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Extraction of Oil from *Tetrapleura Tetraptera (*Akpangbo*)* Seeds: A Kinetic and Thermodynamics Study.

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***Abstract-** Extraction of oil from Tetrapleura Tetraptera (Akpangbo) seeds using n- hexane as solvent was carried out at a temperature range between 45°C to 65°C while time was varied from 20 - 100 minutes. The results obtained showed that the yield of oil increased with increase in time and temperature at all experimental conditions. The maximum oil yield from seeds was 40.32%. Predictive models of Power and Exponential laws were used to evaluate the kinetic order and model of the extraction process parameters using experimental data. Power law model predicted the experimental results at temperatures of 45°C to 55°C with better sum of squares of residuals (R^2) maximum value of 0.99 while the sum of squares of residuals value for Exponential law model had a maximum value of 0.95. However, at a temperature range between 60°C to 65°C, exponential law model gave a better result and higher values of R^2 (0.99) than the Power law model (0.98) which goes to show that lower temperature favors power law model while higher temperature favors exponential law model as seen within the context of this study. The maximum deviation of power law model from experimental yield is 2.03% while that of exponential law is 1.58%. Hence, it is inferred that exponential law model is the most suitable model for predicting the yield of oil. Thermodynamic analysis was done in order to determine the enthalpy, entropy and Gibbs free energy of the seed which shows ΔH=13.89kJ/mol, ΔS=0.0423kJ/mol and ΔG= -1.0412kJ/mol. The findings suggest that oil extraction from Tetrapleura Tetraptera seeds is enhanced by increased temperature and time, consistent with an endothermic process. Thus, this study has provided broadened knowledge on the effects of kinetics and thermodynamic parameters aimed at minimizing the loss of oil and time, amount of energy expended and cost of production during solvent extraction processes.*

***Keywords:** Extraction, Tetrapleura Tetraptera, Kinetics and Thermodynamics*

1. INTRODUCTION

Tetrapleura Tetraptera (T.T) commonly known as Akpangbo in Ikwerre language, Aidan in western Nigeria, is a tropical deciduous forest tree with characteristics distinction. The fruit consists of woody shell, a fleshy pulp, and small brownish-black seed (Pamela *et al.* 2011). So many African countries have different names for *Tetrapluera Tetraptera(*Akpangbo*)*. For example, in Ghana it is called Preskese, the Yorubas in Nigeria call it Aidan, Efiks call it Edeminang, the Igbo part of Nigeria call it Osakirisa or Obogolo, the Ibibio call it Uyayak, Hausas call it dawo. It is a tropical forest tree that sheds seasonally at a certain stage of development in its life cycle. It is brownish black in color. The seed has medicinal values as seen in literature. It is used for the control of an array of human hypertension, epilepsy, asthma, diabetes among others (Asad Samani *et al.,* 2015).The plant stretches to the height of (25 - 30m) and has greenish fair resembling orchids with scented heady taste. It is steamy workshop and its underground stem is second hand for cooking and curative purposes (Mishra & Gangele, 2013).

Extraction is the process of separating one or more components from a liquid mixture with the aid of a solvent that preferentially dissolves the required component (Agu *et al*., 2020). Oil extraction from seed is influenced by several factors like temperature, time, particles size and solvent volume/Feed ratio (Wami & Ezenwankwo, 2022). There are different methods of extraction used in extracting oil from seeds. However, in this study, soxhlet extraction was used because it is simple, allows the use of batch process and a low absorption. In addition, it improves yield and quality of the extract thus making it better for commercial use (Vaibhav & kakasaheb, 2019).

Several parameters such as particle size, volume of solvent/Feed ratio, operating temperature, and extraction time have been found to affect the percentage yield of oil from the seeds and fruits. Therefore, the determination of the effects of these parameters on oil yield from *Tetrapleura Tetraptera (*Akpangbo*)* fruits and seeds is important to minimize the loss of oil, amount of energy expended overtime and finally the cost of extraction. To meet the desired yield, the process variables can be optimized.

There are factors to be considered in choosing extraction methods which include stability to heat, the type of plant materials to be extracted, nature of solvent, duration of extraction and final volume required (Azwanida, 2015; Agyemang & Abdel-Samie, 2021). The type of material will also determine the extraction method to be used; heat stable materials use soxhlet extraction or microwave-assisted extraction, whereas plant materials that are not heat stable are extracted using maceration or percolation. (Pandey & Tripathi 2014).

The solvent, n-hexane is often used for *T.T* oil extraction because of its lower boiling point and non-polar nature for easy separation after extraction and it has low toxicity comparing to other solvents. The yield % of oil obtained by using n-hexane is high compared with ethanol from Neem oil. Several parameters such as Particle size, Volume of solvent/Feed ratio, operating temperature, and extraction time have been found to affect the percentage yield of oil from the seeds (Pandey & Tripathi, 2014).

Therefore, the determination of the effects of these parameters on oil yield from *T.T* Seeds is important to minimize the loss of oil, amount of energy expended overtime and finally the cost of extraction. To meet the desired yield, the process variables can be optimized. Also, knowing the kinetic and thermodynamics of *T.T* oil extraction process can be expended by reducing time and cost. Hence the current study includes extraction of oil from *T.T* Seeds using n-hexane to determine the time, volume of solvent/feed ratio, particle size and detail the kinetic and thermodynamic properties of T.T oil through Soxhlet extraction method. Below are the fruits and seeds pictures of T.T.

Tetrapleura Tetraptera fruit

Tetrapleura Tetraptera Seeds

Medicinal plants such as *T.T* are at great interest to the researchers in the field of living organisms or component to produce useful usually commercial products (biotechnology), as most of the drug industries depend on them for the production of Pharmaceutical compounds. *T.T* has so many health benefits and is used as a remedy for several diseases such as anti-inflammation properties (Ibiari *et al.,* 2010), contraceptive properties (Farah *et al.,* 2013), management of leprosy (Hasene *et al.,* 2017), management of convulsion and support for the cardiovascular system (Koma *et al.,* 2016). It is also used to prepare several traditional dishes as popular seasoning spice in Nigeria. In most African countries, one is often asked to bathe with water soaked with the fruit of *T.T* in order to be relieved from feverish conditions (Erukainure, 2017).

Amin *et al.,* (2010), considered the thermodynamic and kinetic modeling *of Jatropa Curcas* seed using hexane solvent, also Wami & Ezenwankwo (2022) investigated the kinetics and thermodynamics of cashew nut seed at different extraction temperatures and time. Balaji *et al.* (2014) reported how effective ethanol is compared to n-hexane, petroleum ether, and chloroform in extraction of oil from Tamarindus indica edible fruit. Further studies by Nwabanne (2012), on kinetics and thermodynamics of oil extraction from fluted pumpkin seed shows the influence of various parameters such as temperature, time, volume of solvent and particle size on oil yield with increase in temperature, time and volume of solvent but decreased with increase in particle size. Babar *et al* (2015) were able to show the possibility of extracting Thevetiaperuviana oil from its seeds by cold and soxhlet extraction methods, using petroleum ether as extracting solvent at room temperature and 40 to 60°C respectively. Characterization of the oil showed that the percentage oil yield, acid value, saponification value and viscosity increased as temperature of soxhlet extraction increased. A research work on extraction of oil from bitter gourd seeds using soxhlet apparatus with n-hexane as a solvent said that the effect of extraction time, volume of solvent and particles size of bitter gourd on oil yield was estimated and observed that, the oil yield was increased with increase in time and volume of solvent but decreased with increase in particle size and the kinetics and the thermodynamic studies revealed that the process is endothermic and requires energy (Umamaheshwari & Dinesh 2014).

Samuel & Francis (2019) studied kinetics and thermodynamics on Moringa oleifera seeds which produces oil from extraction and converted to biodiesel. However, the kinetics and thermodynamics of this viable was seldomly investigated. It was observed that the biodiesel kinematic viscosity (3.75 ± 0.04 mm^2/s), cetane number (67.12), oxidative stability (15.2 ± 0.5), acid value (0.012 mg/KOH), pour point (9°C) and carbon residue satisfied the (ASTM) limits.

The production of oil from *Tetrapleura Tetraptera* seeds using n-hexane and its characterization as well as the development of kinetic and thermodynamic models are lacking in literature. This undoubtedly is the knowledge gap this paper tends to bridge.

The objective of this study is to develop the kinetics and thermodynamics models taking into consideration the extraction of oil from T.T seeds using n-hexane as solvent. The study considered the estimation of kinetic model for predicting the yield of oil for power and exponential models via curve fitting technique while the thermodynamic analysis was carried out to determine the enthalpy, entropy and Gibbs free energy of *T.T* seeds during oil extraction.

II. MATERIALS AND METHODS

2.1Materials

The following materials were used during this work; thimble, scissors, whatman filter paper, grinded T.T, sample bottles, pycnometer, cellotape, clamp/retort stand, oven, weighing balance, soxhlet extractor, heating mantle, condenser, grinder, capillary viscometer, dean and stark apparatus, stop watch, beaker, round bottom flask (500ml), distilled water, n-hexane

2.2 Methods

2.2.1 Sample Collection and Preparation

Tetrapleura Tetraptera(Akpanbo) was procured from Rumuwoji market popularly known as mile 3 market Port Harcourt, Rivers State of Nigeria. The seeds were cut in piecemeal, washed thoroughly with distilled water, oven dried for 24 hours. It was cooled in a dessicator after which was ground using a grinding machine to a particle size and sieved through different Tyler screens to get the particle size of 1.88mm. The samples were stored in a separate air tight containers and labeled adequately.

2.2.2 *Soxhlet Extraction Experiment*

The experiment was carried out in a Soxhlet Extractor using n-hexane as solvent. The sample was weighed in 10g, 15g, 20g, 25g and 30g and was charged each time in the thimble of the Soxhlet Extractor. The condenser ensures that the solvent vapour cools and drips back down into the chamber housing the solid material. The heating mantle is set at a specified temperature which varied from 45, 50, 55, 60 and 65^{0}C at a different extraction temperature and at different extraction time between 20 - 100 minutes. When the liquid solvent level reaches the top of the siphon tube, the solvent - solute mixture (the extract), is siphoned. The kinetic and thermodynamics of *T.T* is studied at constant particles size of 1.88mm, and 250ml volume of solvent, while the temperature varied at 45, 50, 55, 60 and 65°C. The extracted oil plus solvent were collected and distilled at 60-80^{0}C to separate the solvent from extracted oil (Wami and Ezenwankwo, 2022). N-hexane with lower boiling point temperature distilled out leaving only the oil in the distillation flask. The recovered oil was weighed and the yield determined as given in equation (1) below:

$$\%\,yield = \frac{\text{weight of oil extracted (g)}}{\text{weight of initial sample (g)}} \text{ x } 100 \quad (1)$$

2.2.3 Development of models

2.2.3.1 *Power Law Model*

The power law model expressed is consistent with literature (Wami & Ezenwankwo, 2022):

$$y = kt^n \, (2)$$

$$where\ y = percentage\ of\ oil\ yield\ (\%)$$
$$t = extraction\ time\ (\text{min})$$
$$k = extraction\ rate\ constant\ (\ min^{-1})$$
$$n = power\ index$$

The rate constant and power index were determined by linearizing Equation (2) as follows:

$$\ln y = \ln k + nlnt \qquad (3)$$

A plot lny against lnt gives a slope equivalent to n and $intercept = lnk$; where *k* is the characteristic constant incorporating the active coefficients, while the power index n, is the diffusion exponent, which indicates the transport mechanism of oil and it is less than 1 $(n < 1)$ in most oil extraction process (Agu *et al.*, 2020).

2.2.3.2 Exponential Law Model

A generalized exponential law model equation which took into account the variation of the extraction rate constant (*k* values) with extraction temperature and time was obtained from the plot of Equation (3) at the various extraction temperatures. This is expressed as.

$$y\ = k = \ Ke^{-nt} \qquad (4)$$

where $y = percentage\ of\ oil\ yield\ (\%)$

$$k = extraction\ rate\ constant\ (min^{-1})$$
$$t = extraction\ time\ (\text{min})$$
$$n = power\ index$$
$$y = ke^{-nt}$$

Linearizing equation (4) gives

$$\ln y = \ln k - nt \qquad (5)$$

2.2.3.3 Thermodynamics of Oil Extraction

The thermodynamic parameter such as entropy, enthalpy and Gibbs free energy were evaluated to determine the thermodynamics of *Tetrapleura Tetraptera* (Akpangbo*)* extraction. The relationship between entropy, enthalpy and the Gibbs free energy can be stated as follows:

$$\Delta G\ =\ \Delta H - T\Delta S \qquad (6)$$

where

$$\Delta G\ = change\ in\ Gibbs\ free\ energy\ (KJ/mol)$$
$$\Delta H = change\ in\ enthalpy\ (KJ/mol)$$
$$\Delta S = change\ in\ entropy\ (KJ/mol.K$$
$$T = Temperature\ (K)$$

But from thermodynamics relation, ΔG can be expressed as

$$\Delta G = -RTInK_e \qquad (7)$$

where

$$R = Gas\ constant\ (8134J/molK$$
$$k_e = equilibrium\ constant$$

Hence combining equation (6) and (7) gives

$$RTlnk_e\ =\ -\Delta H + T\Delta S \qquad (8)$$

Dividing all terms in equation (8) by *RT* gives

$$lnk_e\ = \frac{\Delta S}{R} - \frac{\Delta H}{R}\left(\frac{1}{T}\right) \qquad (9)$$

A plot of lnk_e versus $\frac{1}{T}$gives slope = - $\frac{\Delta H}{R}$ and intercept = $\frac{\Delta S}{R}$

k_e will be obtained as the ratio of oil yield at temperature, T, to the percentage of yield left after

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extraction at the same temperature (Nwabanne, 2012; Jaba, *et al* 2015; Umamaheshwari & Dinesh 2014). This is expressed as:

$$k_e = \frac{y_o}{y_c} \qquad (10)$$

The percentage of yield left after extraction at temperature T, is obtained as:

$$Yield\,(\%) = \frac{weight\ of\ cake\ after\ extraction\ (g)}{weight\ of\ particle\ (g)}\ x\ 100 \quad (11)$$

III. RESULTS AND DISCUSSION

3.1 *Yield of Oil Extracted from Tetrapleura Tetraptera (Akpangbo) Seeds at different Temperatures using n-hexane as Solvent*

Table 1: shows the yield of oil extracted from *T.T* Seeds using n-hexane

Time (min)	45°C	50°C	55°C	60°C	65°C
20	0.1225	0.1502	0.1603	0.1923	0.2036
40	0.2139	0.2345	0.246	0.2515	0.2613
60	0.2714	0.2819	0.2936	0.3013	0.3212
80	0.3346	0.3418	0.3525	0.3616	0.3719
100	0.3817	0.4018	0.4225	0.4343	0.4532

Table 1 shows the tabulated values of the yield at different temperatures and time whereas Figure 1 shows the profiles of the percentage yield of oil extracted from (*T.T)* seeds using n-hexane as solvent with sample weight (10g, 15g, 20g, 25g, 30g) at different extraction times (20, 40, 60, 80, 100 minutes) and temperatures (45°C, 50°C, 55°C, 60°C, 65°C) respectively. It was observed that the yield of the oil extracted increased as the extraction time increased. At a temperature of 45°C the yield of oil increased from 12 to 38% after 20 minutes to 100 minutes. At a temperature of 50°C, the yield of oil increased from 15 to 40%. At 55°C, the yield of oil increased from 16 to 42%. At 60°C, the yield of oil increased from 19 to 43%, and at 65°C, the yield of oil increased from 20 to 45%. This is consistent with literature (Koma *et al.*, 2016).

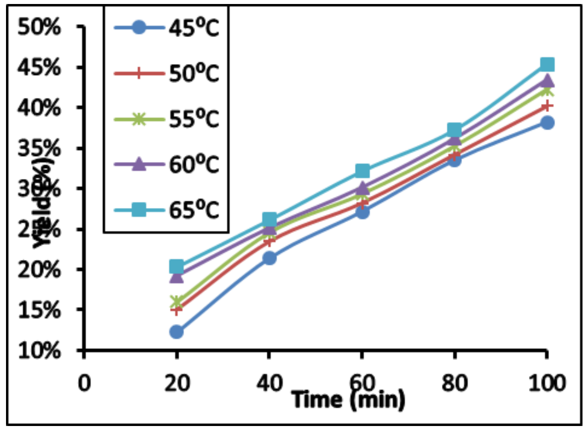

Figure 1: Percentage yield of oil extracted from (*T.T)* seeds using n-hexane solvent

3.2 Kinetic Analysis

3.2.1 Power law model

Figure 2: shows the Evaluation of Power Law Model Constants for Oil Extracted from *T. T* Seeds using n - hexane Solvent, The power law index n and the constant coefficient k, were determined by fitting experimental results into the power law model equation and then linearizing the model equation which was then plotted as shown in Figure 2 for temperature range of 45°C to 65°C. The trend in this result is in line with literature (Wami & Ezenwankwo, 2022).

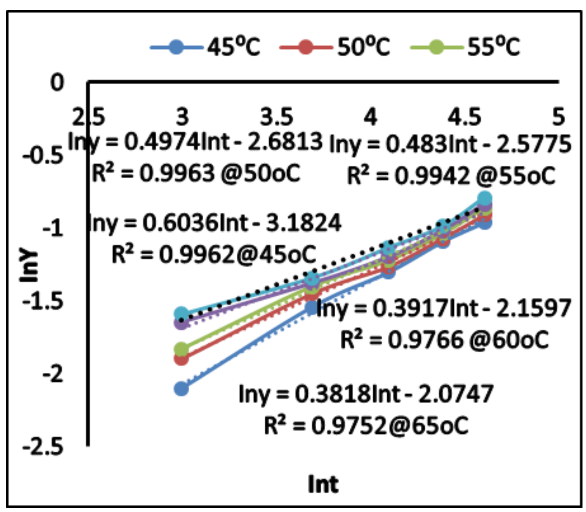

Figure 2: Power Law Model Constants for Oil Extracted from *T. T* Seeds using n - hexane Solvent

3.2.2 *Power Law Kinetic Parameters*

Table 2 below summarizes the values of the kinetic parameters obtained from the power law kinetic law

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during the extraction of oil from T.T Seeds using n-hexane solvent.

Table 2: Values of power law kinetic parameters obtained from T.T. seeds

Temp (oC)	K	n	R2
45	0.0415	0.6036	0.99
50	0.0686	0.4974	0.99
55	0.0760	0.483	0.98
60	0.1154	0.3917	0.98
65	0.1256	0.3818	0.98

3.2.3*Exponential Law Model*

Figure 3 shows the Exponential Law Model Evaluation for *T.T* seeds using n-hexane solvent. The exponential law index n and the constant coefficient k, were determined by fitting experimental results into the power law model equation and then linearizing the model equation which was then plotted as shown in Figure 4 for temperature range of 45°C to 65°C.

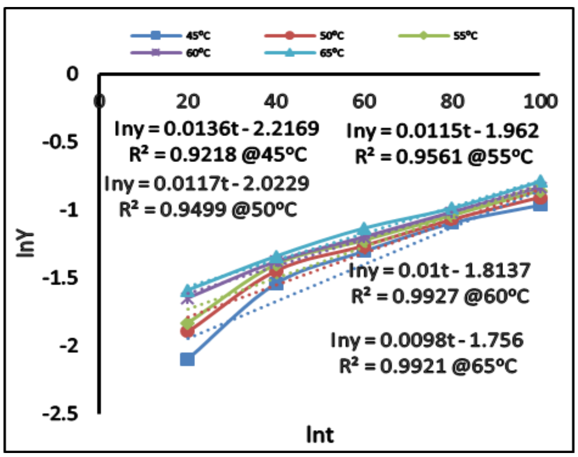

Figure 3: Exponential Law Model Constants from *T.T. s*eeds using n-hexane solvent

Table 3 below summarizes the values of the Exponential law kinetic parameters obtained from oil extraction from *T.T* seeds using n-hexane solvent.

Table 3: Values of Exponential law kinetic parameters obtained from T.T seeds

Temp (°C)	k	N	R^2
45	0.1089	0.00136	0.93
50	0.1323	0.0117	0.95
55	0.1406	0.0115	0.96
60	0.1630	0.0100	0.99
65	0.1727	0.0098	0.99

3.3*Comparison of Prediction Model with Experiment for Yield of Oil*

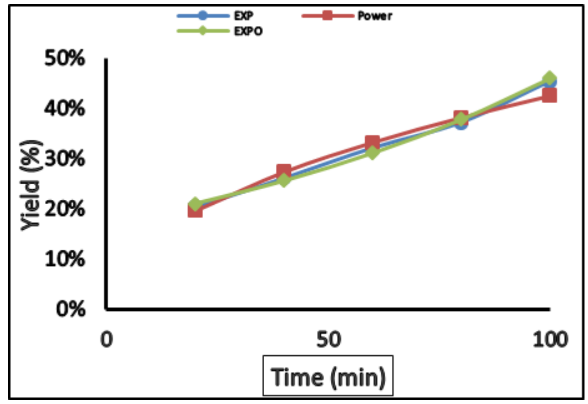

Figure 4: shows the degree of oil yield model prediction comparison with experimental yield of oil extracted from *T.T* seeds using n-hexane solvent at 65°C.

The profile plot indicates clearly that both power law and exponential law model fits closely with the experimental yield. The maximum deviation of power law model from experimental yield is 2.03% while that of exponential law is 1.58%. Hence, it is inferred that exponential law model is the most suitable model for predicting the yield of oil.

3.4 Thermodynamics Analysis

Oil extraction involves heating and hence there is exchange and transfer of heat from the solvent to the solid seed particles. Therefore, the thermodynamic analysis was carried out to determine the enthalpy, entropy and Gibbs free energy of the seed which shows ΔH=13.89kJ/mol, ΔS=0.0423kJ/mol and ΔG= -1.0412kJ/mol. The values of the enthalpy and entropy changes were all positive. A positive enthalpy value shows that the process is endothermic and indicates that heat is absorbed into the seeds to release the oil. The positive value of enthalpy indicates that the extraction of *T.T* fruit is an endothermic process, also, positive entropy implied that the extraction process was irreversible. The trend in this result is in line with literature (Wami & Ezenwankwo, 2022).

IV. CONCLUSION

Extraction of oil from Tetrapleura Tetraptera (Akpangbo) seeds using n- hexane as solvents was carried out at temperature range between 45°C to 65°C while time was varied from 20 – 100 minutes. The results obtained showed that the yield of oil increased with increase in time and temperature at all experimental conditions. Predictive models of Power and Exponential laws which were used to evaluate the kinetic parameters as well as the thermodynamic model results showed good agreement when compared with experimental data.The profile plot indicates clearly that both power law and exponential law model fits closely with the experimental yield. The maximum deviation of power law model from experimental yield is 2.03% while that of exponential law is 1.58%. Hence, it is inferred that exponential law model is the most suitable model for predicting the yield of oil. The findings suggest that oil extraction from *Tetrapleura Tetraptera* seeds is enhanced by increased temperature and time, consistent with an endothermic process. Thus, this study has provided broadened knowledge on the effects of kinetics and thermodynamic parameters aimed at minimizing the loss of oiland time, amount of energy expended and cost of production during solvent extraction processes.

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