\text{FFA (as oleic)} = \frac{\text{Titre} \times M \times 28.2}{\text{weight of sample}} \dots 2.1$$

Where M= Molarity of base

The calculation is done in appendix A and the result is as showed in table 3.2

### Acid Value (AV)

The method used for the determination was that of British Standards Institute No. 684. 1g of the palm kernel oil was placed in a 250ml conical flask and warmed. 25ml of methanol was added with thorough stirring, followed by 2 drops of phenolphthalein indicator and a drop of 0.14M NaOH solution. The contents were then titrated with 0.1N KOH solution until a light pink color which persisted for one minute was seen. The end point was recorded and used to calculate the

A.V as thus;

$$\text{Acid Value} = \% \text{FFA (as oleic)} \times 1.99 \dots 2.2$$

The calculation is done in appendix A and the result is showed in table 3.2

### Saponification Value (SV)

The method used was that of the British Standards Institute 1995. The palm kernel oil (2g) was placed in a 250ml conical flask and 25ml of 0.5M ethanol potassium hydroxide solution was added. A reflux condenser was attached and the flask content refluxed for 30 minutes on a water bath while swirling until it simmered. The mixture was then titrated against 0.5M HCl using phenolphthalein indicator while still hot. A blank determination was also carried under the same conditions and the saponification value calculated as thus;

$$S.V = \frac{(B-S) \times 28.05}{W} \dots 2.3$$

Where B= titre value of blank, S= titre value of sample and W= weight of palm kernel oil

The calculation is done in appendix A and the result is showed in table 3.2

#### **Iodine Value (IV)**

The method used was that of Hanus method (Association of Analytical Chemist. 1975). 1g of the palm kernel oil was placed in a 250ml conical flask followed by 30ml Hanus solution and the flask stopper. The contents were mixed and placed in the drawer for exactly 30 minutes. It was then titrated against 0.1N  $\text{Na}_2\text{S}_2\text{O}_3$  until the solution became light yellow. 2ml of 1% starch indicator was added and the titration continued until the blue color just disappeared. A blank determination was also carried out under the same conditions and the I.V calculated as thus;

$$IV = \frac{(B-S) \times 12.69 \times N}{W} \dots 2.4$$

Where;

B= blank titre

S= sample of titre

N= normality of  $\text{Na}_2\text{S}_2\text{O}_3$

W= weight of palm kernel oil

The calculation is done in appendix A and the result is showed in table 3.2

#### **Cloud Point (C.P)**

An improvised method was used for this determination. The cylindrical test tube was filled with the palm kernel oil to a specific level (5ml) and clamped with a wooden clamp bearing the

#### **Viscosity ( $\mu$ )**

The viscometer was placed in the measuring cylinder filled to the mark with water and regulated to the appropriate temperature. The tube was the filled up to the graduation mark over the left storage bulb with palm kernel oil. The palm kernel oil was the sucked up to fill the higher storage bulb in the right left of the tube and then released. The time taken for the palm kernel oil to flow from the upper mark to the lower was

thermometer. The test tube was placed in the ice/salt bath and the set up inspected at intervals for cloud formation. The temperature at which a distinct cloudiness appeared at the bottom of the test tube was observed and recorded as the cloud point of the palm kernel oil. The result is showed in table 3.2.

#### **Pour Point**

An improvised method was used for this determination. A cylindrical test tube was filled with palm kernel oil to a specific level (5ml) and clamped with a wooden clamp bearing the thermometer. The sample was then allowed to cool below  $0^\circ\text{C}$  in the ice/salt bath. At this point it is was removed and tilted on a clamp and the set up observed at intervals. The lowest temperature at which the palm kernel oil was observed to flow was recorded as the pour point. The result is showed on table 3.2.

#### **Density**

The weight of a small empty bottle was determined using electronic weighing balance. The bottle was filled to the brim with the palm kernel oil and the weight of the bottle and the palm kernel oil determined. The density was calculated using the formula;

$$\text{Density } (\rho) = \frac{W_2 - W_1}{V} \dots 2.5$$

Where;

$W_2$ =weight of bottle + sample

$W_1$ =weight of empty bottle

V=volume of sample

The calculation is done in appendix A and the result is showed in table 3.2

observed and calculated. The viscosity of the palm kernel oil was calculated using the formula below;

$$\text{Absolute viscosity} = \frac{t - t_0}{t_0} \dots 2.6$$

Where;

t=time of flow of sample

$t_0$ =time of flow of the reference (water in this case)

The result is presented in Table 3.2

## RESULTS AND DISCUSSION

The results obtained in this investigation are presented in this section.

### Results

**Table 3.1:** Present the percentage yield obtained from palm kernel extraction

Std	Run	Factor 1 A: Time/min	Factor 2 B: solute/sol	Response Yield Wt/wt(%)
11	1	75	0.30	33
12	2	75	0.02	23.66
10	3	120	0.10	23.28
7	4	75	0.58	23.92
3	5	75	0.30	8.12
4	6	75	0.30	26.36
13	7	138.64	0.30	23.92
2	8	120	0.50	26.36
8	9	30	0.50	12.02
6	10	30	0.10	28.19
5	11	75	0.30	3
9	12	11.36	0.30	23.68
1	13	75	0.30	12.95

**Table 3.2:** The results of physiochemical analysis of palm kernel oil

Parameters	Results	Literature (Viele, <i>et al</i> 2014)
Appearance	Free flowing liquid	Free flowing liquid
Color	White to yellow	white to yellow
Free fatty acid	0.1128%	0.0583%
Saponification value	206.17	232.81
Iodine value	20.050	41.24
Cloud point	-2°C	
Density	0.9103g/cm <sup>3</sup>	0.8809g/cm <sup>3</sup>
Viscosity	12.cp	
Acid value(% oleic acid)	0.81mmKOH/g	11.60mmKOH/g
Pour point	-5°C	-9°C

**Table 3.3:** The Results of Analysis of variance (ANOVA)3

Source	Sum of squares	Df	Mean square	F-value	p-value	
Model	562.14	2	281.07	7.03	0.0124	significant
A-Time	5.53	1	5.53	0.14	0.7178	
B-weight	556.61	1	556.61	13.92	0.0039	
<b>Residual</b>	<b>399.73</b>	<b>10</b>	<b>39.97</b>			
Lack of Fit	19.17	6	3.19	0.034	0.9996	not significant
Pure Error	380.56	4	95.14			
<b>Cor Total</b>	<b>961.87</b>	<b>12</b>				

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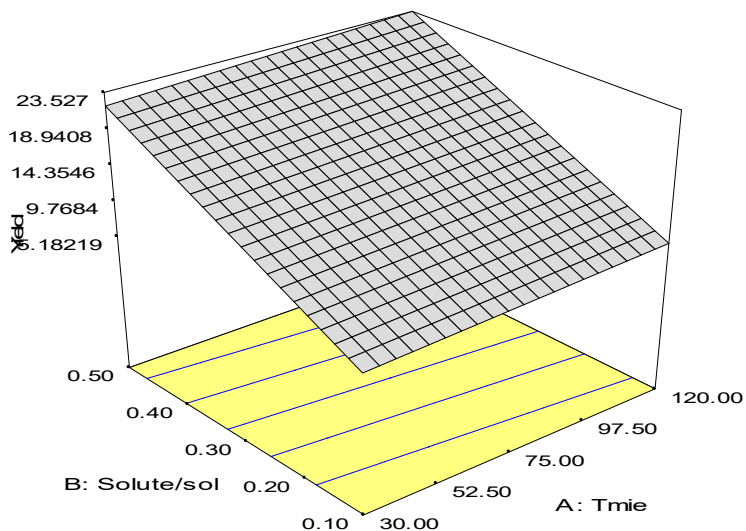
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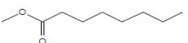

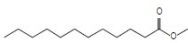
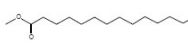

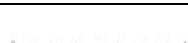
DESIGN-EXPERT Plot

Yield  
 X = A: Tmie  
 Y = B: Solute/sol



**Figure 3.1:** Response surface plots representing the effects of solute/solvent and the extraction time on palm kernel oil yield at constant particle size

**Table 3.4:** The GC-MS analysis of palm kernel oil

peak #	Retention time	Name of component	Molecular weight	Structural formula	Structure	% area under spectrum
1	15.559	Methyl capilate	158	C <sub>9</sub> H <sub>18</sub> O <sub>2</sub>		1.10
2	15.736	Capric acid, methyl ester	186	C <sub>11</sub> H <sub>22</sub> O <sub>2</sub>		7.10
3	17.469	Lauric acid, methyl ester	214	C <sub>13</sub> H <sub>26</sub> O <sub>2</sub>		50.12
4	17.653	Myristic acid, methyl ester	242	C <sub>15</sub> H <sub>30</sub> O <sub>2</sub>		3.84
5	19.336	Palmitic acid, methyl ester	270	C <sub>17</sub> H <sub>34</sub> O <sub>2</sub>		36.38
6	21.017	15-octadecenoic acid, methyl ester	296	C <sub>19</sub> H <sub>36</sub> O <sub>2</sub>		1.45

**DISCUSSION**

The application of response surface methodology in extraction using N-hexane was

conducted the relationship between response (palm kernel oil yield) and two independent factors (mass of sample and extraction period) were

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studied in order to optimize the oil extraction conditions via the Central Composite Design with two-level-two factors in this research. Table 5.1 shows the actual factors considered in this study and the experimental results. Regression analysis is the conventional approach to fit the empirical model via the collected response variable data. Design Expert 6.06 Trial software was employed to evaluate and determine the coefficients of the full regression model equation and their statistical significance. The final equation in terms of coded factors and actual factors for the response surface linear model are in Equation 4.1 and 4.2 below

$$\text{Yield} = +14.35 + 0.83 * A + 8.34 * B \dots 4.1$$

$$\text{Yield} = + 0.45745 + 0.018470 * \text{time} + 41.7063 * \text{solute/sol} \dots 4.2$$

The results of Analysis of Variance (ANOVA) for the response surface model showed that the Model F-value of 7.03 implies the model is significant. The p-values,  $p = 0.0124$  indicate model terms are significant. In this B is the only significant model terms while A is insignificant in this model because it is greater than 0.05. Lack of fit F-value was also significant as it had the F-value of 765.99 implying that the lack of fit was significant in the ANOVA analysis which is an indication that the model actually represented the relationships of independent factors (solute/solvent and extraction time) considered in this study. The effect of solute/solvent and extraction time on the oil yield at constant particle size is presented in Figure 3.1. It is evident that solute/solvent of the palm kernel had greater effect on the oil yield than the time. Although the ANOVA analysis had suggested that both significantly affected the oil yield. The result of the physicochemical analysis of the palm kernel oil extracted is shown on Table 3.2.

The density is a measure of the specific gravity of the oil. Most vegetable oil has density between 0.8767 and 0.8811 kg/m<sup>3</sup>. The density of Palm kernel oil obtained = 0.9103kg/m<sup>3</sup> indicated that it complied with biodiesel specification according to ASTM, 1996. The kinematic viscosity measures the flow resistance of the fuel. Ordinarily, vegetable oils have higher viscosity

which is reduced by trans-esterification processes. Most vegetable oils have kinematic viscosity between 2 and 5.7. (Viele, et al 2014). However, ASTM specification is 1.9 - 6.5 cSt, hence the palm kernel oil conformed to the standard having a viscosity of 12mm<sup>2</sup>/s at 40°C. Iodine value measures the amount of iodine required to saturate the olefinic bonds. It is an indicator of the unsaturation of the fuel, which has been linked with formation of engine deposits and problems in storing the fuel (Meher LC, Vidya SD, Naik SN, 2006). The iodine value of palm kernel oil extracted was 20.050. The saponification value of the palm kernel oil extracted (206.17mHCl/g) was found to correspond with the one in the literature review (Viele, 2014), indicating high concentration of triglycerides and hence palm kernel oil can be a suitable feedstock for the production of biodiesel. Acid value or neutralization number, is expressed in mg KOH required to neutralize 1 g of fatty acid methyl esters and is set to a maximum value of 60.5 mg KOH/g in the European norm, EN14214 (Ramos MJ et al. 2009). The acid value of the palm kernel oil was well below this maximum limit i.e. Acid value=0.253mg KOH/g, making the oil a good candidate for biodiesel production. The FFA obtained for the palm kernel oil was 11.28 (% w/w) which is well above the FFA in the literature (Viele, 2014), also the pour point and cloud point were determined to be 5°C and -2°C corresponding to the values in the literature. (Viele, 2014)

Gas Chromatography mass spectrometry analysis was carried to ensure the quality of palm kernel oil. The results showed that the palm kernel oil is highly saturated and it can be seen from the Figure 5.2 that the seven components are present in the palm kernel oil which are all saturated acids having lauric acid with the highest percentage as the main component present in most palm kernel oils, the percentage of the lauric acid is 50.12% compared to the literature value (Viele, 2014), which ranges between 45-50% implying that it is within the range required for palm kernel

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## CONCLUSION

1. The GC-MS analysis showed that the oil composed of Lauric acid with a percentage value of 50.12%.
2. The physicochemical analysis indicated that the density of palm kernel was found to be 0.9103g/cm<sup>3</sup>.
3. Model for the prediction of palm kernel seed oil extraction using n-hexane was developed by application of response surface methodology.
4. Yield= +14.35 + 0.83 \*A + 8.34 \*B
5. Yield= + 0.45745 + 0.018470 \* time + 41.7063 \* solute/sol
6. Analysis of variance (ANOVA) indicated that the model F-value of 7.30 or P-value = 0.0124. Significant model terms were (time) and (solute/solvent)
7. The predicted values (by the model) and experimental values of the response agreed closely.

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