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Research Article

Optimization and Thermodynamics of the Extraction of Yellow Oleander Seed Oil Using Soxhlet Extractor

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Abstract

Optimization of yellow oleander (*Thevetia peruviana*) oil extraction from yellow oleander seeds was done using the soxhlet extractor. The following parameters were studied in order to measure their influence on batch extraction using petroleum ether (60 – 80℃); particle size, solid-solvent weight ratio, the effect of particle size and time, and temperature on yield, The maximum yield, and optimal conditions were obtained when using petroleum ether at a temperature of 343K with a contact time of 36 hours, average particles size >1 mm and meal to solvent ratio of 1:6. The thermodynamics study of the extracted oil showed that ΔE_a, ΔG and ΔH were positive while ΔS was found to be negative, indicating that yellow oleander oil extraction process is not a spontaneous process at all temperatures.

Key words: *Extraction, Yellow oleander seeds, Yellow oleander oil, Kinetics, Thermodynamics*

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1. Introduction

Thevetia peruviana is a perennial plant and all its parts are toxic due to the presence of cardenolic compounds and cianhidric acid. This non-edible oil has been identified as an excellent raw material for the production of biodiesel [1]. Ibiyemi *et al* [9] report that the yellow oleander kernel contains more than 62 % oil. Obasi *et al*., also reported that this plant contained between 45 and 72 % oil depending on season and location [2].

1.1 Petroleum Ether as an Extraction Solvent

Petroleum ether can be used as an extraction solvent because it is non-polar, cheap and flammable [3]. It also has a high solvent extraction capacity, does not affect the oil properties, is nontoxic, volatile and stabile [3]. Other researchers have observed that the efficiency of a solvent during extraction can be enhanced by decreasing the particle size of the meal through grinding [3]. There are several factors that can affect the efficiency of a solvent during the extraction of a vegetable oil. These include; the type of solvent used, the temperature used in the extraction, solvent-to-meal ratio and the number of cycles used in the extraction process. In order to achieve a high yield in the oil extraction process, the extraction process is always performed using grounded meal under optimum conditions [4].

The aims of this work were to study the optimization and thermodynamics of extraction of yellow oleander oil from dried powdered and sieved yellow oleander seed meal. This was done using petroleum ether (60 – 80℃), the parameters used in this research included; particle size, solid-liquid weight ratio, temperature, contact time and their effects on the extracted oil yield. This was essential in order to evaluate the best parameters to use in the yellow oleander oil production using the soxhlet extractor.

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2. Materials and Methods

2.1 Collection of Samples

Sampling of yellow oleander seeds took place in Gem Constituency, Siaya County, situated in Nyanza, Kenya. Its geographical coordinates are 0° 33' 36" North, 34° 17' 10" East for Yellow Oleander seeds. Sampling was done for a period of four months (January to April 2015). Siaya County: 842,304 (Male - 47 % Female - 53 %); Poverty Rate (based on KIBHS %): 35.3, Gem Constituency: 160,675, Poverty Rate: 42.0 [14].

2.2 Chemicals and Reagents

All chemicals and solvents used were of analytical grade, they were procured from commercial suppliers including Travotech Agencies Limited and Kobian Kenya Limited in Nairobi. The chemicals were used without further treatment.

2.3 Yellow oleander seeds Kernel Sample Preparation

Freshly matured yellow oleander seeds were handpicked in Gem Constituency, Siaya County, Kenya and transported in a plastic bag to the laboratory in The Technical University of Kenya in Nairobi. These seeds were weighed using an electronic balance JT601N and the average wet weight was recorded. The fleshy cover of the kernel containing the seeds was removed, discarded and the seeds sundried for at least 2 weeks in order to allow the biodegradation process to take place. The dried seeds were then weighed and grounded to a powder using a manual grinding machine [15].

2.4 Solvent Extraction

The solvent extraction method of extraction can give a higher percentage yield and less turbid oil than mechanical extraction [5]. 250 cm3 of petroleum ether was delivered into the Soxhlet extractor. 25 gms of powdered yellow oleander seed on Whatman No 1 filter paper was placed in the thimble of the Soxhlet extractor. The heating mantle was set at a specified temperature for the experiment and the extraction was carried out for a 3 hours' period. After the extraction with petroleum ether, the mixture of petroleum ether and yellow oleander seed oil obtained was poured into the distillation flask and placed on the rotatory distiller. The rotatory distiller was set at 80 ℃ which is the maximum boiling point of petroleum ether. After distillation, the extracted yellow oleander oil was weighed.

3. Effective Parameters Investigation

3.1 Particles Size

Particle size is an important parameter in the extraction of oil process. Smaller meal particles have a greater interfacial area between the meal and petroleum ether and therefore increased the yield of yellow oleander oil [6]. 25 g of yellow oleander meal at a temperature of 80℃ for 180 minutes for particle sizes of < 1 mm, 1 mm and > 1 mm, in order to investigate the best particles size which would give the highest yellow oleander oil extracted.

3.2 Solvent-Seeds Ratio

The effect of the volume of petroleum ether on oil yield was carried out using 25 g the meal, the average particles size of 1 mm were taken and treated with different ratios of petroleum ether to powdered yellow oleander meal in order to determine the best ratio of petroleum ether to meal to give the best yield of the oil.

3.3 Temperature

The effect of temperature on the yield of yellow oleander oil was tested by using 250 ml of petroleum ether, 25 g of the meal of particle sizes; unsieved, < 1, and > 1 mm, with the Soxhlet extractor operated at a temperature of 323, 333, 343 and 353 K for 180 minutes.

3.4 Time

The effect of time on oil yield of yellow oleander oil extracted was investigated using 250 ml of petroleum ether, 25 g of powdered yellow oleander meal of unsieved, < 1, and > 1 mm particle sizes at a temperature of different temperatures for 30, 60, 120, 150 and 180 minutes.

4. Results and Discussion

4.1 Effect of Solid/Solvent Ratio

The result obtained for the effect of volume of solvent on the oil yield is shown in Figure 1. As the meal/solvent ratio increased from 1:2 to 1:9, the amount of oil extracted also increased progressively from 37.2 to 65.51 %, then decreased thereafter. Nwabanne also studied the kinetics and thermodynamics of oil extraction from olive cake [7].

An increase in the volume of the solvent acted as a driving force in the extraction process. This increased the washing of the oil extracted away from the meal particle surface as a result of the yield increased [7]. In this experiment, the yield of yellow oleander oil decreased after 4.411 minutes with a yield of 65.51% because of the 1:6 meals to solvent ratio was the optimum condition for the extraction.

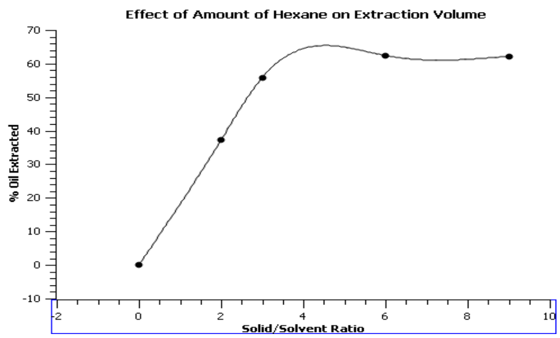


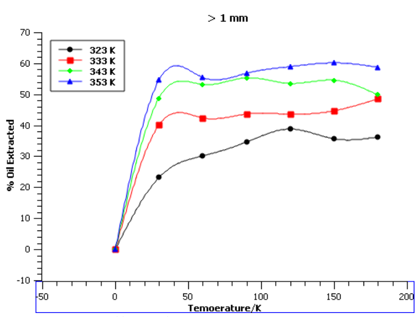
Figure 1. Effect of solid/Solvent ratio

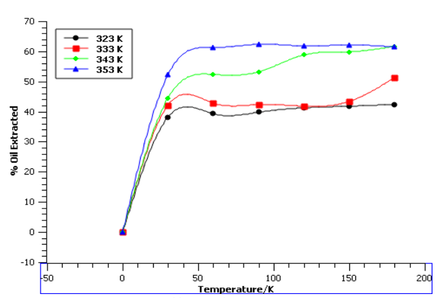
4.2 Effect of Particle Size and Temperature

The effect of the meal particle size and temperature was studied to investigate the optimum conditions for the extraction of yellow oleander oil. The results in figures 2-1 to 2-3 shows that as the particle size decreased to >1 mm, the extraction yield increased. Reduction in size to < 1 mm resulted in less yield. This can be attributed to the fact that smaller particle sizes always lead to agglomeration and this minimizes the contact surface area between the meal and the petroleum ether [8].

Yellow oleander oil yield increased from 24 to 49.6 % as the particle size decreased from unsieved to >1 and < 1 mm. The highest oil yield was obtained with a particle size of 1 mm. Goodrum and Kilgo (1987) [7] found out that total the peanut oil recovery increased from 36 to 82 % when the particle size range was decreased from (3.35 – 4.75 to 0.86 – 1.19 mm) [7]. Smaller particles have a larger amount of surface area combined with an increased in the number of ruptured cells and this resulted in a high oil concentration at the particle surface with little diffusion into the meal particles surface [7]. Large particles have smaller surface areas and this hinders solvent intrusion into the meal and reducing oil diffusion (Sayyar *et al* (2009) [7]. Size >1 mm meal size is recommended as the best size in the extraction of yellow oleander oil.

The effect of temperature on the rate of extraction on yellow oleander seed oil yield is also shown in figures 2-1 to 2-3. The best working temperatures were found to be 343-353 K, higher temperatures increased the yield of the oil extracted. Sayyar observed that the extraction of Jatropha oil increased with the increase of temperature of extraction [9]. Temperature increase increases the diffusion of the oil while decreasing its viscosity. Similar results were reported by Hickox [10] for cottonseed oil [10]. The cell walls of the meal ruptured at high temperature, this creates a void and a migratory space for the contents of the oil-bearing cells [10].





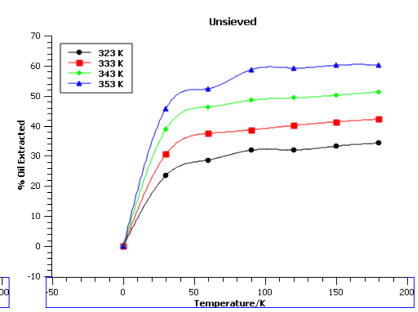


Figure 2-1. Effect of particle size and temperature on yield. **Figure 2-2.** Effect of particle size and temperature on yield. **Figure 2-3.** Effect of particle size and temperature on yield

4.3 Effect of Particle Size and Time

The effect of particle size on yellow oleander seed oil yield is shown in Figure 3. The yellow oleander yield increased with the extraction time [8].

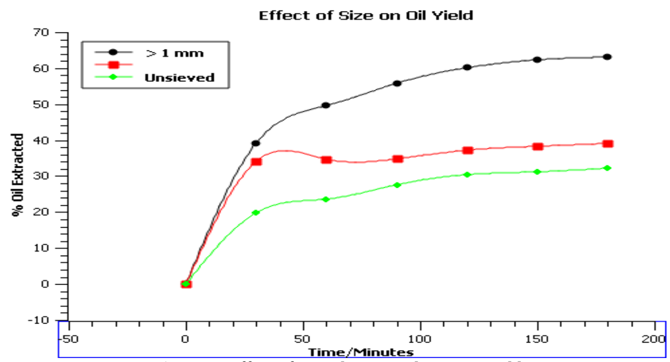


Figure 3. Effect of particle size and time on yield

A similar trend was observed by Nwabanne [7]. The yellow oleander oil yield increased from 37.2 to 55.8 % as time increased from 30 to 180 minutes. The highest percentage oil extracted is obtained at 180 minutes from the > 1 mm size powdered yellow oleander seed. Below 1 mm though the surface area was high but some oil was lost during grinding. The unsieved powdered yellow oleander seed gave the lowest yield due to low surface area. It was recommended that >1 mm meal size with a working extraction time of 180 minutes was the best size.

4.4 Kinetics of Extraction of Yellow Oleander Oil

Table 1. Summary of kinetics of extraction of yellow oleander oil

Temp/ ℃	% Yield	$\frac{dy}{dt}$	$\ln\frac{dy}{dt}$	lnY	Yu	K	lnK	T	$\frac{1}{T}$	conc g/l	$\frac{t}{C_t}$	t/min
30	23.14	0.7713	-0.2596	3.1416	76.86	0.3033	-1.193	303	0.0033	375.3	0.0799	30
40	26.02	0.8673	-0.1423	3.2592	73.98	0.3517	-1.045	313	0.00319	409.9	0.1464	60
50	31.41	1.047	0.0459	3.4471	68.59	0.4579	-0.7811	323	0.0031	472.1	0.1906	90
60	40.42	1.347	0.2981	3.6993	59.58	0.6784	-0.388	333	0.003	507	0.2367	120
70	52.14	1.738	0.5527	3.9539	47.86	1.0894	0.0857	343	0.00292	496.2	0.3023	150
80	65.92	2.197	0.7872	4.1884	34.08	1.9342	0.6597	353	0.00283	522.8	0.3443	180

4.4.1 The Rate of Dissolution

The rate of diffusion of the yellow oleander oil from the meal into the solution can be described by using the second order rate equation; $\frac{dC_t}{dt} = k(C_e - C_t)^2$ ……………………(1)

Where;

K = The second order extraction rate constant (l/g-min)
C_e = The concentration of yellow oleander oil in the solution at equilibrium (g/l)
C_t = The concentration of yellow oleander oil in (g/l) in the solution at any time, t (min)

The second order kinetic model is one recommended as the most suitable module in predicting the rate of dissolution of oil contained in solid to solution [6]. Now considering the initial concentration at $t = 0, C_t = 0,$ integrating this equation will give the following equation; $C_t = \frac{C_e^2 kt}{1+C_e kt}$ ……..(2); Which can be rewritten as can follows; $\frac{C_t}{t} = \frac{kC_e^2}{1+C_e kt}$ ……..(3). Now, when $t \rightarrow 0$, the LHS of equation (3) will be initial extraction rate, E_i, then; $E_i = kC_e^2$………(4). $\Rightarrow$ $\frac{C_t}{t} = \frac{KC^2{}_e}{1_i} + \frac{k\,C^2{}_e}{C_e kt} \Rightarrow \frac{C_t}{t} = \frac{E_i}{1} + \frac{C_e}{t}$. Rearranging this equation, gives the following linear equation; $\frac{t}{C_t} = \frac{1}{E_i} + \frac{t}{C_e}$ ……(5).

The initial extraction rate, E_i, the concentration of solution at equilibrium, C_e and the second order extraction constant, k, can be calculated from the experimental data by plotting $\frac{t}{c_t}$ against t. From figure 4; $\frac{1}{E}$ = 0.03312, $E_i = \frac{1}{0.03312} =$ 30.19, therefore, E_i the initial concentration of yellow oleander oil was 30.19 g/l., and C_e the concentration of yellow oleander oil at equilibrium, $\frac{1}{C_e} = 0.001748, C_e = \frac{1}{0.001748} = 570.08\ g/l$. Since the initial concentration was positive it implies that the dissolution of yellow oleander by hexane was spontaneous.

4.4.2 A plot of $\frac{dy}{dx}$ Against lny

By plotting $ln\frac{dY}{dt}$ versus lnY applying the differential Method [11], the plot resulted in a straight line with ($ln\ y$) as intercept and n as the slope , this is shown in figure 5. The reaction order is the relationship between the concentrations of species and the rate of a reaction.

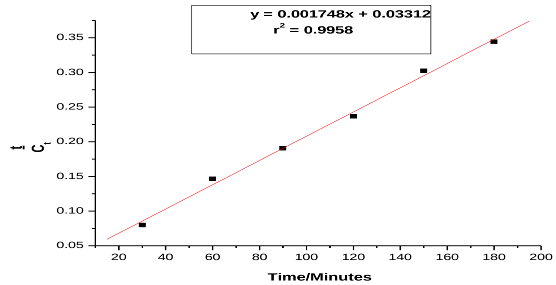


Figure 4. Rate of dissolution of yellow oleander oil

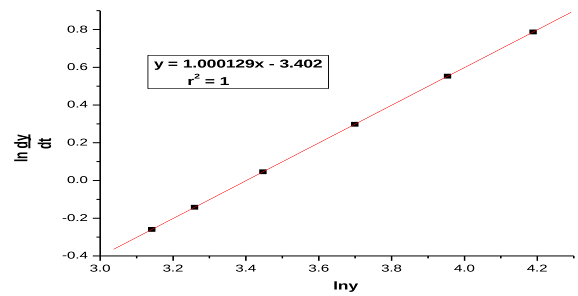


Figure 5. A plot of $\frac{dy}{dx}$ against lny

The percentage yield of the yellow oleander oil was determined using the expression: $Y = \frac{W_o}{W} x\ 100 \ldots..(1)$. Where, Y is the yield of yellow oleander oil extracted (%) and W_o is the weight of yellow oleander meal that remained after extraction and W is the weight of meal used. The rate of extraction was obtained using the equation; $\frac{dY}{dt} = ky^n \ldots\ldots\ldots..(2)$. This equation can also be rewritten as; $ln\frac{dY}{dt} = n\ lnY + ln\ k \ldots\ldots.(3)$. Where, t is the time of extraction in minutes, K is the rate of extraction constant and n is the slope of the graph [11]. From the figure 5 above, the linear regression equation was; $y = 1.0012x - 3.405$, r² = 1 This means that the y intercept is -3.405 = $ln\frac{dy}{dx}$; $\frac{dy}{dx} = ln^{-1}\ -3.402 = 0.0333.\ \frac{dy}{dt} = 0.0333, \Rightarrow 0.0333\ x\ 10 = 0.333\ \%$. This was the yield of oil extracted at the beginning of the experiment. Hence the rate of extraction constant was $0.0333\ \%\ Min^{-1}$. The reaction order is the relationship between the concentrations of species and the rate of a reaction. The order of the reaction $n = 1.00013\ \approx 1$, this means the extraction was of order one. The amount of oil extracted was directly proportional to the concentration of oil extracted.

4.5 Thermodynamics of Yellow Oleander Oil Extraction

The thermodynamics parameters (ΔH, ΔG, and ΔS) for the extraction of yellow oleander oil using petroleum ether as solvents can be estimated using following equations [12], $lnk = -\frac{\Delta G}{RT} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \ldots\ldots\ldots(4)$. Where, k is the equilibrium constant, ΔG is the Gibbs free energy (KJ/mol), ΔH is the enthalpy (KJ/mol), ΔS is the change in entropy (KJ/mol), R is the Universal gas constant (8.314 KJ/K mol) and T is the temperature in Kelvin [12]. The equilibrium constant k = $\frac{Y_T}{Y_U}$ ………....(5). Where Y_T, the percentage of yellow oleander oil that is extracted at temperature, T and Y_u is the percentage of the un-extracted yellow oleander oil. Equation (4) is Van't Hoff reaction and by plotting a graph of lnk against $\frac{1}{T}$ gives a straight line with $\frac{\Delta H}{R}$ as the gradient and $\frac{\Delta S}{R}$ as the y - intercept. Using these, the unknown values of ΔH, ΔG, and ΔS can be determined [12].

4.5.1 A Plot of lnk Against $\frac{1}{T}$

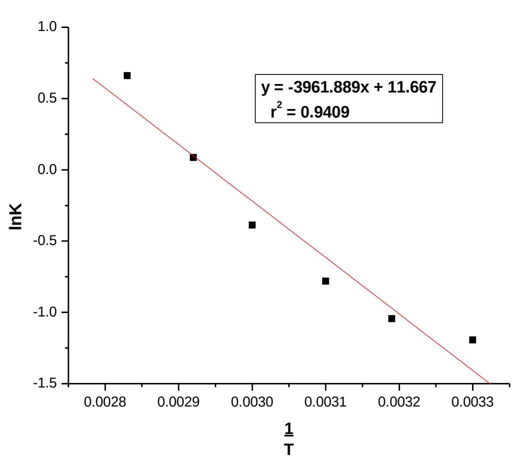


Figure 6. A plot of lnk against $\frac{1}{T}$

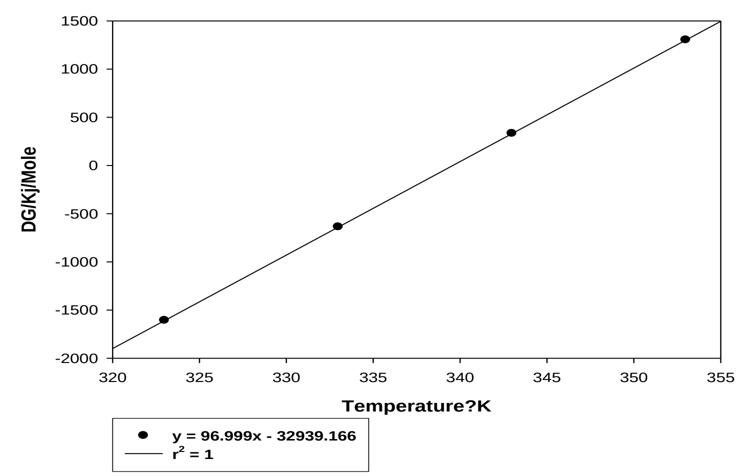


Figure 7. Effect of temperature ΔG values

From figure 6 above; $\frac{\Delta H}{R}$ = -3961.889, $\Delta H = -3961.889\ x\ R = -3961.889\ x\ 8.314 = -32\ 939.145\ KJ/mole$ or $-32.939\frac{MJ}{mole} = 32.939\frac{Mj}{mole}\ x\ \frac{1}{863}\ x\ \frac{1000}{1\ kg} = 38.168\ Mj/kg$. This value was found to be higher than the observed value of the calorific value of 33.345 Mj/kg. But, was less than the expected range of $39.000 - 48.000\ Mj/Kg$ [23]. Duraisamy *et al.,* [42], found a calorific value of $40.148\ MJ/Kg$ for yellow oleander oil, and this was found to be in line with the results in this research. Gravalos *et al.,* [22] also found out that the net calorific values of fats and oils that ranged from 36.247 to 37.294 J/g. In this research we found an experimental value of $33.345\ MJ/kg$ for yellow oleander oil, which was actually lower than the recommended standards.. $\frac{\Delta S}{R} = 11.667$, $\Delta S = 11.667\ x\ R = 11.667\ x\ 8.314 = 96.999\ Kj/mole$. $\Delta G^{\circ} = \Delta H^{\circ} + T\Delta S^{\circ}$. For the above extraction, $\Delta G^{\circ} = -32\ 939.145 + 96.999\ T$. [figure 7].

The change in temperature is directly proportional to the $\Delta\ G$ values. Positive values indicate a non-spontaneous reaction while the negative value indicates a spontaneous process. From 323 to 333 K the extraction is spontaneous, above this temperature the extraction of yellow oleander oil becomes an endergonic (not a spontaneous) process. The value of ΔH° was negative indicating that the extraction was exothermic and that the process was irreversible.

4.5.2 Determination of Activation Parameters

From figure 6 above, it seems intuitive that a reaction goes faster at higher temperature, as more reactant molecules have the energy needed to overcome the activation barrier to the reaction. The Arrhenius equation relates the reaction rate constant (k) and temperature. One of the forms of the Arrhenius equation is given below: $lnk = \frac{-E_a}{RT} + lnA \ldots\ldots\ldots\ldots\ldots\ldots\ldots(7)$. where E_a is the activation energy for the reaction, T is the absolute temperature (in Kelvin) at which a corresponding k is determined, R is the gas constant, and A is a pre-exponential factor. The activation energy may then be extracted from a plot of ln k vs.$\frac{1}{T}$, which should be linear. This plot is called an "Arrhenius plot.". From figure 6 above, $\frac{-E_a}{R} = -3961.889$, $-E_a =$ -3961.889 x R = -3961.889 x 8.314 = -32 939.145 KJ/mol. E_a = 32 939.145 KJ/mol. Since the activation energy is high and positive, it implies that the extraction process is not automatic. Some amount of energy has to be applied for the extraction to be effective. $\ln A = 11.667$, ln^{-1}A = 116 657.78 Kj/Mole or 116.66 Mj/mole.

4.5.3 Application of the Eyring Equation

The general form of the Eyring–Polanyi equation somewhat resembles the Arrhenius equation: $k = \frac{kbT}{h}\ e^{-\frac{\Delta G^{\circ}}{RT}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(8)$. where ΔG° is the Gibbs energy of activation, k_B is Boltzmann's constant, and *h* is Planck's constant. It can be rewritten as: $k\ \frac{k_bT}{h}\ e^{\frac{\Delta S^{\circ}}{R}}\ e^{-\frac{\Delta H^{\circ}}{RT}}$…….(9). To find the linear form of the Eyring-Polanyi equation: The Eyring equation is: $\ln\left(\frac{k}{T}\right) = \frac{-\Delta H^{\circ}}{RT} + \ln(\frac{k_b}{h}) + \frac{\Delta S^{\circ}}{R} \ldots\ldots\ldots\ldots(10)$. where k, T and R are the same as in the Arrhenius equation, k_B is Boltzmann's constant, h is Planck's constant and ΔH° and $\Delta S^{\circ}$are the enthalpy and entropy of activation, respectively. A certain chemical reaction is performed at different temperatures and the reaction rate is determined. The values for $\Delta H^{\circ}$and ΔS° can be determined from kinetic data obtained from an $\ln\left(\frac{k}{T}\right)$ vs. $\frac{1}{T}$, plot. The Equation is a straight line with negative slope, $\frac{-\Delta H}{R}$, and a y-intercept, $\ln(\frac{k_b}{h}) + \frac{\Delta S^{\circ}}{R}$.

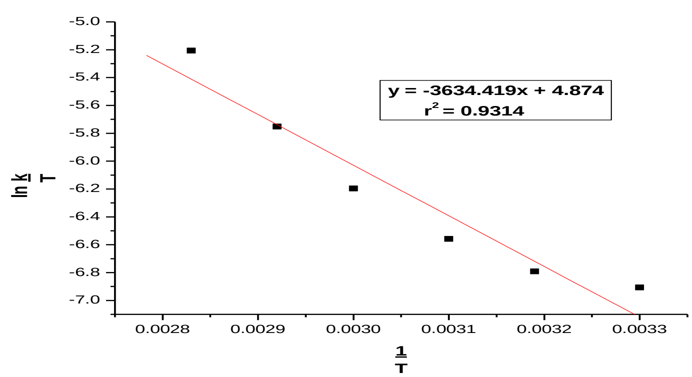


Figure 8. The Eyring curve for yellow oleander oil extraction

From figure 8 above; $\frac{-\Delta H}{R} = -3634.42, -\Delta H = -3634.42\ x\ R = -3634.42\ x\ 8.314 = -30\ 216.568\frac{KJ}{mol}$. Hence ΔH = 30 216.568 or 30.217 MJ/mol = $30.217\frac{Mj}{mole}\ x\ \frac{1}{863}\ x\ \frac{1000}{1kg} = 35.014\ Mj/kg$. And; $ln\frac{k_b}{h} + \frac{\Delta S}{R} = 4.874,\ \frac{\Delta S}{R} = 4.874 - ln\frac{k_b}{h},\ 4.874 - \ln(\frac{1.38064\ x\ 10^{-23}}{6.627\ x\ 10^{-34}})$. $4.874 - \ln 2.083\ x\ 10^{11} = 4.874 - 26.062 = -21.188$. $\frac{\Delta S}{R}$= -21.188, $\Delta S = -21.188\ x\ 8.314,\ \Delta S = -176.157\ KJ/mol$. From these results, $\Delta H > 0\ and\ \Delta S < 0$, which means that the extraction of yellow oleander oil was not a spontaneous process.

Conclusion

The optimum conditions for high oil yield include grinding the solid seeds particles to 1 mm size, the mass of petroleum ether has to be four times the mass of the seeds, heating the mixture up to the boiling point of petroleum ether and extraction time of 36 hours. The petroleum ether extraction process of yellow oleander oil follows the third order kinetic.

Yellow oleander seed oil extraction followed second-order kinetics. It was also found that HΔ is positive, ΔS is negative, and ΔG is positive indicating that this process is not spontaneous at all temperatures. ΔE_a, the activation energy was high this confirms that extraction of yellow oleander oil from the seeds is not a spontaneous reaction.

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