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Kinetics and Thermodynamics of Oil Extraction from Nigerian Cashew Nut Seed Using Petroleum Ether

Emenike N. Wami[1*] and Angela C. Ezenwankwo[1]
[1]*Department of Chemical and petrochemical Engineering, Rivers State University, Nkpolu-Oroworukwo, Port Harcourt, Rivers State of Nigeria*
**Corresponding Author: Angela C. Ezenwankwo*

ABSTRACT
The kinetics and thermodynamics of Cashew Nut Seed oil extraction was investigated at different extraction temperatures and times. The power model and generalised modified power model (MPM) were used to study the oil yield. Thermodynamic properties of the oil extraction process were studied to determine the possibility and disorderliness of the process. The percentage yield of Cashew Nut Seed Oil was favoured by increase in extraction time and temperature. The optimal yield (43.77%) was recorded at temperature of 60 ºC and extraction time of 60 min. Both models fitted the experimental data, but the MPM proved to be more suitable to apply in oil seed extraction because it incorporated extraction temperature and time. The thermodynamic analysis revealed that cashew nut oil extraction has positive enthalpy (27.62kJ/mol), implying an endothermic process, while positive entropy (0.87kJ/mol.K) showed the extraction process is irreversible. The negative Gibbs free energy (-0.02 to -1.32kJ/mol) shows extraction of oil from cashew nut seed is possible. Therefore, application of the conventional and modified power models can be useful during design analysis of extraction system and also reduce cost, time wastage man hour loss.
KEYWORDS: Cashew Nut Seed Oil, Petroleum Ether, Extraction, Kinetics, Thermodynamics

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I. INTRODUCTION

Plants contain liquids that are useful for medicinal and food applications. Most of these liquids are refined to act as raw material for the production of domestic and industrial products. Meanwhile, the seeds or nuts of plants are thrown away after the edible parts are eaten. Interestingly, most plant seeds have high oil content that can be harnessed and utilised in the industry or domestically as raw material for production of beautification products and food processing [1]. Application of plant oil, otherwise known as vegetable oil, is gaining tremendous attention in recent times. Vegetable oil may be saturated or unsaturated compound with triglycerides of different chain lengths. Saturated triglycerides are single bond compounds of carbon and hydrogen atoms, while in unsaturated triglycerides are double bond compounds of carbon and hydrogen atoms [2].

Vegetable oils are classified as edible and non-edible. Edible oils are chiefly used for food preparation and production of pharmaceutical products, while the non-edible oils are generally used as raw materials for production of biodiesel [3], soap, perfumes and cosmetics [4], antioxidants [5] and bioactive compounds [6]. Vegetable oils produced for human consumption are extracted from non harmful sources such as soybean, sunflower, rapeseed, canola, cotton seed, avocado seed, peanut, coconut, shea butter and palm kernel seed among others plant's seeds [7][8][9], while non-edible oil from *Jatropha curcas L.*, *Pimenta racemosa*, *Lavender*, *Calotropis procera*, Sterculia apetala, *Rhus typhina L.* are used to produce biodiesel, perfumes, cosmetics, bio-lubricant and antioxidant among others [3][4][5][6][10][11][12]. Edible oils have equally been used to produce biodiesel and other non edible products [13][14].

Vegetable oil yield may vary between 3% and 70% of the total weight of seed, nut, kernel, or fruit pulp [15]. Seed processing can be beneficial to individual, organisations, and even to national economy as both the seed oil and the cake are of great commercial value [2].

However, oil extraction from seed is influenced by several factors like temperature, time, particles size and solvent volume/feed ratio. To meet the desired yield, the process variables can be optimised. Also, understanding the kinetics and thermodynamics of vegetable oil extraction process can equally reduce time and cost. Therefore, the kinetics and thermodynamics of cashew nut seed oil extraction was studied using the soxhlet extraction method at various extraction temperatures and times.

II. MATERIALS AND METHODS

The materials used in the experiment include cashew nut seeds, petroleum ether (solvent) with boiling point 60-80 ºC and distilled water. Also, the following laboratory equipment and apparatus were used in the study: soxhlet extractor, steam distillation apparatus, heater, water bath, G-clamp, flasks, beakers, pipette, burette, sample bottle, specific gravity bottle, weighing balance, grinder, Gas Chromatography Mass Spectroscopy (GC-MS) detector, pH meter, thermometer and stop watch, while the reagents used include acetic acid, hydrochloric acid, chloroform, potassium iodide, carbon tetra chloride (CCl_4), sodium thiosulphate, phenolphthalein, starch indicator, hydrochloric acid, sodium hydroxide and potassium hydroxide.

2.1 Sample Collection and Preparation

Cashew nut seeds were collected from Agbiligba Nanka community in Anambra State, transported and prepared at Chemical/Petrochemical Engineering Laboratory, Rivers State University, Port Harcourt, Rivers State of Nigeria. The nuts were cut open to expose the seeds and sundried for a week. Thereafter, the seeds were grinded to powdered form and sieved to a uniform particle sizes of 1.8 mm.

2.2 Procedures of Oil Extraction

The experimental procedures were adopted according to the work of Nwabanne [16]. The oil extraction process was carried out in soxhlet apparatus using petroleum ether as solvent. The kinetics and thermodynamics of cashew nut oil extraction process was studied at constant seed particles of size 1.8 mm and solvent volume of 300 ml, while temperature was varied from 45, 50, 55 and 60 ºC. 20 samples weighing 50 g each were prepared and divided into 4 groups with five samples each. All samples in Group I were heated to 45 ºC. Also, all samples in Group II, III and IV were heated to 50 ºC, 55 ºC and 60 ºC respectively at different extraction times of 20, 30, 40, 50 and 60 minutes. Firstly, one of the samples in Group I was placed in the thimble of the extractor, and 300 ml petroleum ether was poured into a round bottom flask. The flask was heated via a mantle set at 45 ºC to extract the oil in the seed particles. The extraction was stopped at 20 minutes. The extracted oil + solvent was collected and distilled at 70 – 80 ºC temperature to separate the solvent from the extracted oil. Petroleum ether with lower boiling point temperature was distilled out leaving only the oil in the distillation flask. The recovered oil was weighed and the yield determined. This process was repeated on the remaining four samples in Group I (samples heated to 45 ºC), but stopped at different extraction times of 30, 40, 50 and 60 minutes.

The procedure was repeated for samples in Group II, but heated to 50 ºC, while the extraction was stopped at 20, 30, 40, 50 and 60 minutes. Similarly, Group III samples were heated to 55 ºC while Group IV samples were heated 60 ºC, but the extractions were stopped at 20, 30, 40, 50 and 60 minutes. At end of each extraction, the distillation of the oil-solvent mixture was carried out to recover the extracted oil, which was weighed and the oil yield calculated. The oil yield in each experimental run was calculated using the formula stated in Equation (1).

$$Yield\ (\%) = \frac{weight\ of\ pure\ oil\ extracted\ (g)}{weight\ of\ particle\ (g)} \times 100\ \% \tag{1}$$

2.3 Kinetics and Thermodynamics of Oil Extraction

Various kinetic models have been proposed for the study of oil extraction from plants seeds. In this study, the power model and first order kinetic model were used to study the extraction of Cashew Nut Oil. Also, the thermodynamics of the extraction process was evaluated using established models.

2.3.1 Power Model

The power model is expressed as:

$$Y = kt^n \tag{2}$$

where: Y = Percentage of oil yield (%)

t = Extraction time (min)

k = Extraction rate constant (min^{-1})

n = Power index

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

$$\ln Y = \ln k + n \ln t \tag{3}$$

A plot $\ln Y$ against $\ln t$ gives a slope equivalent to n and intercept as k.
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 processes [17].

2.3.2 Modified Power Model

A generalised power 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:

$$Y = (aT + b)t^n \tag{4}$$

where: Y = Percentage of oil yield (%)
T = Extraction temperature (ºC)
a = Constant coefficient of temperature
b = Constant
t = Extraction time (min)
n = Power index

2.4 Thermodynamics of Oil Extraction

The thermodynamic parameters such as entropy, enthalpy and Gibbs free energy were evaluated to determine the thermodynamics of Cashew Nut oil extraction. The relationship between entropy, enthalpy and the Gibbs free energy can be stated as follows:

$$\Delta G = \Delta H - T\Delta S \tag{5}$$

where: ΔG = Change in Gibbs free energy (kJ/mol)
ΔH = Change in enthalpy (kJ/mol)
ΔS = Change in entropy (kJ/mol.K)
T = Temperature (K)

But from thermodynamics relation, ΔG can be expressed as:

$$\Delta G = -RT \ln K_e \tag{6}$$

where: R = Gas constant (8.134 J/mol.K)
K_e = Equilibrium constant (-)

Hence, combining equations (5) and (6) gives

$$RT \ln K_e = -\Delta H + T\Delta S \tag{7}$$

Dividing all terms in equation (7) by RT gives

$$\ln K_e = \frac{\Delta S}{R} - \frac{\Delta H}{R}\left(\frac{1}{T}\right) \tag{8}$$

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

K_e was obtained as the ratio of oil yield at temperature, T, to the percentage of cake left after extraction at the same temperature [16][18][19]. This is expressed as:

$$K_e = \frac{Y_o}{Y_c} \tag{9}$$

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

$$Cake\ Yield\ (\%) = \frac{weight\ of\ cake\ after\ extraction\ (g)}{weight\ of\ particle\ (g)} \times 100\ \% \tag{10}$$

III. RESULTS AND DISCUSSION

The kinetics and thermodynamics of cashew nut oil extraction process have been studied at constant particle size of 1.8mm, 300ml solvent volume and various extraction times and temperatures.

3.1 Yield of Cashew Nut Oil at Different Temperatures

Figure 1 shows the profiles of the percentage yield of cashew nut oil obtained at extraction times of 20 to 60 min and temperatures of 45ºC to 60ºC. The amount of cashew nut oil, evaluated in terms of percentage of oil recovered after extraction/distillation processes, increased as extraction time was increased. Also, for a given extraction time, the yield of oil increased with increase in temperature. Thus, the oil yield obtained between extraction time of 20 min and 60 min at 45ºC ranged between 27.29% and 34.58%. Similarly, at temperatures of 50, 55 and 60ºC, the yield of oil obtained ranged from 30.04 – 40.02%, 33.22 – 42.12% and 34.72 – 43.77% respectively, at extraction time of 20 to 60 min. This implied that the highest percentage of oil yield was obtained at 60 ºC and 60 min extraction time.

Between 50 and 60 min extraction time, particularly at extraction temperatures of 55 ºC and 60 ºC, the difference in the amount of oil recovered was only about 3 – 4%. This indicated that allowing the extraction proceed beyond 60 min may not resulted to significant yield at higher temperatures. Thus, some studies have reported insignificant yield of oil within 60 min of extraction [20][21]. Within 20 min, the yield of oil from fluted pumpkin seed was reported almost exhausted at 80 to 100 ºC [16]. However, a significant yield of oil was recorded beyond an hour by some authors [18][19][22].

Also, the effects of temperature and extraction time on the oil yield as recorded in this work agreed with some previous studies on oil extraction from seed and nut of different plant species [23][24][25]. However, the maximum oil yield recorded after 60 minutes and temperature of 60 ºC for cashew nut oil extraction was less than the 49.1% reported by Akinhanmi *et al.* [26], but above the percentages reported by some authors [27][28][29]. This implied that there is variation in the amount of oil yield from same type of plant seed, leaf, nut or fruit. This scenario was observed in oil extracted from jatropha seeds collected from different geographical locations in Ethiopia, which was attributed to genetic variations and/or relative differences in the environmental conditions surrounding the seed [30].

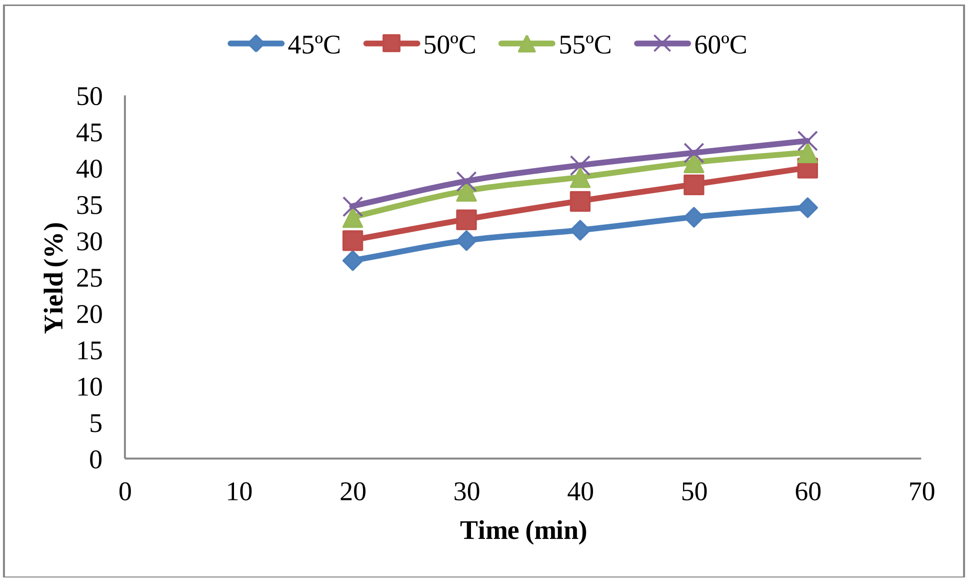

Fig. 1: Oil yield versus extraction time at different temperatures

3.2 Kinetic Analysis of Oil Extraction from Cashew Nut

The extraction of vegetable oil from plant seeds and nuts has been widely studied using mathematical expressions, but to apply the models, the constant coefficients need to be evaluated.

3.2.1 Evaluation of Power Model Constants

The power index, n and the constant coefficient, k, were determined by fitting experimental results into equation (3) and then, plotted as shown in Figure 2. The values of n and k were obtained by comparing the linear regression equation in Figure 2 with Equation (3) at temperatures of 45 – 60ºC. Thus, at extraction temperature of 45ºC, n and k were obtained as 0.2124 and 14.4776min^{-1} respectively. After substituting into equation (2), gave the percentage yield of oil from the cashew nut seed at 45ºC was expressed as: $Y = 14.4776\, t^{0.2124}$.

At 50ºC extraction temperature, *n* and *k* were obtained as 0.2593 and 13.7138min^{-1}. Hence, The percentage yield of cashew nut seed oil at 50ºC is $Y = 13.7138\ t^{0.2593}$. At 50ºC extraction temperature, *n* and *k* were obtained as 0.2145 and 17.5772min^{-1} respectively, which upon substitution into equation (2) gave the percentage yield as: $Y = 17.5772\ t^{0.2145}$. Finally, at 60ºC extraction temperature, *n* and *k* were obtained as 0.2085 and 18.6734min^{-1} respectively, which upon substitution into equation (2) gave the percentage yield of cashew nut seed oil as: $Y = 18.6734\ t^{0.2085}$. The characteristic constant, *k*, and the diffusion exponent, *n*, varied with temperature. Hence, they are temperature dependent. Also, from the kinetic evaluations, it can be seen that the correlation coefficients, R^2 at the various temperatures are above 99%. The high correlations are indication of good agreement between the fitted experimental data and the model.

The value of power index, *n* has been reported differently by different authors. A range of 3 – 4 was reported in some studies for pumpkin seed [16] and aegyptiaca kernel [22] though, at higher extraction temperatures or longer extraction time. At extraction temperature of 60ºC and 60 min extraction time, Dagde and Okure [31] obtained 0.84 and 0.81 as the power index and characteristic constant, *k* at 0.8917 and 1.2575min^{-1} for *Luffa cylindrica* oil and *Hura crepitan* seed oils respectively.

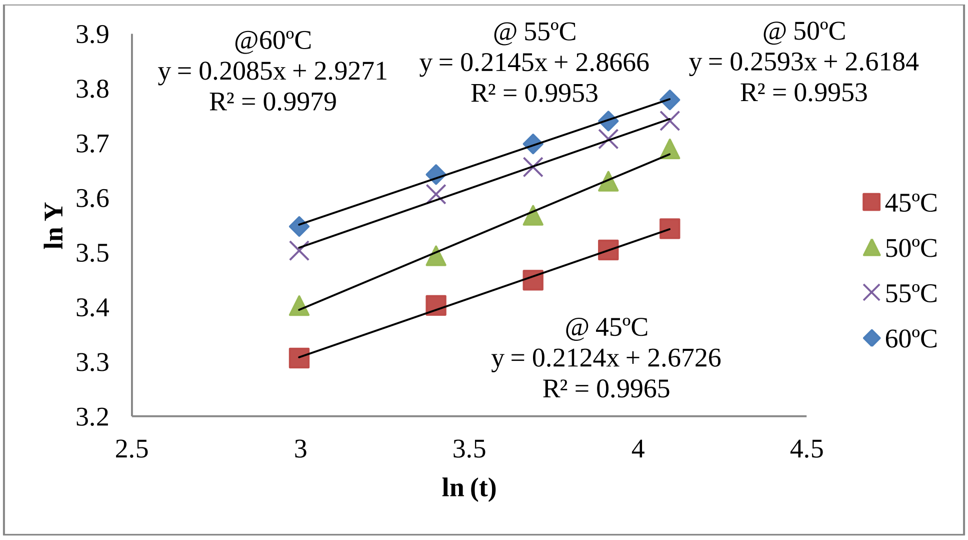

Fig. 2: Plot for evaluation of power index and constant coefficient

3.2.2 Modified Power Model

By plotting the different values of characteristic constant, *k* obtained at the various extraction temperatures in Figure 3, the relationship between *k*-values and extraction temperature and time was readily established to obtain a modified power model equation. Thus, by comparing equation (4) and the regression equation in Figure 3, the modified power model equation (MPM) was obtained as: $Y = (0.284\ T + 1.761)t^{0.21}$. This is a convenient generalised expression that enables the prediction of extraction yield at different temperatures and extraction time.

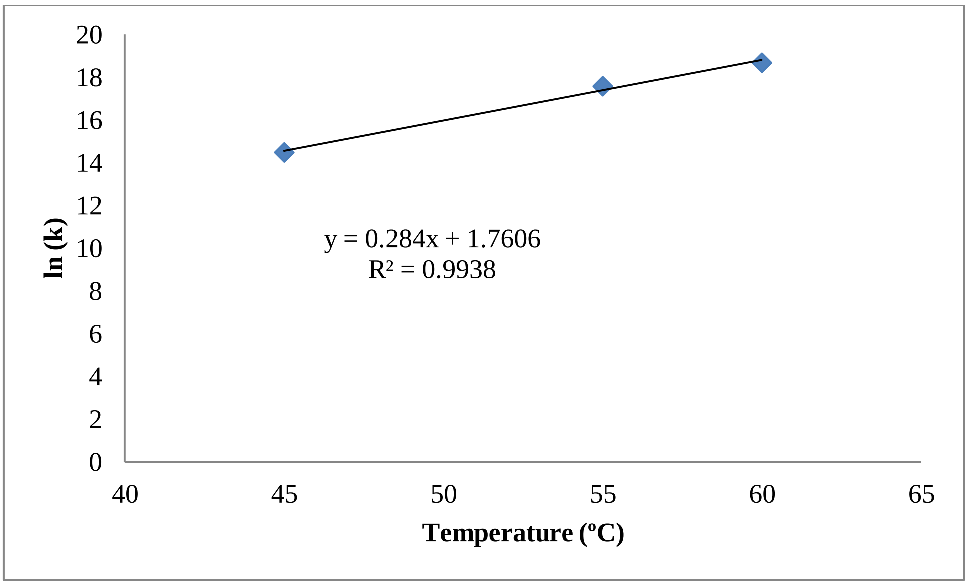

Fig. 3: Plot for evaluation of constant coefficient in the modified power model equation

3.3 Comparison of Model of Predicted Oil Yield

Table 1 shows the predicted yields and deviations from the experiment at extraction temperature of 60 ºC and extraction times 0f 20 – 60 minutes.

Table 1: Comparison of Power and 1st Order Kinetic Models Prediction at 60ºC

Time	Y (%)			Deviation (%)	
(min)	Expt.	Power	MPM	Power	MPM
20	34.72	34.68	35.27	0.12	1.58
30	38.18	37.76	38.4	1.10	0.58
40	40.38	40.11	40.8	0.67	1.04
50	42.11	42.04	42.75	0.18	1.52
60	43.77	43.68	44.42	0.22	1.49

The degree of oil yield predictions by the power model and modified power model (MPM) equation after the determination of the constants and subsequent substitutions were tested at temperature of 60ºC and then, compared with the measured oil yield (Table 1). Figure 4 showed the profiles of the percentage yield against extraction time. The profiles indicated that both models are effective for the prediction of oil yield with time, and over 95% of yield calculated from the experiment were predicted by the models. The deviation between the values predicted by the power model and the experiment ranged from 0.12 – 1.10%, while that between the values predicted by the MPM equation and the experiment ranged from 0.58 – 1.58%. The modified power model which incorporated the effect of temperature variation had comparable error margin just like the conventional power model, but because it incorporated the two parameters that influenced the extraction process, it is most suitable for prediction application.

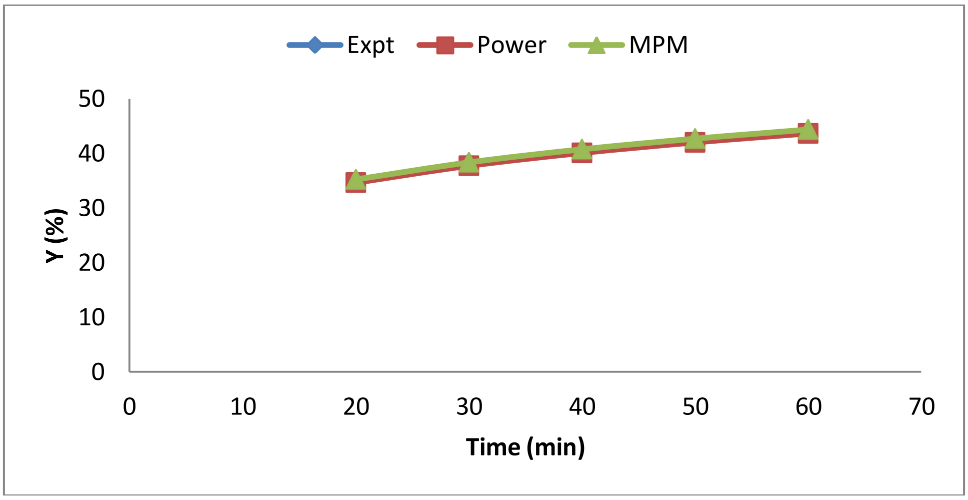

Fig. 4: Comparison of the degree of model predictions

3.4 Thermodynamic Analysis of Oil Extraction from Cashew Nut

Oil extraction involves heating, and hence, there is exchange and transfer of heat from the solvent to the solid seed particles. Therefore, the thermodynamics oil extraction was studied to describe the process enthalpy and entropy. Before the evaluation of the thermodynamic properties, the equilibrium constant was first determined using equation (14). The change in enthalpy (ΔH) and change in entropy (ΔS) were determined from the linear equation in Figure 5. Thus, by comparing equation (8) with the equation in the graph and equating the slope with the ratio of enthalpy to the universal gas constant, R, the value of ΔH was obtained as 27.62kJ/mol, while from the intercept, ΔS was obtained as 0.087kJ/mol.K. From the values of the enthalpy and entropy changes, the Gibbs free energy (ΔG) was evaluated for the given temperatures, which ranged from -0.0190 to -1.3228kJ/mol. The changes in enthalpy and entropy obtained in this study are close to values reported by Jabar *et al.* [18] for *Thevetia peruviana* seed oil. The corresponding values of the parameters for the thermodynamic evaluations are presented in Table 2.

The positive value of enthalpy indicates that the extraction of cashew nut oil is an endothermic process, which implies that energy was absorbed into the seed particle to release the oil. Also, the positive entropy implied that the extraction process was irreversible and there was increase in disorderliness of the liquid molecules in the process, while the negative values of Gibbs free energy shows that cashew nut oil extraction was spontaneous, and the extraction process was possible at the temperature range [17][20]. Various authors have reported different values of enthalpy, entropy and Gibbs free energy for different seed oils, but some authors reported positive values for enthalpy and entropy, and negative values for Gibbs free energy [16][18][19]. The changes in enthalpy, entropy and Gibbs free energy from literatures are summarized in Table 3. However, the changes in the thermodynamic properties differ according the operational parameters.

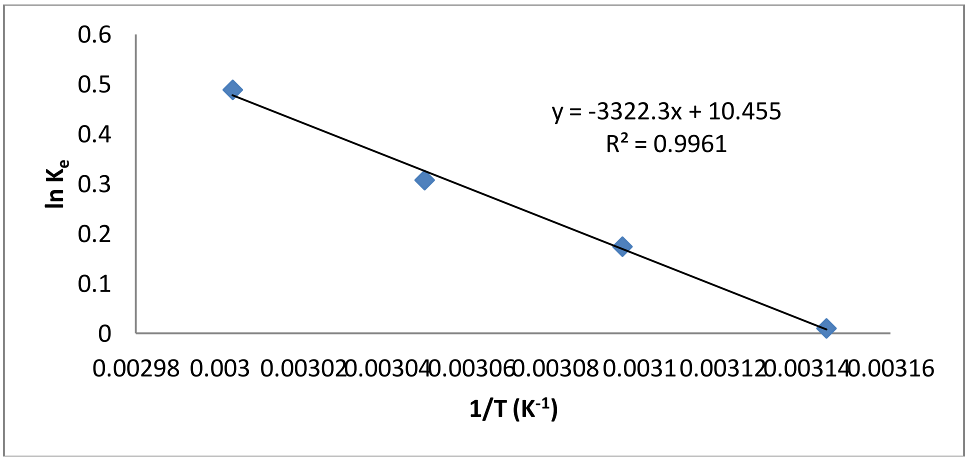

Fig. 5: Plot of lnK_e versus 1/T

Table 2: Thermodynamic Parameters at 60 Minutes Extraction

T (K)	Y (%)	Ys (%)	K= Y/Ys	1/T (K^{-1})	lnK	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (kJ/mol.K)
318	34.58	34.24	1.01	0.00314	0.00995	-0.019	27.6216	0.08692
323	40.02	33.63	1.19	0.00310	0.17395	-0.4536	27.6216	0.08692
328	42.13	30.98	1.36	0.00305	0.30748	-0.8882	27.6216	0.08692
333	43.77	26.85	1.63	0.00300	0.48858	-1.3228	27.6216	0.08692

Table 3: Ranges of Thermodynamic Properties for Seed Oil Extraction

ΔH (kJ/mol)	ΔS (kJ/mol.K)	ΔG (kJ/mol)	Oil Source	Author(s)
28.17	0.234	-3.90 to -8.91	Pumpkin seed	[16]
11.19	0.033	0.24 to 0.57	Coconut Seed	[32]
7.83	26.62	-0.86 to -1.31	watermelon seed	[33]
29.20	0.092	0.45 to -13.83	*Thevetia peruviana* seed	[18]
15.02	45.52	1.62 to -0.10	Jatropha seed	[20]
11.70	0.26	-2.49 to -1.95	Bitter gourd seed	[19]
372.05	1.29	-26.64 to -52.35	*Colocynthis vulgaris Schrad* seed	[34]
27.62	0.087	-0.02 to -1.32	Cashew nut seed	This work

IV. CONCLUSION

The extraction kinetics and thermodynamics of cashew nut seed oil was studied at different extraction temperatures and times. The study showed that the yield of cashew nut oil was favoured by increase in extraction time and temperature. The kinetics of cashew nut seed oil extraction is dependent on temperature and time. The power model and the modified model were capable of predicting the percentage of oil recovered from the extraction, but the incorporation of temperature and extraction time in the modified power model made it more suitable for prediction of extraction yield. Also, the thermodynamic analysis revealed that enthalpy change in the extraction process was positive, which implied that the cashew nut seed oil extraction was endothermic, while the positive entropy showed the extraction process is irreversible and disorderly. The negative Gibbs free energy implied the extraction of oil from cashew nut seed is possible. Therefore, kinetics and thermodynamics application in vegetable oil extraction are useful for prediction of time and temperature required for optimum oil yield and understanding the changes in thermodynamic properties.

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