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RESPONSE SURFACE OPTIMIZATION OF AVOCADO PEAR (*PERSEA AMERICANA*) SEED OIL EXTRACTION AND CHARACTERIZATION AS A POTENTIAL INDUSTRIAL FEEDSTOCK

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ABSTRACT

The present work is concerned with the study of the effect of temperature, time and their combined interactive effects on oil extraction yield from Avocado Pear seed using response surface methodology (RSM) in a soxhlet extraction apparatus with petroleum ether as solvent. The extracted avocado oil was also characterized to determine its suitability for relevant industrial application. The optimum extraction conditions were temperature of 60 °C, time of 5 hr with a corresponding oil yield of 17.9 wt%. Model validation using coefficient of determination (R^2) gave a value of 0.9211. Physicochemical properties of the oil such as acid value (6.89 mg KOH/g), peroxide value (3.7 meq/kg), saponification value (189 mg KOH/g), iodine value (62.18 g I₂/100g) establishes the potential of the oil for paint making, soap production and a potential feedstock for biodiesel production.

Keywords: Avocado pear, extraction, response surface, optimization, model

INTRODUCTION

Vegetable oil of plant and animal origins has been known to be an important feedstock for renewable energy production. It is used mainly as a viable feedstock for biodiesel production. Vegetable oil is also playing noteworthy role in the agro-industrial activities globally. The oils are used in the production of cosmetics, drugs and food substance. These oils are known to be potential source of energy in human and safeguard of internal organs. Most developing countries of the world are presently in short supply of edible oils and fats which is putting a heavy strain on the foreign exchange position of most African countries. This situation, therefore, calls for concerted research effort to increase edible oil production in Africa. In order to overcome this challenge, new sources of oil bearing crops are being exploited (Musa *et al.*, 2015).

The Avocado pear (*Persea Americana*) is a nutritious and valuable fruit tree found in the tropics. The species belongs to the family *Lauraceae*. *Persea Americana* is well known in many parts of the tropical world including Nigeria (Akachukwu, 2006). Avocado oil is the major avocado product, although it is rarely been explored in Nigeria. Only a few countries are actually involved in the production of oil namely Mexico (34 %), USA (8 %), Israel (4 %), South Africa (<2 %) and New Zealand (<1 %) and these are also the countries involved in growing, trading of the fruit to the cosmetics and pharmaceutical industry and as a lubricant. The emerging market for avocado should be acknowledged and taken advantage of as it presents many opportunities for relevant exploration of the surplus fruits for the production of value added product (Kuinimeri, 2011). Avocado's oil is mostly composed of monounsaturated fat. The oil is rich in Vitamin B, E and K. Avocado oil is well known for its anti-bacterial, anti-wrinkle and healing properties. The multiple properties of avocado oil such as excellent stability, emollient, effective skin penetration, softening and moisturizing ability which facilitate its broad application for cosmetic products synthesis (http://www.olivado.com/avocado_cosmetics.htm).

Solvent extraction is a viable means of extracting oil from the plant seeds. The process leaves a residue of less than 1% oil. It is one of the most efficient method uses for oil extraction from its precursor. This unit operation produces high quality oil at relatively low cost (Ochigbo and Paiko, 2011). Several studies have been documented on the extraction of oil from avocado pear (Botha, 2004; Ikhuria and Maliki, 2007; Mostert, 2007; Akpabio *et al.*, 2011; Adama and Edoga, 2011; Gatbonton *et al.*, 2013; Costagli and Betti, 2015).

Studies of the effects of process variables on the extraction of Nigeria avocado pears using Response surface methodology are rarely reported in literature. Response surface methodology (RSM) is a statistical technique used for the design of experiments and optimization of complex systems. RSM is an efficient and effective way to optimize the parameters that affect the process. This technique is used in order to find the optimize conditions when a number of factors are involved in process. It is characterize with several advantages such as it reduces process cost and process time through a reduction in the number of experimental runs. RSM is used to identify the response by using given process variables and fit an empirical model (Bokhari *et al.*, 2012). The aim of this work is to study the optimization of the Avocado oil seed extraction using Response Surface Methodology (RSM) studying the effect of temperature and time on oil yield.



METHODOLOGY

Materials

Avocado pear, petroleum ether, Acetic acid, Chloroform, Phenolphthalein indicator, Potassium hydroxide, Starch solution, Sodium thiosulphate, Potassium Iodide were all of analytical grade.

Methods

Raw material preparation

The avocado seed were distilled washed manually with water and sun dried for 5 days followed by oven drying. It was then ground to fine powdered particle. The powdered/ crushed seeds were sieved to a particle size of 500 micrometer sample and was stored in a polythene bag and kept in a cool place for further analysis.

Experimental design

A 2² RSM experimental design was used to determine the optimum yield, two variables (time and temperature of extraction) was studied at both high and low levels. The upper and lower levels were chosen considering the range commonly employed in literature.

Table 3.2: Matrix of Experimental Design

| Code | Level | | | | |
|-----------|------------|----|----|----|------------|
| | - α | -1 | 0 | 1 | + α |
| Time (hr) | 2.59 | 3 | 4 | 5 | 5.41 |
| Temp (C) | 47.93 | 50 | 55 | 60 | 62.07 |

Oil extraction from seed

Ten (10) g of dried seeds was placed onto a thimble and the thimble was put into the sohxlet apparatus. The solvent (Petroleum ether) was poured into three-neck- round bottom flask that is connected to the extractor and flask along with the condenser on the top to avoid any solvent losses due to evaporation. The entire experimental set up was then placed on the temperature controller heater to provide the required temperature. The temperature was measured by a thermometer that was inserted in one of the necks of the round bottom flask. After certain interval of the time the experiment was stopped and the oil extracted by the solvent was separated using rotary evaporator under vacuum at temperature of 65 °C. The oil obtained after evaporation was weighed. The yield was calculated using equation (1).

$$\text{Oil yield (\%)} = \text{Mass of oil extracted} / \text{mass of seed} \quad (1)$$

Characterization of Avocado seed Oil

The crude oil samples were subjected to various physicochemical analyses accordingly to determine its properties such as saponification value, iodine value, peroxide value, free fatty acid value, acid value, specific gravity, refractive index and viscosity using AOACS standard.

RESULTS AND DISCUSSION

Table 2: RSM Oil Extraction

| Run | Temp($^{\circ}$ C) | Time (hr) | Yield (%) |
|-----|---------------------|-----------|-----------|
| 1 | - | - | 8.75 |
| 2 | + | - | 13.5 |
| 3 | - | + | 16.5 |
| 4 | + | + | 17.9 |
| 5 | - α | 0 | 10.6 |
| 6 | + α | 0 | 12.5 |



| | | | |
|----|---|------------|-------|
| 7 | 0 | - α | 9.45 |
| 8 | 0 | + α | 17.4 |
| 9 | 0 | 0 | 15.5 |
| 10 | 0 | 0 | 15.23 |
| 11 | 0 | 0 | 16.89 |
| 12 | 0 | 0 | 15.7 |
| 13 | 0 | 0 | 15.67 |

Table 3: Analysis of Variance for Petroleum Ether

| Source | SS | DF | MS | F- Valu e | Prob > F |
|----------------|-------|----|-------|-----------------|-------------|
| Model | 103.3 | 5 | 20.66 | 16.35 | 0.001 |
| A- temp | 9.76 | 1 | 9.76 | 7.73 | 0.0273 |
| B-time | 68.4 | 1 | 68.4 | 54.15 | 0.0002 |
| AB | 2.81 | 1 | 2.81 | 2.22 | 0.1798 |
| A ² | 20.23 | 1 | 20.23 | 16.01 | 0.0052 |
| B ² | 4.1 | 1 | 4.1 | 3.25 | 0.1146 |
| Lack of Fit | 7.21 | 3 | 2.4 | 5.9 | 0.0596 |

Where SS=Sum of Squares, DF=Degree of Freedom, MS=Mean Squares

The experimental result for the optimization of oil extraction using a 2² RSM design is shown in Table 2. The result of statistical analysis of variance (ANOVA) carried out for the response evaluated to obtain the quadratic model is also depicted in Table 3. The response surface quadratic model equation for the weight percentage of oil yield as a function of temperature and time for coded factors and with actual factors is shown in the equation 2 and 3 respectively below. For coded factors

$$T = 0.3 + 4A + 3B + 2C + 22D \quad (2)$$

For actual factors

$$T = 1.031 + 3.4A + 0.31B + 0.231C + 0.120D + 0.13E + 0.21220F \quad (3)$$

From the coded factors, it can be seen that only A and B has positive coefficients, which indicates that they affect the yield of oil positively. That is, the factors A and B are directly proportional to the oil yield. However, the factors, such as A², B² and AB all have negative coefficients; therefore will have an inverse effect on the oil yield. From Table 3 the model F and p- value of 16.35 and 0.001 respectively is an indication that the model is very significant. When the values of probability >F is less than 0.0500 it is an indication that model terms are significant. In this case A, B and A² are significant model terms while if the values are greater than 0.100 it is an indication that the model terms are not significant in this case AB and B² are not significant model terms. The result revealed that both temperature and time shows a significant influence on the oil yield from extraction.



Table 4: Model Response

| Parameter | Values |
|----------------|--------|
| Std. Dev. | 1.12 |
| Mean | 14.28 |
| R-Squared | 0.9211 |
| Adj R-Squared | 0.8648 |
| Adeq Precision | 13.016 |

The Analysis of Variance shows that the model was sufficient to express the actual relationship between the yield and the significant value, with a satisfactory coefficient of determination, $M = 4$ which indicates 92 % of variability can be adequately explained by the model. It is also important to state that there is a quantitative agreement between R- squared and the adjusted R-squared value.

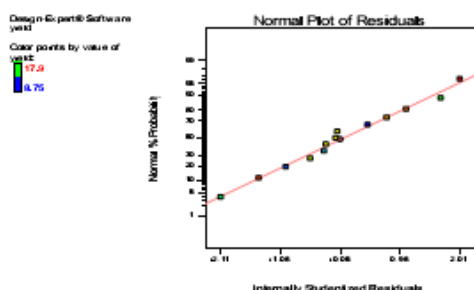


Figure 1: Model diagnostic plot

The central composite design normal probability plot of the residuals is usually an indication of the level normality of the response surface model developed. From Figure 1 shown above the approximately linear data points shows the normality of the model. According to Bokhari *et al.* 2012 when non-linear pattern are observed its signifies abnormality in the error term which may be corrected by transformation. The result obtained in this study shows no sign of any abnormality in this model.

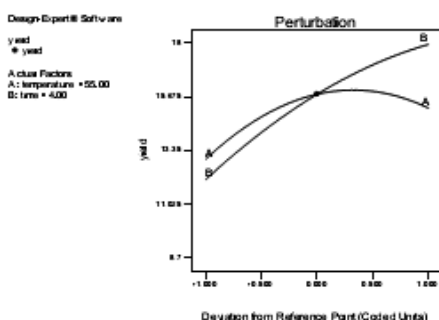


Fig 2: Perturbation Plot

Perturbation provides the outline views of the response. Perturbation plot shows how the response changes as any of the parameters moves from the reference point, with all other factors held constant at the reference value (Bokhari *et al.*, 2012). Figure 2 show that extraction time has the most pronounced effect of oil yield from the extraction process than temperature. This can be explained from the steep nature of slope when compared to slope of temperature. The yield decreases after a yield of 15 % and begins to reduce again.



Fig. 3: 3-D plot of yield with respect to extraction time and temperature

Figure 3 shows the 3D plot of the factors with respect to the yield. From the result the extraction time is shown to be the most important factor that affects the oil yield. The oil yield increases as times increases from low to high as higher extraction time results in more oil yield. Also an increase in temperature also leads to an increase in oil yield. However, when the temperature was increased beyond the boiling point of the solvent used a reduction in oil yield was experienced due to possible loss of the solvent. It is important to note simultaneous increases in both temperature and time will simultaneously inversely affect the yield of oil.

3.2.5 Properties of Avocado Seed Oil

Table 5: Properties of from Avocado pear oil

| Properties | Value |
|--|--------|
| Saponification value (mg KOH/g) | 198 |
| Iodine value (gI ₂ /100g) | 62.18 |
| Peroxide value (Meq/kg) | 3.07 |
| Free fatty acid value (%) | 5.4 |
| Acid value (mgKOH/g) | 6.8943 |
| Specific gravity (@ 25 ^o C) | 0.9162 |
| Refractive index | 1.465 |
| Viscosity (mm ² s ⁻¹) | 7.1 |

The oil content of the avocado seed was 17.9 wt%. This shows that only a slight fraction of the fruit is made up of oil. The saponification value obtained for this work is 198 mg KOH/g. Musa *et al* (2015) reported that oil with saponification value of 200 mg KOH/g indicates a high value of fatty acids with low molecular weights. This kind of oil will be good for the production of soap, shampoos and shaving cream.

The iodine value of avocado seed oil is 62.8 gI₂/100g. This value signifies high level of unsaturation. The oil could be classified as non-drying oil, since its iodine value is less than 100 gI₂/100g (Musa and Aberuagba, 2012). According to Akintunde *et al.* (2015) the European standard recommended a maximum iodine value of 120g I₂/100g oil for feedstock to be used for biodiesel production. The value obtained for this study indicates that avocado oil is a viable raw material for biodiesel synthesis. This iodine value also establishes its potential for paint making (Akpabio *et al.*, 2011)

The Free Fatty Acid (FFA) value of avocado seed oil is 5.4 %. The FFA of the oil is higher compared with 2.4 % for native pear oil (Akpabio *et al.*, 2011). The free fatty acid is important in determining the suitability of oil as edible oil. The lower the free fatty acid content, the more appealing the oil (Ikhuoria and Maliki 2007). The low free fatty acids of the oils (<5 %) makes them suitable as edible oils. Although, the value was slightly higher than 5 % it could also be used as edible with further refining. The free fatty acids value is low when compared to *Jatropha* oil and other conventional oil. The low free fatty acids content is an obvious indication of low enzymatic hydrolysis. According to Mahale and Goswami-Giri, (2012) oil with high free fatty acids develops off flavour during storage due rancidity. The oil from this study can be used for cooking and for the production of biodiesel and biodegradable lubricating oil.

Peroxide value is a valuable measure of oil quality and an indication of the ability oil to resist oxidation. The peroxide value obtained for the oil was 3.07



Viscosity is an important property that measures the resistance of flow of liquids (Musa and Aberuagba, 2012). The viscosity of oil is 7.1. The low viscosity of oil bears close similarity to the viscosity of biodiesel thereby establishing the ease of by which the oil can be effectively converted into biodiesel.

Refractive index is used to check purity. The avocado pear oil had a slightly higher refractive index of 1.421. Other physical properties such as viscosity, density and moisture content were $7.1 \text{ mm}^2\text{s}^{-1}$, 0.9kg/m^3 and 20 % respectively.

CONCLUSION

RSM was successfully used to study the effect of temperature and time on oil extraction from avocado pear seed using the soxhlet apparatus. An empirical equation was developed for oil yield as a function of variables investigated. The optimal condition for the extraction of oil from avocado seed was at the temperature of $60 \text{ }^\circ\text{C}$ and time of 5 hours, particle size of $500 \text{ }\mu\text{m}$. Oil yield in the soxhlet apparatus was 17.9 wt % The oil can be used for paint making due to its low iodine value. However, the free fatty acid value and peroxide value makes the oil a good candidate for domestic use as edible oil and also commercially for soap making, lubricant and biodiesel production.

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