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OPTIMIZATION AND CHARACTERIZATION OIL EXTRACTION FROM TIGER NUT (*CYPERUS ESCULENTUS L.*) USING RESPONSE SURFACE METHODOLOGY

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Abstract

Oil extracted from brown tiger nut (Cyperus esculentus) tubers, were optimized via soxhlet apparatus using n- hexane as solvent. Central composite design (CCD) in response surface methodology (RSM) was applied to this experimental layout and effect of Time (sec) Temperature (°C), and Liquid to solid Ratio (ml/g) on the oil extraction were evaluated, characterized using physicochemical analysis and Fourier transform infrared (FT-IR). It was found that within the given rang, the highest oil yield of 24.3 was achieved at a temperature of 80°C, reaction time 360 min and 7 ml/g of liquid to solid ratio. The optimized condition was validated with the actual oil yield of 24.3%. The FT-IR analysis confirm the composition of tiger nut oil and physicochemical properties of the oil showed it to be liquid/golden yellow, specific gravity, 0.91 g/cm^3, acid value,2.92 mg KOH/g oil, Saponification value, 216.48 mg KOH/g oil, Peroxide value, 5.90 (M mol/kg), Iodine value, 11.086 g I2/100 g oil, FFA, 1.459 (%), Refractive Index 0.907. Based on the available data, tiger nut oil has been established as an oil of good nutritional value which may be exploited to the great benefit of growers, processors (industries) and dealers of the tuber.

Keywords:
Optimization, Solvent extraction, Tiger nut oil

Introduction

Vegetable oils play an active role in nourishment and industrial applications, among several oil bearing plant tuba tiger nut (Cyprus esculents) is gaining attention due to its high oil content and nutritional benefits. Tiger nut is a perennial grass like plant commonly cultivated in Europe, Asia and Africa. Africa particularly in Nigeria, where it is locally named Aya (Hausa), Imumu (Yoruba), and Ofio (Igbo) (Adejuyitan, *et al* (2011).

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Oil from tiger nut is reported to be rich in oleic acid, a monounsaturated fatty acid known to decrease bad cholesterol and promote heart health. In addition, tiger nut oil contains natural antioxidants, vitamin E, and phytosterols, which make it suitable not only for nutritional purposes but also for cosmetic and pharmaceutical applications (Maduka & Ire, 2018). Despite these benefits, the crop remains under-utilized due to limited awareness, lack of commercial processing, and inefficient extraction techniques. (Adejumo et al., 2013). The need for unique methods lies in the fact that traditional techniques have different shortcomings like additional energy, more time, low yield, and less environmentally friendly. However, the physical methods of oil extraction can only recover about 80% of oil present in oleaginous material; hence, to recover the remaining 20%, different technology has to be applied. Solvent extraction is commonly adopted owing to the uncomplicatedness and economy of the process. The frequently used solvents are hexane and n-hexane because they result in the highest (95%) yield. Mostly, n-hexane is chosen due to its superior attributes such as simple recovery, low latent heat of vaporization (330 kJ/kg), narrow range of boiling point (63 to 69 ◦C), high solubility, and its nonpolar nature (Peter *et al.,* 2021).Tiger oils is more stable, has a pleasant flavor and suitable for both edible and cosmetic purposes. However, oil yield from tiger nut is affected by factors such as particle size, solvent type, extraction time, and temperature. Hence, optimization of the oil extraction process is necessary to achieve maximum yield and quality (Li H, *et al.,* 2021). Conventional edible oils like groundnut, soybean, and palm oil are widely used, but environmental and health concerns call for alternative oils. Tiger nut oil, despite its potential, is underutilized due to lack of optimization and awareness. Efficient extraction and full characterization can improve its acceptability and use. However, this study addresses these challenges by optimizing extraction parameters, fully characterizing the extracted oil and its applications.

METHOD

A pre oven dried t**hi**mble was weighed with analytical weighing balance (w_1). 10g of tiger nut flour sample was added to it (w_2), and put inside the Soxhlet apparatus. 500 cm^3 boiling flask was filled to 2/3 with solvent (Hexane) and fitted to the Soxhlet carrying the condenser to cool the solvent. However, three-factor test to study the effect of Time (min) (A), temperature ^0C (B) and liquid to solid ratio (C), yields was investigated using central composite rotatable design (CCRD). The CCRD used for this work contains twenty (20) experimental runs using 2k+2k+n, where k is the number of factors and n is the number of center point. Besides, there are eight (8) factorial point (2k), six (6) axial point (2k) and six (6) replicated point (n=6). Here k is the independent variable and k=3 (Muhammad et al., 2019). The limitation of apparatus and extraction condition used in tiger nut oil extraction were carefully used in determining the factors level for factorial

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design. The liquid to solid ratio 3 and 7 ml/g, a high and low level of 80°C and 40 °C and 60 and 360 minutes was reported by Xiaoshuang et al, (2022). Table1 shows range level of process parameter in design for tiger nut oil extraction. The set-up for continuous Soxhlet apparatus for oil extraction is shown in plate I.



Plate I: The: Set-up of Continuous Soxhlet Apparatus for Tiger Nut Oil Extraction Process.

Table 1; Range of Level of Process Parameter in Design

Independent Factors	Symbol	Low level (-)	Medium level (0)	High level (+)
Time (mins)	A	60	210	360
Temperature (°C)	B	40	60	80
Liquid to solid ratio (ml/g)	C	3	5	7

Physicochemical Characteristics of Tiger Nut Oil

The results of the physicochemical characterization of the oil extracted from tiger nut are presented in Table 2. The iodine value displays the level of unsaturation of the oil. It is used in determining the drying property of the oil low iodine value (11.086 g/100 g) which

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confirmed it as non-drying oil. Other oils classified as non-drying are macadamia, olive, and peanut oils. Low iodine values for tiger nut oil (31.74 g/100 g) was also obtained by (Nina et al., 2020). However, the iodine value of tiger nut oil reported by El-Naggar ., (2016) was 87.08 gI2/100g which is high and high value of Iodine implies high degree of unsaturation of the oil. High saponification value indicates that the oil will be more suitable for soap and cosmetic. The saponification value 216.48 mg KOH/g of oil was achieved and it is in close agreement with 192.88 mg KOH/.g of oil reported by El-Naggar, (2016) which are high when compare with the value of 160.01 mg of KOH/g of oil reported by Kesin and Yilmaz. (2024) who studied Cold pressed tiger nut (Cyperus esculentus L.) oils Chemical and aromatic profiles, sensory properties, and consumer preferences. However, the Acid value is a measure of the free fatty acids in oil. The higher the acid value found, the higher the level of free fatty acids which translates into decreased oil quality. The free fatty acids and the peroxide values are valuable measures of oil quality. The tiger nut oil showed 1.459% of free fatty acids and 5.90 m mol.kg–1 of peroxide value. These values are not considered high since crude vegetable oil maximum contents of 5% of free fatty acids and 10 m mol.kg–1 of peroxide value may be verified (Nina et al., 2020). Besides, the lower the acid numbers the better the oil for cosmetic.

Table 2: Physicochemical Characteristics of Tiger Nut Oil

Parameter	This Study	Tiger nut oil[a]	Tiger nut oil[b]	Tiger nut oil[c]
Iodine Value (g I_2 100g)	11;09	82.00	87.08	31.74
Peroxide Value (M mol/kg)	5.90	2.64	-	4.43
Specific Gravity	0.91	-	-	-
Saponification Value (mgKOH/g)	216.48	-	-	-
Density g/cm3	0.91	1.01	0.92	0.89
FFA (%)	1.46	1.80	-	0.75
R. Density	0.91	-	-	-
R.Index	1.47	-	-	-
Oil yield (%)	24.3			

[a]Kesnin and Yilmaz(2024) [b]El-Naggar et al (2016) [c]Nina et al (2020)

Fourier Transform Infrared Spectroscopy (FTIR) Spectrum Analysis of Tiger Nut Oil

The Figure 1 shows the analyzed sample and is compared with known signature of identified materials in the FTIR library. The FT-IR bands of 2922 cm-1, observed in brown tiger nut oil are the region of unsaturated fatty acid. The asymmetrical and symmetrical modes of vibration of –CH2-exhibit very strong and sharp bands at 2870 cm-1, tiger nut oil. The band at 1778 cm-1 is very strong and sharp band due to ester carbonyl functional

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group of the triglycerides i.e. C=O stretching vibrations no absorption, This shows that the fatty acids is exclusively in ester forms. The vibration frequency at, 998 cm-1, 862 cm-1 and 840 cm-1 for tiger nut oil is finger print of the stretching vibration of the C-O ester group. The frequencies of the in-and out-of plane rocking of the cis-olefinic CH_2 group are at 634 cm-1, 412 cm-1, 394 cm-1 and 238 cm-1 for tiger nut. The observed differences in frequency positions of the ester carbonyl, and the inactivity or absence of some in-plane bending are attributed to the variation in fatty acids composition, chain length, degree and position of double bonds in the triacylglycerols . However, polymeric applications of quality characterized plants oils in general found useful in polymer preparations.

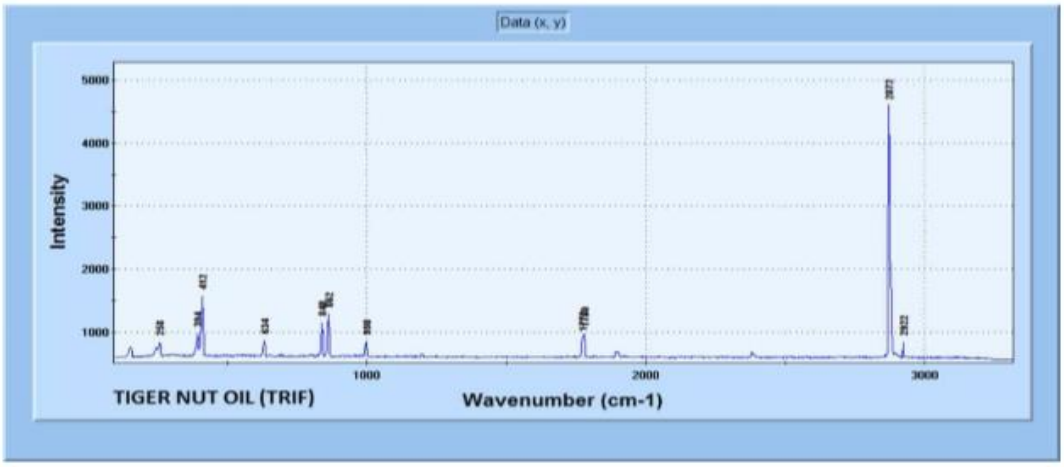


Figure1; FTIR Spectrum of Tiger Nut Oil

Optimization of Process Variables for the Yield of TIGER NUT OIL

The optimization process result for tiger nut oil yield is shown in table 3; the design expert was used to analyze the result. Time (sec) Temperature (°C), and Liquid to solid Ratio (ml/g) of oil extraction were all fixed between 40 - 360min, 40 -80°C, and 3-7 ml /g, respectively. It was found that within the given rang, the highest oil yield of 24.3 was achieved at a temperature of 80°C, reaction time 360 min and 7 ml/g of liquid to solid ratio.

Table 3: Experimental Yield of tiger nut oil extraction for CCDR Arrangement

Runs	Time (min)	Temp (°C)	Liq-Sold ratio	Yield (%)
1	60	40	3	23.1
2	360	40	3	24.0
3	60	80	3	23.2
4	360	80	3	24.25
5	60	40	7	22.9

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Runs	Time (min)	Temp (^{0}C)	Liq-Sold ratio	Yield (%)
6	360	40	7	23.9
7	60	80	7	23.25
8	360	80	7	24.3
9	0	60	5	22.1
10	462	60	5	23.9
11	210	26	5	23.0
12	210	94	5	23.7
13	210	60	2	23.55
14	210	60	8	23.5
15	210	60	5	23.52
16	210	60	5	23.45
17	210	60	5	23.4
18	210	60	5	23.35
19	210	60	5	23.4
20	210	60	5	23.45

The Analysis of Variance (ANOVA) for tiger nut oil Yield

The summary of P-values and model statistics are shown in table 4 and 5. A linear model was suggested from the CCD module with a high adjusted and predicted R^2 values of 0.7796 and 0.6774 respectively. However, the ANOVA carried out confirmed the adequacy of linear model.

Table 4: ANOVA for Response Surface Linear

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.84	3	1.28	23.40	< 0.0001	Significant
A-Time	3.45	1	3.45	63.14	< 0.0001	
B-Temperature	0.3806	1	0.3806	6.96	0.0179	
C-Liq-Solid Ratio	0.0060	1	0.0060	0.1106	0.7438	
Residual	0.8750	16	0.0547			
Lack of Fit	0.8580	11	0.0780	22.83	0.0014	significant
Pure Error	0.0171	5	0.0034			
Cor Total	4.71	19				

The ANOVA result in Table 4.4 show that the Model F-value of 23.40 is high with lowest P-value (P<0.0001), this implies that the model is significant. However, the larger the magnitude of F- value and smaller the magnitude of P-value, the higher will be the significance of the corresponding coefficient (Xiaoshuang *et al*., 2022). The values of

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"Prob > F" less than 0.0500 indicate model terms are significant (Table 4.4). In this case A and B are significant model terms while C is insignificant model term. The value of regression coefficient $R^2$for the model is 0.8144, this indicate that the model adequately represents the experimental results. This is in agreement With Audu *et al*., (2017). A linear model was suggested from the CCD module with high adjusted and predicted R-squared values of 0.7796 and 0.6774 respectively shown in Table 4.4.

Table 5: Regression Analysis

Parameter	Value
Std. Dev.	0.2339
Mean	23.46
C.V. %	0.9968
R^2	0.8144
Adjusted R^2	0.7796
Predicted R^2	0.6774
Adeq Precision	15.3096

Adequate Precision measures the signal to noise ratio and it compare the range of the predict value at design point to the average prediction error. The adequate precision value greater than 4 is desirable for sufficient model efficiency. Therefore, the obtained adequate precision value of15.310 indicate an adequate signal. The fitness of the model equation was validated using the coefficient of regression, R^2. The high R^2value of 0.8144 implies that 81.44% of the variability in the response can be explained by the model, this also confirmed that the model can be used to steer the design space. However, the higher the value of the adjusted coefficient of determination, the more the significant the fitted model. In this work, the coefficient determination is higher which shows that the fitted model is significant. The final equation in term of coded factors was obtained and shown as equation 4.1 respectively.

Final Equation In Terms of Coded Factors

$$Yield = 23.45 + 0.5199A + 0.1662B - 0.0220C$$

Equation 1 above shows the coefficient of the full regression, numerical significant and the exact impact on each model expression on the yield. The above positive and negative sign before each term shows synergistic or antagonistic effect respectively. The temperature, Time, has synergistic effect on the tiger nut oil yield while Liquid to solid ratio has antagonistic effect on yield. The model was found more significant at 81.44% percent

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confident level for computed F values (23.40) with very low probability value ($p < 0.0001$). However, all terms in the model was found to be significant at 81.44percent confident level as the computed F values for the particular terms were higher. Nevertheless, the smaller the p-value for a parameter the more important the parameter and therefore it shows the relative significance of the term related with the parameter (Xiaoshuang *et al*., 2022). The predicted vs actual plots in figure 4.2 were used to check the close distributions of the points along the straight lines indicate the excellent relationship between the experimental and predicted values of the response. These plots also validate that the chosen model was sufficient for prediction of the response variables in the experimental values.

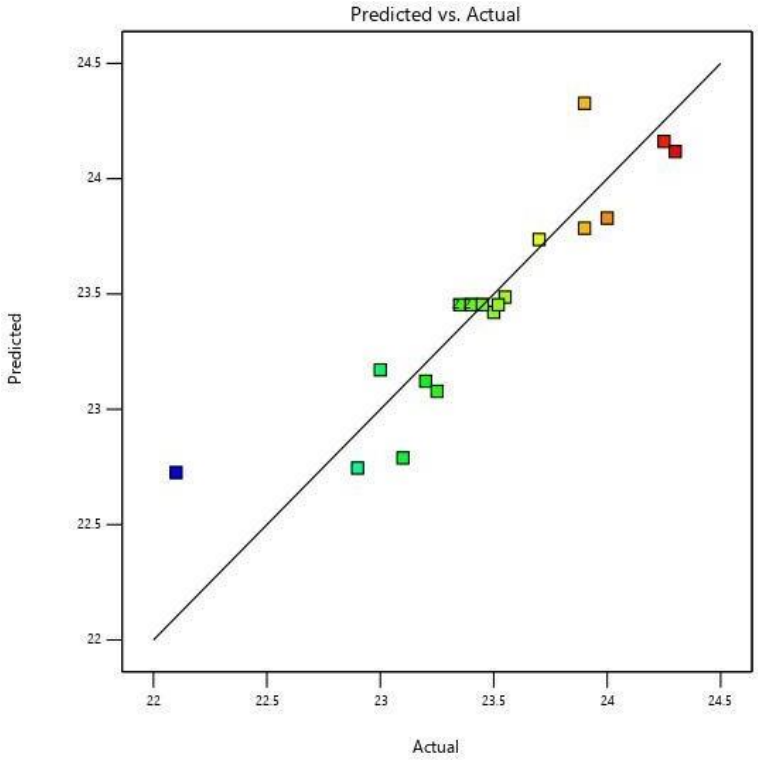


Figure 2: A Graph of Predicted Yield against Actual Yield

Response Surface Measurements

The 3-dimensional (3D) surface plot of the linear model shown in Figure 4.3, and 4.4 helps to understand better the interactive effect of the variables on the yield of tiger nut oil and how oil yield varied as a function of any two factors selected for display. The nature of the response surface curves reveals the communication between the variables. An elliptical shape in a curve shows worthy interaction between the variables. The elliptical nature of

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the contour in the graphs also indicates that there was a relative significant interaction between every two variables.

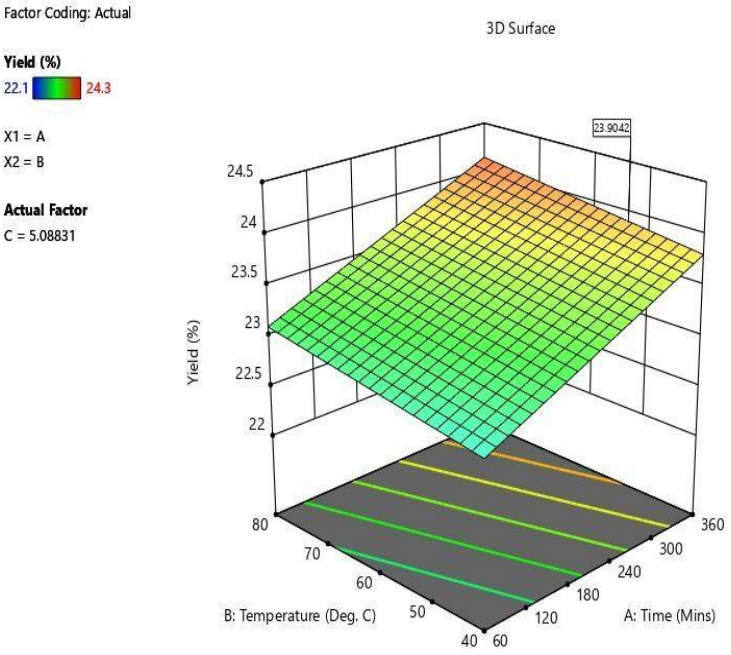


Figure 3: 3D Surface Plot Showing Combined Effect of Temperature and Time

Figure 3 shows the interactive effect of temperature and time on tiger nut oil yield. It can be seen that the tiger nut oil yield increases as temperature increases which might be due to increase in the reaction speed resulting to higher conversion of the oil. The increasing effect of temperature shows that 24.30 % yield was recorded at temperature of 80°C, reaction time of 360 mins and liquid to soild ratio 7.

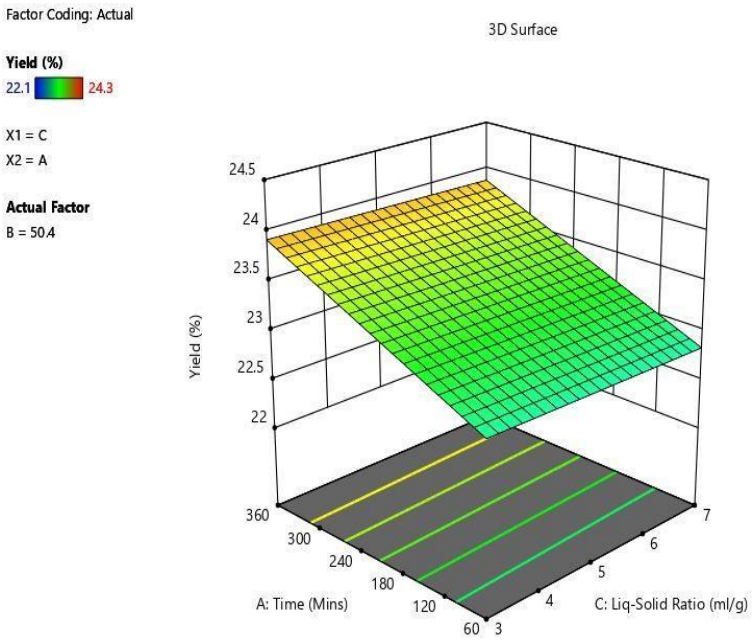


Figure 4: 3D Surface Plot Showing Combined Effect of Time and Liquid- Solid Ratio

Figure 4 shows the interactive effect of time and liquid to solid ratio on the yield of tiger nut oil. There was no significant effect of the increase in liquid to solid ratio on the yield, the 24.30 % yield was recorded at temperature of 80°C, liquid to solid 7 and reaction time of 360 min

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CONCLUSION

The following conclusions were made from the results of the research carried out:Tiger nut oil showed high oil content (24.30%). Tiger nut has sufficient oil content and the oil has suitable properties which are similar with other vegetable oils. However, the physicochemical properties of tiger nut oil are in close agreement with that of El-Naggar *et al*. (2016). Information provided by present study is of great importance and revealed industrial utilization of the tiger nut oil as feedstock production.

FTIR analysis result shows the appearance of the band peaks of 2922 cm^{-1}, 2872, 17780 cm^{-1}; and 258 cm^{-1}, which were are present in the tiger nut oil which shows that the oil in cosmetic industries.The optimization of extraction via. Vital process variable on the yield of tigernut oil. Was made possible by three-factorial central composite design using response surface methodology (RSM) in 20 experimental runs. A linear model capable of predicting the tiger nut oil yield based on the process variables was developed. 24.30 % optimum yield was achieved with desirability of 1.000 at optimum conditions of temperature at 80°C, reaction time at 360 minutes. Statistical analysis of variance (ANOVA) of results show that temperature and time has a positive effect on the tiger nut oil yield, liquid to solid ratio effect is less significant yield.

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