Kinetics and Thermodynamic Studies of Beta Carotene Extraction from Palm Oil by Solvent Extraction

Samuel O. Egbuna[1], Donatus C. Onwubiko[1] and Christian O. Asadu[1*]

[1]*Department of Chemical Engineering, Enugu State University of Science and Technology, P.M.B. 01660, Enugu, Nigeria.*

Authors' contributions

This work was carried out in collaboration among all authors. Authors SOE and DCO designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors COA and DCO managed the analyses of the study. Author COA managed the literature searches. All authors read and approved the final manuscript.

Article Information

Original Research Article

ABSTRACT

This study evaluated the kinetics and thermodynamics of the extraction of beta-carotene from palm oil using acetone and ethanol. Six kinetics models which include; first order, second order, pseudo-first order, pseudo-second order, intra-particle diffusion model and power law model were used for the study. Pseudo-second order was found to fit the experimental data better than the rest of the models with correlation coefficient (R^2) of above 0.99. The thermodynamics study of the process was done at the temperature of 35°C, 40°C, 45°C and 50°C. From the result of the parameters, it was discovered that the process was endothermic, non-spontaneous and randomized (disorderly).

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*Corresponding author: Email: aasadu@yahoo.com;

1. INTRODUCTION

Beta-carotene is among the major products of food industry that has been widely employed as nutrients and additives. The global market demand for carotenoids indicates a gradual annual increase. In recent time, there have been concerns regarding the source of the ingredients together with awareness about potential harmful synthetic compounds from the consumer. These rising consumer awareness and concerns have necessitated the global demand for naturally produced carotene to rise appreciably. Hence, industries are constantly looking for new sources as well as introducing new methods to produce beta-carotene. Beta-carotene is a pigment dominant in plants that give yellow and orange fruit, vegetables their colour. Carotenoid is a precursor of vitamin-A [1]. It is a powerful antioxidant that plays a vital role in maintaining vision, skin and neurological function. Beta-carotene is a natural occurring retinol precursor obtained from some fruits and vegetables with potential anti-neoplastic and chemo-preventive activities [2]. As an antioxidant, beta-carotene inhibits free radical damage to DNA. This agent also induces cell differentiation and apoptosis of some tumour cell types, particularly in early stages of tumorigenesis and enhances immune system activity by stimulating the release of natural killer cells. As a precursor of Vitamin-A, it is administered to reduce the severity of photosensitivity reactions in patients with erythropoietic protoporphyria [3]. Beta carotene may be used for prevention of Vitamin A deficiency states in most individuals, [4]. Vitamin A deficiency may occur as a result of inadequate nutrition or intestinal mal-absorption but does not occur in healthy individuals receiving an adequate balanced diet. There are more than 500 carotenoids in nature; they consist of two classes of molecules known as carotenoids and xanthophylls [2]. Commercially, carotenoids are used in food coloration, vitamin supplement, pharmaceutical and cosmetics products.

Beta-carotene that belongs to the carotene group is the most widespread in foods with chemical formula $C_{40}H_{56}$ and composed of eight isoprene units with specific retinyl rings. It has a molecular weight of 536.88 g/mol.

Palm oil is one of the natural oils that are interesting alternatives to the problems of natural sources of carotene. It is a natural source of carotene that is known to contain high concentration of natural carotenoids. The main carotenoids of palm oil are alpha carotene and beta carotene, (Ooi et al. 1994).

2. MATERIALS AND METHODS

2.1 Materials

The freshly produced crude palm oil used for this study was procured from a local palm oil processing mill in Amator-Amuzu in Aboh Mbaise L.G.A Imo State Nigeria.

2.2 Methods

2.2.1 Determination of the physiochemical properties of the oil samples

The specific gravity, viscosity, peroxide value, acid value, iodine value, saponification value and free fatty acid (FFA) of the oil samples were determined as prescribed by AOAC (2005) and are presented in Table 1.

Table 1. Physiochemical properties of palm oil

Parameters	Values
Specific gravity (mg/cm^3)	0.900
Viscosity (cp$_s$ at 25°C)	0.056
Peroxide value (meq/cm^3)	4.9806
Acid value (mg KOH/cm^3)	1.662
Iodine value (wijs)	44.9136
Saponification value (mg/cm^3)	210.22
FFA (%)	0.831

2.2.2 Extraction and determination of beta carotene

For each of the oil samples (palm &soybean oils), 10 cm^3 was measured into a conical flask and 30 cm^3 of 95% ethanol was measured and added into the conical flask and maintained at room temperature in a water bath for 20 mins with periodic shaking. The supernatant was decanted and allowed to cool. The ethanol concentration of the mixture was brought to 85% by adding 5 cm^3 distilled water and further cooled for about 5mins. The mixture was transferred into a separating funnel and 15 cm^3 of pet-ether; and 10 cm^3 of ethanol added into it. It was allowed to stand until two layers were formed. The button layer was turned off into a beaker while the top layer was collected into a 250 cm^3 conical flask, the bottom layer was transferred into a funnel and re-extracted with 15 cm^3 of pet-ether ones to get a fairly yellow extract. The entire pet-ether was collected into 250 cm^3 conical flask and transferred into a separating funnel for another

re-extraction with 15 cm^3 of 80% ethanol. The extraction formed was measured and poured into a sample bottle for further analysis using a U-V-spectrophotometer at 390 Nm.

2.2.3 Effect of temperature on the extraction of beta carotene

The effect of temperature on the extraction of beta carotene was done by employing the procedure in 2.2.2, while keeping dosage of the oil sample constant at 10 cm^3, solvent volume at 30 cm^3, time at 20 mins, and temperature varied from 35°C, 40°C, 45°C and 50°C.

This was done to determine the effect temperature has on the extraction of beta carotene using acetone and ethanol as shown in Fig. 2.

2.2.4 Effect of time on the extraction of beta carotene from palm oil

The extent of extraction of beta carotene from palm oil using acetone and ethanol was studied as a function time. The substrate volume was kept constant at 10 cm^3, solvents volume was kept constant at 30 cm^3. Time of extraction was varied at the range of 10, 20, 30,40,50,60 and 70mins at difference temperature of 35°C, 40°C, 45°C and 50°C. This is shown in Fig. 3.

2.3 Kinetics Models for the Extraction of Beta Carotene from Palm Oil

Six kinetics models have been used to study the extraction of beta-carotene from palm oil. These include; first-order kinetic model, second order kinetic model, pseudo-first-order model, pseudo-second-order-kinetic model, intra-particle diffusion model and power law model.

2.3.1 First order kinetic model

$$\text{Log } C_t = \frac{K_1}{2.303} \ (t) + \text{Log } C_0 \qquad (2.1)$$

Where C_t and C_0 are the concentrations of the solute at time t and initial concentration (mg/cm^3), respectively and K_1 is the first order rates constant, (Min^{-1}). If the first-order kinetics is applicable to the extraction, then the plot of log C_t versus t of equation (2.1) above will give a linear relationship with $\frac{K_1}{2.303}$ and Log C_0 as slope and intercept as shown in Fig. 4.

2.3.2 Second-order kinetic model

The second-order rate equation in solution systems is given as;

$$\frac{dct}{-K_2 C_t^2} = - K_2 \ C_t^2 \qquad (2.2)$$

Integrating equation (2.2) with the boundary conditions of Ct = 0 at t = 0 and C_t = C_t at t = t to yielded;

$$\frac{1}{C_t} = K_2(t) + \frac{1}{C_0} \qquad (2.3)$$

Where C_0 and C_t (mg/cm^3) are the concentration of solute at equilibrium and at time t (min), respectively, and K_2 (mg/cm^3. min) is the rate constant of second oder. If the second-order kinetics is applicable to the system, then the plot of I/Ct versus t of (2.3) will give a linear relationship with K_2 and 1/C_0 as slope as shown in Fig. 5.

2.3.3 Pseudo-first order model

First order rate equation was used to describe kinetic process;

$$\frac{dqt}{qt} = Kp_1 \ (q_e \text{ - } q_t) \qquad (2.4)$$

Where q_e and q_t (mg/cm^3) are the extraction capacities at equilibrium and at time t (min) respectively. Kp_1 (min) is the pseudo-first-order rate constant for the kinetic model.

Integrating equation (2.4) above with the boundary conditions of $q_{t=}$ = 0 at t = 0 and q_t = $q_{t'}$ at t = t, yields.

$$\text{In}q_e \text{ – In } (q_e \text{ -}q_t) = Kp_1 \ t \qquad (2.5)$$

This can be re-arranged to give,

$$\text{Log } (q_e \text{-}q_t) = \text{log } q_e -\frac{Kp_1}{2.303} \ (t) \qquad (2.6)$$

If the first-order kinetics is applicable to the system, then the plot of log $(q_e^- \ q_t)$ versus t of equation (2.6) will give a linear relationship with $\frac{Kp_1}{2.303}$ and Log q_e as slope and intercept respectively Fig. 6.

To distinguish kinetic equations based on extraction capacity from solution concentration,

Lagerangrean's first order rate equation has been called pseudo first-order.

2.3.4 Pseudo-Second-Order model

According to Sudipta Das et al, (2014), kinetic process of extraction can be described using pseudo second order model where the driving force $(q_e^- \ q_t)$, is proportional to the available fraction of active site. It follows that;

$$\frac{dq_t}{dt} = Kp_2 (q_e - q_t)^2 \qquad (2.7)$$

Re-arranging equation (2.7) above gives;

$$\frac{dq_t}{(q_e - q_t)} = Kp_2 dt \qquad (2.8)$$

Integration of equation (2.8) with the boundary condition of $q_t = 0$ at $t = 0$ and $q_t - q_t$ at $t = t$, yields,

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + K_{P2} (t) \qquad (2.9)$$

Equation (2.9) can be re-arranged to give,

$$\frac{t}{q_t} = \frac{1}{Kp_2 q_e^2} + \frac{1}{q_e} + (t) \qquad (2.10)$$

Where, $Kp_2 q_2^2 (\text{mg/cm}^3 (\text{min})$ is the initial extraction rate. If the kinetics is applicable to the system, then the plot of $\frac{t}{q_t}$ versus t of equation (2.10) above will give a linear relationship with $\frac{1}{q_e}$ and $1/_{Kp_2 q_e^2}$ as slope and intercept respectively. This is shown in Fig. 7.

2.3.5 Intra-particle diffusion model

$$\frac{q}{t} = K_{id} t^{1/2} \qquad (2.11)$$

The logarithm form of the equation 2.11 is given as:

$$\text{Log } q_t = \text{Log } K_{id} + 0.5 \text{ Log } t \qquad (2.12)$$

Where K_{id} is the intra-particle diffusion rate constant. According to equation (2.12), a plot of Log q_t versus 0.5 log t gives a straight line with a positive intercept for intra particle diffusion controlled process. It is essential for the plot of q_t versus $t^{1/2}$ to go through the origin if the intra particle diffusion is the sole rate limiting step Fig. 8.

2.3.6 Power law model

Power law model is used for the diffusion of an active agent through non-smelling devices and is described by equation 2.1.3

$$q = B. \qquad (2.13)$$

Logarithmic form;

$$\text{loq } q = \text{Log } B + n \text{Log } t \qquad (2.14)$$

Where B is a constant which is related with the characteristics of the carrier-active agent system q is yield, n is the diffusion exponent and t is time

of activity (min). In case of extraction from vegetable component n is always less than 1.

2.3.7 Determination of the thermodynamics parameters, ΔG^0, ΔH^0, ΔS^0

The above listed thermodynamic parameters were determined by employing the equations; 2.14 – 2.16

$$\Delta G = - RT \text{ Ln } Kc \qquad (2.15)$$

$$\text{Ln} Kc = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \qquad (2.16)$$

$$\Delta G_{ext} = \Delta H_{ext} - T\Delta S_{ext} \qquad (2.17)$$

Where

ΔS = Entropy
R = Gas Constant
ΔH = Enthalpy
T = Absolute temperature (K)
ΔG = Gibb's Free energy

By plotting lnKc Vs $\frac{1}{T}$, the values of $\Delta H°$ and $\Delta S°$ were estimated from the slopes and intercepts, hence ΔG was calculated from equation (2.17). Similarly, the activation energy was estimated through Arrhenius equation, respectively.

$$K = A_{exp} (-\Delta E/RT) \qquad (2.18)$$

$$\text{ln}K = \text{ln}A - \frac{\Delta E}{RT} \qquad (2.19)$$

By plotting lnK Vs 1/T, the activation energy and the pre-exponential function (A) were estimated from the slope and intercept.

3. RESULTS AND DISCUSSION

3.1 Concentration of Beta-carotene at Different Temperatures and Time

The concentration of the extracted beta carotene from palm oil using acetone and ethanol was studied as a function of time and temperature. The substrate volume was kept constant at 10cm^3, solvents volume was kept constant at 30cm^3. Time of extraction varied at the range of 10, 20, 30, 40, 50, 60 and 70 mins, at different temperature of 35°C, 40°C, 45°C, and 50°C.

Figs. 1 (a & b) shows the effect of extraction time on the extraction capacity of acetone and ethanol at various temperatures. It was observed that the concentration increased as time and temperature in creased for palm oil on both solvents resulting

from increase in time of agitation. Fig. 1 (a) shows clearly that concentration was highest at temperature 45°C. At temperature 50°C, concentration was higher than that at 40°C, but as time progressed, precisely after 50mins, concentration at 40°C became higher than that at 50°C. This suggests that, the extraction of beta-carotene from palm oil using acetone can best be achieved between the temperature of 40°C and 45°C Fig. 1(b) also shows a very close competition in the concentration of extracted beta-carotene from temperature with 50°C slightly higher and followed by 45°C. This also suggests that the extraction of beta-carotene from palm oil using ethanol can be best between the temperature of 45°C and 50°C. The increase in the concentration of the extracted beta-carotene with increase in temperature was also observed to go along with increase in time. The increase in the concentration with increase in the temperature is due to increase in mass transfer rate with temperature. Increase in concentration with increase in time was as a result of longer time of shaking (agitation) which allowed for adequate mixing.

3.2 Effect of Temperature on the Extraction of Beta-carotene from Palm oil Using Acetone and Ethanol

The effect of temperature on the extraction of beta-carotene from palm oil using acetone and ethanol was evaluated based on the concentration at temperature range of 35°C, 40°C, 45°C and 50°C at a dosage of 10 cm^3, solvent volume of 30 cm^3 and constant time of 20 mins. It is clear from the result as shown in Fig. 2 that as the temperature increases, the concentration of the extracted beta carotene increased. This is due to increased mass transfer rate with increase in temperature (Kalmalpreet and Reighvan, 2012). When temperature is increased, it facilitates the breakdown of cell walls and improves extraction with solvent. As reported by Oliverira et al. [5], that carotenoid degradation occurs in temperature close to 40°C.

Therefore temperature up to 45°C resulted in a decrease in the yield of carotenoids constant. Norshizila et al. [6] reported that carotenoids concentration decreased at the temperature at 45°C. This present study is in agreement with Norshazila *et al.,* [6] in the case of evident in Fig. 2. Mustapha [7] reported that the extraction of beta carotene from palm oil using R134a, that the extraction of beta carotene was maximum at the temperature of 80°C. In this present study,

the temperatures studied were maximum at 50°C. Also, the increase in concentration as temperature increased shows that the process is an endothermic process [8]. The decline in concentration at 45°C using acetone could be attributed to the volatile nature of acetone.

3.3 Effect of Time on the Extraction of Beta-carotene from Palm Oil

The degree of extraction of beta-carotene from palm oil using acetone and ethanol was studied as a function of time. The volume (dosage) of the oil sample was kept constant at 10 cm^3 for both acetone and ethanol. Solvent volume was kept constant at 30 cm^3 and temperature was kept constant at 35°C. Fig. 3 shows the concentration of beta-carotene with time of extraction increased. The increase in concentration with increase in time as observed, could be attributed to the fact that as the time of extraction is increased, it allows for longer time of shaking (agitation) which allows for adequate mixing of the substrate and solvent (Moldoveanu and David, 2015).

3.4 Kinetics of Beta Carotene Extraction from Palm Oil Using Acetone and Ethanol

The study of the kinetics of extraction of beta carotene from palm oil is very vital as it provides clear understanding of the reaction pathway and mechanisms of the extraction process.

3.4.1 First order model

The first order kinetics model was used to study the extraction mechanism. The experimental data was tested with the first order model. The values of the first order rate constant for palm oil using acetone and ethanol were calculated from the slope of the graphs plotted and presented in Table 2. The correlation coefficient (R^2) obtained at different temperature were low, which showed poor agreement of the first order model with the experimental data.

3.4.2 Second order model

The values of the second order rate constant (K_2), C_0 and the correlation coefficient (R^2) as calculated are presented in Table 2. The value of the correlation coefficient at the studied temperatures, were small compared to the pseudo-second order, and this showed that, the model did not fit the experimental data well.
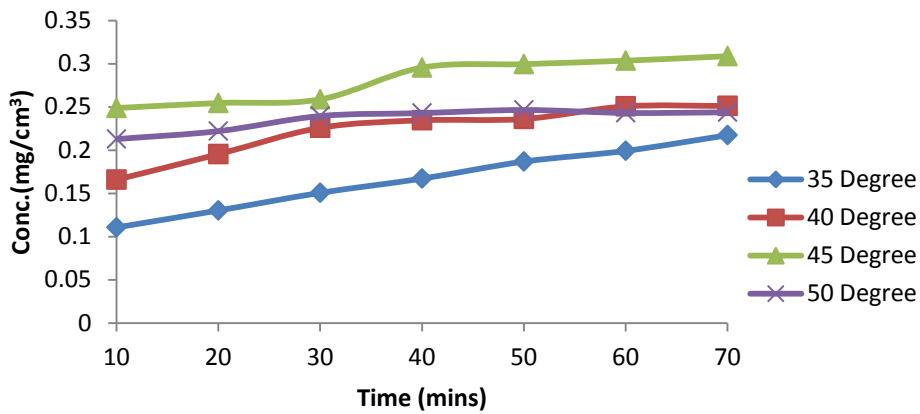
Fig. 1a. Concentration of beta-carotene at different temperatures and time using acetone
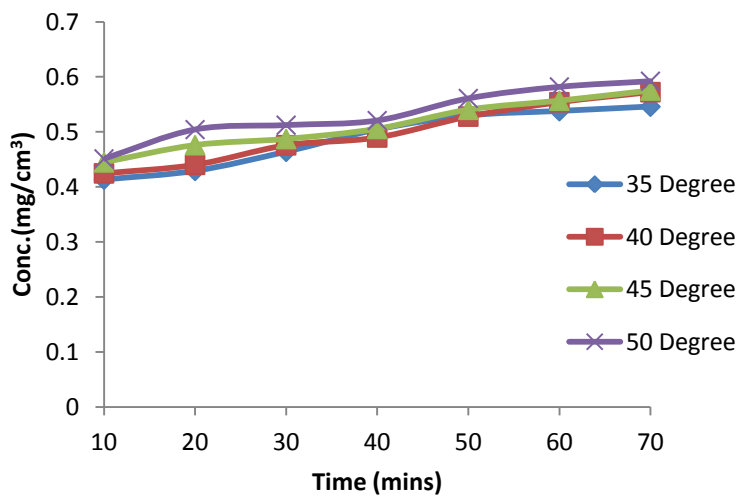

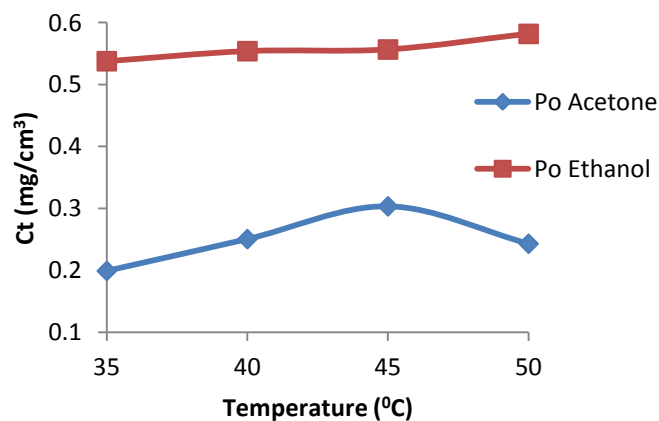**Fig. 1b. Concentration of beta-carotene at different temperatures and time using ethanol**

Fig. 2. Effect of temperature on the extraction of beta carotene from palm oil

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Table 2. Kinetics parameters

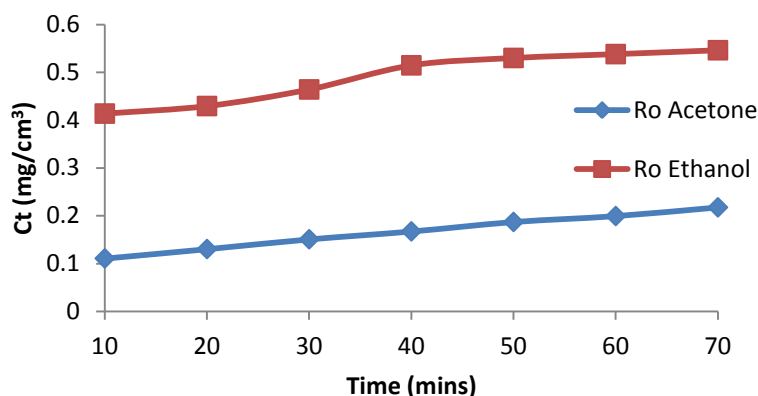| | First order | | | | | | Second order | | | | | |
| | Acetone | | | Ethanol | | | Acetone | | | Ethanol | | |
$T(^0C)$	K_1	C_0	R^2	K_1	C_0	R^2	K_2	C_0	R^2	K_2	C_0	R^2
35	0.0111	0.1044	0.9784	0.0051	0.3986	0.9198	-0.0711	0.1090	0.9424	-0.0106	0.4024	0.9102
40	0.0064	0.1709	0.8262	0.0053	0.4055	0.9644	-0.0307	0.1723	0.7898	-0.0107	0.4104	0.9543
45	0.0041	0.2380	0.8887	0.0041	0.4331	0.9764	-0.0148	0.2397	0.885	-0.0083	0.4367	0.9672
50	0.0023	0.2157	0.708	0.0041	0.4482	0.9372	-0.0096	0.2157	0.7044	-0.0079	0.4515	0.9216
	Pseudo-first order						Pseudo-second-order					
	Kp_1	q_e	R^2	KP_1	q_e	R^2	Kp_2	q_e	R^2	Kp_2	q_e	R^2
35	-0.0089	0.1867	0.9987	-0.0120	0.2136	0.9371	-0.5885	0.4008	0.9959	-1.3854	0.0512	0.9579
40	-0.0071	0.1542	0.875	-0.0119	0.2334	0.981	-0.9144	0.3630	0.9992	-1.7589	0.0293	0.8158
45	-0.0059	0.1476	0.8954	-0.0106	0.2085	0.9883	-0.9426	0.3021	0.9977	-2.0610	0.0268	0.7929
50	-0.0028	0.1169	0.7253	-0.0103	0.2089	0.9558	-2.5633	0.3784	0.9998	-6.4998	0.0110	0.9661
	Intra-particle diffusion						Power law model					
	Kid	R^2	Kid	R^2			B	N	R^2	B	n	R^2
35	0.5649	0.9321	0.0402	0.8994			-0.124	0.7243	0.9321	-0.6979	0.8079	0.8994
40	0.5851	0.9769	0.0155	0.8333			-0.1164	0.6132	0.9769	-0.9056	2.1213	0.8334
45	0.6062	0.8482	0.0196	0.8139			-0.1164	0.6132	0.9769	-0.9056	2.1213	0.8331
50	0.8011	0.8832	0.0029	0.6833			-0.0481	0.4666	0.8831	-1.2672	4.6153	0.6834

Fig. 3. Effect of time on the extraction of beta carotene from palm oil using acetone
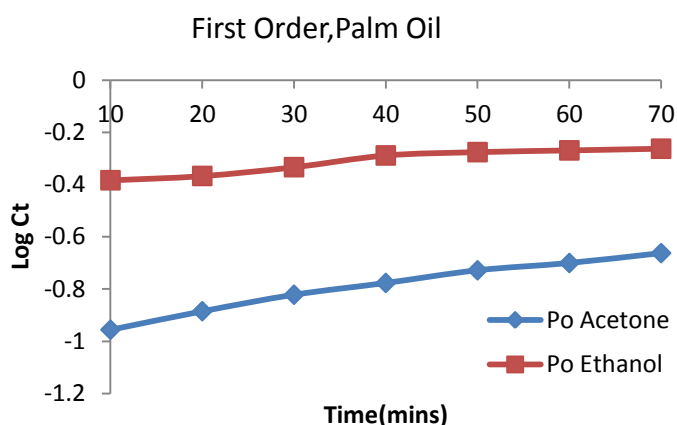
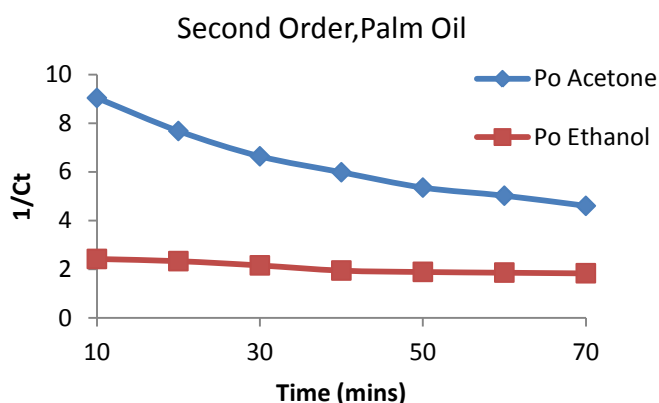

Fig. 4. First order model plot

Fig. 5. Second order plot

3.4.3 Pseudo first order

The pseudo-first order rate constant (K_{P1}), calculated q_e, which were determined from the slopes and intercepts of the plot of log $(q_e - q_t)$ against the time (t) are presented with the correlation (R^2) in Table 2. The R^2 values gotten were small in all the temperature studied and experimental qe values did not agree with the calculated values generated from the linear plots.

3.4.4 Pseudo-second order

The values of qe and pseudo-second order rate constant (K_{P2}) were obtained from the shape and intercept and are presented in Table 2. It can be

seen from the result that the correlation coefficient for pseudo-second order model studied. Furthermore, there was a good agreement between the experimental q_e values and the calculated q_e values. This goes to shows that, the kinetics of extraction of beta-carotene from palm oil is better represented by pseudo-second order model when compared to the other models studied at the selected temperatures (Sudipta Das et al., 2014). The plots of the pseudo-second order model at the studied temperatures are shown in Fig. 7(a) –7(d).

3.4.5 Intra-particle diffusion model

The intra-particle diffusion model was proposed to identify the extraction mechanism and to predict the rate controlling step, if the plot of log qt versus 0.5 log t is linear and passes through the origin. From Table 2, the correlation coefficient (R^2) for the extraction of beta-carotene from palm oil using acetone and ethanol are much smaller than those gotten in pseudo

second order model. Also, the plot of intra-particle diffusion model did not pass through the origin which proves that intra-particle diffusion model is not the rate controlling step of the extraction of beta-carotene from palm oil using acetone and ethanol.

3.4.6 Power law model

The values of B which represent the power law rate constant, the correlation coefficient (R^2) are presented in Table 2. The correlation coefficients generated for the palm oil are similar to those from intra-particle diffusion model. This low correlation coefficient shows that power law model does not fit the experimental data.

3.5 Thermodynamics of the Extraction of Beta-carotene from Palm Oil Using Acetone and Ethanol

Gibbs free energy combines enthalpy and entropy into a single value. It is the energy
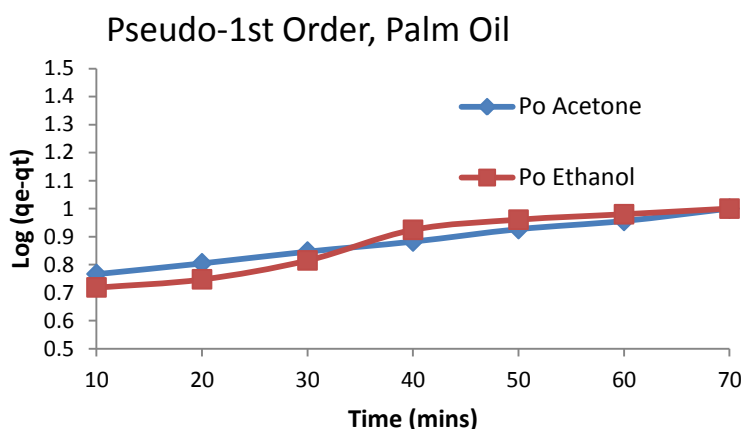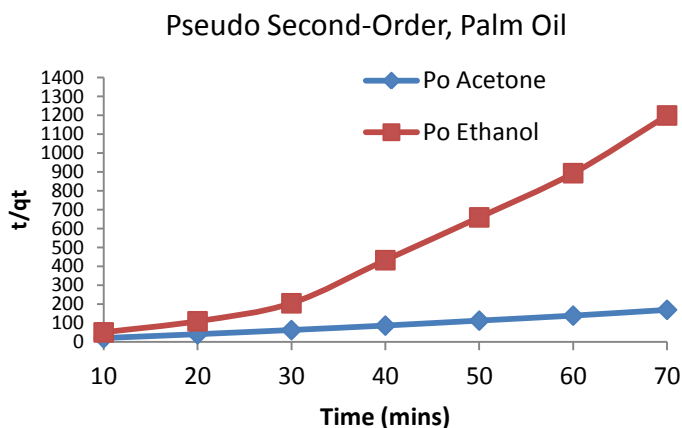

Fig. 6. Pseudo-1st order plot
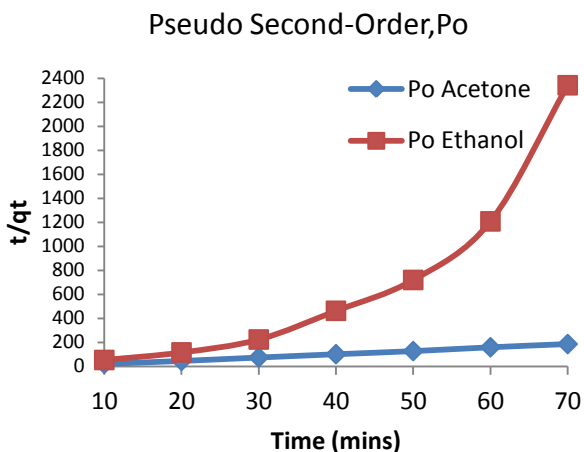

Fig. 7a. Pseudo second-order at 35^{U}C

Pseudo Second-Order,Po

Fig. 7b. Pseudo second-order at 40^{0}C

Pseudo Second Order,Po

Fig. 7c. Pseudo second order at 45ºC

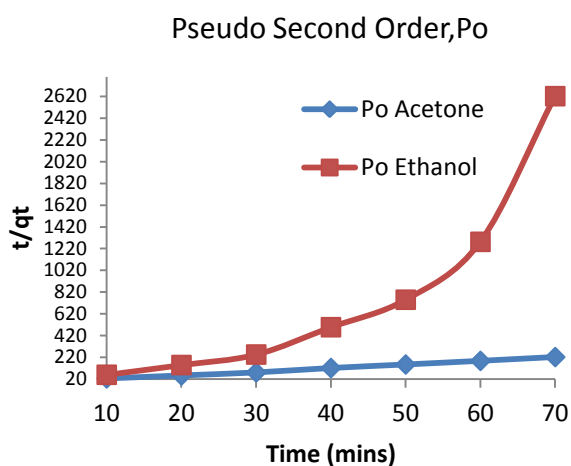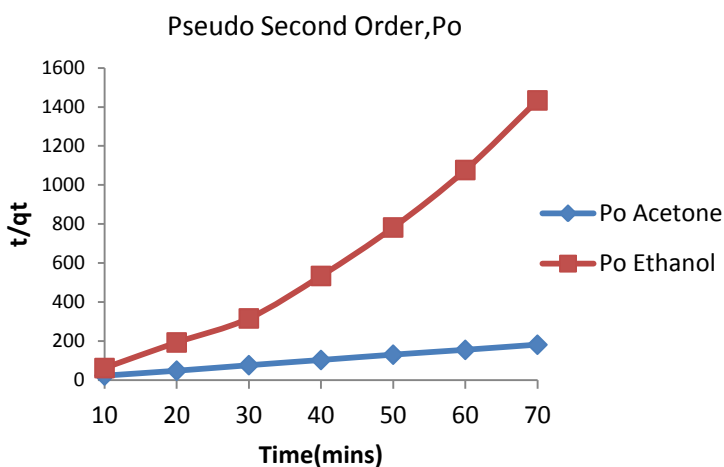## Pseudo Second Order,Po

Fig. 7d. Pseudo second order at 50^{0}C

associated with a chemical reaction that can do useful work. It equals the enthalpy minus the product of the temperature and entropy change of the system.

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \qquad (2.20)$$

Enthalpy (H) is a measure of how much energy is released or absorbed during a chemical reaction. Energy in the form of heat is leased in a exothermic reaction, and the change is enthalpy is negative ($-\Delta H$). On the other hand, energy in the form of heat is absorbed in an endothermic reaction, and this makes the change in enthalpy to be positive ($+\Delta H$). Entropy is a measure of disorder or randomness in the system. When disorder increases, entropy is positive ($+\Delta S$), (Rajput, 2014). The thermodynamics parameters, $\Delta G°$, $\Delta H°$, ΔE and $\Delta S°$ were determined using equation 2.15 and presented in Table 2. The values of ΔH and ΔS were calculated from the slopes and the intercepts at the selected temperatures. The rate constant (K used for the plot of lnK vs 1/T was that of pseudo-second order kinetics model which best fitted the experimental data. From Arrhenius equation, by the plot of ln KVs 1/T, the values of activation energy and the pre-exponential function were determined from the slope and intercept respectively.

From Table 2, the activation energies were positive indicating that the process is an endothermic process. The differential enthalpy (ΔH) and differential entropy (ΔS) were positive indicating that the extraction process is endothermic and irreversible (randomized). The values of Gibbs free energy obtained at the extraction of beta carotene from palm oil using acetone and ethanol as solvents is non-spontaneous. This is in agreement with Silmaria et al., (2015).
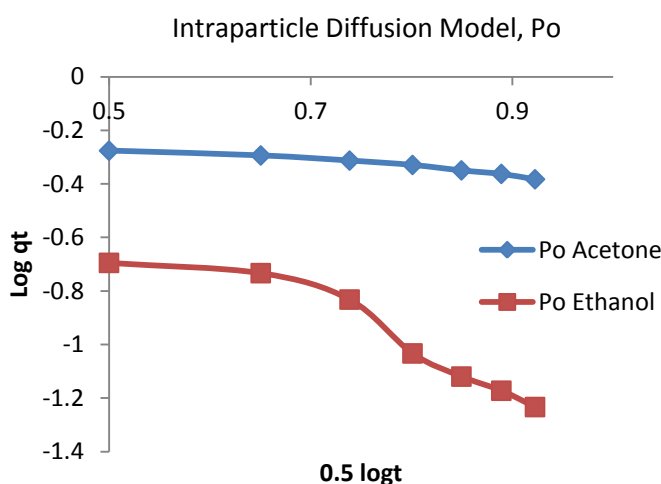

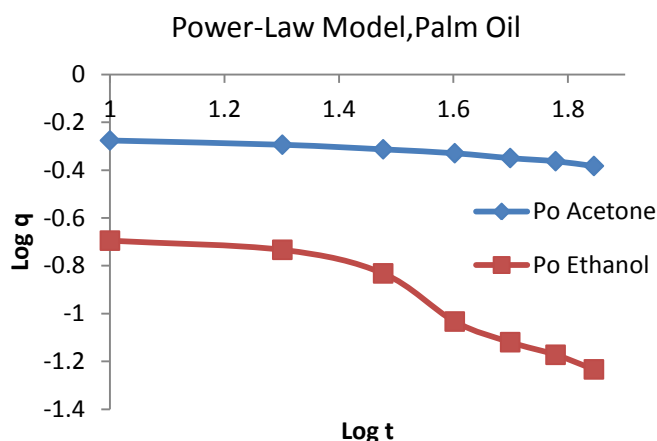**Fig. 8. Intra-particle diffusion model plot**

Fig. 9. Power law model plot

Table 3. Thermodynamics parameters

Samples	ΔE^0 (J/mol)	A	ΔH^0 (J/mol)	ΔS^0 (J/mol.K)	ΔG^0 (J/mol.K)			
					308K(35⁰C)	313K(40⁰)	318K(45⁰)	323K(50⁰)
P_0 Acetone	73,226.3864	1.433×10^{12}	73,226.3864	27.991	64,605.1584	64,465.2034	64,325.2484	64,185.2934
P_0 Ethanol	78,849.1446	2.7385×10^{10}	78,849.1446	30.941	69,319.3166	69,164.6116	69,009.9066	68,855.2016

4. CONCLUSION

Extraction of beta-carotene from palm oil by solvent method using acetone and ethanol were done. The kinetics and thermodynamics of process was studied. Among the six kinetic studied, which includes, First-order, second-order, pseudo-first-order, pseudo-second order, intra-particle diffusion and power law models, pseudo-second order was found to best fit the experimental data obtained with correlation coefficient of above 0.99. From the data of the thermodynamics parameters obtained, the process was seen to be endothermic, non-spontaneous, randomized (disorderly).

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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