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Ultrasound Assisted Synthesis of 1-Butoxynaphthalene Under Liquid-Liquid Multi-Site Phase-Transfer Catalysis Condition and Their Kinetics

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Abstract: ***In this research work, the convenient synthesis of 1-butoxynaphthalene from cheaply available starting materials under multi-site phase-transfer catalyst viz., 1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dichloride and assisted by sonication (40 kHz, 300 W) using solution of sodium hydroxide in liquid–liquid biphasic (LL-PTC) system was reported. The reaction is greatly enhanced in the liquid–liquid system, catalyzed by multi-site quaternary ammonium salt (MPTC) and ultrasound irradiation in a batch reactor. The reaction is carried out under pseudo first-order condition and which is sufficient to describe all the results, such as the effects of agitation speed, different ultrasonic frequency, different phase transfer catalysts and the effect of organic solvents, the amount of MPTC, effect of various temperature, concentration of butyl bromide, sodium hydroxide concentrations, on the conversion of butyl bromide and the apparent rate constant (K_{app}) were investigated in detail.***

Keywords: Multi-site phase-transfer catalyst, sonication, liquid-liquid PTC, α-naphthol, butyl bromide, kinetics

1. Introduction

The phase-transfer catalysis is widely recognized as a versatile and important technique in organic synthesis, nucleophilic substitution, addition and elimination reaction etc. The advantage of PTC technique is, the reactions were carried out between immiscible reactants which reside in different phases in an excellent yield, with high conversion and good selectivity [1]-[4]. Generally used PT catalysts are quaternary ammonium and phosphonium salts, crown ethers, and cryptands in which quaternary ammonium salts are most widely used catalyst by several researchers. This green methodology advantageously resulted in usage of less hazardous solvents, ease of separation and higher selectivity etc and it is a powerful tool in the manufacture of fine chemicals and pharmaceuticals [1],[2]. The economical scale and efficiency of catalysts are improved by multi-site phase-transfer catalyst [5]. The total weight of the MPTC required in these reactions is less compared to the single site PTCs analogue [5]. To improve the reaction rate, the development of new multi-site PT catalyst [6], [7] and the use of non-conventional techniques such as sonication [6],[7] or microwave irradiation [8] and combination of ultrasound and MPTC [9].

Ultrasonic irradiation in the liquid-liquid heterogeneous system can increase interfacial area coupled with local hot-spot generation, and has been resulted in the enhanced rate of the reaction [10],11].Ultrasonic irradiation combined with liqid-liquid PTC has enhance the formation of catalytic intermediate and thus, the rate of reaction is increased. In addition, MPTC combined with sonication reveals, the overall rate of the reaction effectively increased [6],[7],[9].

In the present study, multi-site phase-transfer catalyst (1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dichloride) in liquid-liquid PTC under ultrasonic irradiation for the preparation of 1-butoxynaphthalene was developed and the highly added value chemical such as ether obtained as a single product in mild conditions under greener methodology aspects. The kinetic aspects such as, the effects of agitation speed, different ultrasonic frequency, different phase transfer catalysts and the effect of organic solvents, the amount of MPTC, effect of various temperature, concentration of butyl bromide, sodium hydroxide concentrations, on the conversion of butyl bromide and the apparent rate constant (K_{app}) were investigated in detail.

2. Experimental Section

2.1. Materials

All the reagents, including 1,4-diazabicyclo[2.2.2]octane, benzyl chloride, α-naphthol, butyl bromide, biphenyl, tetrabutylammonium bromide (TBAB), tetrabutylammonium chloride (TBAC), tetrahexylammonium bromide (THAB) and tetraoctylammonium bromide (TOAB), sodium hydroxide, n-hexane, toluene, chlorobenzene, diethyl ether and other reagents for synthesis were guaranteed grade (GR) chemicals and were used without further purification.

2.2. Instrumentation

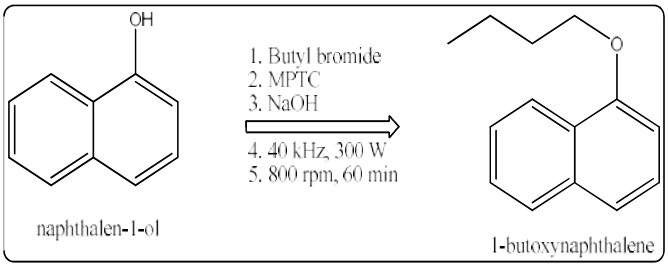
^{1}H NMR and ^{13}C spectra were recorded on a Bruker 300 MHz and 75 MHz respective using TMS as an internal standard. Gas chromatography was carried out using a GC-Varian 3700 model. Ultrasonic water bath, Equitron, Media Instrument Manufacturing Company, Chennai, India-600 004. The ultrasonic generator was a thermostatic bath equipped with dual frequencies (28/40 kHz) and electric power 300 W with 0.0126 W/mL of power density. Details of ultrasonic process equipment are well explained in our previous report [6]. The multi-site phase-transfer catalyst (MPTC) is prepared according to our previous report [12]

3. Synthesis of 1-butoxynaphthalene

To the 15g NaOH in 20 mL water, the α-naphthol (7.2g, 49.8mmol, excess agent) was added under overhead stirring for few minutes to generate the naphthyl anion. Then synthesized MPTC (0.3g) and butyl bromide (2g, 7.1mmol) in toluene (30 mL) were added slowly. The reaction mixture was heated at 50^{o}C for an hour with vigorous stirring. The product formed was evidenced by thin layer chromatography (TLC). The crude product was isolated by simple extraction with Ethyl acetate (3 x 25 mL). The organic layer was collected and the solvent was evaporated under reduced pressure. The crude product was subjected to chromatography (SiO_2) employing hexane: ethyl acetate (9.9:0.1) as an eluent to obtain pure Products as colorless viscous liquid (scheme 1). The obtained product was characterized by ^{1}H and ^{13}C NMR spectra.

1-butyloxynaphthalene:
^{1}H NMR ($CDCl_3$): δ 1.020-1.553(3H, t, -**CH_3**), 1.572-1.646 (2H, m, -**CH_2**-CH_3), 1.869-1.938 (2H, m, -O-CH_2-**CH_2**), 4.111-4.143 (2H, t, -O-**CH_2)**, 6.779-6.797 (1H, d, **Ar-H**), 7.330-7.768 (4H,m, **Ar-H**), 7.789-7.790 (1H, d, **Ar-H**), 8.278-8.298 (1H, d, **Ar-H**).
^{13}C NMR ($CDCl_3$): δ 13.98, 19.55, 31.43, 67.83 (Aliphatic-**C**), 104.53, 119.94, 122.13, 125.06, 125.79, 125.95, 126.33, 127.44, 134.53, 154.93 (Aromatic-**C**).

Scheme 1
Synthesis of 1-butoxynaphthalene



4. Kinetics of etherification reactions

The reaction was conducted on a 250 mL three-necked Pyrex round-bottom flask which permits agitating the solution, inserting the water condenser to recover organic reactant and taking samples and feeding the reactants. This reaction vessel was supported at the centre of the sonicator. Known quantities of toluene (30 mL, solvent), sodium hydroxide (15g in 20 mL water) and 0.2 g biphenyl (IS-internal standard) were introduced into the reactor. Then, 7.2g of alpha-naphthol (49.8mmol) and 2 g of buty bromide (7.1mmol), 0.3g of MPTC were introduced to the reactor to start the reaction. The reaction mixture was stirred at 800 rpm. The phase separation was almost immediate on arresting the stirring process. Samples were collected from the organic layer at 10, 20, 30, 40, 50 and 60 minutes into the 10 mL vials and pinch of anhydrous sodium sulphate was placed in the sample vials to absorb any moisture present in the organic layer. The kinetics was followed by estimating the amount of butyl bromide (limiting reagent) that disappeared and measured by a gas Chromatography (GC-Varian 3700 model). The analyzing conditions were as follows; Column, 30 m x 0.525 mm i.d. capillary column containing 100% poly(dimethyl siloxanen); injection temperature, 250^{0}C; FID detector (300^{0}C). Yields were determined from standard curve using biphenyl as an internal standard.

5. Results and discussion

The kinetic experiments of etherification were carried out under pseudo first order conditions, taking excess of alpha-naphthol and sodium hydroxide under the combined effect of sonication (40 kHz, 300 W) and MPTC. The k_{app} value was obtained by plotting -ln (1-X) versus time.

5.1. Effect of Varying Stirring Speeds

The effect of varying stirring speed on the rate of etherification reaction between α-naphthol and buty bromide was studied in the range 0–1000 rpm and the ultrasonic energy used is 40 kHz (300 W) for throughout the reaction. From the plots of -ln(1-X) vs. time, the pseudo-first order rate constants were evaluated. The experimental results show that the apparent rate constants increase with the increase of stirring speed from 0 to 400 rpm. Further increasing the agitation speed does not increase the reaction rate (Figure 1). Therefore, the reaction kinetics is controlled by the chemical reaction for stirring speed greater than 400 rpm. Hence, the stirring speed was set at 800 rpm for further experiments.
The reactions are carried out in three different conditions such that,

1) Stirring without sonication (800 rpm only): k_{app} = 8.01 x 10^{-3}, min^{-1}
2) Sonication without stirring (40 kHz, 300 W only): k_{app} = 4.21 x 10^{-3}, min^{-1}
3) Combination of both (800 rpm and 40 kHz, 300 W): k_{app} = 26.22 x 10^{-3}, min^{-1}

These results portray the combination of both ultrasound and stirring shows higher kapp value (condition 3) than the other conditions and the kapp value for condition (3) is approximately 3.3 times higher than the condition (1). It may be due to by the presence of ultrasonic wave increase the collision rate between the reactants [13].

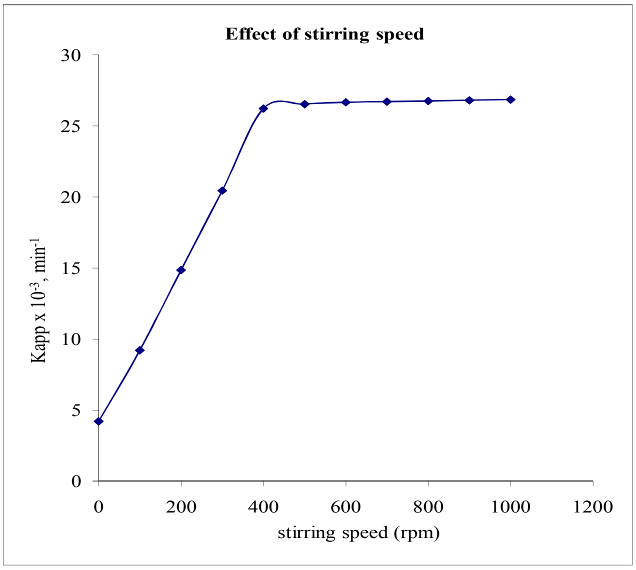


Figure 1: Effect of stirring speed: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of

alpha-naphthol, 7.1mmol of butyl bromide, 0.3g of MPTC, 30mL of toluene, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.2 Effect of Different Ultrasound Frequencies

The chemical effects of ultrasound attributed to intense local conditions generated due to cavitation bubble dynamics, i.e., the nucleation, formation, disappearance, and coalescence of vapour or gas bubbles in the ultrasonic field [14]–[18]. However in phase-transfer catalyst reaction rate enhancements are typically due to mechanical effects, mainly through an enhancement in mass transfer. The presence of ultrasound, a number of phase-transfer catalyzed reactions have also been shown to benefit from the mass transport enhancement by sonication [18]. Recently, many studies have reported a dramatic rate increase in phase-transfer catalysis under ultrasound irradiation [18].

To study the kinetic experiments, two different low frequency ultrasounds are used under the 300 W power output. The reactions are carried out in 28 kHz and 40 kHz having the 300 W and silent condition (0 kHz) under similar reaction conditions. The kinetic profile of the reaction is obtained by plotting –ln (1-X) against time. The obtained k_{app} values are

(i) For 28 kHz, 300 W, the k_{app} value is 17.51 x 10^{-3}, min^{-1}
(ii) For 40 kHz, 300 W, the k_{app} value is 26.22 x 10^{-3}, min^{-1}
(iii) For 0 kHz (silent condition), the k_{app} value is 8.01 x 10^{-3}, min^{-1}

Hence, the overall k_{app} value was increased by increasing the ultrasonic frequency in the order of 0 kHz (silent condition) < 28 kHz (300 W) < 40 kHz (300 W) for our system (Table 1) .Similar trend was observed by Entezari coworkers [19],[20].

Table 1: Effect of ultrasonic frequency

Ultrasonic frequency (kHz)	k_{app} x 10^3, min^{-1}
0	8.10
28	17.51
40	26.22

Influence of ultrasonic frequencies: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide,0.3g of MPTC, 30mL of toluene, 800 rpm, 50^0 C;

5.3. Effect of temperature

To find out the effect of temperature on the reaction of alpha-naphthol with butyl bromide was studied under otherwise similar conditions. The temperature was varied from 30^0C to 70^0C.The kinetic profile of the reaction is obtained by plotting -ln (1-X) versus time. It is obvious that the reactivity is increased with an increase in the temperature along with ultrasonic effect [6]. The reason behind this is,

1) The number of reactant molecules which possess higher activated energy at a higher temperature and thus the ultrasonic wave easily passes through the reactor.
2) The collision of the reactants at higher temperature is also increased.

Hence, the apparent rate constant is increased at higher temperature. Arrhenius plots were made in Figure 4 of –lnk_{app} against 1/T to get activation energy o f 50.35 $kJ.mol^{-1}$. This higher activation energy demonstrates that, this reaction was kinetically controlled and the mass-transfer resistance between phases was unimportant. In addition ultrasound is used to overcome the mass-transfer resistant and avoid the use of higher temperature for the reaction system. It is evident from higher activation energy and higher stirring speed that the present reaction system follows the interfacial mechanism [21],[22].

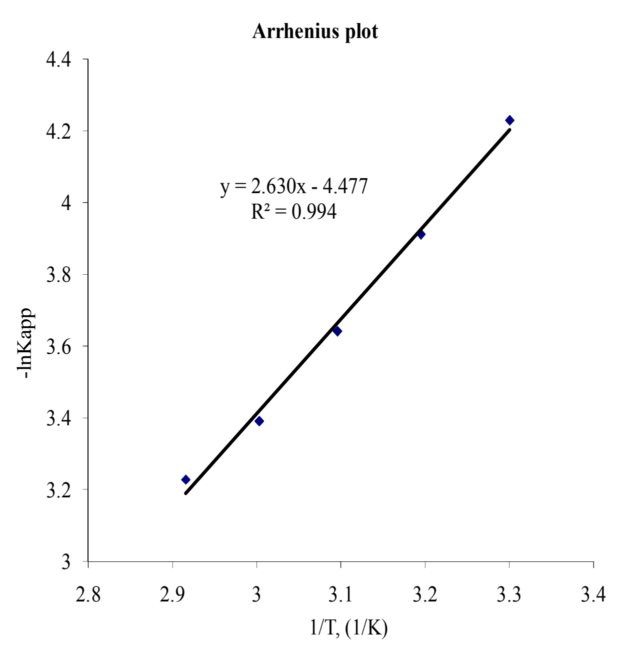


Figure 4: Arrhenius plot: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide, 0.3g of MPTC, 30mL of toluene, 800 rpm, ultrasound condition (40 kHz, 300 W).

5.4 Effect of MPTC Amount Variation

These experiments were conducted by varying the amounts of MPTC (1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dichloride) by keeping other experimental parameters are constant. The effect of the amount of MPTC on the etherification reaction has been carried by varying amount of MPTC from 0.1 g to 0.3 g under ultrasound irradiation (40 kHz, 300 W). The apparent rate constants were evaluated from the plot of -ln(1-X) versus time. As shown in Figure 2, the rate of the reaction increased with increase in the amount of MPTC along with ultrasound irradiation (40 kHz,300 W).The kapp values are linearly dependent on the amount of multi-site phase-transfer catalyst. The increase in the kapp value is may be attributed to the change in the size, surface area and morphology of MPTC due to the positive effect of ultrasound might be enlarged [23].

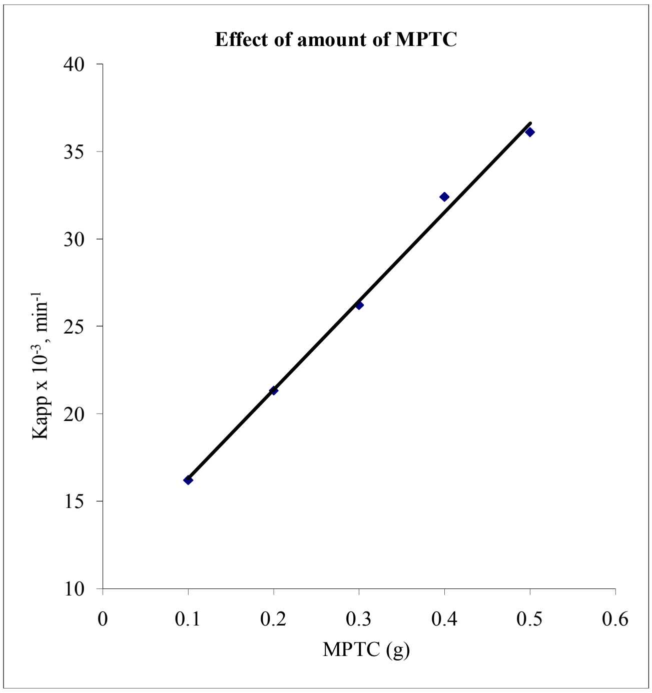


Figure 2: Effect of MPTC: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide, 30mL of toluene, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.5 Effect of various butyl bromide concentrations

We investigate the influence of butyl bromide concentrations on the synthesis of 1-butoxynaphthalene under ultrasonic irradiation condition (40 kHz, 300 W) in the presence of MPTC (1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dichloride). For this study, the amount of butyl bromide was varied from 1 g to 3 g keeping other experimental parameters as constant. The results are shown in Table 2. The data clearly indicates that the kapp value increases with increasing the amount of butyl bromide. When the butyl bromide concentration increased, the probability of finding the substrate with active-site of the catalyst is enhanced and thereby the rate of the reaction was increased [23]. Ultrasound irradiation resulted in enhanced kapp values and it may be due to it reduces the surface area between the aqueous and organic phases and hence more reactants collide with each other simultaneously we get higher kapp value [23].

Table 2: Effect of amount of butyl bromide

Butyl bromide (BB), g	$k_{app\ x}$ 10^3, min^{-1} (40 kHz, 300 W)
1	16.43
1.5	21.52
2	26.22
2.5	30.30
3	34.78

Effect of amount of benzyl chloride (BC): 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 0.3g of MPTC, 30mL of toluene, 800 rpm, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.6. Effect of Organic Solvents

The three different organic solvents employed in this study are toluene, chlorobenzene and n-hexane. The influence of these three organic solvents on the rate of etherification of α-naphthol with butyl bromide was followed under otherwise standard reaction conditions. From the plot of –ln (1-X) against time, the k_{app} values are obtained. Among these solvent chlorobenzene possesses a higher k_{app} value, due to its higher dielectric constant. The order of the solvents are chlorobenzene > toluene > n-hexane (Table 3). This result reveals that the combination of ultrasonic irradiation and polarity of solvent enhances the reaction rate.

Table 3: Effect of organic solvents

Solvent	Dielectric constant (ε^a)	K_{app} x 10^3, min^{-1}
chlorobenzene	5.6	30.25
Toluene	2.4	26.22
Hexane	2.2	7.5

Influence of organic solvents: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide, 0.3g of MPTC, 30mL of toluene, 800 rpm, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.7. Effect of volume of toluene

The conversion or the reaction rate is directly proportional to the concentration of the reactants in 60 min of reaction. A dilute concentration of the reactant is obtained using a large amount of organic solvent. The conversion of butyl bromide is increased with the decrease in the volume of toluene. The k_{app} value is inversely proportional to the volume of toluene, as expected (Figure 3).

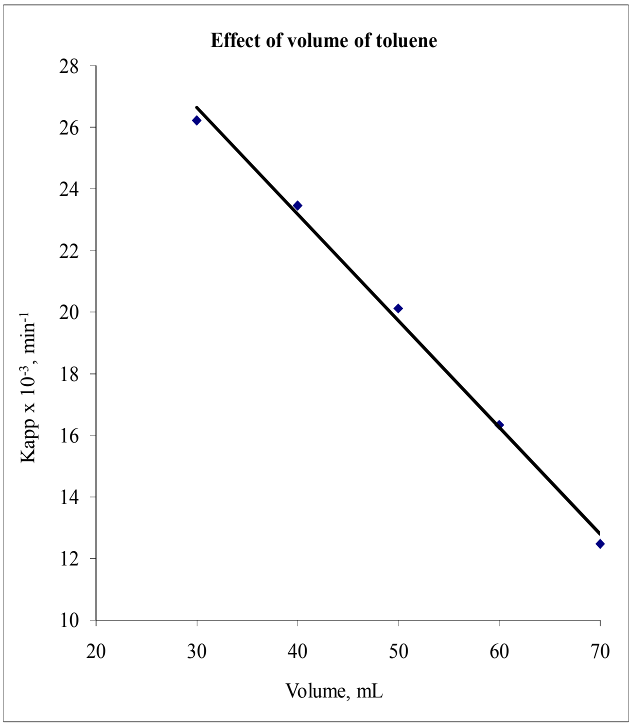


Figure 3: Effect of volume of toluene: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of

alpha-naphthol, 7.1mmol of butyl bromide, 0.3g of MPTC, 50^0 C, 800 rpm, ultrasound condition (40 kHz, 300 W).

5.8. Effect of MPTC and phase-transfer catalysts

In this work, in addition with MPTC, four different phase-transfer catalysts are employed to evaluate their influence in the reaction of α-naphthol and butyl bromide in the presence of sonication keeping similar reaction conditions. The employed MPTC is 1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dichloride and PTCs are TOAB, THAB, TBAB and TBAC. The pseudo-first order rate constant are evaluated for all the catalyst from the plot of –ln(1 - X) versus time. Table 4 depicts the apparent rate constant for these catalyst, in which MPTC's shows higher activity. The order of catalyst efficient activities are MPTC > TOAB > THAB > TBAB > TBAC. These results indicate the liphophilic character of the catalysts and MPTC has the ability to coordinate with more number of anions and transfer the catalytic intermediate to the reactions medium. The results also indicate an additional increase of k_{app} value when the reaction was carried out under MPTC and ultrasound irradiation condition 40 kHz, 300 W (Table 4). It may be due to the change in the size, surface area and morphology of phase-transfer catalysts due to the positive effect of ultrasound.

Table 4: Effect of MPTC and phase-transfer catalysts

PTC (g)	k_{app} x 10^3, min^{-1} (40 kHz, 300 W)
MPTC	26.22
TOAB	23.43
THAB	21.44
TBAB	19.16
TBAC	16.62

Effect of various PTC's: 15g NaOH in 20 mL water, 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide, 30mL of toluene, 800 rpm, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.9 Effect of sodium hydroxide concentrations

The reaction rate is known to be greatly affected by a concentration of the alkaline compound. The rate of reaction strongly depends on the strength of the sodium hydroxide. Kinetic experiments were carried out, employing 10–25g under similar reaction conditions. The kinetic profile of the reaction is obtained by -ln(1-X) against time. The kapp values tremendously increased with increase in basicity of hydroxide ion (Table 5). It suggests that the hydroxide ions are less solvated by water molecules and there by the activity of the hydroxide ion increases [3],[24]. In the present case, extraction of anion of α-naphthol is more effective when the reaction is carried out in the presence of ultrasound irradiation along with higher concentration of sodium hydroxide.

Table 5: Effect of NaOH concentrations

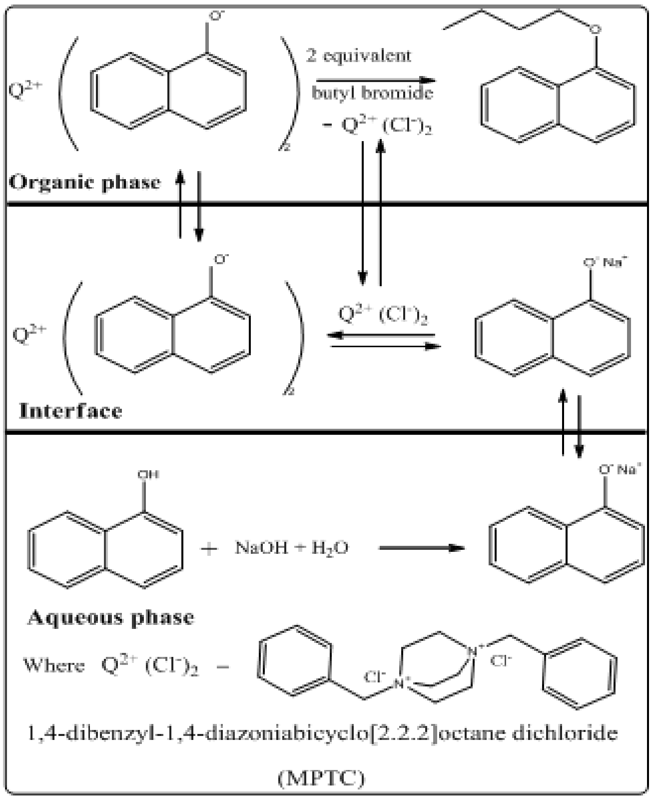
NaOH (g) (20 mL water constant)	k_{app} x 10^3, min^{-1} (40 kHz, 300 W)
10	18.43
15	26.22
20	33.22
25	38.30

Effect of NaOH concentrations: 0.2g of biphenyl (internal standard), 49.8mmol of alpha-naphthol, 7.1mmol of butyl bromide, 0.3g of MPTC, 30mL of toluene, 800 rpm, 50^0 C; ultrasound condition (40 kHz, 300 W).

5.10 Reaction mechanism

The experimental result from the present kinetic study indicates that the dependencies of the kinetic data on the entire stirring speed, concentration of the catalyst, aqueous sodium hydroxide and temperature and higher Ea value are indicative of an interfacial mechanism. Hence we proposed an interfacial mechanism for the current study (Scheme 2). Initially, the hydroxide ion deprotonates the α-naphthol at the interface, forming an ion-pair (ArO^-Na^+). Upon addition of the catalyst, $Q^{2+}(X^-)_2$, ion exchange takes place at the interface $\{Q^{2+}(ArO^-)_2\}$ and the new formed ion pair $Q^{2+}(ArO^-)_2$, which is more organophilicity and hence easily migrates into the organic phase. This ion-pair reacts with the alkylating agent (butyl bromide) in the organic phase resulting in the formation of two equivalent of desired product is obtained.

Scheme 2 (Interfacial mechanism)



6. Conclusion

The following conclusions can be drawn from the etherification reaction catalyzed by MPTC in the presence of ultrasound (40 kHz, 300 W).

(1) Multi-site phase-transfer catalyst has been successfully synthesized and tested for the synthesis of the 1-butyloxynaphthalene from α-naphthol and butyl bromide under ultrasonic irradiation condition.

(2) The efficiency of the MPTC compared with the known phase-transfer catalysts, in which the MPTC shows higher activity.
(3) From the kinetic study, the rate of the reaction is found to be directly dependent on each kinetic variables, viz., [MPTC], [substrate], [NaOH], ultrasonic frequency, stirring speed and temperature.
(4) The rate of the reaction decreases with increase in the volume of toluene.
(5) Based on the experimental result, the interfacial mechanism is proposed.
(6) The combination of ultrasound and MPTC resulted in better efficacy as compared to the individual operation.
(7) The activation energy is calculated from Arrhenius plot.

7. Acknowledgments

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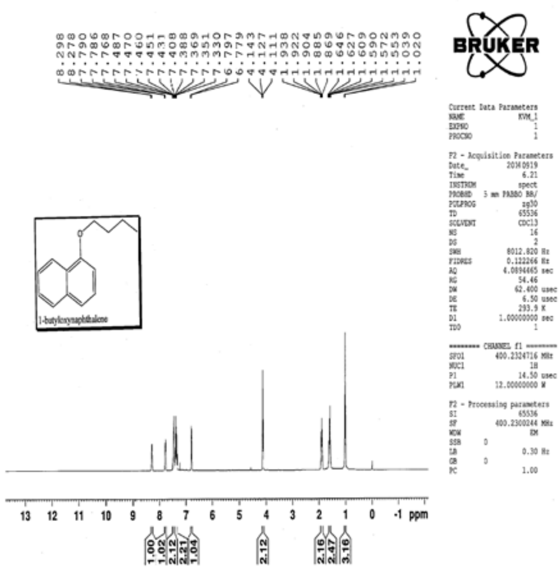
^{1}H NMR spectra of 1-butoxynaphthalene



^{13}C NMR spectra of 1-butoxynaphthalene