

Kinetic Study for Oxidation of Existing Mercaptans in Kerosene Using Impregnated Activated Carbon with MEROX Catalyst in Alkaline Solution

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Received 13 June 2005; accepted 12 July 2005

Abstract

MEROX process is used for oxidation of mercaptans to disulfide from oil cuts using air in the presence of alkaline solution and MEROX catalyst. In this paper, the kinetic of conversion existing mercaptans in kerosene is studied for better understanding of MEROX process. Impregnated activated carbon with powder of MEROX catalyst was analyzed in laboratory scale. The results indicate that in laboratory scale, because of pore diffusion resistance, the impregnation efficiency is low. Also, optimum conditions for mercaptan extraction by using of soda solution obtained. Meanwhile the kinetic of oxidation reaction is considered in the presence of MEROX catalyst and activated carbon and also, impregnated activated carbon with MEROX catalyst.

The results, indicate for mercaptan decreasing, in similar conditions the efficiency of Impregnated activated carbon with MEROX is better than other catalysts.

Also, the kinetic model related to catalytic reaction for mercaptan conversion to disulfide in the presence of impregnated activated carbon with MEROX catalyst, is represented.

Key words: MEROX, mercaptans, activated carbon, kinetics, oxidation, kerosene

Introduction

With due attention to population in industrial area, environmental problems, due to presence of compounds contain of sulfur, Nitrogen and oxygen in oil products, is considered more than ever.

In recent years, the refining industry is under immense pressure to produce cleaner fuels because of stricter environmental regulations.

Light oil cuts, mostly, are contain of mercaptans and should be treated, for presenting as acceptable products.

Mercaptan compounds are corrosive and malodorous. So, removing or conversion of these to harmless materials is important

One of processes used for treating of mercaptans is MEROX process. This technology is licensed by UOP Company and is used for chemical treating of light cuts to middle distillation products.

Since 1975, there were more than 1000 MEROX unit with low operating and maintenance costs and therefore had been very effective and consequent.

In this paper, the kinetic of mercaptans oxidation by using of impregnated activated carbon with MEROX catalyst and also, influence of activated carbon and MEROX catalyst on reaction are considered.

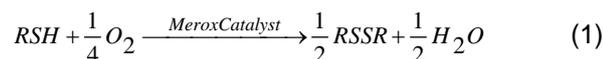
Also, The summary of analysis doing on MEROX catalyst and activated carbon and kerosene will be represented.

In this study, kerosene product from Shiraz (Iran) refinery (atmospheric distillation product) has selected for treating by using of impregnated activated carbon with MEROX catalyst.

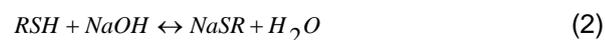
(1) Theory ^[1]

MEROX process is used for mercaptans separation or conversion to disulfide in the presence of soda solution and MEROX catalyst. Mercaptans and especially, mercaptans with low molecular weight, are solved in soda solution. Transferring of mercaptans from an organic phase to an aqueous (soda) phase is named extraction. Mercaptans transferred to soda phase is converted to sodium mercaptid that is converted to disulfide using MEROX catalyst.

Total interaction is as follows:



Above interaction is resulted of two following interaction:



by using of MEROX catalyst, Direction oxidation of mercaptan by using of oxygen (air) for conversion to disulfide is accelerated and will be possible in industrial scale.

As well as MEROX, activated carbon also is reaction catalyst.

Produced disulfides from reaction (3) are insoluble in soda solution and separate from it, easily. Therefore, soda will be reused for mercaptan extraction from hydrocarbon feed. The rate of above reaction increases by increasing of air quantity, system temperature, mixing speed and increasing of MEROX catalyst concentration.

There is two type of process for MEROX consist of sweetening and extraction. Sweetening process is used in two options: (1) solid bed sweetening and (2) liquid-liquid sweetening.

The objective of sweetening options is conversion of mercaptans (RSH) to disulfides not separated from Hydrocarbon. but, In Extraction, mercaptans separate from Hydrocarbon. in solid bed sweetening, MEROX catalyst is placed on activated carbon bed, but In liquid-liquid sweetening and extraction, MEROX catalyst is used in the form of liquid.

Extraction process is used for treating of oil cuts with final boiling point less than 205°C, whereas liquid-liquid sweetening process is suitable for treating of cracked cuts with final boiling point less than 205°C and Gasoline with approximate boiling point less than 135°C.

If treating of oil cuts by above options isn't possible, catalyst fixed bed process is an economical solution.

(2) Experimental studies and experiment options

(2-1) Analysis of MEROX catalyst, activated carbon and kerosene

• MEROX catalyst

First, solid MEROX catalyst (powder), analyzed by method of Energy Dispersion X-Ray (EDXR). MEROX catalyst is a powder. EDXR couldn't distinguish the amount of carbon (C) and Nitrogen and hydrogen (H), so, the amount of (CHN) was determined in standard laboratory. Therefore, presented percents, are based on total existing components.

If percent of CHN is deducted of these presented percents, actual amount of components will be obtained. Weight percent of CHN in solid MEROX catalyst is shown in table 1.

Table 1. weight percent of CHN in solid MEROX catalyst.

Component	Wt%
Nitrogen (N)	11.2
Carbon (C)	38.8
Hydrogen (H)	2.2
CHN	52.2

The results of MEROX catalyst analysis is shown in table 2:

Table 2. MEROX catalyst analysis results

Component Name (Formula)	Component (wt%)	Actual Component (wt%)
Cobalt (Co)	11.9582	5.716
Iron (Fe)	307 ppm	146.746 ppm
Copper (Cu)	389 ppm	185.742 ppm
Calcium (Ca)	0.5002	0.23909
Molybdenum (Mo)	7.6924	3.67697
Sulfur (S)	40.5471	19.38151
Sodium (Na)	1.7412	0.83229
Oxygen (O)	34.4721	16.4776

For determination the amount of MEROX catalyst placed on activated carbon, amount of existing cobalt in MEROX catalyst is considered as a base for measurement. Cobalt percent achieved via atomic absorption, atomic absorption laboratory announced the amount of cobalt in solid MEROX catalyst equal 5.8 weight percent.

MEROX catalyst is a blue heterogeneous powder and a mixed of cobalt sulfonite phthalocyanine derivatives (COPC (NaSO₃)₂ COPC(Na₂SO₃)) with sodium and calcium salts.

• **Activated carbon**

Porous activated carbon is known as the base of MEROX catalyst physical properties that are given in table 3.

Table 3: physical properties of activated carbon

Property	
Particle Bulk Density	0.31 g/cm ³
Particle area	0.882 m ² /g
Mean particle diameter	0.77 mm

• **Kerosene**

The properties of kerosene are given in table 4

Table 4. Kerosene properties

Property	
Molecular Weight	172.2
Specific Gravity	0.8155
Initial Boiling Point (IBP)	177 °C
Final Boiling Point (FBP) (97% Distillation)	263 °C
Viscosity (30°C)	1.3 cp
(40°C)	1.13 cp
(50°C)	0.98 cp
Initial existing mercaptan concentration in kerosene	193 ppm

(2-2) Experimental tests and options

Tests was done in autoclave with volume 500 ml and design pressure 136 bar and design temperature 320°C. This Set-up system is shown in figure 1.

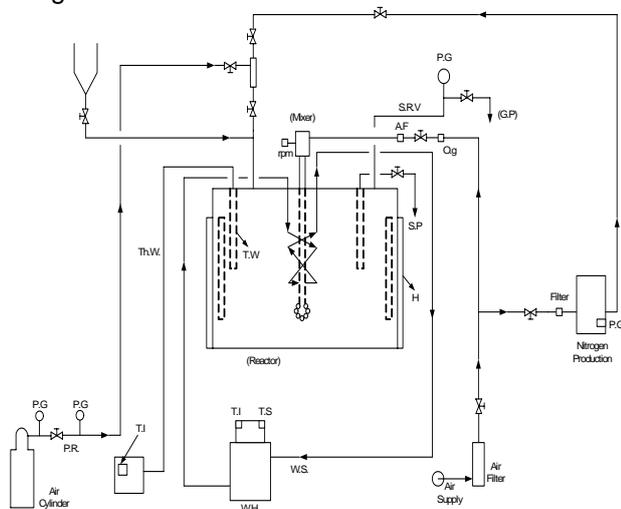


Figure 1. Set-up system for experimental tests

(2-2-1) Condition development for mercaptan extraction by using of soda solution

Optimum operating conditions for tests are as follows:

- Initial kerosene volume = 400 ml
- Temperature = T=30°C
- Pressure = P = 5-6 kgf/cm²
- Mixer speed = 2000-2050 rpm

For achieving of maximum oxygen solubility in aqueous phase, the pressure of mercaptan oxidation reaction in industrial scale is 6 kgf/cm². for this reason, the pressure of extraction reaction has been selected in limit 5-6 kgf/cm².

For elimination of mercaptan mass transfer from organic phase (kerosene) to aqueous phase (soda solution), Mixer speed has been selected equal 2000 rpm.

The extraction tests were done in different volume and concentration of soda solution and the results illustrate that optimum conditions is 5 ml of 10%wt soda solution.

Different cases was compared in figure 2. in this figure, mercaptan concentration (ppm) was plotted against time (minuets) for three different cases.

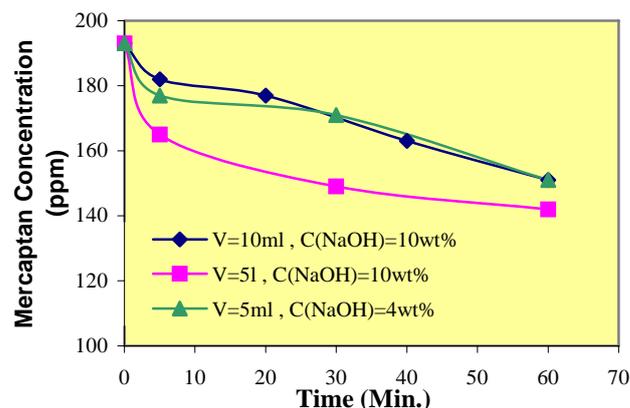


Figure 2. Comparison different cases of soda solution

Tests for oxidation of mercaptan to disulfide by using of air in the presence of MEROX catalyst (on base of activated carbon) and soda solution.

The reaction for oxidation of mercaptan to disulfide is possible in the presence and absence of oxidation – regeneration catalyst^[2]. In any case, alkaline aqueous solution (NaOH) is an essential material for treating. because, mercaptid ion (RS⁻) is active ion of reaction, so, soda solution dispersing in hydrocarbon is important.

Activated carbon , alone , is a catalyst for oxidation of mercaptan to disulfide^[3]. but impregnated activated carbon with MEROX have

catalytic property rather than catalytic components.

(2-2-2) Pore diffusion resistance study

For studying pore diffusion resistance, impregnated activated carbon with MEROX was crushed. The results were shown catalyst, has lost, catalytic efficiency. so, existing of pore diffusion resistance is demonstrated via following formula:

$$\frac{(-r_A)_{obs} L^2}{D_e C_{AL}} \approx 1.0 \quad (4)$$

(2-2-3) Impregnation of activated carbon by using of MEROX catalyst [4,5]

Impregnation of activated carbon with MEROX using methanol solution was done in laboratory scale.

Nothing that 5.4 mg of MEROX should be placed on one-gram of activated carbon and because of laboratorial impregnation, more quantities of MEROX and methanol solvent was selected so that MEROX concentration was 1.4 times of industrial concentration. Impregnation stages consist of contacting of methanol MEROX solution with activated carbon particles within 2-3 days, methanol discharging using water washing and drying of particles by nitrogen flow.

The amount of MEROX placed on activated carbon, by above option, was 9.65 mg for one gram of carbon.

Because, it was possible that one of controller factors for MEROX placing is pore diffusion resistance of MEROX in activated carbon, so activated carbon particles was crushed to size smaller than 182 micrometer.

The results obtained from impregnation of activated carbon with similar concentration of MEROX in methanol and only by using of a little changes in option (consideration of mixer for mixing and hokhner funnel for sending out of methanol) was shown that, MEROX concentration on activated carbon have been changed (12.06 mg for one gram activated carbon), That indicate existing pore diffusion resistance in activated carbon.

(2-2-4) The effect of pressure on catalytic reaction for oxidation of mercaptan using of air.

The effect of pressure in aforesaid reaction considered via reducing of pressure from 6 bar to 4 bar . The results was shown that pressure variations haven't effect on variations of mercaptan concentration with time. So, in this

pressure limit, reaction order related to oxygen can be take equal zero.

(2-2-5) Tests for achieving to kinetic model in with impregnated activated carbon with MEROX catalyst

For determination of kinetic model (variations of reaction rate with concentration) in batch reactor, variations of mercaptan concentration was measured according to time. For achieving to above objective, a test is done in temperature of 30°C by using of impregnated activated carbon with MEROX and then, for calculating constant of kinetic model, aforesaid test is done in other temperatures. Tests conditions are as follows:

- type of catalyst : impregnated activated carbon with MEROX catalys
- catalyst weight : 0.5 g.
- mean particle diameter : less than 0.8 mm.
- mixer speed : 2000 rpm.
- Pressure : 6 atm and
- Temperatures : 30, 40, 50°C.

The tests related to each stages have been repeated twice and mean mercaptan concentration according to each time was shown in table 5. Meanwhile, it is reminded that in each stage of test, 25 ml of kerosene is used for mercaptan analysis and initial volume during all tests is 400 ml .

The results of tests (variation of mercaptan concentration with time) in temperatures of 30, 40, 50°C under foresaid conditions is shown in table 5.

Table 5. variations of mercaptan concentration with time

Time (min.)	Mercaptan conc. (ppm) T=30°C	Mercaptan conc. (ppm) T=40°C	Mercaptan conc. (ppm) T=50°C
0	193	193	193
5	152	142.5	133.5
30	65.5	50.5	38.5
60	31.5	22.5	18
90	22.5	14.5	14
120	14.5	12.5	10

The results of above tests have shown in figure 3. just as specified from figure 3, conversion of mercaptan increases with increasing of temperature.

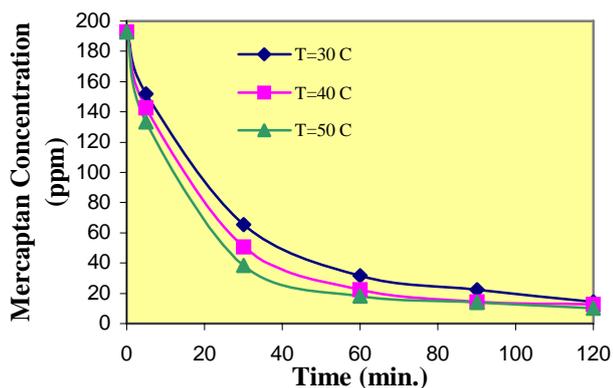


Figure 3. Variation of mercaptan concentration with time also, variations of conversion according to temperature is shown in figure 4.

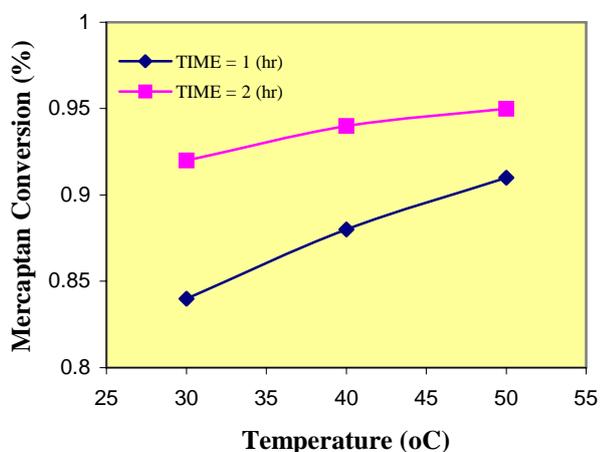


Figure 4. Variation of mercaptan conversion according to temperature

Catalytic reaction in temperature of 30°C is compared with mercaptan extraction by using of soda solution in figure 5.

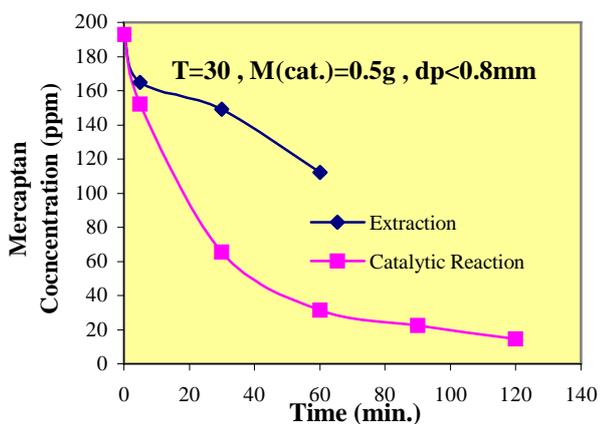


Figure 5. Comparison of catalytic reaction and mercaptan extraction

The effect of catalyst presence on variations of mercaptan concentration is obvious from above figure, completely.

(2-3) Tests in the presence of activated carbon and MEROX catalyst

Activated carbon and MEROX are catalysts for oxidation of mercaptan to disulfide, alone. Different tests were done by using of different weight of activated carbon and MEROX.

The results of consideration the effect of three type of catalyst, activated carbon, MEROX and Impregnated activated carbon with MEROX on catalytic reaction is shown in figure 6.

test conditions are consist :

- Temperature : 30°C
- Pressure : 6 atm
- Mixer speed : 2000 rpm and
- Catalyst weight : 0.5 g

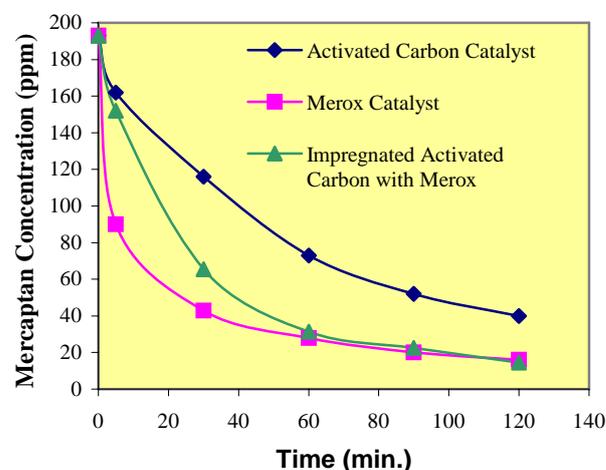


Figure 6. Effect of catalyst type on catalytic reaction

The above results, illustrate that the efficiency of Impregnated activated carbon with a little MEROX is better than activated carbon, alone. i.e. with Impregnation a little of MEROX (9.65 mg of MEROX in one gram activated carbon) , increase the efficiency of catalyst and the variations rate of mercaptan concentration according to time.

With comparing the efficiency of Impregnated activated carbon with MEROX (activated carbon particle size = 0.77 mm) with MEROX solution, it is specified that because of presence of pore diffusion resistance, variations of mercaptan concentration with time in case of dispersed MEROX in kerosene is better than Impregnated activated carbon with MEROX. for comparison of catalysts efficiency, time is important factor. because, it is possible that a catalyst, just in specific time period, has best efficiency.

The effect of temperature on catalytic reaction was considered and for achieving of maximum conversion, 50°C was selected as

optimum temperature. The conversion of mercaptan in temperatures of 30, 40, 50°C is 95%, 93%, 92%, respectively.

The effect of increasing of catalyst weight was studied. The results was shown that by increasing of catalyst weight, the variations rate of mercaptan concentration increase .

(2-3-1) kinetic equation modeling of mercaptan oxidation reaction to disulfide, in the existence of soda solution and impregnated activated carbon with MEROX catalyst.

(2-3-1) Model presentation for variations of mercaptan concentration according to time.

The test for determination of foresaid reaction model in temperatures of 30, 40, 50°C 0.5 gr of catalyst was done and and then, variations of the ratio of mercaptan concentration (the ratio of mercaptan concentration to initial mercaptan concentration) in every time, was plotted against time.

Laboratory data fitting in one model was done using "Eureka" software program (by using of least square method). This program don't determine, fitting equation, and only considers information on it and presents model parameters, fitting error, the curve of variations of function according to related variables and variations of function in every other point.

Following relationship is presented for variations of mercaptan concentration ratio according to time (hour).

$$f(t) = \frac{C_A}{C_{A_0}} = a_0 \text{EXP}(a_1 t) + a_2 + \frac{a_3}{a_4 + a_5 t} \tag{5}$$

in above relationship , a_0 to a_5 are model parameters. these parameters, change in different temperatures from 30°C to 50°C.

Presented model had minimum error and it's form is combined of exponentially form (variations of concentration form for first order) and fractional form (variation of concentration form for second order reaction). parameters of model for temperatures of 30, 40, 50°C are given in table 6.

Table 6. Parameters of model for different temperatures

Coefficien t	T=30°C	T=40°C	T=50°C
a_0	0.31090	0.63215	-0.66438
a_1	-2.72716	-2.8286	-1.130
a_2	0.021567	0.041718	-0.1258
a_3	0.35417	0.13322	0.78177
a_4	0.49903	0.40856	0.43673

a_5	1.5437	3.1965	1.35425
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Obtained Values from Model and experiment in temperature of 30°C have shown in figure 7.

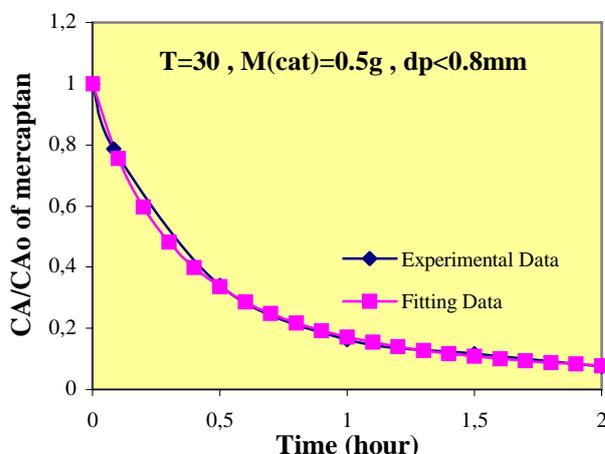


Figure 7. Mercaptan Concentration curve fitting in oxidation reaction

(2-3-3) Rate of reaction calculations and kinetic model presentation

The function of mercaptan concentration variations according to time obtained now, by differentiation from this function (df(t)/dt), we can obtain rate of reaction by multiply df(t)/dt by ratio of kerosene volume to catalyst weight (0.5 g).

$$f(t) = \frac{C_A}{C_{A_0}} \tag{6}$$

$$\frac{df(t)}{dt} = \frac{1}{C_{A_0}} \frac{dC_A}{dt} \tag{7}$$

$$\text{Rate} = \frac{1}{C_{A_0}} \frac{dC_A}{dt} \times \left(\frac{V}{M_{cat.}} \right) \times C_{A_0} \tag{8}$$

Nothing to calculated values, we will obtain variations of rate of reaction according to mercaptan concentration in every time.

Ny plotting these variations, an equation, will fitted by "Eureka" software program. Obtained equation is superficial kinetic of mercaptan oxidation.

By consideration of pore diffusion resistance , actual kinetic model will obtain. For determination of activation energy, reaction was done in temperatures of 30, 40, 50°C .

After obtaining superficial order of reaction, superficial kinetic coefficients will obtain, via Eureka software program. After correction of kinetic coefficients, it's logarithm was plotted against reversed temperature (1/T).

Slope of obtained line, shows the ratio of activation energy to gases constant (-Ea/R).

We can calculate the variations of V/M according to reaction time based on variations of kerosene volume.

Initial mercaptan concentration (C_{A0}) obtains according to kerosene Density and mercaptan molecular weight (equivalent with kerosene and sulfur molecular weight).

Data fitting procedure on a model using "Eureka" software program is as follows:

1. In first stage, numerical information related to variations of rate of reaction ($-r_A$) according to mercaptan concentration were entered.
2. The equation for variations of rate with concentration was presented ($-r'_A = K_m(C_A)^n$) and then, Eureka software program determine parameters of model (n, K_m). The results, confirmed that foresaid model has sufficient accuracy.
3. in other stage, parameters of model in different temperatures obtained. because of difference in order of reaction in deferent temperatures, so, mean of orders has been considered as superficial order of reaction.

Obtained mean order of reaction is equal to 1.661. Also, superficial kinetic coefficient (k_m) was obtained in every temperature. It is illustrated that pore diffusion resistance is controller of reaction, via following relationship :

So , obtained kinetic will be superficial kinetic in above relationship:

$$\frac{(-r'_A)_{obs} L^2 f(C_{AL})}{2D_e \int_{C_{Ae}}^{C_A} f(C_{AL}) dC_A} > 1.0 \quad (9)$$

If above relationship is true, pore diffusion resistance will be intense.

With consideration of pore diffusion resistance for temperature 30oC during 0.08333 hour (5.0 min) :

$$T=30^\circ\text{C}, C_{AL}=0.60805 \text{ mol/m}^3$$

$$-r'_A = 0.354056 \times 10^{-3} \text{ mol/ kgcat.sec}$$

If in above relationship, equilibrium concentration of mercaptan is assumed zero ($C_{Ae}=0$) as :

$$\frac{2.6614 \times (-r'_A)_{obs} L^2}{2D_e C_{AL}} > 1.0 \quad (10)$$

After calculation of efficient diffusion coefficient and equivalent length of catalyst , it is demonstrated that above relationship is true.

Molecular diffusion coefficient of mercaptan will obtain by wilke – change equation :

$$D_e = 7.8 \times 10^{-8} \times \left(\frac{\Psi_B \times M_B}{V_A^{0.6}} \right) \left(\frac{T}{\mu} \right) \quad (11)$$

The relationship between coefficients are as :

$$(k) (V_r) = (K_m)(m_{cat}) = (kg)(S_{cat}) \quad (12)$$

Initial volume of kerosene was 400 ml and therefore, the value of K by using of K_m value can be calculated. For example :

K_{m1} in temperature of 30°C will obtain as follows :

$$K_{m1obs} = 0.00080591 \frac{m^{4.98}}{\text{mol}^{0.66} \cdot \text{Kg}(\text{cat.}) \cdot \text{Sec}} \quad (13)$$

and K_{1obs} will obtain according to K_{m1obs} as follows:

$$K_{1obs} = 0.0011245 \frac{m^{1.9842}}{\text{mol}^{0.66} \cdot \text{Sec}} \quad (14)$$

According to value of K_{1obs} and existing relationship, K_1, K_{m1} will obtain as follows:

$$K_1 = 3.4447 \times 10^{-4} \frac{m^{3.9684}}{\text{mol}^{1.32} \cdot \text{Sec}} \quad (15)$$

$$K_{m1} = K_1 \left(\frac{V_r}{m_{cat.}} \right) = 2.397 \times 10^{-4} \frac{m^{6.9884}}{\text{mol}^{1.32} \cdot \text{Kg}(\text{cat.}) \cdot \text{Sec}} \quad (16)$$

Therefore, other values of k_m in temperatures of 40 , 50°C, also, will obtain as above stages. the values of k_m in temperatures of 30, 40, 50°C are given in table 7. and kinetic model equation for temperature 30°C was presented in figure 8.

Table 7. Coefficients in T=30, 40, 50 °C

Coefficient	T=30°C	T=40°C	T=50°C
$K_{m(obs)}$	0.008059	0.001068	0.001301
$K_{obs} \cdot 10^3$	1.1245	1.5442	1.8162
$K \cdot 10^4$ (actual)	3.344	5.336	6.195
$K_m \cdot 10^4$ (actual)	2.39	3.82	4.44

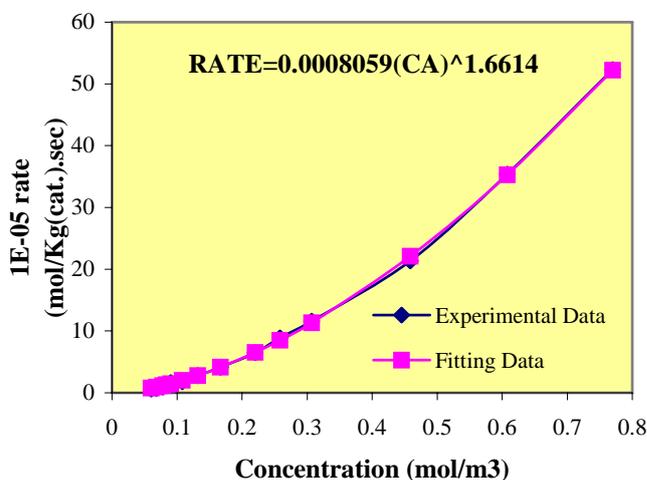


Figure 8. Reaction rate curve fitting

(2-3-3) Activation energy of reaction and presentation of actual kinetic model

With attention to "arrhenius" relationship ($K_m = K_{m_o} \cdot \text{EXP}(\frac{-E}{RT})$) and it's logarithm ,

($\ln(K_m) = \ln(K_{m_o}) - \frac{E}{R} \cdot (\frac{1}{T})$) With plotting of $\ln(K_m)$ against $1/T$, activation energy will obtain.

In figure 9, $\ln(K_m)$ against $1/T$ was plotted.

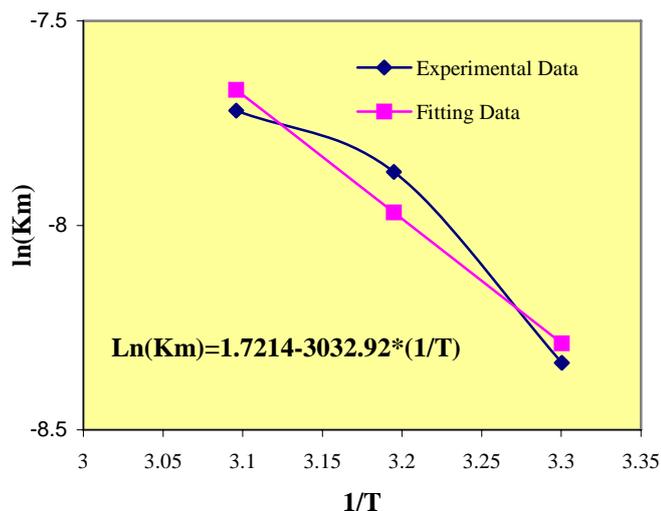


Figure 9. Activation energy of mercaptan oxidation

By using of figure 9, activation Energy is :

$$E = 6.035 \text{ kcal/mol and}$$

$$k_m = 5.5925$$

Kinetic model of reaction is presented as follows:

$$K_m = 5.5925 \exp\left(\frac{-3032.92}{T}\right) \quad (17)$$

$$-r'_A = K_m C_A^{2.3228} \quad (18)$$

Nomenclature

$a_0 - a_5$: parameters of function $f(t)$

A : Notation for mercaptan molecule

B : Notation for kerosene molecule

C_A : Mercaptan concentration in every time (mol/m^3)

C_{A0} : Initial mercaptan concentration (mol/m^3)

C_{AL} : Bulk concentration of mercaptan

C_{Ae} : Equilibrium concentration of mercaptan

dp : Particle diameter (mm)

D_e : Efficient diffusion coefficient of mercaptan

$-E_a$: Activation energy (Arrhenius)

$f(t)$: Function with variable time (hr)

K_1 : Kinetic constant refer to catalyst volume

K_m : Superficial kinetic constant $\left(\frac{\text{m}^{1.98}}{\text{mol}^{0.66} \cdot \text{s}}\right)$

K'_m : Observation kinetic constant

$\left(\frac{\text{m}^{4.98}}{\text{mol}^{1.66} \cdot \text{kg}(\text{cat.}) \cdot \text{s}}\right)$

$K_{m1(obs)}$: Observation kinetic constant refer to catalyst

$\text{weight} \left(\frac{\text{m}^{4.98}}{\text{mol}^{0.66} \cdot \text{kg}(\text{cat.}) \cdot \text{s}}\right)$

L : Catalyst length characteristic

$M_{cat.}$: Mass of catalyst (g)

M_B : Kerosene molecular weight

n : Order of reaction

P : Pressure

R : Gas constant

r_A : Rate of reaction refers to kerosene volume (m^3)

$-r'_A$: Rate of reaction refers to catalyst weight

$\left(\frac{\text{mol}}{\text{kg}(\text{cat.}) \cdot \text{s}}\right)$

$(-r'_A)_{obs}$: Observation reaction rate (corresponding catalyst volume)

$S_{Cat.}$: catalyst area (m^2/g)

T : Temperature ($^{\circ}\text{C}$)

V_A : Molar volume of soluble (mol/m^3)

V_r : Volume of kerosene (ml)

V_t : Fluid volume

Ψ_B : non-aggregation coefficient (is equal 1 for kerosene)

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