

PTC Oxidation of Alcohols: Design of Experiments

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ABSTRACT

Factorial design approach helps in better experimentation of the process. This approach was used in the phase transfer catalytic oxidation of n-amyl alcohol and n-hexanol by potassium permanganate under heterogeneous conditions. Tricapryl methyl ammonium chloride (Aliquat 336) was used as the phase transfer catalyst. A baffled batch reactor was used for the studies. The rate of oxidation/reaction was determined by changing the concentration of alcohol, concentration of the catalyst and temperature. Two values, a high and low of these parameters/factors were fixed which entails to a 2³ factorial design approach. This leads to eight treatment combinations of the experiments. These treatment combinations include main effects of the factors/parameters (concentration of alcohol, concentration of catalyst, temperature) and the interaction effects among these factors. For both the systems, the rate of oxidation was found to be dependent on the main effects. A regression model was fit and the response surface is plotted.

Keywords: Phase transfer catalyst, Aliquat 336, n-Amyl alcohol, n-Hexanol.

I. INTRODUCTION

The factorial design technique is used in the phase transfer catalytic oxidation of higher alcohols. Phase transfer catalysis is a technique, in which by the addition of a catalytic amount of phase transfer catalyst, the rate of reaction is enhanced between the reactants present in two immiscible phases. The phase transfer catalyst (PTC), tricapryl methyl ammonium chloride (Aliquat 336), was used in the oxidation of n-amyl alcohol and n-hexanol under heterogeneous conditions by potassium permanganate. Factorial experiments were performed to evaluate the effects of process parameters.

Changlu Wang et al, conducted single factor experiments to find the effects of the parameters like extraction duration, solvent-solid ratio, ethanol volume fraction and extraction times on Total Flavonoids (TF) from *Toona Sinensis* Leaves (TSL). Factorial experiments and response surface methodology were used to optimize the extraction parameters. The analysis of variance indicated that the linear coefficient extraction duration and solvent-solid ratio had high significant effects on the TF yield [1]. Pranabendu Mitra et al, performed experiment on the extraction of Kaempferol, a strong antioxidant. It was extracted from *Cuscuta reflexa* (a medicinal plant) using supercritical CO₂. A rotatable central composite design was used to determine the influence of process variables and arrived at optimal processing conditions in the supercritical CO₂ extraction process of kaempferol. The kaempferol yield was effectively

modeled as a function of the independent variables like temperature, time and pressure [2]. Frank Yates made significant contributions, particularly in the analysis of designs, by the Yates analysis. Aliquat 336 is a salt which is not soluble in the aqueous phase and exchange the anions at the interface.

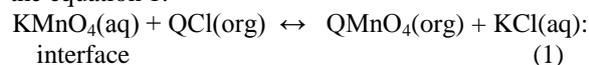
Ozbay et al [3] investigated the removal of Remazol yellow dye from aqueous solutions by adsorption on activated carbon prepared by chemical activation of sunflower red cake. The effect of pH, adsorbent dosage and initial dye concentration were investigated by full factorial design method and adsorbent dosage was found as the most significant factor. A two level factorial experimental design technique was used by Al-Ashah et al [4] to investigate the influence of the operating parameters on the production of tomato powder from tomato paste during the spray drying operations. The results showed the effect of air flow rate was relatively insignificant, while the effects of feed total solids, feed flow rate and inlet air temperature were at the same significance level. Most interactions between the studied parameters were insignificant. Jessica et al [5] showed the sequential use of two-level and three-level fractional factorial designs in the screening for important drugs and drug interactions and as well in determining the potential optimal drug dosages through the use of contour plots.

Herriott and Picker [6] studied the extractability of permanganate ion from aqueous solution into benzene with various onium salts, like Aliquat 336,

triethyl benzyl ammonium chloride (TEBAC), tributyl ammonium bromide (TBAB) and cetyl tri methyl ammonium bromide (CTMAB). Menger et al [7] reported the two-phase permanganate oxidation of piperonyl-2 piperonylic acid to yield 65% when CTMAB was present in the reaction mixture. Weber and Shepherd found that when TEBAC was used as catalyst and the temperature was maintained near 0°C, internal olefins were oxidized by basic permanganate in dichloro ethane to the corresponding cis-glycols in moderate yields [8]. Sam and Simmons [9] found that diclohexyl-18-crown-6 could solubilize solid potassium permanganate in benzene to the extent of about 0.06 molar. The resulting purple benzene solution was used to oxidize a number of organic substances in good to excellent yield. Similar results were reported by other investigators [10, 11]. The performance of the oxidation of 2-ethyl-1-hexanol by potassium permanganate with solid supported catalyst was compared with unsupported catalyst and without the catalyst by Sankarshana et al [12]. The present study involved the oxidation of n-amyl alcohol and n-hexanol by potassium permanganate in a two liquid phase system. 2³ factorial design experiments were performed with three factors at two levels. The factors are alcohol concentration, aliquat 336 concentration and temperature. The experimental runs or treatment combinations were analyzed using the analysis of variance.

II. EXPERIMENTAL

The experimental set up consisted of a 250 ml baffled cylindrical reactor with a stirrer. The reactor was kept in a water bath to maintain constant temperature during the reaction. In the present investigation the catalyst employed was aliquat 336. Known concentration of alcohol in 100 ml of benzene solution along with known quantity of the phase transfer catalyst, aliquat 336 constituted the organic phase. The aqueous phase consisted of 100 ml potassium permanganate solution of 3.165 x 10⁻² gmol/l concentration. The two liquids maintained at a particular temperature were charged into the reactor and the reaction was conducted by mixing the two liquids vigorously and by maintaining the stirrer speed around 400 rpm. Aliquat 336 (QCl) being insoluble in aqueous phase, reacts with potassium permanganate (KMnO₄) at the interface according to the equation 1.



Quaternary ammonium permanganate, QMnO₄ thus formed diffuses from the interface into the bulk and reacts with alcohol (ROH). Of course, the reaction location depends on the type of reaction regime it falls. The reaction of QMnO₄ and ROH occurs as per equation (2).



As the reaction progresses with time the MnO₄⁻ from the aqueous phase is consumed and then its concentration falls. At a particular time for sampling, the agitation was stopped and the contents of the reactor were allowed to separate into two layers. A sample was withdrawn from the aqueous layer to determine the concentration of potassium permanganate, C_{KMnO₄}. The combined operation of allowing the contents to separate out into two layers and taking the sample from the aqueous layer, took a total time of about 45 to 50 seconds during which time any reaction may be neglected. After the sample was taken the experiment was continued by restarting the agitation. The analysis of the sample was done by observing the change in concentration of the permanganate ion. The concentration of the permanganate was calibrated against the absorbance in a uv-spectrophotometer. At different time intervals the fall in concentration was measured by taking the samples from the reactor. The absorbance of KMnO₄ was measured at 525 nm from which the concentrations in gram moles per liter units were calculated. C_{KMnO₄} in aqueous phase was kept at the same value and so also the stirrer speed and the volume of aqueous and organic phases.

The concentration of the catalyst, C_{QCl}, concentration of the alcohol, C_{Am}/C_{Hex} both in organic phase and the temperature are the three factors. These three factors were fixed at two levels. Therefore, a 2³ factorial design was planned for both the systems. The experimental runs were made in duplicate.

Following are the experimental conditions:

2.1 Oxidation of n-amyl alcohol: V_{aq} = 100 ml, V_{org} = 100 ml, C_{KMnO₄} = 3.165x10⁻² gmol/l, speed = 400 rpm, Factor A = C_{Am}, gmol/l, Factor B = C_{QCl}, gmol/l, Factor C = T, °C.

Factor	Low Level (-)	High Level (+)
A=C _{Am} , gmol/l	0.138	0.689
B=C _{QCl} , gmol/l	2.261x10 ⁻³	1.131x10 ⁻²
C=T, °C	30	50

2.2 Oxidation of n-hexanol: The oxidation of n-hexanol has been carried out in similar manner as that of n-amyl alcohol and the runs were made in duplicate with the conditions given below.

V_{aq} = 100 ml, V_{org} = 100 ml, C_{KMnO₄} = 3.165x 10⁻² gmol/l, speed = 400 rpm

Factor A = C_{Hex} gmol/l, Factor B = C_{QCl} gmol/l, Factor C = T °C.

Factor	Low Level(-)	High Level(+)
A=C _{Hex} , gmol/l	0.120	0.602
B=C _{QCl} , gmol/l	2.261x10 ⁻³	1.131x10 ⁻²
C= T, °C	30	50

III. RESULTS AND DISCUSSION

In the oxidation of alcohols with PTC under heterogeneous conditions, several factors are to be considered. Since, it is a two phase liquid system, the respective volumes of aqueous and organic phases, V_{aq} and V_{org} , C_{KMnO_4} , the stirrer speed, the catalyst concentration in the organic phase, C_{QCl} , C_{Am}/C_{Hex} , T and the time of reaction, t are the factors to be considered.

The change in the volumes of the aqueous and organic phases entails the change in the interfacial area and hence, the transfer of permanganate ion from the aqueous phase to the organic phase. However, as reported by Sankarshana et al [13], the reaction between $KMnO_4$ in aqueous phase and QCl in organic phase is very fast, equation (2). This will not be affected irrespective of the ratio of volumes of both the phases in the system, as long as sufficient amount of $KMnO_4$ is maintained in the aqueous phase. In all the runs made, C_{KMnO_4} was large compared to C_{QCl} . Thus, the concentration of $QMnO_4$, C_{QMnO_4} remains constant. Hence, the V_{org} , V_{aq} and C_{KMnO_4} will not affect the rate of oxidation. It is also reported by Sankarshana and Rao [14] that, beyond an agitation speed of 350 rpm the system was found

to fall in very slow reaction regime which implies that the diffusional effects are unimportant. In the present investigation, the speed of agitation was maintained at 400 rpm. So the three factors, C_{QCl} , C_{Am}/C_{Hex} and T, all at two levels were considered. 8 experiments in each of the oxidation of n-amyl alcohol and n-hexanol were performed. Duplicate runs of these experiments were made.

3 1 Oxidation of n-amyl alcohol.

Table 1 gives the concentration change of $KMnO_4$ in the aqueous phase, from which the conversion of $KMnO_4$ is calculated. Thus, the average rate of oxidation (rate of extraction), R_{Am} is determined. I and II refers to the duplicate runs of the experiments. It can be seen that the R_{Am} remained same for different reaction times. So, for a particular experiment, the average R_{Am} is calculated, and can be inferred that the time of reaction is not affecting it. Hence, R_{Am} is a function of C_{QCl} , C_{Am} and T.

The results of the runs for different treatment combinations for the oxidation of n-amyl alcohol are given in Table 2. R_{Am} s are observed to be same for different t for each of the run.

Table 1. Effect of factors, A at low (-), B at low (-), C at low (-)

S. No	t, min	$C_{KMnO_4} \times 10^4, \text{gmol/l}$	(x), %	$R_{AmI} \times 10^7, \text{gmol/l min}$	$C_{KMnO_4} \times 10^4, \text{gmol/l}$	(x), %	$R_{AmII} \times 10^7, \text{gmol/l min}$
1	00	5.129	00.00		5.065	00.00	
2	20	4.812	06.19	1.59	4.735	06.50	1.65
3	40	4.506	12.16	1.56	4.412	12.89	1.64
4	60	4.188	18.35	1.57	4.065	19.74	1.67
5	80	3.859	24.77	1.59	3.776	25.44	1.61
6	100	3.600	29.82	1.53	3.459	31.71	1.61
Average				1.57			1.64

$C_{KMnO_4} = 3.165 \times 10^{-2} \text{ gmol/l}$, $V_{aq} = 100 \text{ ml}$, $V_{org} = 100 \text{ ml}$, Factor A = $C_{Am0} = 0.138 \text{ gmol/l}$, Factor B = $C_{QCl} = 2.261 \times 10^{-3} \text{ gmol/l}$, and Factor C = T = 30°C and A = -, B = - and C = -.

Table 2. Effects of treatment combinations on R_{Am}

S. No	Treatment combination	Factorial Effects				$R_{AmI} \times 10^7$	$R_{AmII} \times 10^7$	$R_{sum} \times 10^7$
		I	A	B	C	gmol/(l min)	gmol/(l min)	gmol/(l min)
1	(1)	+	-	-	-	1.57	1.64	3.20
2	a	+	+	-	-	2.57	2.42	4.99
3	b	+	-	+	-	3.56	3.56	7.12
4	ab	+	+	+	-	5.25	5.34	10.59
5	c	+	-	-	+	6.29	6.11	12.40
6	ac	+	+	-	+	8.40	8.50	16.90
7	bc	+	-	+	+	9.81	9.44	19.24
8	abc	+	+	+	+	12.60	12.89	25.49

$C_{KMnO_4} = 3.165 \times 10^{-2} \text{ gmol/l}$, $V_{aq} = 100 \text{ ml}$, $V_{org} = 100 \text{ ml}$, speed = 400 rpm, Factor A = $C_{Am0} = 0.138$ (-,low level), 0.689 (+,high level), gmol/l, Factor B = $C_{QCl} = 2.261 \times 10^{-3}$ (-,low level), 1.131×10^{-2} (+,high level), gmol/l, Factor C = T = 30 (-,low level), 50 (+,high level), $^\circ\text{C}$

The main effects A, B, C and the interaction effects AB, AC, BC and ABC are calculated as shown below:

Main effect of A is given by the expression:

$A = (1/4n)[a(-1) + ab - b + ac - c + abc]$ and rearranging it gives:

$A = (1/4n)[-(-1) + a - b + ab - c + ac - bc + abc]$ and on substituting the values, $A = 2.000 \times 10^{-7}$. Similarly,

$B = (1/4n)[(-1) - a + b + ab - c - ac + bc + abc] = 3.119 \times 10^{-7}$

$C = (1/4n)[(-1) - a - b - ab + c + ac + bc + abc] = 6.016 \times 10^{-7}$

$AB = (1/4n)[(1) - a - b + ab + c - ac - bc + abc] = 4.290 \times 10^{-8}$

$AC = (1/4n)[(1) - a + b - ab - c + ac - bc + abc] = 6.865 \times 10^{-8}$

$BC = (1/4n)[(1) + a - b - ab - c - ac + bc + abc] = 7.398 \times 10^{-8}$

$ABC = (1/4n)[(-1) + a + b - ab + c - ac - bc + abc] = 7.000 \times 10^{-10}$

From the above, it can be inferred that only main effects A, B, C like C_{Am} , C_{QCl} and T, have effect on the oxidation of n-amyl alcohol. The interaction effects of these factors are relatively less.

Further F-test performed on these effects also gave the same inference. F values obtained for each of the effects are $F_A = 17.49$, $F_B = 42.54$, $F_C = 158.3$, $F_{AB} = 0.81$, $F_{AC} = 2.1$, $F_{BC} = 2.4$, $F_{ABC} = 0.0002$

For 8 run experiment, the half-normal probability plot will give a better interpretation of the effects (15). This is a plot of Effects against the half-normal % probability and is shown in the Figure 1. The plot very clearly shows the deviation of A, B and C from the straight line, indicating only main effects A, B, and C are affecting R_{Am} .

The results of the experiment is expressed in terms of regression model, an empirical fit to the oxidation of alcohol. In the present discussion the response variable is related to the dependent variable which is as follows:

$$R_{Am} = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 + \beta_{123}x_1x_2x_3 \quad (\text{for } 2^3 \text{ factorial design}) \quad (3)$$

where, R_{Am} is the response, and x_1, x_2, x_3 are the coded variables. β 's are the regression coefficients.

The coded variable x is given as

$$x = [v - (v_H + v_L)/2] / (v_H - v_L)/2$$

where v is the variable

β_0 is the grand average of all observations and the regression coefficients β_1, β_2 etc. are one half of the corresponding factor effect estimates.

Since, R_{Am} is dependent only on main effects A, B and C, the regression model reduces to:

$$R_{Am} = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 \quad (4)$$

Thus, $\beta_0 = (R_{sum} / 8n) = 6.21 \times 10^{-7}$, $\beta_1 = A/2 = 1.00 \times 10^{-7}$, $\beta_2 = B/2 = 1.56 \times 10^{-7}$, $\beta_3 = C/2 = 3.09 \times 10^{-7}$ and,

$$R_{Am} = 6.21 \times 10^{-7} + 1.00 \times 10^{-7} x_1 + 1.56 \times 10^{-7} x_2 + 3.09 \times 10^{-7} x_3 \quad (5)$$

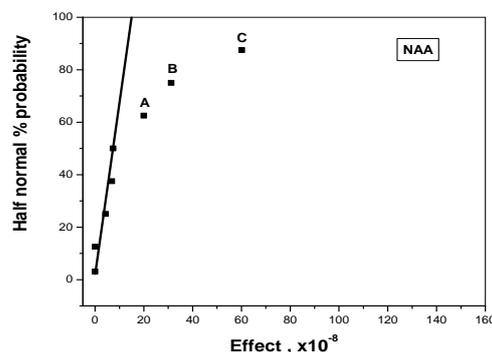


Fig1. Plot of Half-normal % probability versus effects for n- amylalcohol.

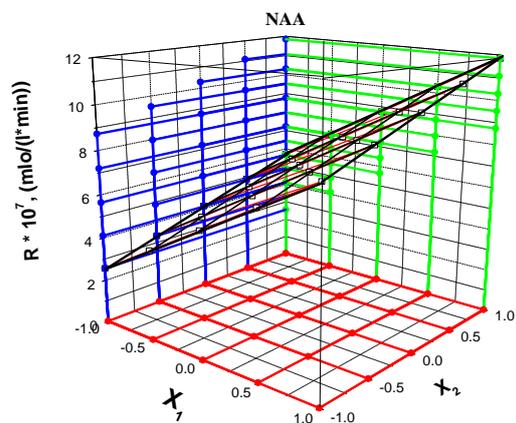


Fig 2. Plot of Response surface for the oxidation of n-amyl alcohol.

Figure 2 gives the response surface for the oxidation of n-amyl alcohol. According to equation 6, R_{Am} increases with temperature for which the coded variable is x_3 . The response is plotted for $x_3 = 1$ for different values of x_1 and x_2 .

3.2 Oxidation of n-hexanol.

The main effects A, B, C and interaction effects AB, AC, BC and ABC are calculated and are given below

$A = 1.70 \times 10^{-7}$, $B = 2.85 \times 10^{-7}$, $C = 6.00 \times 10^{-7}$, $AB = 2.63 \times 10^{-8}$, $AC = 3.99 \times 10^{-8}$, $BC = 1.04 \times 10^{-8}$ and $ABC = 4.24 \times 10^{-8}$.

Even for this system, the main effects A, B and C are large compared to the interaction effects AB, AC, BC and ABC. The F-test also justifies this. The values are as follows: $F_A = 1484$, $F_B = 4157$, $F_C = 18489$, $F_{AB} = 35.3$, $F_{AC} = 81.8$, $F_{BC} = 553.2$ and $F_{ABC} = 92.2$. The half normal probability as shown in Figure 3 also indicates that main effects A, B and C

deviates from the straight line relation implying their effects on the rate of oxidation. The regression model

for the rate of oxidation of n-hexanol, R_{Hex} is given below with,

Table 3. Effect of factors, A at low (-), B at low (-), C at low (-)

S. no	t,min	$C_{KMnO_4} \times 10^4$, gmol/l	x, (%)	$R_{HexI} \times 10^7$ gmol/l min	$C_{KMnO_4} \times 10^4$ gmol/l	x, (%)	$R_{HexII} \times 10^7$ gmol/l min
1	00	5.412	00.00		5.406	00.00	
2	20	5.141	05.00	1.35	5.112	05.44	1.47
3	40	4.900	09.46	1.28	4.941	08.60	1.16
4	60	4.706	13.04	1.18	4.718	12.73	1.15
5	80	4.412	18.43	1.25	4.382	18.93	1.28
6	100	4.147	23.37	1.26	4.106	24.05	1.30
average				1.26			1.27

$C_{KMnO_4} = 3.165 \times 10^{-2}$ mol/l, $V_{aq} = 100$ ml, $V_{org} = 100$ ml, Factor A = $C_{Hex0} = 0.120$ gmol/l, Factor B = $C_{OCl} = 2.261 \times 10^{-3}$ gmol/l, and Factor C = $T = 30^\circ C$ and A = -, B = - and C = -.

$$\beta_0 = (R_{SUM} / 8n) = 5.91 \times 10^{-7}, \beta_1 = A/2 = 0.88 \times 10^{-7}, \beta_2 = B/2 = 1.42 \times 10^{-7}, \text{ and } \beta_3 = C/2 = 3.002 \times 10^{-7}$$

$$R_{Hex} = 5.91 \times 10^{-7} + 0.88 \times 10^{-7} x_1 + 1.42 \times 10^{-7} x_2 + 3.00 \times 10^{-7} x_3 \quad (6)$$

The response plot is given in Figure 4, for $x_3 = 1$ with x_1 and x_2 ranging between, -1.0 to 1.0

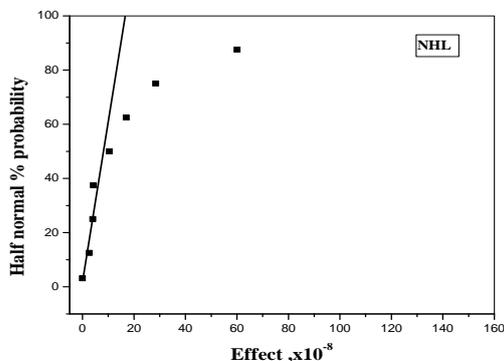


Fig 3. Plot of Half-normal % probability versus effects for n-hexanol

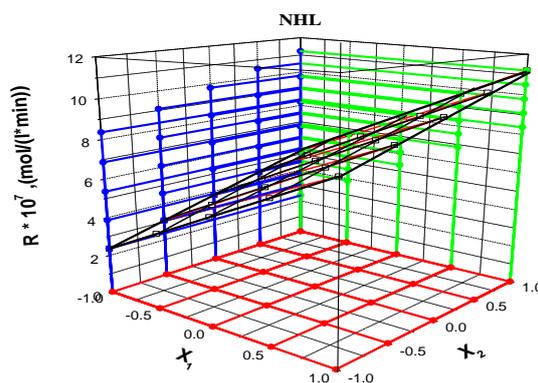


Fig 4. .Pot of Response surface for the oxidation of n-hexanol.

Table 4. Effect of treatment combinations R_{Hex}

S. no	Treatment combination	I	Factorial Effects			$R_{HexI} \times 10^7$	$R_{HexII} \times 10^7$	$R_{sum} \times 10^7$
			A	B	C			
1	(1)	+	-	-	-	1.264	1.272	2.54
2	A	+	+	-	-	2.748	2.715	5.46
3	B	+	-	+	-	3.238	3.238	6.48
4	Ab	+	+	+	-	4.397	4.360	8.76
5	C	+	-	-	+	6.228	6.288	12.52
6	Ac	+	+	-	+	7.782	7.562	15.35
7	Bc	+	-	+	+	9.332	9.582	18.92
8	Abc	+	+	+	+	12.288	12.200	24.49

$C_{KMnO_4} = 3.165 \times 10^{-2}$ gmol/l, $V_{aq} = 100$ ml, $V_{org} = 100$ ml, speed = 400 rpm, Factor A = $C_{Am0} = 0.120$ (-, low level), 0.602 (+, high level), gmol/l, Factor B = $C_{OCl} = 2.261 \times 10^{-3}$ (-, low level), 1.131×10^{-2} (+, high level), gmol/l, Factor C = $T = 30$ (low level), 50 (+, high level), $^\circ C$

IV. CONCLUSION

1. The average rate of extraction (reaction) was found to be same for different reaction times, for a particular catalyst concentration and also for alcohol concentration for both oxidation processes.
2. For both systems, the main effects A, B and C were found to affect the rate of reaction. The interaction effects AB, BC etc. were relatively less on the rate of reaction.
3. An empirical regression model is fit to both systems and the response surface is plotted.

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Nomenclature

- C_{KMnO_4} = Concentration of potassium permanganate, gmol/l
 C_{Am} = Concentration of n-amyl alcohol, gmol/l
 C_{Hex} = Concentration of n-hexanol, gmol/l
 C_{QCl} = Concentration of catalyst, gmol/l
v = Coded Variable
 v_H = Coded Variable at high level
 v_L = Coded Variable at low level
 V_{aq} = Volume of the aqueous phase, ml
 V_{org} = Volume of the organic phase, ml
Factor A = Concentration of n-amyl alcohol/n-hexanol
Factor B = Concentration of catalyst, aliquat 336
Factor C = Temperature
Minus (-) = Low Level.
Plus (+) = High Level.
R = rate of extraction, gmol/(l*min).
T = Temperature, °C
t = Time, min