

Comparative Kinetic Study on Alkylation of *p*-cresol with *tert*-butyl Alcohol using Different SO₃-H functionalized Ionic Liquid Catalysts

Pandian Elavarasan, Kishore Kondamudi, Sreedevi Upadhyayula

Abstract—Ionic liquids are well known as green solvents, reaction media and catalysis. Here, three different sulfonic acid functional ionic liquids prepared in the laboratory are used as catalysts in alkylation of *p*-cresol with *tert*-butyl alcohol. The kinetics on each of the catalysts was compared and a kinetic model was developed based on the product distribution over these catalysts. The kinetic parameters were estimated using Marquadt's algorithm to minimize the error function. The Arrhenius plots show a curvature which is best interpreted by the extended Arrhenius equation.

Keywords—Alkylation, *p*-cresol, *tert*-butyl alcohol, kinetics, activation parameter, extended Arrhenius equation.

I. INTRODUCTION

ALKYLATION OF phenols with *tert*-butyl alcohol is an industrial important reaction for production of fine chemicals and anti-oxidants. In particular, alkylation of *p*-cresol with *tert*-butyl alcohol is an important product called BHT (butylated hydroxyl toluene), it has many industrial applications, namely, as antioxidants in food industry as well as in jet fuels, petroleum products, cosmetics, pharmaceuticals, rubber, and embalming fluid, antiseptic, polymerization inhibitor and UV absorber [1-4]. Investigation of both homogeneous and heterogeneous catalysts for this typical Friedel-Crafts reaction resulted in different selectivities and activities based mainly on the acidity of the catalysts used. Catalysts used for the production of alkylated *p*-cresols include Lewis acids (AlCl₃, FeCl₃ and ZnCl₂) [5], Bronsted acids (H₃PO₄, H₂SO₄, HF, HClO₄) [6], cation-exchanged resins [7], mesoporous materials [8], zeolites [9], sulfated zirconia [10], heteropolyacids [11] and also ionic liquids. The liquid acid catalysts cause equipment corrosion and environmental pollution while solid acids deactivate rapidly. Although cation-exchanged resins showed promise, thermal stability and fouling of the resins pose major problems for their commercialization [7].

Sreedevi Upadhyayula is with the Indian Institute of Technology Delhi, New Delhi, 110016 India (corresponding author to provide phone: +91-11-26591083; fax: +91-11-26591120; e-mail: sreedevi@chemical.iitd.ac.in).

Pandian Elavarasan is with the Indian Institute of Technology Delhi, New Delhi, 110016 India. On leave from Department of Chemical Engineering, Annamalai University, Annamalai nagar, 608002 india(e-mail: arasu2@gmail.com).

Kishore Kondamudi is with the Indian Institute of Technology Delhi, New Delhi, 110016 India (e-mail: withkishore@gmail.com).

Last decade, there has been an increasing interest in developing catalytic processes with minimum environmental threats and maximum economic benefits. Room temperature ionic liquids are finding growing applications as alternative reaction media for organic transformations and separations. They possess important attributes, such as negligible vapor pressure, excellent chemical and thermal stability, potential recoverability and ease of separation of products from reactants [12, 13]. Bronsted acidic ionic liquids as novel benign catalysts have been reported for similar acid catalyzed reactions [14-16]. Here, we report the comparative alkylation of *p*-cresol with *tert*-butyl alcohol using acidic ionic liquid catalysts which gave high *p*-cresol conversion and selectivity to monoalkylated product. A kinetic model was developed and the rate parameters estimated and the extended Arrhenius equation was used to interpret the kinetics of this reaction well.

II. EXPERIMENTAL

A. Catalyst Preparation

SO₃H-functionalized Bronsted acidic ionic liquids were prepared in the laboratory following the procedure outlined in literature [14-16]. N-methyl imidazole, pyridine, triethylamine and 1,4-butane sultone were purchased from Sigma Aldrich Chemicals Pvt. Ltd, India. *p*-cresol and *tert*-butanol were purchased from Ranbaxy Ltd., New Delhi, India. In a typical ionic liquid preparation procedure, N-methyl imidazole was mixed with 1, 4-butane-sultone stirring at 313-353 K for 12-24 hours. After solidification, the zwitterions mass was washed three times with ethyl ether and then dried under vacuum (393 K, 0.01 Torr). Stoichiometric amount of sulfuric acid was then added to the precursor zwitterions. The mixture was stirred at 353 K for 8h to obtain the ionic liquid. The structures of the prepared ionic liquid are shown in Fig. 1. All the chemicals were research grade and were used without further purification unless otherwise stated.

B. Activity testing

The activity testing of the ionic liquids catalysts in this reaction was carried out in a 30 ml stainless steel autoclave lined with a Teflon bomb and equipped with a magnetic stirrer under autogeneous pressure. A typical batch consisted of 10

mmol each of *p*-cresol, TBA and ionic liquid. Reaction temperature was maintained at 70°C and reaction mixture was stirred for 8 hours. A qualitative product analysis was conducted with a GC-MS and quantitative analyses were conducted with a NUCON GC supplied by AIMIL India Ltd. using a CHROMSORB-WHP (2 m x 3.175 mm x 2 mm) column and flame ionization detector.

III. RESULTS AND DISCUSSION

A. Comparison of activity of the ionic liquids catalysts

The activity of the prepared ionic liquid catalysts was compared in this reaction and shown in Fig. 2. From the figure, it is clear that all ionic liquid catalysts gave high *p*-cresol conversion at a temperature as low as 343K. During reaction phase separation into phases, an IL rich phase and a product rich phase was observed. Phase separation is because cause of lipophilic nature of products, water being one of the byproducts. Table 1 shows the product distribution over the IL catalysts. The influence of reaction time on conversion of *p*-cresol and the selectivity towards TBC & DTBC was investigated in the range of 323-363K and different reactant mole ratios. Fig. 2 shows the conversion of *p*-cresol at 343 K, 1:1 molar ratio of *p*-cresol / TBA and 1:1 molar ratio of ILs / *p*-cresol. The alkylation of *p*-cresol with TBA in the ionic liquid shows high selectively to TBC and 2, 6-di-*tert*-butyl-3-methyl phenol (DTBC). With increase in reaction time, the conversion of *p*-cresol and selective yield of TBC increased rapidly and reached a steady value after 8 h. All three ionic liquid catalysts, showed 80% of *p*-cresol conversion and 90% selectivity to TBC. In all the cases, it was observed that 90% of *p*-cresol conversion was achieved within the first hour of the reaction time. The increase in the selectivity to TBC led to decrease in selectivity to DTBC with time but the combined selectivities of TBC & DTBC almost remained constant over the time period investigated.

B. Reaction mechanism

Based on the experimental results, detailed reaction mechanism has been proposed in fig. 3. The reactant added to the system, TBA instantaneously participates in hydrolysis to form *tert*-butylium (carbocation) and iso-butylene. The *tert*-butylium (carbocation)/iso-butylene react with *p*-cresol which gives the mono-alkylated (TBC) products. There is no oligomerization reaction occurring due to solvent activity of ionic liquids in reaction temperature. The intermediate O-alkylation may be possible, but it was not detected throughout the reaction. Further, realkylation of mono-alkylated products to di-alkylated (DTBC) products was observed.

IV. KINETIC MODELING

A. Kinetic modeling

The kinetic runs were carried out at five different temperatures, 333, 343, 353, 363 and 363 K respectively in a batch reactor at autogeneous pressure. Table 3 shows the final concentration of reactants and products at various

temperatures. Detailed reaction mechanism of *tert*-butylation of *p*-cresol using functional ionic liquid catalyst was studied. The system can be described by the reaction scheme given in figure 8. Based on the product distribution, the reaction mechanism was formulated and the kinetic model was developed based on the following assumptions:

1. Formation of *tert*-butylium/iso-butene gas is negligible due to fast hydrolysis of TBA.
2. The amount of TBA and water calculated from mass balance of the major products.
3. *tert*-butylation of *p*-cresol is considered as irreversible.
4. TBC to DTBC reaction is considered as reversible.
5. There is no consideration of intermediate formations (O-alkylation).

B. Batch reaction kinetic model

The mechanism of the reaction is detailed in fig. 3. From this reaction mechanism, a second-order rate equation is formulated. The intrinsic kinetics of phase separation reaction in the liquid phase is extremely difficult to explain, hence, here the activity co-efficients of the reactants and products were assumed to be unity and the rate constant estimated. The rate of formation of different components can be expressed as follows:

Alkylation of *p*-cresol

$$\frac{dC_C}{dt} = -k_1 * C_C * C_{TBA} \quad (1)$$

Rate of conversion of *tert*-butyl alcohol

$$\frac{dC_{TBA}}{dt} = -k_1 * C_C * C_{TBA} - k_2 * C_{TBC} * C_{TBA} + k_3 * C_{DTBC} * C_W \quad (2)$$

Rate of formation of 2-TBC

$$\frac{dC_{TBC}}{dt} = k_1 * C_C * C_{TBA} - k_2 * C_{TBC} * C_{TBA} + k_3 * C_{DTBC} \quad (3)$$

Rate of formation of 2, 6-DTBC

$$\frac{dC_{DTBC}}{dt} = k_2 * C_{TBC} * C_{TBA} - k_3 * C_{DTBC} * C_W \quad (4)$$

Rate of formation of water

$$\frac{dC_W}{dt} = k_1 * C_C * C_{TBA} + k_2 * C_{TBC} * C_{TBA} - k_3 * C_{DTBC} * C_W \quad (5)$$

Where, *C* is the concentration of respective components mol / L, *t* is the batch reaction time in sec, *k* is the rate constant of respective reaction in L / mol⁻¹ sec⁻¹. A nonlinear regression algorithm was used for parameter estimation for the above batch model eq. (1-5). A software for parameter estimation in dynamic model algorithm was followed as given in fig. 4 [17]. The optimum values of the parameters were estimated by minimizing the objective function given by

$$f = \sum_{i=1}^n \left[(x_{pred})_i - (x_{exp})_i \right]^2 \quad (6)$$

Batch reaction rate constants were optimized shown in table 4. The standard error of estimates for the rate reaction from concentration of reactant and products was of the order of 10⁻⁵ given by equation (6). The experimental and the predicted yield of reactants and products were plotted in fig. 5 at 343 K

for IL-2. It shows that the proposed reaction rate expression predicts the alkylation values comparable with the experimental ones. Similarly, all rate constant were estimated and reported in table 2.

Evaluated kinetic rate constants at various temperatures were used to determine the activation energy and frequency factor using Arrhenius relationships as shown in fig. 10 & 11. The activation energy and frequency factor from fig. 10 and 11 are calculated to be 14.46 kcal/mol and 5.19×10^6 for IL-2 and 13.65 kcal/mol and 1.87×10^6 for IL-3 in the temperature range 323 – 363 K respectively. The activation energy values for various reactions compare well with the values for same reactions in IL-1. The activation energy and frequency factor from previous data 15.63 kcal/mol and 2.65×10^7 in the temperature range 323 – 363 respectively [14]. As compared to the batch alkylation of *p*-cresol using IL-1 catalysts, the activation energy using IL-2 and IL-3 are low showing that the reaction is intrinsically kinetic controlled. The activation energy of the system decreases from IL-1 < IL-2 < IL-3 which may be attributed to the properties of the IL due to change in organic cations which vary the physical properties of ILs [12, 13]. The plots for the rate constants k_2 and k_3 show a curvature, this deviation from the Arrhenius relation is attributed to heat and mass transfer resistances at low temperature and also the solvolysis effect in the reaction [18]. In order to incorporate these effects, a modified empirical form of Arrhenius equation is applied as given below

$$\ln k = A + B(1/T - 1/T_0) + \varepsilon \quad (7)$$

$$\ln k = \ln(kT/h) - \Delta H/RT + \Delta S/R \quad (8)$$

$$E_a = -RB \quad (9a)$$

$$\Delta H = -R(B + T_0) \quad (9b)$$

$$\Delta S = R(A - \ln(k_B T_0/h) + \Delta H/RT) \quad (9c)$$

The empirical Arrhenius equation for the temperature dependent rate constant and its interpretation by the transition state theory using the parameter A and B was reported in literature for similar reactions [41]. The empirical Arrhenius equation given by the linear relationship with the error function 'ε' in eq. (7) and (8) interpret the reaction rate constant in terms of transition state theory. Linear regression analysis was used to estimate the parameters A, B, activation energy, enthalpy of activation and entropy of activation calculated from the eq. (9) and valid only in an interval around the temperature T_0 K. The parameters, enthalpy of activation and entropy change during activation are valid only in the transition state theory and the values are given in table 3.

Extended Arrhenius equation

$$\ln k = A' + B'(1/T - 1/T_0) + C'(1/T - 1/T_0)^2 + \varepsilon \quad (10)$$

$$\Delta C_p = R(C'/T_0^2 - 1) \quad (11a)$$

$$\Delta H = -R(B' + T_0) \quad (11b)$$

$$\Delta S = R(A' - \ln(k_B T_0/h) + \Delta H/RT) \quad (11c)$$

Most kinetic data can be adequately described by the

empirical Arrhenius equation. In this case, the enthalpy and entropy of activation given in table 5 suggest that the extended Arrhenius equation needs to be used to interpret the kinetics better. The extended Arrhenius equation is in the form of second-order quadratic expression (10). Non-linear regression analysis was used to solve for parameters A', B' and C' with the error estimate in the range of 10^{-5} . The activation parameter, enthalpy change and the entropy change were calculated from eq. (11c) and the values are given in table 4.

TABLE I PERCENTAGE YIELD OF REACTANTS AND PRODUCTS IN ALKYLATION OF *p*-CRESOL WITH TBA AT VARIOUS TEMPERATURES USING IL-2 AND IL-3 AS CATALYSTS

Temp. K	conversion of <i>p</i> -cresol	yield				
		<i>p</i> -cresol	TBA	TBC	DTBC	water
IL-2						
323	65.7	34	28	54	11	77
333	77.6	22	15	71	7	85
343	85.1	14	5	78	9	95
353	87.1	12	1	81	9	99
363	88.1	11	2	83	7	98
IL-3						
323	65.7	36	29	50	10	71
333	77.6	24	21	66	6	79
343	85.1	20	11	71	8	90
353	87.1	18	10	72	9	90
363	88.1	18	9	74	7	91

TABLE II OPTIMIZED PARAMETERS OF SECOND-ORDER RATE CONSTANTS, APPARENT ACTIVATION ENERGIES AND PRE-EXPONENTIAL FACTORS FOR ALKYLATION OF *p*-CRESOL WITH *tert*-BUTYL ALCOHOL IN IL-1 AND IL-2 CATALYSTS

Temperature °C	Rate constant (L / mol s)		
	$k_1 (x 10^{-4})$	$k_2 (x 10^{-4})$	$k_3 (x 10^{-3})$
IL-2			
363	0.86	93.00	16.05
353	1.60	5.76	2.04
343	3.29	1.24	0.18
333	6.67	5.23	0.29
323	9.29	11.24	0.98
Activation energy (E_a), kcal/mol	14.41	-	-
Pre-exponential factor	5.19×10^6	-	-
IL-3			
363	0.98	31.85	53.84
353	2.02	4.08	10.97
343	4.17	0.89	0.82
333	7.49	4.18	1.13
323	9.36	6.64	3.37
Activation energy (E_a), kcal/mol	13.65	-	-
Pre-exponential factor	1.87×10^6	-	-

TABLE III ESTIMATED PARAMETERS IN EMPIRICAL ARRHENIUS EQUATION
AT $T_0 = 70^\circ\text{C}$

reaction rate constants	k_1	k_2	k_3
IL-2			
A	-5.44	-5.05	-4.94
B (10^3)	-7.28	5.41	9.18
E_a , kcal/mol	14.46	-10.7	-18.2
ΔH , kcal	13.8	-11.4	-18.9
ΔS , cal	-29.5	-102.2	-123.8
IL-3			
A	-5.31	-5.48	-5.85
B (10^3)	-6.87	3.93	9.45
E_a , kcal/mol	13.64	-7.81	-18.82
ΔH , kcal	13.0	-8.50	-19.51
ΔS , cal	-31.6	-94.5	-127.1

TABLE IV ESTIMATED PARAMETERS IN EXTENDED ARRHENIUS EQUATION
AT $T_0 = 70^\circ\text{C}$

Reaction rate constant	k_2	k_3
IL-1		
A'	-6.21	-5.9
B' (10^3)	-4.82	-0.11
C' (10^7)	9.83	8.93
ΔC_p , kcal/mol K	1.6	1.45
ΔH , kcal	8.84	-0.48
ΔS , cal	-45.5	-71.85
IL-3		
A'	-6.41	-6.75
B' (10^3)	-4.33	1.44
C' (10^7)	7.94	7.70
ΔC_p , kcal/mol K	1.30	1.263
ΔH , kcal	7.89	-3.54
ΔS , cal	-48.67	-82.18

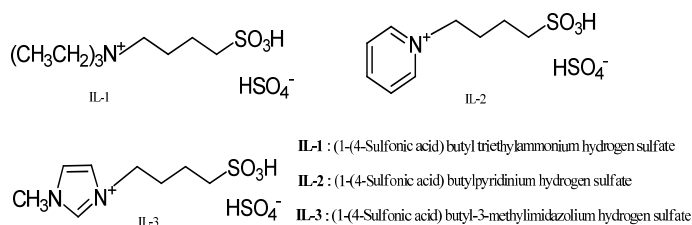


Fig. 1: Structure of ionic liquids.

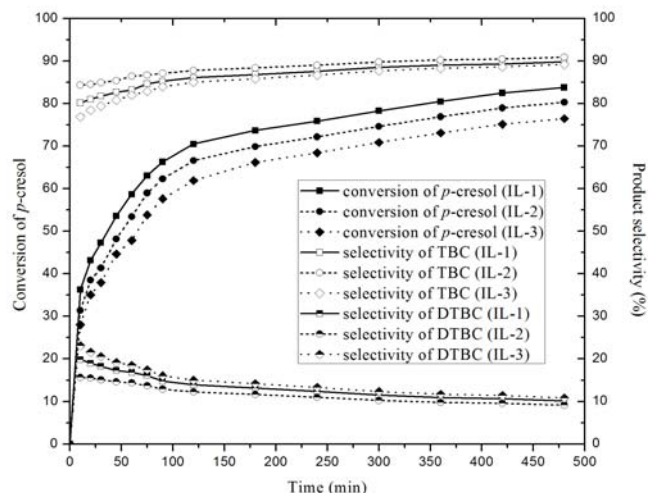


Fig. 2: Effect of reaction time on ionic liquids. Reaction conditions: *p*-cresol (10 mmol); TBA: IL molar ratio of (1:1:1), 343 k and autogeneous pressure at 800 rpm.

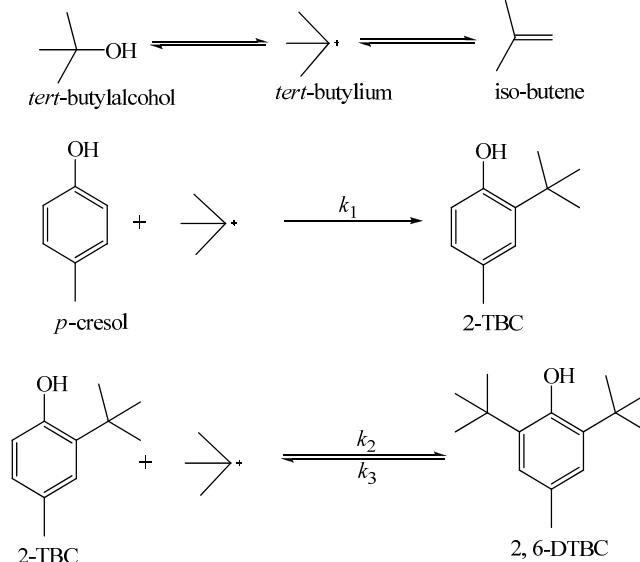


Fig. 3: Possible reaction mechanism for alkylation of *p*-cresol with *tert*-butyl alcohol in ionic liquids.

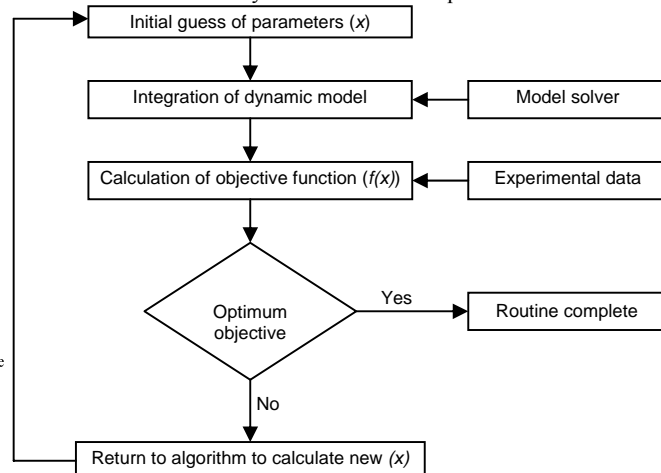


Fig. 4: Iteration algorithm of parameter estimation.

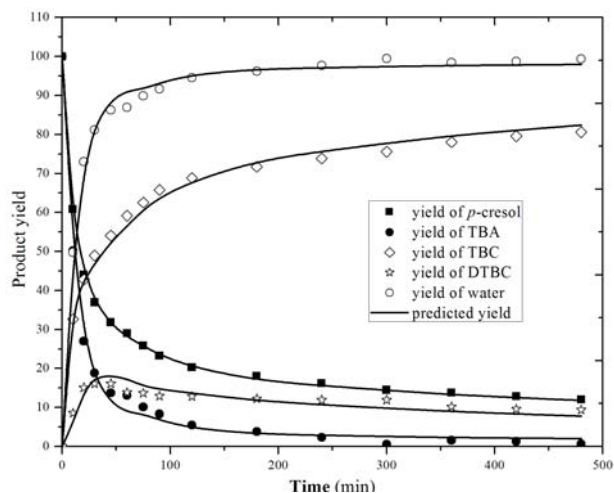


Fig. 5: Comparison of experimental and predicted product yields for alkylation of *p*-cresol with *tert*-butyl alcohol using IL-2 at 343 K.

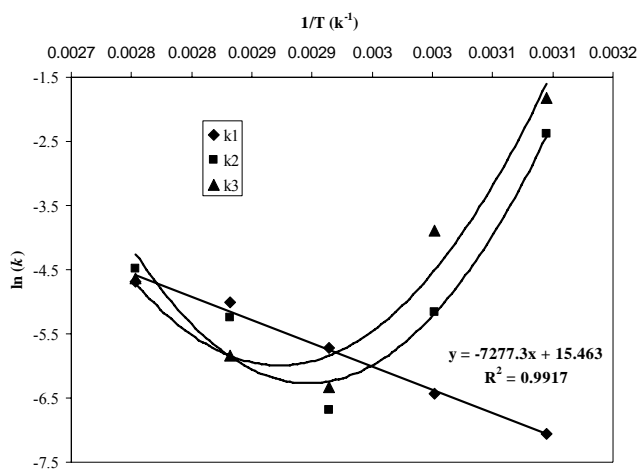


Fig. 6: Arrhenius plot for alkylation of *p*-cresol with *tert*-butyl alcohol using IL-2 catalyst.

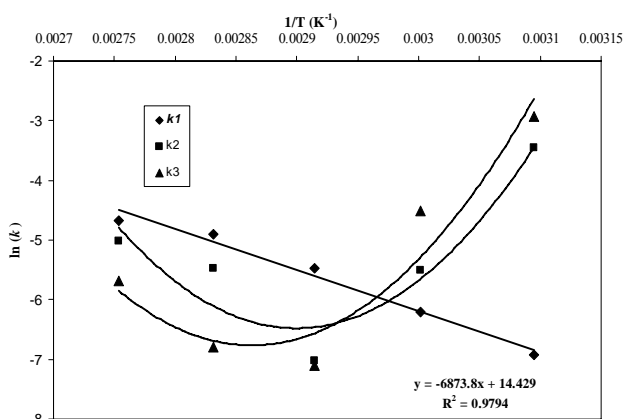


Fig. 7: Arrhenius plot for alkylation of *p*-cresol with *tert*-butyl alcohol using IL-3 catalyst.

V. CONCLUSION

The kinetics of alkylation of *p*-cresol with *tert*-butyl alcohol using different ionic liquids in a batch reactor was studied. All the three ionic liquids gave more than 80 % *p*-cresol conversion and 90% selectivity to TBC. Although, the *p*-cresol conversion was almost similar in all ionic liquids, the activation energy varied due to change in the physicochemical properties of ILs. The kinetics of the reaction are well interpreted using the extended Arrhenius equation. The activation parameter, enthalpy of activation and entropy changes were estimated for the reaction in IL-2 and IL-3. The experimental yields of the products match well with model predicted yield suggesting that the reaction rate model is appropriate.

NOMENCLATURE

Symbol	Quantity	Unit
C_C	yield of <i>p</i> -cresol	
C_{DTBC}	yield of 2,6-di- <i>tert</i> -butyl- <i>p</i> -cresol	
C_{TBA}	yield of <i>tert</i> -butyl alcohol	
C_{TBC}	yield of 2- <i>tert</i> -butyl- <i>p</i> -cresol	
C_W	yield of water	
E_a	activation energy.	kcal/mol
k_1, k_2 and k_3	second-order rate constant.	L/mol s
A', B' & C'	parameters in expansion of Arrhenius equation	
ΔC_p	activation parameter,	kcal/mol
ΔH	enthalpy of activation,	K
ΔS	entropy of activation,	kcal
A & B	parameters in empirical Arrhenius equation	cal
\mathcal{E}	error function	
k_B	Boltzmann's constant	
h	Planck's constant	
BHT	butylated hydroxytoluene	
DTBC	2,6-di- <i>tert</i> -butyl- <i>p</i> -cresol	
ILs	Ionic liquids	
IL-1	N-(4-sulfonic acid)butyl triethylammonium hydrogen sulfate	
IL-2	1-(4-sulfonic acid) butyl pyridinium hydrogen sulfate	
IL-3	1-(4-sulfonic acid) butyl-3-methyl imidazolium hydrogen sulfate	
TBA	<i>tert</i> -butyl alcohol	
TBC	2- <i>tert</i> -butyl- <i>p</i> -cresol	
x_{exp}	experimental yield	
x_{pred}	predicted yield	

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Pandian Elavarasan received his master's degree in Chemical engineering, Annamalai University, Annamalai nagar, India in 2005, and is presently pursuing Ph. D. degree at Department of Chemical Engineering, Indian Institute of Technology Delhi, New Delhi, India. He is lecturer in the Department of Chemical Engineering, Annamalai University, Annamalai nagar, India. He has worked on mass transfer on waste water treatment, ionic liquids, catalysis and reaction engineering.

Kishore Kondamudi received his master's degree in Chemical engineering with Process Engineering and Design, Indian Institute of Technology Delhi, New Delhi, India in 2008, and is presently pursuing Ph. D. degree at Department of Chemical Engineering, Indian Institute of Technology Delhi, New Delhi, India. He has worked on Catalysis and reaction engineering, ect.

Dr. **Sreedevi Upadhyayula** received her master's degree in Chemical engineering with Petroleum Refinery Engineering and Petrochemical Technology as specialization, Indian Institute of Technology, Kharagpur, India in 1993, and received Ph. D. degree NCL, Pune & Indian Institute of Technology, Kharagpur, India in 2003. presently she is is an Assistant Professor in the Department of Chemical Engineering, Indian Institute of Technology Delhi, New Delhi, India. She was Assistant Professor, Department of Chemical Engineering, IIT Kharagpur, West Bengal, India in 2004-2006. She has worked on rational design of novel catalytic materials and catalytic conversion technologies, solid acid catalysis, ionic liquids and reaction engineering, ect.