Kinetic Study on Synthesis of Alkanolamides of Fatty Acids of Mangokernel Oil

(MangiferaIndica Linn)

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Abstract **- The kinetics of alkanolamide synthesis from Mango kernel oil *(MangiferaIndica Linn)*fatty acids and diethanolamine was studied using conventional reactor and microwave synthesis reactor. The experimental results were found to fit *Pseudo* first order kinetics. The influence of the temperature on the rate constant were determined for both reactors by fitting the results into the Arrhenius equation. The maximum rate of reaction was found at 150^{O} C. The activation energy for the reaction was determined using Arrhenius equation. Based on the experimental results a fatty acid to diethanolamine ratio of 1:2 (w/w) and 1:5 (5.37) (molar basis) and a temperature of 150 ^{O}C were found suitable for the synthesis of fatty acid alkanolamides of mango-kernel oil. The results obtained from microwave synthesis reactor were better than the conventional reactor due to effective heat transfer in microwave synthesis reactor.**

Keywords **- Kinetics, mango-kernel oil fatty acids, diethanolamide, microwave synthesis.**

I. INTRODUCTION

Alkanolamides are condensation products of the reaction of a primary or a secondary alkanolamine with a fatty acid, methyl ester or a triglyceride. Their chemical properties vary, depending on the length of their hydrocarbon chain and the nature of substituent on nitrogen atom [1]. Fatty alkanolamides are compounds that exhibit low reactivity and high thermal stability. Since the amide linkages are very stable chemically and not easily degraded in alkaline media, they are of great interest for application requiring relatively stable emulsifier [2]. Alkanolamides have a broad spectrum of uses such as in shampoos, detergents, cosmetics, lubricants etc [3].

The kinetic study of fatty alkanolamide is important, since it gives idea about the rate of reaction and factors affecting the rate of reactions. Further it helps in deciding the parameter such as time and temperature that can be used for obtaining the optimum yield of fatty alkanolamide. The determination of correct reaction order is of great importance, since the calculation of activation energies must be related to correct order of reaction and knowledge of activation energies is of value in the design and control of the reaction.

Not much more attention has been given for the kinetic study of fatty alkanolamide by the researchers. However KuanJu Line et al 2001 studied the synthesis and kinetic analysis of hexanonyldiethanolamide (HADEA), lauryl diethanolamide (LADEA) and oleoyldiethanolamide (OADEA) [4]. Candia anterctica lipase (Novozym G35) was used to catalyze the amidation of fatty acids with diethanolamine. Kinetic analysis showed that the yields of HADEA and LADEA in lipase catalyzed reaction were largely associated with the forward reaction constant.

The objectives of this present research is to study the kinetics of synthesis of fatty alkanolamides from nontraditional oil i.e., mango kernel oil *(Mangiferaindica Linn)* through measuring acid values of the amide for its yield. Also to compare the performance of conventional reactor and microwave synthesis reactor for this reaction. The physicochemical analysis of the mango kernel oil is also studied.

II. MATERIALS AND METHODS

2.1. Materials

The mango kernel oil*(Mangiferaindica Linn)*was procured from a Charbhuja Industries Pvt. Ltd, Maharashtra Industrial Development Corporation (M.I.D.C) area , Nagpur, (Maharshtra state).

All solvents used were of analytical grades. Diethyl ether, petroleum ether, ethanol, diethanolamine, HCl, H_2SO_4, NaOH, and KOH etc. were obtained from S.D. Fine Company and Qualigen Company, Mumbai.

2.2. Methods

2.2.1. Physio-chemical analysis of Mango-kernel oil (*Mangiferaindica Linn*)

2.2.1.1. Moisture content and specific gravity

The AOCS official method was followed for determination of the moisture content and specific gravity [5] of the oil at 30^{0}C.

2.2.1.2. Refractive Index
The AOCS official method was followed for determination of the refractive index [5] of the oils at 30^0C, by Abbes' refractometer in which the angle of total light reflectance from the surface is measured directly.
2.2.1.3. Flash and Fire Point
The flash and fire point of oil was determined by standard method [6] by using Pen sky-Marten's closed cup apparatus.

2.2. Chemical characterization
The chemical parameters such as acid value, saponification value, iodine value, peroxide value, unsaponifiable matter, were determined according to AOCS official methods [5].

2.2.3. Preparation of fatty acid
The mixed fatty acid were prepared by saponifying 100 gms of oil with 5N alcoholic potassium hydroxide. The content was refluxed for 2-3 hours on water bath. After saponification, the excess alcohol was distilled off and soap was dissolved in 150-200 ml hot distilled water. The mixed fatty acids were liberated by acidifying the soap solution with 1:1 H2SO4 using methyl red indicator. The acid was added till pink color was developed; the content was then boiled for 5-10 minutes and transferred to a separating flask. The fatty acid and glycerol layer formed were separated. Fatty acid layer was dissolved in solvent ether and washed with hot water 2 to 3 times in a separating flask and the mixture is allowed to settle in flask. Two layers were obtained, top layer containing fatty acids bottom containing glycerol. The fatty acid layer was separated and dried over anhydrous sodium sulphate [6].

2.2.4. Chemical characterization of mixed fatty acids of mango kernel oil
The chemical parameters such as acid value, saponification value, iodine value of mixed fatty acids prepared were determined according to AOCS official methods [5].

2.2.5. Fatty acid composition of Mango kernel Oil (*MangiferaIndica Linn*)
Fatty acid composition was determined by preparing methyl ester of fatty acid, which was then subjected to gas chromatography.
2.2.5.1. Preparation of Methyl Ester
A 100 mgm of mixed fatty acid was taken in a round bottom flask. Measured amount of boron tri fluoride-methanol solution (BF3- 125gms /liter of methanol) were added to mixed fatty acid. The mixture was heated and refluxed for 25-30 minutes at steady temperature. A 5 ml of heptane was added through condenser and the content was boiled for 5 minutes till it dissolved .The content was transferred to a separating flask. The lower layer formed was removed from the bottom of separating flask. To the upper layer anhydrous sodium sulphate was added and traces of moisture were removed. This dried heptane solution was subjected to Gas chromatography. The ester sample was injected into Gas Chromatography apparatus (Model GC-14 of Shinadzu, Singapore). Packed Column was used, its film thickness was 0.31µm, and column length was 2 meter. The temperature was from 160^O C heated to 250^OC (1.5^OC/min): injector 250 ^{O} C, detector 270 ^{O} C; carrier gas 4.0ml/min hydrogen; 6ml/min air and 4 ml/min nitrogen; manual injection volume < 1 µl. The peak area was computed by integration software of packed column and percentage of fatty acid methyl esters were obtained as weight percent by direct internal normalization [7].

2.2.6. Kinetics of synthesis of fatty alkanolamide (diethanolamide)
2.2.6.1. Conventional Method
A 40 grams of mixed fatty acids of Mango Kernel (MK) oil and 80 grams of diethanolamine (ratio : 1:2 w/w and approximately mole ratio of 1:5) was charged into 250ml three necked glass reactor placed in heating mental. The reaction was carried out first at 100^O C. The content was stirred continuously by regulating the speed of stirrer. When the reaction mixture was thermostatically adjusted the samples were withdrawn at regular intervals of time i.e. at 1, 3, 5, and 7 hours and acid value and nitrogen content of the amide formed of each sample was determined according to AOCS official methods (10). The same procedure was repeated by conducting the reaction at 120^OC and 150 ^{O} C.
2.2.6.2. Microwave synthesis method
The diethanolamine and mixed fatty acids were mixed properly in the ratio of (1:2w/w and on mole basis 1:5). The whole mass was fed to the vial (10 ml,) of the microwave synthesis reactor (Model CEM-3011, CEM Corporation USA) batch wise. The vial was filled up to prescribed mark and kept into the microwave synthesis reactor. The reaction temperature in the microwave synthesis reactor was first maintained 100^O C. The vials were withdrawn at regular intervals of time at 3, 5, 7, and 10 minutes. The acid value and nitrogen content of fatty diethanolamide thus prepared at different time intervals were then determined using AOCS official methods (10). The same procedure was repeated for 120^O C and 150^OC temperature.

III. Result And Discussion

3.1. Physicochemical Characteristics of Mango Kernel oil
The physiochemical characteristics of the mango kernel oil procured was compared with that reported by J.M. Nzikou et.al [9], Saiprabha M. Mahale and A.S. Goswami-Giri [10] and T.Anwer, et.al, [11] andare given in Table-1. The oil used in the present study was having pale yellow color with moisture (volatile matter) content- 1.12%, refractive index 1.4578, Acid value 2.41, Iodine value 47.3, Saponification value 192.4, Unsaponifiable matter 2.33 and peroxide value 3.09 with flash point more than 140 ^{0}C and fire point 166^O C. It was observed that the reported values are different [9], [10] and [11] from those obtained in the present study. Even the values are different for the Indian mango-kernel oil obtained from different source [10].

Such variation in the values of these parameters may be attributed to possible changes in environmental and geological conditions of the region as well as the particular species of the mango.

Table 1 Analysis of mango kernel oil and its mixed fatty acids

Se. No.	Particulars	Results Obtained		Reported values		
		Crude oil	Mixed fatty acids	Ref. [9]	Ref.[10]	Ref. [11]
1	Moisture (Volatile matter) %	1.12	0.90	---	0.53	---
2	Color	Pale yellow	Dark Reddish	----	Pale yellow	----
3	Acid Value (mg KOH/g)	2.41	188.33	7.86	2.22	0.38 ± 0.01
4	Iodine Value (wiji's)	47.3	49.13	43.0	45.55	50.09 ±1.95
5	Saponification Value	192.4	196.3	206	173.3	189 ±3.68
6	Unsaponifiable Matter %	2.33	0.98	4.35	3.45	1.11±0.04
7	Peroxide Value (m. eq/kg)	3.09	--	0.20	2.5	2.10±.04
8	Flash point ^{0}C	>140 ^{0}C	--	----	------	---
9	Fire Point ^{O}C	166 ^{O} C	--	-----	-------	----
10	Refractive Index at 40 ^{0}C	1.4578	--	----	------	1.461±0.04
11	Specific gravity at 30 ^{0}c	0.901	-----	----	0.9100±.03	---

3.2. Preparation and Characterization of mixed fatty acids of the Mango Kernel Oil:

The yield of mixed fatty acid obtained was 89.61%. Chemical parameters such as acid value, iodine value, saponification value and unsaponifiable matter of the mixed fatty acid determined by AOCS method [5] and physical parameters like moisture and color are reported in Table- 1. Mixed fatty acid was found to have dark reddish color with less moisture content than the mango kernel oil. Except unsaponifiable matter all other values such as free fatty acid (acid value), iodine value and saponification value were more than mango kernel oil.

3.3. Fatty acid composition of Mango Kernel Oil (*MangiferaIndica Linn*)

The fatty acid composition of mango kernel oil used in the present study and the composition of the mango kernel oils reported in the literature are given in Table-2.

Table 2 Fatty acid composition of Mango (*MangiferaIndica Linn*) Kernel Oil

Se. No.	Name of Fatty acids	Formula	Structure	Weight %	Reported Values			
1	**Saturated Fatty acids**				Ref[8]	Ref[9]	Ref[13]	
							(a)	(b)
	Palmitic acid	$C_{16}H_{32}O_2$	16:0	6.68%	6.38±0.82	6.43	7.18	7.50
	Stearic Acid	$C_{18}H_{36}O_2$	18:0	41.43 %	37.51±1.20	37.73	38.9	38.70
2	**Unsaturated Fatty acids**							
	Oleic acid	$C_{18}H_{34}O_2$	18:1	46.083%	46.67±0.32	46.22	42.6	43.20
	Linoleic acid	$C_{18}H_{32}O_2$	18:2	4.631%	7.21±0.88	7.33	5.7	6.20
	Linolenic acid	$C_{18}H_{30}O_2$	18:3	0.369%	2.22±0.53	2.30	5.3	2.90
	Arachidic acid	$C_{20}H_{40}O_2$	20:0	0.347%	-----	-----	-----	------
	Gadoleic acid	$C_{20}H_{38}O_2$	20:1	Traces	-----	------	-----	------

Variety Chausa

Variety Dusheri

The major saturated fatty acids in the mango kernel oil are stearic (41.43%) and palmitic (6.68%) acids and the main unsaturated fatty acids are oleic (46.083%), linoleic (4.631%) acids. Bligh E.G. and W.J. Dyes (1959) [8] , J.M.Nzikou et.al [9] and andDhingra S. and A.C. Kapoor [13] reported saturated fatty acids, stearic acid(37.51±1.20),(37.73%),(38.9), (38.7) and palmitic acid(6.38±0.82), (6.43%), (7.18), (7.50) respectively and unsaturated fatty acids, oleic (46.67±0.32), (46.22%), (42.6), (43.2) and linoleic (7.21±0.88), (7.33 %), (5.7), (6.2) respectively. The proportion of unsaturated fatty acids was found to be greater than saturated fatty acids from our study which is in agreement with result reported by Bligh E.G. and W.J. Dyes [8], Nzikou et.al [9] and Dhingra S. and A.C. Kapoor [13]. The greater proportion of oleic acid (46.083%), an unsaturated fatty acid helps for amidification reaction.

3.4. Kinetics of synthesis of fatty alkanolamide

The reaction of diethanolamine with fatty acid to give the fatty alkanolamide contains considerable amount of unreacted (Diethanolamine) DEA which accounts for aqueous solubility of the product [14]. This makes it suitable for use in shampoos, detergent and cosmetics.

The amidification reaction taking place is as follows:

Fatty acid + Diethanolamine→ Fatty alkanolamide + H_2O.

The ratio of fatty acid to diethanolamine reported in the literature was 1 mole: 6 mole, where as in the current investigation the mole ratio used was 1 mole: 5.37 mole and on weight basis 1:2 for the kinetic study of fatty alkanolamide. The use of excess amine improves the yield of product. (Gregorio C. Garvajio, (2005), [14].

The composition of unsaturated fatty acid in mango kernel oil used was oleic acid 46.08 %, linoleic acid 4.63% and linolenic acid 0.369%. This unsaturated fatty acids which reacted with excess diethanolamine (DEA) to produce fatty alkanolamide.

Table 3 Variation in acid value and rate constant k with temperature and time (Conventional Reactor Method)

Se. No.	Time in minutes	Acid value			k, minute $^{-1}$		
		100 ^{O}C	120^{O}C	150^{O}C	100 ^{O}C	120^{O}C	150^{O}C
1	0	64.66	64.66	64.66	--	--	--
2	60	57.23	56.36	49.25	$2.03*10^{-3}$	$2.29*10^{-3}$	$4.538*10^{-3}$
3	180	52.46	42.74	27.27	$1.1617*10^{-3}$	$2.30*10^{-3}$	$4.795*10^{-3}$
4	300	49.22	36.72	19.01	$0.9090*10^{-3}$	$1.88*10^{-3}$	$4.080*10^{-3}$
5	420	44.72	27.33	11.77	$0.8776*10^{-3}$	$2.05*10^{-3}$	$4.05*10^{-3}$

Table 4 Variation in acid value and rate constant k with temperature and time (Microwave Synthesis Reactor Method)

Se. No.	Time in minutes	Acid value			k, minute $^{-1}$		
		100 ^{O}C	120^{O}C	150^{O}C	100 ^{O}C	120^{O}C	150^{O}C
1	0	64.66	64.66	64.66	--	--	--
2	3	54.91	46.43	30.86	0.0544	0.1104	0.2462
3	5	47.72	37.27	17.89	0.0607	0.1192	0.2570
4	7	43.24	29.21	12.37	0.0574	0.1135	0.2362
5	10	36.52	24.16	06.17	0.0571	0.09845	0.2349

Table 5 Effect of Temperature on rate constant, 'k'

Se. No.	Temperature in ^{O} K	' k' by Conventional method	' k' by Microwave Synthesis method
1	373	$1.2445*10^{-3}$	0.0574
2	393	$2.1300*10^{-3}$	0.1104
3	423	$4.3657*10^{-3}$	0.2435

Table 6 Effect of time and temperature on Nitrogen and Amide content of fatty alkanolamide (Conventional Reactor)

Se. No.	Time in minutes	Nitrogen Content in %			Amide Content in %		
		100 ^{O}C	120^{O}C	150^{O}C	100 ^{O}C	120^{O}C	150^{O}C
1	0	1.62	1.62	1.62	11.6	11.6	11.6
2	60	3.79	4.55	5.40	27.1	32.5	38.6
3	180	6.87	7.55	10.13	49.1	53.9	72.4
4	300	8.23	9.01	11.96	58.8	64.4	85.4
5	420	9.02	10.44	12.42	64.4	74.4	88.7

Table 7 Effect of time and temperature on Nitrogen and amide content of fatty alkanolamide (Microwave Synthesis Reactor)

Se. No.	Time in minutes	Nitrogen Content in %			Amide Content in %		
		100 ^{O}C	120^{O}C	150^{O}C	100 ^{O}C	120^{O}C	150^{O}C
1	0	1.89	1.89	1.89	13.49	13.49	13.49
2	3	3.69	6.55	8.89	26.35	46.78	63.49
3	5	7.93	8.56	11.96	56.64	61.13	85.42
4	7	8.77	9.21	12.42	58.8	65.77	88.70
5	10	9.67	11.89	13.26	69.06	84.91	94.70

The kinetic study results obtained using conventional reactor and microwave synthesis reactor. (Table-3 and Table-4) were tried to fit in the rate equation of first order and second order reactions. Since the results obtained fits into first order rate equation, kinetic model for the first order equation is developed.

3.5. KINETIC MODEL

The kinetic model used in this work is based on following assumptions.

The rate of amidification reaction under the operating conditions used was controlled by chemical reaction,

The rate of byproduct formation was negligible compared to fatty alkanolamide formation.

The diethanolamine to mixed fatty acid ratio (2:1 w/w, 5:1 on mole basis) used was high enough for the diethanolamine concentration to remain constant throughout the process.

Under these conditions, the reaction was assumed be *pseudo*-homogeneous, first order reaction and hence confirm the following kinetics.

$$A+B \xrightarrow{k_1} C+D \text{ --------- (1)}$$

Where,

A → Fatty acid.
B →Diethanolamine.
C →Diethanolamide.
D → water.

Let-

C_A → Concentration of Fatty acid (FA) (acid value in mg KOH/ gm)
C_B → Concentration of Diethanolamine (DEA) .
C_c → Concentration of Diethanolamide.
C_d → Concentration of Water.

The rate of reaction (1) is given by

$$\frac{-dC_A}{Dt} = k_1 C_A . C_B \quad (2)$$

C_B remains constant as it is in excess

$$\frac{-dC_A}{Dt} = k_1 C_B . C_A \text{ ---------- (3)}$$

$$\frac{-dC_A}{dt} = k . C_A \text{ ---------- (4)}$$

Ask$_1C_B$= k = New rate constant.

But $C_A = C_{AO}(1-X_A)$ ------ (5) Where C_{AO}→ Initial concentration of fatty acids

Therefore $dC_A = -C_{AO}dX_A$--- (6) Where X_A→ Fractional conversion of fatty acids.

$$C_{AO}\frac{dX_A}{dt} = k . C_{AO}(1-X_A)$$

i.e.

$$\frac{dX_A}{(1-X_A)} = k . dt \text{----------(7)}$$

On integration equation (7) gives

$$-\ln(1-X_A) = k\,t \text{----------(8)}$$

The kinetic study results obtained, (Table -3 and Table -4) fits into equation (8) confirms that alkanolamide synthesis reaction is of first order and since two reactants are involved in the synthesis, more precisely is pseudo first order reaction.

The value of 'k' for each of the reaction that is mixed fatty acids of mango kernel oil and diethanolamine to get fatty acid diethanolamide and water 100, 120 and 150^0 C was calculated using equation (8).Since the data obtained at 100 ^{0} C fits in to equation (8) the reaction may be a first order reaction and also two reactants are involved in the reaction indicating that it is pseudo first order reaction.

3.6. Effect of temperature on the rate constant 'k':

The effect of temperature on fatty alkanolamide synthesis reaction was studied by both conventional and microwave synthesis reactor method, of mixed fatty acids of mango kernel oil. It is observed that for all these reactions the value of rate constant increases with temperature, in both conventional and microwave synthesis reactor (Table-5). That is the rate of reaction increases with temperature as reported in the literature [15].

Further it is observed that the values of the rate constants k at temperature 100 ^{O}C, 120 ^{O}C and 150 ^{O}C obtained in microwave synthesis reactor method were more compared to conventional reactor. This might be due to effective heat transfer at molecular level taking place in microwave synthesis reactor. Therefore the time required for the completion of the reaction is less in microwave synthesis reactor. Since the rate of all these reactions was maximum at 150 ^{0} C, the fatty acids diethanolamide from the mixed fatty acids of these oils were prepared at this temperature.

3.7. Activation Energy:

Activation energy varies widely for different chemical reactions, from a few joules per mole to 10 joules/mole. For the same chemical reaction, the value of activation energy depends on the form of distribution function of the molecules with respect to the energies of their translational motion and with respect to the internal degrees of freedom (electronic, vibrational, and rotational).

The activation energy for fatty acid diethanolamide synthesis from mango kernel oil, was determined using Arrhenius law:

$$k = k_O e^{-E/RT} \text{ ------(9)}$$

$$\ln k = \ln k_o^{-E/RT} \text{----------(10)}$$

Where k→Reaction rate constant , k_o→Frequency factor
T →Absolute Temperature, E → Energy of Activation.

The activation energy was determined by plotting a graph between ln K versus 1/T . The activation energy obtained using conventional reactor is 37.413 kJ. $mole^{-1}$(Figure-1) and that using microwave synthesis reactor is 36.283 kJ $mole^{-1}$(Figure 2). Conventional heating used in the conventional reactor is inefficient and time consuming where as in the microwave heating is efficient and deep within the reactants and therefore for the same reaction time required is in minutes. Hence activation energy might be low for the amidification reaction carried out in the microwave reactor than that in the conventional reactor. However, reaction rate constant is also more in microwave reactor.

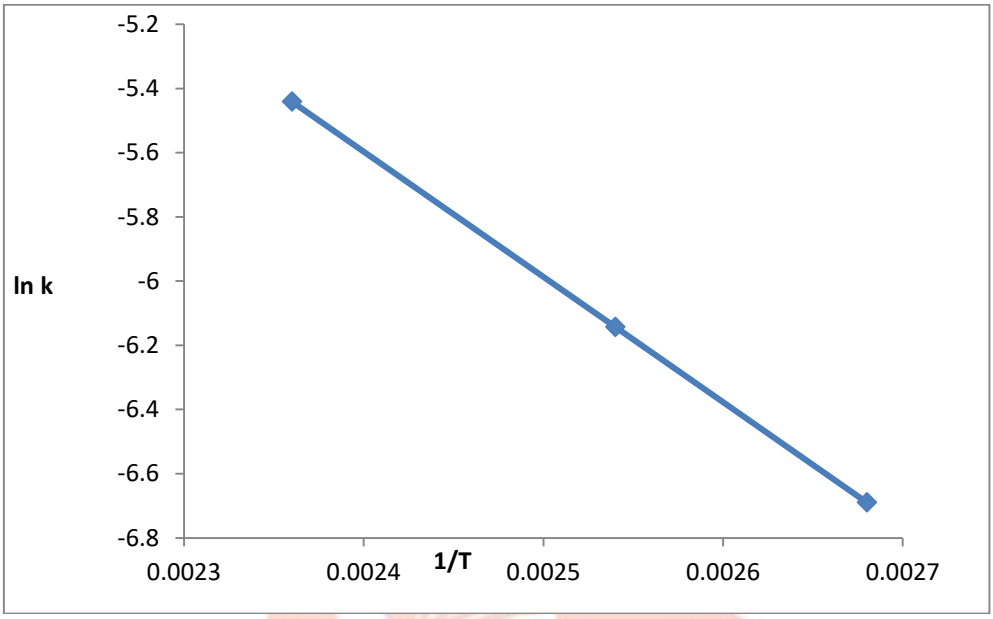

Figure-1 Activation energy (Conventional reactor)

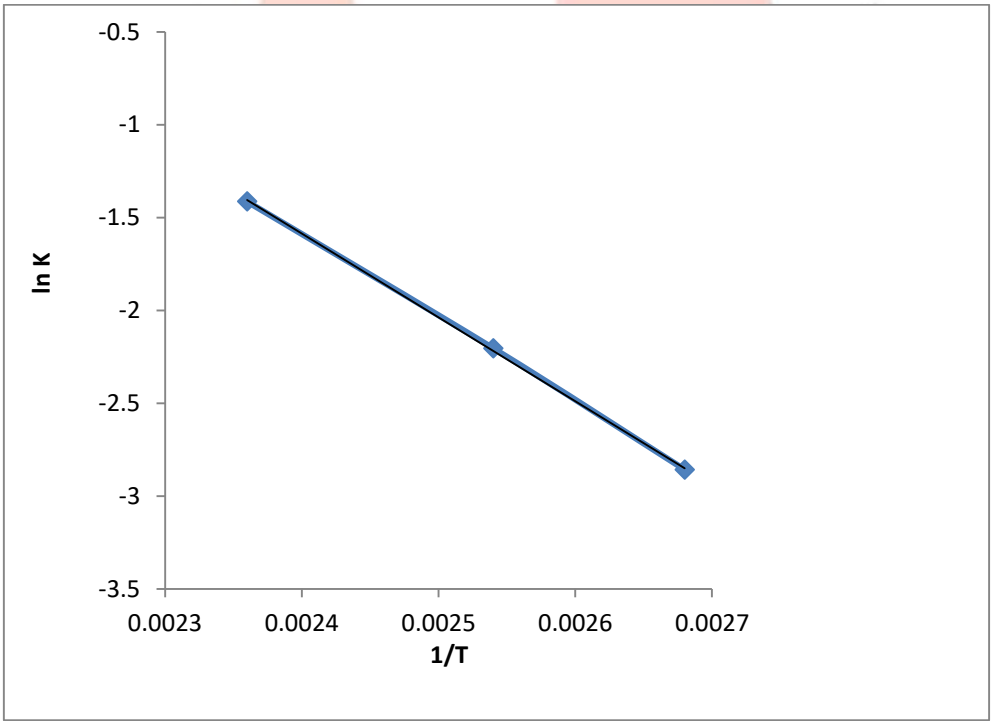

Figure-2 Activation energy (Microwave synthesis reactor)

3.8. Effect of temperature and time on the rate of formation of product

Effect of temperature on the rate of formation of product fatty alkanolamide was studied by both by conventional and microwave synthesis reactors. The amide content of fatty alkanolamide formed was determined by estimating the nitrogen content at different time intervals and temperatures. The results obtained by conventional reactor and microwave synthesis reactor are shown in Table-6 and Table-7 respectively.

It was found that the amide content of fatty diethanolamide was increased with the reaction time and increase in temperature. The final amide content was also increased for both conventional and microwave synthesis reactor. However, the amide content was found to be more in the fatty alkanolamide (94.70% at 150^{O} C) in the microwave synthesis reactor. It is because in microwave heating, microwave energy is unique source, it creates heat deep within the material being processed. This property results in much shorter process time and higher yield of product.

IV. CONCLUSION

Mango kernel oil (*Mangiferaindica Linn*), a non-traditional oil contains stearic acid and oleic acid as the principle fatty acids and the proportion of unsaturated fatty acids is greater than the saturated fatty acids. The amide content of alkanolamide synthesized increases with time and temperature in both conventional and microwave synthesis reactors. The microwave synthesis reactor is better than the conventional reactor as shorter time and higher percentage of amide is obtained in microwave synthesis reactor.
The kinetic study of synthesis of fatty alkanolamide showed that the reaction is pseudo first order reaction and the rate of reaction increases with temperature and the maximum rate of the reactions is at 150 ^{0}C with more percentage of amide.
Thus, kinetic study is important as it gives the information about the process parameters, temperature, time etc. to get the optimum yield of the product.

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