Journal of Basics and Applied Sciences Research (JOBASR)
ISSN (print): 3026-9091, ISSN (online): 1597-9962
Volume 3(2) March 2025
DOI: https://dx.doi.org/10.4314/jobasr.v3i2.1

Physicochemical Properties, Kinetics, and Thermodynamics Study of Oil Extraction from Pearl Millet Bran

Jafar, H.[1*], Kamaluddeen, S. Kabo[2] and Bashir, A[3].
[1]Department of Pure and Applied Chemistry, Faculty of Physical Science, University of Maiduguri, Maiduguri, Borno State, Nigeria.
[2, 3]Department of Chemistry, Faculty of Physical Science, Federal University Dutsin-Ma, Katsina State, Nigeria.
*Corresponding Author Email: jhk40983@gmail.com

Keywords:
Extraction, Oil, Kinetics, Thermodynamics, Physicochemical Properties, Characterization.

ABSTRACT
The increasing demand for edible oils and the need for sustainable waste management have driven research into alternative oil sources such as millet bran. This study focuses on the kinetics and thermodynamics of oil extraction from millet bran using a Soxhlet extractor with n-hexane as the solvent. The maximum oil yield from millet was observed to be 10.78%, 13.23%, and 15.08% at temperatures of 303, 313, and 323 K respectively. The physicochemical properties, including iodine value, ash content, and density, were analyzed alongside FTIR and GC-MS analysis for characterization, confirming the extracted material as oil. The activation energy (Ea) for millet oil extraction was determined to be +7.8990 kJ/mol. Thermodynamic parameters were also assessed, with activation enthalpy (ΔH) recorded at +5413 kJ/mol, activation entropy (ΔS) at -8.634 $kJmol^{-1}K^{-1}$, and Gibbs free energy (ΔG) values indicating an endothermic and non-spontaneous reaction. These findings contribute to optimizing pearl millet oil extraction and its potential application in edible oil production.

INTRODUCTION

Millet has been an essential food source for humans for over 10,000 years, providing significant nutritional value depending on the milling process used. Its bran contains 6–7% oil, making it a viable source for oil extraction (Lu et al., 2005). Despite its potential, millet has historically been overlooked in food markets, research, and commercialization efforts (Rai et al., 2008). However, its nutritional benefits and potential for alternative food products have recently gained interest in scientific studies. Millet ranks among the top five cereal grains produced globally, particularly in semi-arid tropical regions of Asia and Africa, where it serves as a crucial source of energy, protein, vitamins, and minerals for millions of people (Rao et al., 2017). Its bran, often considered an agricultural by-product, has been recognized for its high oil content, leading to research on its potential use in edible oil production and biodiesel (Yu et al., 2018).

The extraction of millet oil is primarily done using mechanical pressing or solvent extraction, with n-hexane being the most common solvent due to its high solubilizing power and cost-effectiveness. However, concerns over hexane's environmental and health impacts have led researchers to explore alternative solvents such as ethanol, isopropanol, and ethyl acetate (Kostic et al., 2014; Kumar et al., 2017). Studies show that solid-liquid extraction is the most efficient method for vegetable and seed oil production, though it requires optimization to balance extraction efficiency, cost, and sustainability (Topalla & Gecgel, 2000).

Millet oil has gained attention in biodiesel production, where it serves as a renewable and biodegradable fuel source. Compared to inedible oils such as jatropha and neem, millet oil is classified as edible and has been widely used in food industries (Azam et al., 2005). However, a major challenge is the competition between edible oil productions for food versus energy use (Gui et al., 2008). Studies indicate that biodiesel from edible oils is of higher quality, yet economic and food security concerns limit its large-scale use (Refaat, 2010). The chemical composition of vegetable oil plays a crucial role in its application for biodiesel production. As shown in Scheme 1, the molecular structure consists of long hydrocarbon chains with hydroxyl functional groups, which influence its solubility and reactivity.

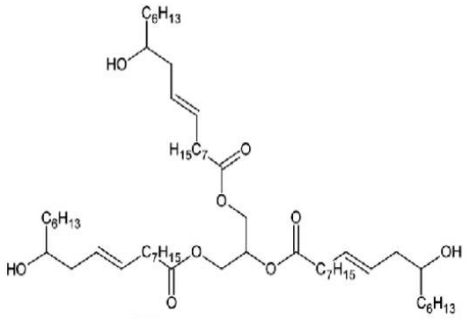

Figure 1: Chemical structure of vegetable oil

Recent research into the kinetics and thermodynamics of millet oil extraction has focused on optimizing yield and efficiency. Factors such as particle size, temperature, solvent-to-sample ratio, and agitation speed significantly influence extraction rates (O'Brien, 2008). Smaller particle sizes increase surface area, improving oil recovery, while higher temperatures accelerate solvent diffusion into the cellular structure (Hussain et al., 2018). Furthermore, thermodynamic studies have shown that millet oil extraction is endothermic and non-spontaneous, requiring external energy input (Menkiti et al., 2015).

By understanding the physicochemical properties, extraction kinetics, andthermodynamicbehavior of millet oil, researchers aim to improve its commercial viability for food and biofuel applications. Future studies should explore green solvents, alternative extraction methods, and energy-efficient processes to enhance sustainability and industrial adoption (Vithanage et al., 2015). This study examines the physicochemical properties, kinetics, and thermodynamics of the oil extraction process in pearl millet, aiming to improve extraction efficiency and sustainability.

MATERIALS AND METHODS

Materials

The chemicals and reagents used were of AR (Analytical reagents) grades. The list of the materials and reagents used include: n-Hexane, cyclohexane, Wijs solution, potassium iodide (KI), distilled water, Na2SO3, starch solution , millet bran, tap water, density bottle, stopper, muffle furnace, Soxhlet apparatus 250 mL and 500mL, filter paper, 250mL, and 500mL glass beakers, weighing balance, forced hot air oven, hot plate, thermometer, stirrer, measuring cylinder, stopper/timer, power supply, 500 mLround bottom flask, pipette, funnel. vacuum, reagent bottles, Sieve/gauge, and water bath.

Methods

Preparation of millet bran

Millet bran is recovered by mixing the millet grains with a small amount of water followed by putting the mixture in a decorticator until the desired amount of the bran is obtained. The bran recovered was dried at 50 ℃under a hot air oven until complete elimination of its moisturecontent before any experimental test. It's then transferred sealed in plastic and preserved inthe fridge for three days to avoid the deterioration of the sample before the extraction process(Topallar and Gecgel, 2000).

Oil Extraction process

The experiment for oil extraction was carried out by weighing a specified wrapped gram of Millet bran in a filter paper and place it inside the thimble chamber of the 250 ml Soxhlet extractor. A round bottom flask containing n-hexane fitted with a condenser was fixed to the extractor. 170ml of n-hexane was measured and poured on the top opening of the condenser until it siphoned into the round bottom flask. Cool water flowing through the condenser was used to condense the evaporating solvent back into the Soxhlet extractor where the sample was packed to ensure sufficient extraction of the oil from the seeds. After a specified time, the mixture of n-hexane and the extracted oil was separated by gently heating off the mixture to evaporate the solvent (Topallar and Gecgel, 2000). At the end of each experiment, the yield of the oil was obtained as the percentage of the extract from the seed using equation (1).

$$Percentage\ yield\ (Y) = \frac{Mass\ of\ the\ oil}{Mass\ of\ the\ sample} \times 100 \quad (1)$$

Characterization of Extracted Oil

Determination of physicochemical parameters of the extracted oils

The extracted oil was analyzed for iodine value, ash content, and density to determine its suitability for edible and industrial applications.FTIRwas used to identify functional groups, while GC-MS determined the composition of fatty acids.

Iodine value determination

A 0.4 g sample of oil was dissolved in 15 mL of cyclohexane and 15 mL of Wijs solution. The mixture was left in the dark for 30 min, followed by adding 20 mL of potassium iodide (KI). After that, 100 mL of distilled water was added to the solution. The solution was titrated with 0.1 M Na2SO3 with a few drops of 1% starch solution until the color disappeared. (Kong S. *et al.*, 2023). The iodine value (IV) was calculated using Equation (2)

$$I.V = 12.69\ M \times (VB - VS)/W \quad (2)$$

Where, VS is the volume of Na2SO3 titrated with the sample (mL), VB is the volume of Na2SO3 used as a blank, M is the molarity of Na2SO3 used, and W is the mass of the sample used (g).

Determination of Density

Density bottle was used to determining the density of the oil. A clean and dry bottle of 25ml capacity was weighed (W_0) and then filled with the oil, stopper inserted and reweighed to give (W_1). The oil was substituted with water after washing and drying the bottle and weighed to give (W_2) (Akpan*et al.*, 2006). The expression for specific gravity (Sp.gr) can be estimated using equation (3):

$$Sp.gr = \frac{W_1 - W_0}{W_2 - W_0} = \frac{\text{Mass of the substance}}{\text{Mass of an equal volume of water}} \quad (3)$$

Determination of Ash content

The ash content was determined by incineration using a muffle furnace according to AOAC 942.05. A clean and dry porcelain crucible was weighed (W_0) and then filled with the dried sample. The sample was gently charred on a low-temperature hot plate before being transferred to a preheated muffle furnace set at 550–600°C. The sample was incinerated for 2 hours until all carbonaceous material disappeared, leaving only ash residue. The crucible with the ash was cooled in a desiccator and reweighed (W1). The ash content was calculated as a percentage using equation (4):

$$\text{Ash content (\%)} = \frac{\text{W1} - \text{W0}}{\text{W2} - \text{W0}} \times 100 \quad (4)$$

where:
W_0 = Weight of the empty crucible
W_1 = Weight of the crucible with ash
W_2 = Weight of the original sample before incineration
The composition of oil was expressed as a percentage (%)

FTIR Characterization

FTIR spectra of both millet extracted oil were analyzed using an FTIR spectrophotometer (Cary 360) and spectra were recorded over a scanning range from 650 to 4000cm^{-1}

GC-MS Characterization

The GC-MS analysis of the extracted oil was performed to separate and identify its chemical components. The analysis was conducted using a Gas Chromatograph (GC) coupled with a Mass Spectrometer (MS) (Agilent 5977B) under the following conditions:

Parameter	**Condition**
Instrument	Agilent 5977B GC-MS system
Column	HP-5 (30 m × 250 µm, 0.25 µm film thickness)
Carrier Gas	High-purity nitrogen at a flow rate of 1.0 mL/min
Injection Mode	Split injection with a split ratio of 10:1
Injection Volume	1 µL of the oil sample diluted in n-hexane
Injection Temperature	250°C
Oven Temperature Program	Initial temperature at 100°C, held for 1 min Increased at 15°C/min to 280°C Held at 280°C for 10 min
Solvent Delay	5 min
Ionization Mode	Electron ionization (EI) at 70 eV
Ion Source Temperature	230°C
Interface Temperature	230°C
Mass Scan Range	m/z 20 – 500

The obtained mass spectra were compared with the NIST Mass Spectral Library to identify the chemical compounds present in the millet bran oil.

KINETICS AND THERMODYNAMICS STUDIES

Kinetics Study

Oil extraction kinetics from millet bran can be described using a generalized kinetic model. The extraction rate depends on the interaction between the solvent and the oil present in the solid matrix. The process is often expressed using the rate equation in equation (5):

$$\frac{dY}{dt} = kY^n \quad (5)$$

Where ***Y*** is the percentage oil yield (%), ***t*** is the time of extraction (min), ***K*** is the extraction constant and ***n*** is reaction order. A plot of ***ln Y*** against ***t*** gives ***lnk*** as the intercept and ***n*** as the slope (Topallar and Gecgel, 1999).

Determination of Activation Energy (Ea) for Millet bran oil

$$k = Ae^{-\frac{E_a}{RT}} \text{ (Arrhenius equation)} \quad (6)$$

where; A = Frequency factor

E_a= Activation energy

R = Gas constant, and T = Temperature

Taking the natural logarithm to both sides the equation (6) becomes:

$$lnk = \frac{E_a}{R}(\frac{1}{T}) + lnA \quad (7)$$

where *lnA* is the intercept.

Thermodynamic Study

Thermodynamic parameters like enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG) can be estimated using a known thermodynamic equation (Menkiti *et al.*, 2015). The thermodynamic parameters (ΔH, ΔS, and ΔG) for the extraction of oil from a millet, using n-hexane as a solvent, can be estimated using Eqs. (9) and (10). However, Eq. (12) is used to calculate the parameters.

$$k = k^{\neq}\frac{k_B T}{h}e^{-\frac{\Delta G}{RT}} \quad (8)$$

Where: k = rate constant

$k^{\neq}$ = transmission coefficient usually takes value of 1

k_B = Boltzmann constant = 1.38×10^{-23} JK^{-1}

h =Planckconstant = 6.63×10^{-34}Js

Recall that: ΔG = ΔH - TΔS (9)

Where: ΔG is the Gibss free change, ΔH is the enthalpy change, and ΔS is the entropy change.

Putting equation (3) into equation (2)

$$k = k^{\neq}\frac{k_B T}{h}e^{\frac{T\Delta S - \Delta H}{RT}} \quad (10)$$

Taking natural logarithm of equation (4)

$$\ln\frac{k}{T} = \frac{\Delta S}{R} - \frac{\Delta H}{RT} + \ln k^{\neq} + \ln\frac{k_B}{h} \quad (11)$$

$$\ln\frac{k}{T} = -\frac{\Delta H}{R}(\frac{1}{T}) + [\ln k^{\neq} + \ln\frac{k_B}{h} + \frac{\Delta S}{R}] \quad (12)$$

RESULTS AND DISCUSSION

Physicochemical Properties and Oil Yield

Physicochemical Properties

The results obtained from the physiochemical properties of millet bran oil are presented in Table 1.

Table 1: Pysiochemical properties of millet bran oil

Components	Value	ASTM 763 standard
Iodine value (mg I_2/100g)	122.3	120
Ash contents (%)	2.6	1.5-4.5
Density (g/cm^3)	0.85	0.8-0.9

The iodine value is an indication of the unsaturation of fats and oils. The greater iodine value shows the higher unsaturation of oils and fats (Hundie*et al.*, 2022). Oils with iodine values greater than 120 are grouped as drying oils; those with iodine values of 60–120 are grouped as semi-drying oils; and those with iodine values below 60 are regarded as non-drying oils. In this study, the iodine values of the millet bran oil were 122.3 mgI_2/g, which is close to the standard specified by ASTM D763 (120 mg I_2/g) for dry oil.

The ash content in oils is to indicate the quality of the oil. An ash content of 2.0% has been recorded for millet bran oil. Small ash content indicated that the extracted oil can be ignitable with a low amount of ash and this result is consistent with the result of Chugtai*et al.*,(2015).

Density is an important parameter for oil used in diesel fuel injection systems. Densities were obtained as 0.85 g/cm^3 for millet bran oil.

CHARACTERIZATION ANALYSIS OF MILLET BRAN OIL

FT –IR Analysis

To characterize the chemical composition of the extracted oil from millet bran, FT-IR spectroscopy was performed. The FT-IR spectra provide insights into the functional groups present, helping to confirm the molecular structure of the extracted oil. Table 2 presents the wavenumber values and their corresponding functional group assignments.

Table 2: FT-IR Spectral Data for the extracted oil of millet bran

Wavenumber(cm^{-1})	Functional Group	Vibration Type	Assignment
1744.4	C=O	Stretching	Ester group
2922.2	=C–H	Asymmetric stretching	Hydrocarbon
2855.1	$–CH_2$	Symmetric stretching	Hydrocarbon
1461.1	$–CH_2$	Bending vibration	Hydrocarbon
1237.5	$–CH_2$	Bending vibration	Hydrocarbon
1162.9	$–CH_3$	Bending vibration	Hydrocarbon

The oil extracted from millet bran comprises long–chain fatty esters, the strong peak observed at 1744.4 cm^{-1} is assigned to C=O, which typically belongs to esters. The peaks located at 2922.2 and 2855.1 cm^{-1} confirmed the presence of =C–H asymmetric stretching vibrations and $–CH_2$ symmetric stretching vibrations, respectively, in the oil. The peaks located at 1461.1 and 1237.5 cm^{-1} indicate bending vibrations of $–CH_2$, and the point located at 1162.9 cm^{-1} indicates the bending vibrations of $–CH_3$, and this result is inconsistent with that of (Velasco *et al.*, 2006).The corresponding FT-IR spectrum for the extracted oil is presented in Figure X, confirming the functional group assignments listed in Table 2.

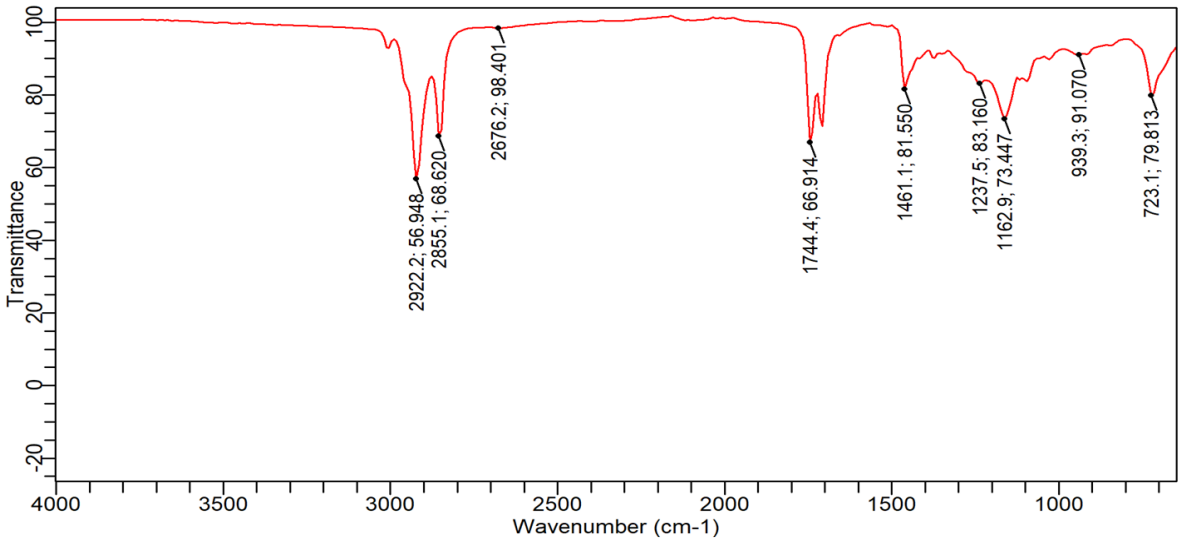

Figure 2: FTIR spectrum for millet bran oil.

GC –MS Analysis of oil from millet bran

The GC-MS chromatogram of millet bran oil provides a visual representation of the detected volatile compounds, highlighting their retention times and relative intensities. By comparing the observed spectral data with the NIST Mass Spectral Library, the chemical constituents of the oil can be identified. The peaks in the chromatogram correspond to various compounds, including fatty acids, ketones, esters, and other volatile compounds, which are further analyzed for their molecular structures and chemical properties (Figure 3).

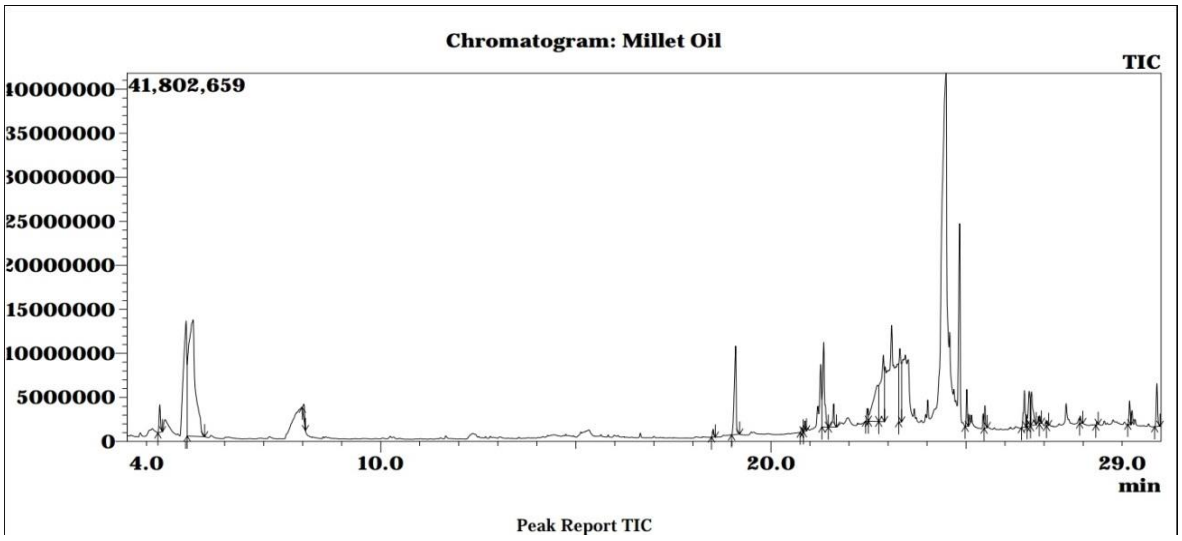

Figure 3. GC-MS Chromatogram of millet bran oil

GC-MS analysis identified 23 volatile compounds in millet bran oil, belonging to different chemical classes, including fatty acids, ketones, esters, aldehydes, benzene derivatives, alcohols, hydrocarbons, acids, and furans. The chromatogram (Figure 3) facilitated the spectral comparison of each compound with NIST library data, confirming their identities. Among the identified constituents, 9,12-Octadecadienoic acid, methyl ester was the predominant fatty acid, aligning with the findings of Felicia and Deborah (2021). The molecular structures of these compounds are depicted in Figure 4, providing fur insight into their chemical composition.

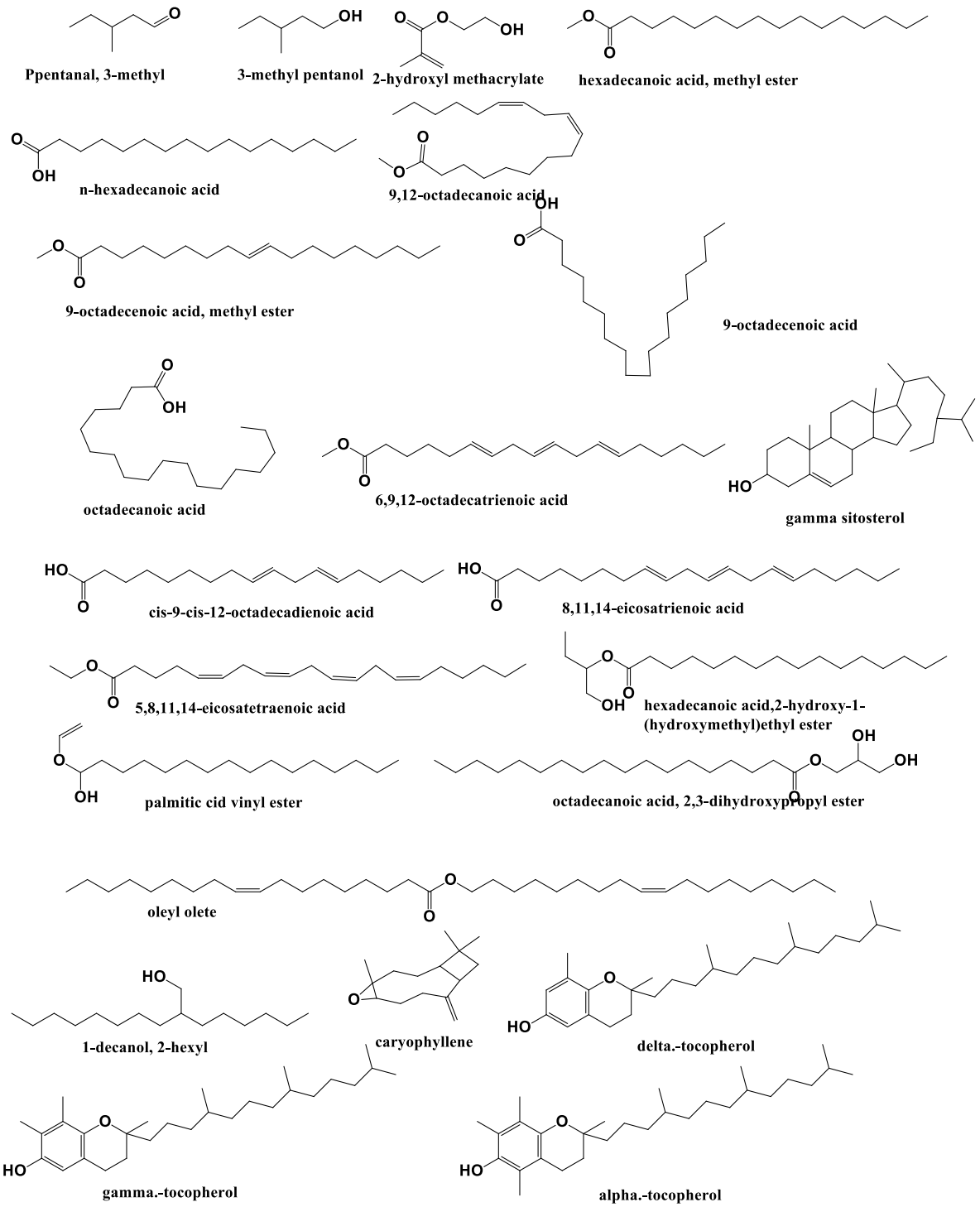

Figure 4: Identified compounds from the GC-MS spectrum of millet bran oil

Oil Yield at different extraction time

The effect of timeon oil yield is shown in Table 3. The oil yield shows a significant increase from 8.50% to 10.78% with a rise of timefrom70minsto 100mins.

Table 3: Oil yield at different temperatures

S/No	Time(mins)	Oil Yield %
1	70	8.50
2	80	9.45
3	90	10.39
4	100	10.78

The oil yield in Table 3, shows a significant increase from 8.50% to 10.78% with the increase in the extraction time from (70min to 100min). After the extraction, the highest yield (10.78%) of oil has been recovered with the possible valid optimum conditions: Amount of substrate 12g, and Time interval of 100min.The result obtained is in correlation with the one reported by Akinhanmi*et al.* (2008).

KINETICS AND THERMODYNAMICS STUDY OF MILLET BRAN OIL EXTRACTION

Kinetics study of millet bran oil extraction

The kinetics of millet bran oil extraction was studied using the optimum conditions of the substrate and varying reaction temperatures while adjusting the time interval (70–100 min). The percentage of oil yield (Y) was determined for each condition. Table 4 presents the oil extraction yield at different reaction temperatures over time, highlighting how both temperature and duration influence extraction efficiency.

Table 4: Percentage of oil extraction yield at various reaction temperatures

Time (min)		% Oil Yield(Y)	
	303 K	313 K	323 K
70	8.50	10.31	11.34
80	9.45	11.48	12.70
90	10.39	12.61	14.03
100	10.78	13.23	15.08

Table 5: Data for determination of rate constant of reaction

Time (min)		ln Y	
	303 K	313 K	323 K
70	2.14	2.33	2.43
80	2.25	2.44	2.54
90	2.34	2.53	2.64
100	2.38	2.58	2.71

The rate constants (k) at different temperatures are acquired from the slope of the plot of ln Y versus time.

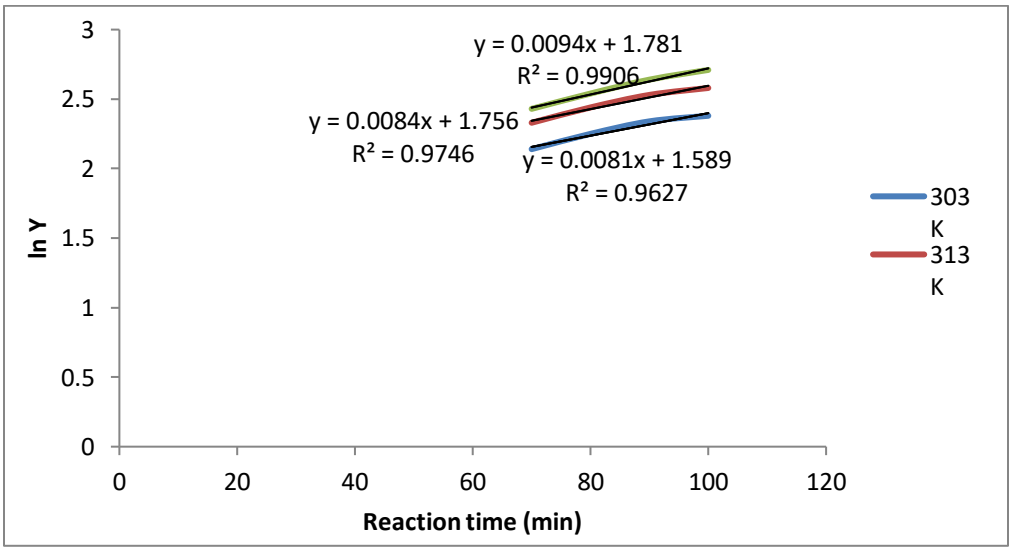

Figure 5: Plot of ln Y against reaction time at 303, 313, and 323 K

From Figure 5 plot, the rate constant at different temperature was determined as follows:
k = 0.0081 at 303K
k = 0.0084 at 313K
k = 0.0094 at 323K

The linear nature of these plots signifies that the reaction follows a pseudo-first-order rate law, which is in **tandem** with what Atkins stated (Atkins *et al.*, 2000).

Table 6: Data for the kinetics study, the rate constants, and R^2 values

Temperature (K)	Time (min)	Yield (%)	ln Y	k	R^2
303	70	8.50	2.14	0.0081	0.9627
	80	9.45	2.25		
	90	10.39	2.34		
	100	10.78	2.38		
313	70	10.31	2.33	0.0084	0.9746
	80	11.48	2.44		
	90	12.61	2.53		
	100	13.23	2.58		
323	70	11.34	2.43	0.0094	0.9906
	80	12.70	2.54		
	90	14.03	2.64		
	100	15.08	2.71		

Activation Energy (Ea) for Millet Bran Oil Extraction

The activation energy (Ea) for millet bran oil extraction was determined using the Arrhenius equation, which describes the temperature dependence of reaction rates. This equation establishes a relationship between the rate constant (k), activation energy, temperature (T), and the universal gas constant (R). A linear form is obtained by taking the natural logarithm of the Arrhenius equation, allowing for the calculation of E_a from the slope of the plot of ln k versus (1/T). The intercept of this plot corresponds to (ln A), where A represents the frequency factor, indicating how often molecules collide with the correct orientation to react. This approach provides insight into the energy barrier that must be overcome for the extraction reaction to proceed efficiently.

Table 7: Data for determination of Activation Energy

Rate constant (min^{-1})	Temperature (K)	ln k	1/T (K^{-1})
0.0081	303	-4.81589	0.00325
0.0084	313	-4.77952	0.00319
0.0094	323	-4.66705	0.00309

Relating the data in Table 7 with equation (7) will allow plotting the graph of the natural logarithm of reaction rate constant with reciprocal temperature to determine the activation energy from the slope.

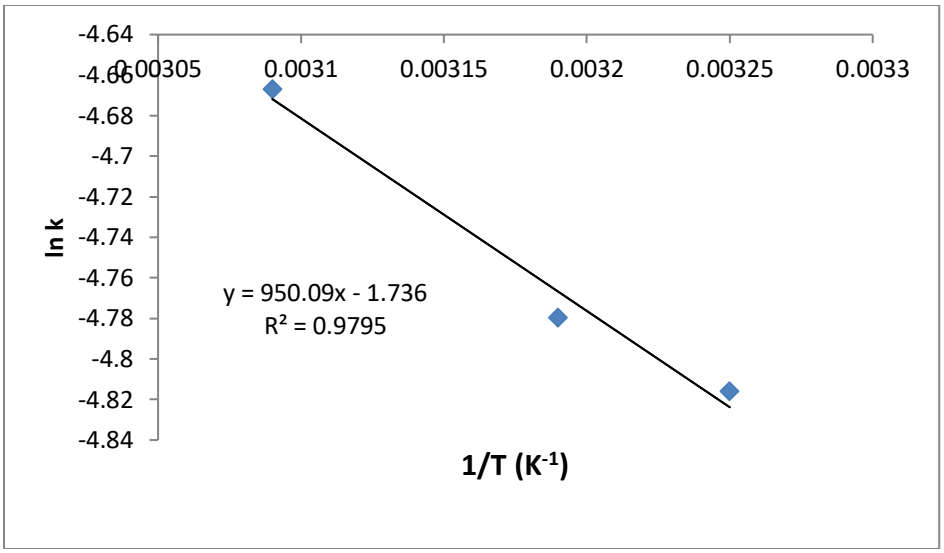

Figure 6: Arrhenius plot of the natural logarithm of reaction rateconstant with reciprocal temperature

Therefore, the activation energy value calculated from the Arrhenius equation is 7.899 kJmol^{-1}.

THERMODYNAMICS OF MILLET BRAN OIL EXTRACTION

The thermodynamic parameters of millet bran oil extraction provide insights into the energy changes associated with the process. These parameters can be determined using thermodynamic equations that relate the rate constant to temperature, enthalpy (ΔH), entropy (ΔS), and other fundamental constants (Menkiti*et al*., 2015).
The transmission coefficient (k*) is typically assumed to be 1, indicating that every successful molecular collision leads to a reaction. The Boltzmann constant (k_B) and Planck's constant (h) are used to describe the molecular interactions during the extraction process.
The equation (12) suggests that changes in temperature influence the reaction rate, which in turn affects the extraction efficiency. The relationship between the natural logarithm of the rate constant and the inverse of temperature allows for the determination of enthalpy and entropy, providing valuable information on the feasibility and spontaneity of the extraction process. Table 8 presents experimental data used to determine the thermodynamic parameters of millet bran oil extraction.

Table 8: Data for determination of thermodynamic parameters

Rate constant (min^{-1})	Temperature (K)	k/T × 10^{-5}	ln (k/T)	1/T (K^{-1})
0.0093	303	3.0194	-10.4079	0.00325
0.009	313	2.8754	-10.4567	0.00319
0.0077	323	2.3839	-10.6441	0.00309

Relating the data in Table 8 with equation (12) will allow plotting the graph of $ln\ \frac{k}{T}$ against $\frac{1}{T}$ to determine the enthalpy ΔH and entropy ΔS.

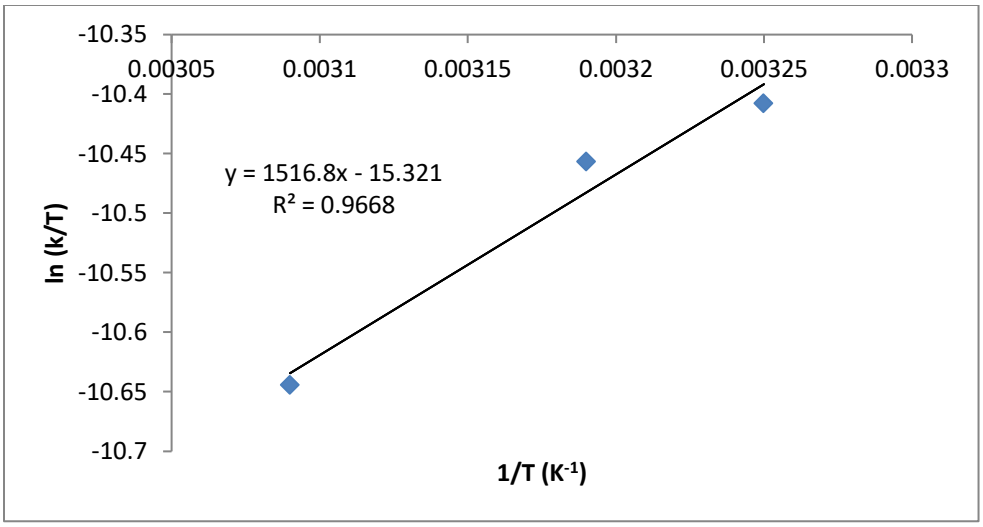

Figure 7: Plot of ln (k/T) versus T^{-1}

From the plot of ln (k/T) versus $T^{-1}$the enthalpy and entropy of the reaction was calculated. The calculated ΔH and ΔS values were +5413 $kJmol^{-1}$ and – 8.634$kJmol^{-1}$, respectively. The positive ΔH value indicates the endothermic nature of the reaction. At the same time, the negative value for ΔS proposes an associative mechanism. The calculated positive ΔG values (+2664.685 $kJmol^{-1}$, +2707.855$kJmol^{-1}$, and +2794.195 $kJmol^{-1}$at 303k, 313k, and 323k)revealed that the process was non – spontaneous signifying that external energy was added for the reaction to proceed. This is following the result obtained by Meziance and Khadi (2008) who studied the kinetics and thermodynamics of oil extraction from olive cake.

Table 7: Summary of kinetics and thermodynamics results

Oil extraction	Temp. (K)	Rate constant	Gibbs free energy, ΔG (kJ/mol)	Enthalpy, ΔH (kJ/mol)	Entropy, ΔS($kJmol^{-1}$ K^{-1})	Activation energy (kJ/mol)
303	0.0081	+2664.685	+5413	-8.634	+7.8990	
313	0.0084	+2707.855				
323	0.0094	+2794.195				

CONCLUSION

This study successfully analyzed some of the physicochemical properties with the kinetics and thermodynamics of pearl millet bran oil extraction. The oil yield, physicochemical properties, and analysis for characterization confirm the potential use of millet oil in industrial applications. The thermodynamic study indicates that the process is endothermic and non-spontaneous, requiring controlled conditions for maximum yield. Future research should explore alternative solvents and methods to enhance extraction efficiency.

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