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Kinetic study of synthesizing *N*-butylphthalimide under solid–liquid phase-transfer catalysis conditions

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Abstract

The synthesis of *N*-butylphthalimide (PTR) from the reaction of 1-bromobutane and potassium salt of phthalimide (PTK) was carried out successfully in a solid–liquid phase-transfer catalytic condition (SL-PTC) instead of reaction in a liquid–liquid two-phase medium. No water is required in the reaction system. The advantage of using SL-PTC to synthesize organic chemicals is to avoid a serious hydration of potassium salt of phthalimide from the reaction of 1-bromobutane and potassium salt of phthalimide in a liquid–liquid two-phase solution. The reaction is greatly enhanced in the solid–liquid solution catalyzed by quaternary ammonium salts. The reaction mechanism is proposed and verified by examining the experimental evidence. A kinetic model is proposed in which a pseudo first-order rate law is sufficient to describe the results. Kinetics of the reaction, including the effects of agitation speed, amount of acetonitrile, organic solvents, amount of tetrabutylammonium bromide, quaternary ammonium salts, amount of water and temperature on the conversion of 1-bromobutane and the apparent rate constant (k_{app}) were investigated in detail. Rational explanations to account for the phenomena on the results were made.
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1. Introduction

In synthesis of organic chemicals from two reactants, which exist in different immiscible phases, it is difficult to obtain a high yield of product or a large conversion of reactant. The main reason is that there is little contact of these two reactants due to the limited interface. Past efforts to enhance the reaction rate, including the use of protic or aprotic solvent, high agitation speed or elevated reaction temperature, intending have produced limited results. In addition, side effects, which cause the reaction be carried out in anhydrous condition for using aprotic solvents and produce byproducts due to side reaction at high temperature, increase the difficulty in separating the product from solution. In contrast, phase-transfer catalysis (PTC) is now well recognized as an invaluable methodology for organic synthesis from two immiscible

reactants, and its scope and application are the subjects of current research [1–3].

Imide derivatives are important substrates for biological and chemical application [4,5]. The synthesis of these derivatives has received much attention in recent years. Those well known methods include dehydrative condensation of an anhydride and amine catalyzed by concentrated sulfuric acid [6], direct alkylation of phthaloyl dichloride with azide in the presence of PPh$_3$ in CH$_2$Cl$_2$ [7] and *N*-alkylation of imides using alcohol promoted by PPh$_3$ and DIAP in THF [8]. These methods are definitely necessary to develop an attractive solvent for the synthesis of imides under mild conditions. Recently, ionic liquids have been employed with success to synthesize imide derivatives [9,10]. However, the cost of ionic liquids limits their industrial application. Potassium salt of phthalimide (PTK) is easily prepared from phthalimide in an alkaline solution of KOH [11]. In general, potassium salt of phthalimide can also react with alkyl halide in an organic solution [12]. However, the yield of product is low

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because potassium salt of phthalimide is insoluble in organic solvents.

In principle, the synthesis of *N*-butylphthalimide (PTR) can be achieved by reacting 1-bromobutane and potassium salt of phthalimide in a liquid (water)–liquid (organic solvent) two-phase medium catalyzed by quaternary ammonium salt. However, the hydration of potassium salt of phthalimide in aqueous solution is serious. Thus, few yields are obtained from the liquid (water)–liquid (organic solvent) solution, even though it is catalyzed by quaternary ammonium salts. In this work, the imide derivatives are synthesized from the reaction of 1-bromobutane and potassium salt of phthalimide in a solid–liquid type catalysis under the phase-transfer catalytic conditions (SL-PTC). The advantage of using a solid–liquid reaction is to prevent the deficiency of hydration of reactant in using liquid–liquid reaction. Nevertheless, the reaction is greatly enhanced in the solid–liquid solution catalysis by quaternary ammonium salt, even though potassium salt of phthalimide is insoluble in organic solvent. With the aid of quaternary ammonium salt, PTK is dissolved in organic solvent. The mechanism of the solid–liquid reaction of alkyl halide and potassium salt of phthalimide in an organic solvent is verified in this work. A pseudo first-order rate law is employed to describe the kinetic behaviors. Effects of the reaction conditions, such as the agitation speed, amount of acetonitrile, organic solvents, amount of tetrabutylammonium bromide, quaternary ammonium salts (TBAB), amount of water and temperature on the conversion of 1-bromobutane, as well as the apparent rate constant were investigated in detail.

2. Experimental

2.1. Materials

Potassium salt of phthalimide, 1-bromobutane; quaternary ammonium salts: tetrabutylammonium bromide, tetrahexylammonium bromide (THAB), tetraoctylammonium bromide (TOAB), benzyl triethylammonium bromide (BTEAB) and tetraethylammonium bromide (TEAB), and 4-(tributylammonium) propansultan (QSO₃); organic solvents: acetonitrile, methylethyl ketone (MEK), benzene, tributylamine, dimethylbutylamine and cyclohexane and other reagents for synthesis are all G.R. grade chemicals.

2.2. Procedures

2.2.1. Kinetics of the reaction in synthesizing PTR

The reaction was proceeded in a 150-ml three-necked Pyrex flask which permits agitating the solution, inserting the thermometer, taking samples and feeding the reactants. A reflux condenser is attached to the port of the reactor to recover organic solvent (acetonitrile). The reactor is submerged in a constant temperature water bath whose temperature can be controlled to ±0.1 °C. To carry out a reaction, known quantities of potassium salt of phthalimide, toluene (internal stan-

dard, IS) and tetrabutylammonium bromide were dissolved in organic solvent (acetonitrile) and introduced into the reactor. To start an experimental run, measured quantities of 1-bromobutane were added to the organic solution. The mixture was stirred mechanically by a two-blade paddle (5.5 cm) at 800 rpm. During the reaction, an aliquot sample of 0.1 ml was withdrawn from the solution at a chosen time. The sample was immediately introduced into an organic solvent (acetonitrile) at 4 °C for dilution in order to retard the reaction, and then analyzed by GC using an internal standard method.

The product *N*-butylphthalimide was purified for identification from the solution by vacuum evaporation to strip off the organic solvent and washing with deionized water several times, then dried, and identified.
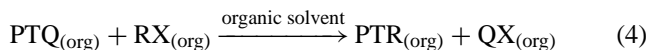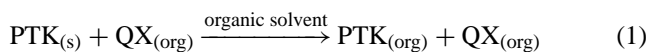
The product *N*-butylphthalimide and the reactants (potassium salt of phthalimide and 1-bromobutane) were identified by NMR and IR. Their concentrations (or contents) were analyzed by GC model GC17A (Shimadzu). The stationary phase was 100% poly(dimethylsiloxane). The carrier gas was N_2 (30 ml/min). The column was db-1 type. The results obtained from NMR and IR are highly consistent with the published data.

3. Reaction mechanism and kinetic model

In a liquid–liquid phase-transfer catalysis, the pseudo first-order kinetics is frequently observed for a molar quantity of the aqueous-phase reactant larger than that of the organic-phase reactant. The rate equation of the intrinsic organic-phase reaction was thus always expressed as a second-order kinetic law. In this work, potassium salt of phthalimide is sparingly soluble in organic solvent, such as acetonitrile. However, the hydration of potassium salt of phthalimide in aqueous solution is serious. Therefore, few products were obtained when the reaction of potassium salt of phthalimide and organic substrate was carried out under liquid (organics solvent)–liquid (water) phase-transfer catalysis conditions (LL-PTC). In this work, the synthesis of *N*-butylphthalimide from the reaction of potassium salt of phthalimide with organic substrate was carried out in a solid–liquid solution (organic solvent) under phase-transfer catalysis. No water was added to the reaction solution to prevent hydration. The main advantage of using a solid–liquid two-phase reaction is that the reaction is greatly enhanced and the yield of the product is increased in the presence of quaternary ammonium salt. First, potassium salt of phthalimide dissolved in the organic solvent with the addition of quaternary ammonium salt, i.e. PTK dissolving in the organic solvent reacts with quaternary ammonium salt to form an active intermediate *N*-(tetrabutylammonium) phthalimide (PTQ). The inorganic salt KX precipitated as a solid form from the organic solution.

The active $PTQ_{(org)}$ then react with 1-bromobutane to produce *N*-butylphthalimide. Thus, the reaction mechanism in

the organic solvent is proposed as:

$$PTK_{(s)} + QX_{(org)} \xrightarrow{\text{organic solvent}} PTK_{(org)} + QX_{(org)} \quad (1)$$

$$PTK_{(org)} + QX_{(org)} \xleftrightarrow{K_1} PTQ_{(org)} + KX_{(org)} \quad (2)$$

$$PTQ_{(org)} + KX_{(org)} \xleftrightarrow{K_2} PTQ_{(org)} + KX_{(s)} \quad (3)$$

$$PTQ_{(org)} + RX_{(org)} \xrightarrow{\text{organic solvent}} PTR_{(org)} + QX_{(org)} \quad (4)$$

where $RX_{(org)}$ and $QX_{(org)}$ represent the organic-phase reactant (1-bromobutane) and quaternary ammonium salt in the organic-phase solution, respectively. The subscripts "s" and "org" denote the species in solid-phase and in organic-phase, respectively.

The overall reaction is expressed as:

$$PTK_{(s)} + RX_{(org)} \xrightarrow{\text{organic solvent, QX}} PTR_{(org)} + KX_{(s)} \quad (5)$$
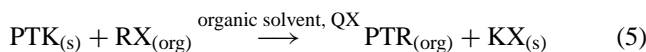
The forward reaction shown in Eq. (2) is fast and reaches to an equilibrium state in a short time. Thus, the equilibrium constant K_1 is defined as:

$$K_1 = \frac{[KX]_{org}[PTQ]_{org}}{[PTK]_{org}[QX]_{org}} \quad (6)$$

As stated, the inorganic salt KX precipitates from the organic solution, i.e.

$$K_2 = \frac{[KX]_s}{[KX]_{org}} \quad (7)$$

The rate equation of the intrinsic reaction is given in Eq. (4):

$$\frac{d[PTR]_{org}}{dt} = -\frac{d[RX]_{org}}{dt} = k_{int}[PTQ]_{org}[RX]_{org} \quad (8)$$

where k_{int} is the intrinsic rate constant.

A material balance for the catalyst is:

$$[QX]_{org,i} = [QX]_{org} + [PTQ]_{org} \quad (9)$$

where $[QX]_{org,i}$ is the initial concentration of QX.

Solving Eqs. (6), (7) and (9), we obtain:

$$[PTQ]_{org} = f_c[QX]_{org,i} \quad (10)$$

where f_c is given as

$$f_c = \frac{1}{1 + \frac{1}{K_1 K_2}\frac{[KX]_s}{[PTK]_{org}}} \quad (11)$$

The concentration of $[KX]_s$ and $[PTK]_{org}$ are retained at constant values after the induction period of the reaction. Therefore, as shown in Eq. (10), $[PTQ]_{org}$ keeps at a constant value. For this, Eq. (7) can be expressed as:

$$-\frac{d[RX]_{org}}{dt} = k_{app}[RX]_{org} \quad (12)$$

where k_{app} is the apparent rate constant of the pseudo first-order rate law.

$$k_{app} = k_{int}[PTQ]_{org} = k_{int} f_c[QX]_{org,i} \quad (13)$$

Eq. (12) is integrated:

$$-\ln(1 - X) = k_{app}t \quad (14)$$

where X is the conversion of 1-bromobutane (RX), i.e.

$$X = 1 - \frac{[RX]_{org}}{[RX]_{org,i}} \quad (15)$$

where $[QX]_{org,i}$ is the initial concentration of RX in the organic-phase solution. From Eq. (14), it is obvious that the reaction follows a pseudo first-order rate law.

4. Results and discussion

As stated, potassium salt of phthalimide is insoluble in organic solvent and is soluble in water. The hydration of potassium salt of phthalimide is serious. Therefore, only a small amount of product was obtained when the reaction is carried out in a liquid (water)–liquid (organic solvent) two-phase solution, even in the presence of quaternary ammonium salt in this work. Thus, only a trace amount of water or no water is added to the reaction system in order to minimize the loss of reactant due to hydration. To enhance the rate, the reaction was carried out in relatively anhydrous condition, i.e. a solid–liquid (organic solvent) solution.

In general, the reaction of a solid reactant and organic substrate was verified by many scientists [1,13–16]. The reaction mechanisms of the solid–liquid phase-transfer catalysis can be classified as the non-soluble system (heterogeneous solubilization) and soluble system (homogeneous solubilization), which depend on the solubility of the inorganic salt in the organic solvent. In this work, potassium salt of phthalimide is insoluble in organic solvent (acetonitrile). Therefore, the reaction mechanism of the present reaction system is classified as the non-soluble system (heterogeneous solubilization). The potassium salt of phthalimide dissolves in the organic solvent in the presence of tetrabutylammonium bromide (TBAB catalyst). In a similar way, Vander and Hartner [15] and Yoel and Zahalka [17] proved that quaternary ammonium salt can be used to dissolve the solid reactant.

In this work, the mechanism of the reaction of potassium salt of phthalimide ($PTK_{(s)}$) (in solid form) with the organic substrate (1-bromobutane, $RX_{(org)}$) (in liquid form) in the presence of quaternary ammonium salt was proposed by Eqs. (1)–(4). Independent experiments were carried out to examine the dissolving $PTK_{(s)}$ in organic solvents (e.g. acetonitrile) in the presence of quaternary ammonium salt. Without adding quaternary ammonium salt, $PTK_{(s)}$ is not dissolved in the organic solvent. This verifies the dissolving solid $PTK_{(s)}$ in the organic solvent in the presence of quaternary ammonium salt, as shown in Eq. (1). After dissolving $PTK_{(s)}$ in
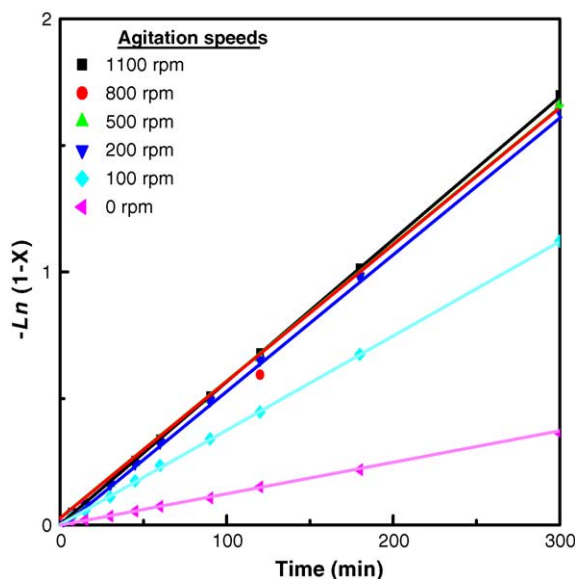
Fig. 1. A plot of $-\ln(1 - X)$ of 1-bromobutane vs. time with different agitation speeds; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 70 °C.

Fig. 2. A plot of $-\ln(1 - X)$ of 1-bromobutane vs. time with different volumes of acetonitrile; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 800 rpm, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 70 °C.

organic solvent with organic substrate (1-bromobutane), a homogeneous solution is formed. Then, $PTR_{(org)}$ is produced from the homogeneous solution. This evidence confirms that proposed reaction mechanism, as shown in Eqs. (2)–(4).

The effects of the reaction conditions on the conversion of 1-bromobutane (X) and the apparent rate constant (k_{app}) are discussed below.

4.1. Effect of the agitation speed

In principle, the homogeneous reaction is independent of the agitation speed. As shown in Fig. 1, the conversion is highly dependent on the agitation speed for agitation speed less than 200 rpm. Macroscopically, this result indicates that the reaction is carried out in a solid–liquid phase. In fact, the reaction proceeds by accompanying with the dissolving potassium salt of phthalimide in acetonitrile. The dissolving rate of potassium salt of phthalimide in acetonitrile is highly influenced by the agitation speed. In general, a high concentration of PTK dissolving in organic solvent is obtained at a high agitation speed. Therefore, the conversion of 1-bromobutane was increased with the increase in the agitation speed up to 200 rpm. For agitation speeds higher than 200 rpm, the conversion is influenced almost not at all by the agitation speed. This verifies that the reaction of potassium salt of phthalimide and 1-bromobutane was carried out in a homogeneous solution, i.e. PTK first dissolves gradually in acetonitrile and then reacts consecutively with 1-bromobutane. The purpose of stirring is to provide a well mixing to dissolve PTK in the acetonitrile in the presence of tetrabutylammonium bromide to form tetrabutylammonium phthalimide. Then, the dissolved PTQ reacted with 1-
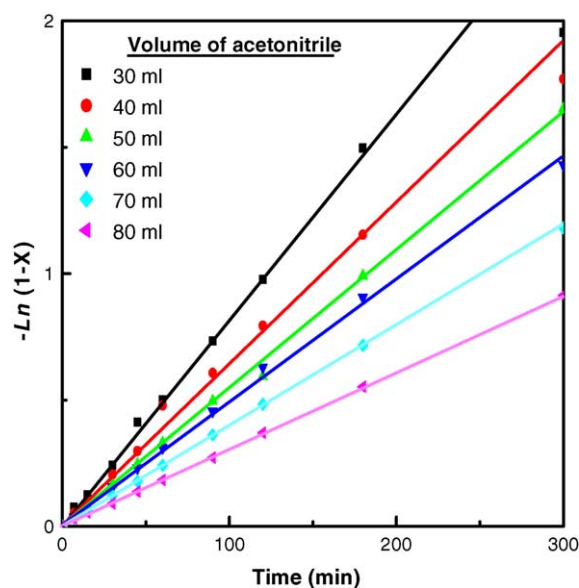
bromobutane to produce *N*-butylphthalimide in the homogeneous solution.

4.2. Effect of the acetonitrile volume

In a homogeneous reaction, the reaction follows the intrinsic kinetic law. The conversion or the reaction rate is directly proportional to the concentration of the reactants. A dilute concentration of the reactant is obtained using a large amount of organic solvent. As shown in Fig. 2, the conversion of 1-bromobutane is increased with the decrease in the volume of acetonitrile. Fig. 3 shows the dependence of the k_{app}-value on the volume of acetonitrile. The k_{app}-value is inversely proportional to the volume of acetonitrile, as expected.

4.3. Effect of the amount of TBAB catalyst

As stated, the reaction of potassium salt of phthalimide and 1-bromobutane was carried out in a homogeneous acetonitrile solution. The potassium salt of phthalimide in solid form first dissolves in acetonitrile in the presence of tetrabutylammonium bromide. Overall, TBAB still has an important role as catalyst in enhancing the reaction rate or the conversion. Fig. 4 shows the effect of the TBAB catalyst amount on the conversion of 1-bromobutane. The conversion of 1-bromobutane is increased with the increase in the amount of TBAB catalyst. The conversion is low in the absence of TBAB catalyst, although the reaction is greatly enhanced by adding a small amount of TBAB catalyst.

As stated, $PTK_{(s)}$ is insoluble in organic solvents, such as acetonitrile. However, it is only soluble in acetonitrile in the presence of tetrabutylammonium bromide. Therefore, the
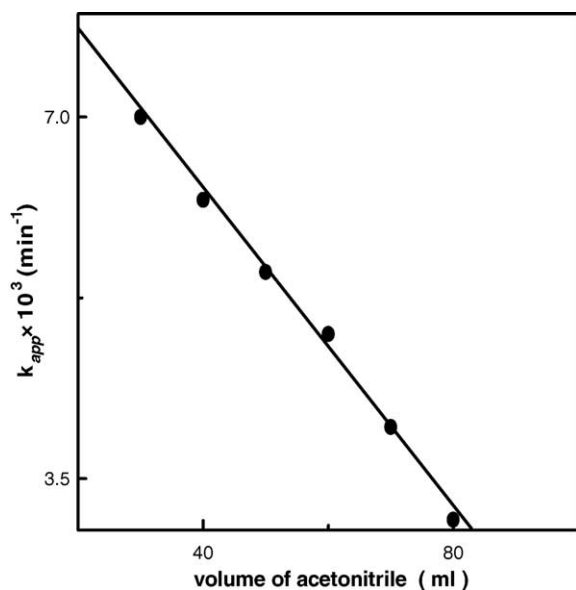
Fig. 3. A plot of the apparent rate constant vs. volume of acetonitrile; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 800 rpm, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 70 °C.

role of TBAB catalyst is not only to increase the dissolving $PTK_{(s)}$ in acetonitrile, but also to enhance the reaction. Fig. 5 shows the effects of the amount of TBAB catalyst on the k_{app}-value, which increases with the increase in the amount of TBAB catalyst. The k_{app}-value is linearly proportional to the amount of the TBAB catalyst up to 0.225 g. This result at low amount of TBAB catalyst is consistent with the theoretical derivation given by Eq. (13). However, the result does not follow the linear relation between k_{app}-value and the amount
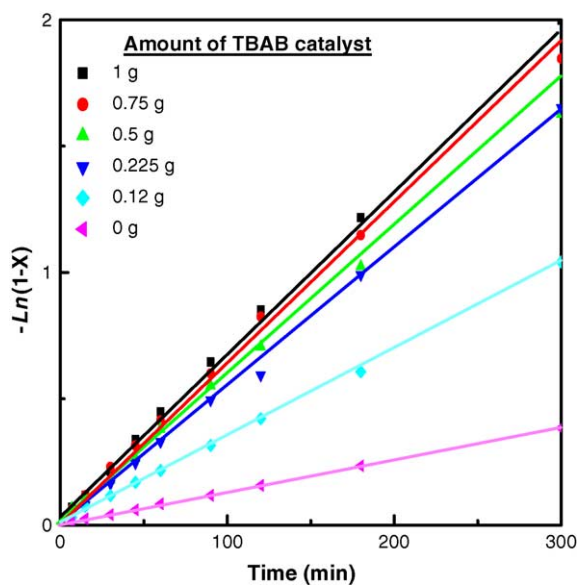

Fig. 4. A plot of $-\ln(1 - X)$ of 1-bromobutane vs. time with different amounts of TBAB catalyst; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 800 rpm, 0.5 g of toluene (internal standard), 70 °C.
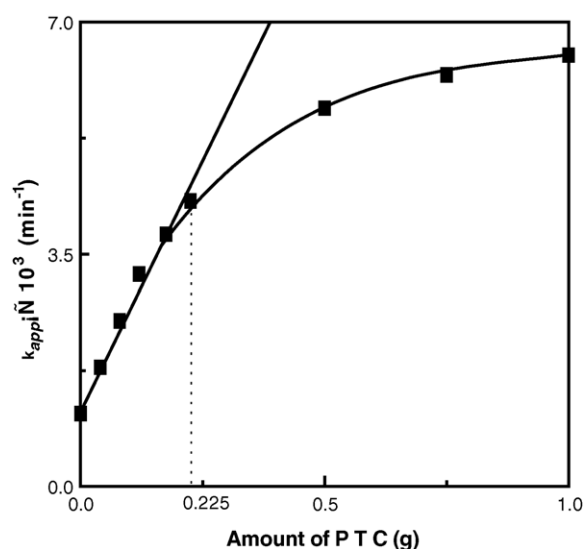

Fig. 5. A plot of the apparent rate constant vs. the amount of TBAB catalyst; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 800 rpm, 0.5 g of toluene (internal standard), 70 °C.

of the TBAB catalyst by using large amount of TBAB catalyst. This phenomenon is the same as that of the result by Yadav and Sharma [16], and Yoel and Zahalka [17]. In addition, Yoel and Zahalka [17] attributed this phenomenon by the salting out effect.

4.4. Effect of the quaternary ammonium salts

Since the reaction was carried out in the organic solvent (acetonitrile), it is clear that the hydrophilic and hydrophobic properties of the quaternary ammonium salt both play important roles in affecting the conversion. The effect of the quaternary ammonium salts on the conversion of 1-bromobutane is shown in Fig. 6. The order of the reactivities of these quaternary ammonium salts are: THAB > TBAB > TOAB > BTEAB > QSO₃. THAB, TBAB and TOAB of appropriate hydrophilic and hydrophobic properties exhibit high reactivity to obtain high conversion of 1-bromobutane. Those TEAB and QSO₃ (4-(trialkylammonium) propansultan), which are more hydrophilic, do not possess high reactivity. Therefore, the conversion of 1-bromobutane is relatively low in using hydrophilic quaternary ammonium salts.

4.5. Effect of the organic solvents

In this work, acetonitrile, methylethylketone, benzene, tributylamine, dimethylbutylamine and cyclohexane were employed as the organic solvents. Fig. 7 shows the influence of the organic solvents on the conversion. The dielectric constants of these six organic solvents are in the following order: acetonitrile (37.5) (70 °F) > methylethylketone (18.4) (72 °F) > benzene (2.3) (68 °F) > tributylamine (2.2) (77 °F) > dimethylbutylamine (2.1) (68 °F) > cyclohexane
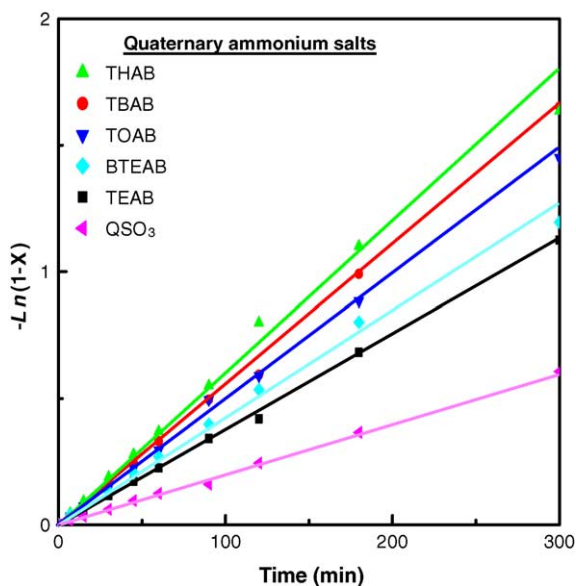
Fig. 6. A plot of $-\ln(1-X)$ of 1-bromobutane vs. time with different quaternary ammonium salts; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 0.7 mmol of PTC, 0.5 g of toluene (internal standard), 800 rpm, 70 °C.

Fig. 8. A plot of $-\ln(1-X)$ of 1-bromobutane vs. time with different amount of water; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 800 rpm. 70 °C.

(2.0) (68 °F). However, the reactivities of these six organic solvents are: acetonitrile > methylethylketone > benzene > tributylamine > dimethylbutylamine > cyclohexane. It is clear that the reactivities of the reactants in these organic solvents are consistent with the dielectric constants of the organic solvents. A high dielectric constant organic solvent leads to a high conversion of reactant.

4.6. Effect of the amount of water

As stated in this work, the reaction was carried out in a solid–liquid (organic solvent) solution in the absence of water. $PTK_{(s)}$ was dissolved in organic solvent in the presence of quaternary ammonium salt without adding water. In this section, only water was added to examine its effect of the conversion of 1-bromobutane. As shown in Fig. 8, the conversion of 1-bromobutane is affected by the water added. A negative effect due to the addition of water on the conversion is obtained, as expected, i.e. the conversion of 1-bromobutane is decreased with an increase in the amount of water. The main reason for this is that the hydration of $PTK_{(s)}$ occurs in the aqueous-phase. The hydration of $PTK_{(s)}$ is serious by adding more water to the reaction system, since the portion of $PTK_{(s)}$ is consumed due to the hydration instead of reacting with 1-bromobutane to produce $PTR_{(org)}$. The values of k_{app} are 5.5×10^{-3}, 3.7×10^{-3}, 1.7×10^{-3}, 1.1×10^{-3}, 0.6×10^{-3} and 0.3×10^{-3} min^{-1} for 0, 5, 10, 15, 20 and 25 ml of water, respectively. Therefore, the apparent rate constant is decreased with the increase in the amount of water. Without adding water, a large value of the apparent rate constant is obtained.

4.7. Effect of the temperature

As expected, the conversion of 1-bromobutane is large at a higher temperature. Fig. 9 shows the effect of temperature on the conversion. An Arrhenius plot of $-\ln(k_{app})$ versus $1/T$ is used to obtain an activation energy 21.84 kcal/mol from which the values of k_{app} are 0.07×10^{-3}, 0.13×10^{-3},
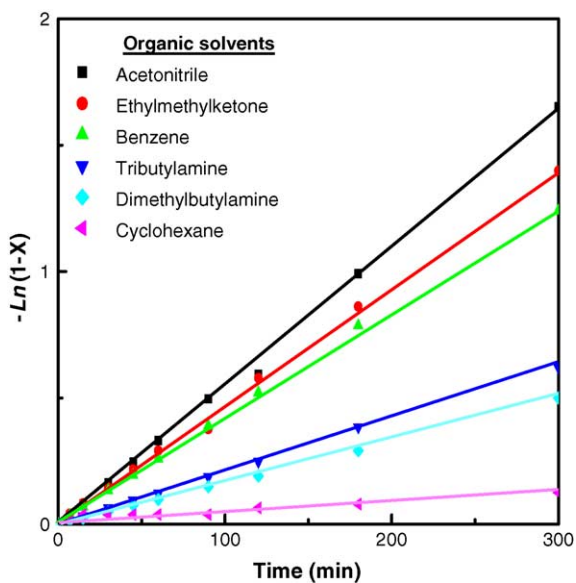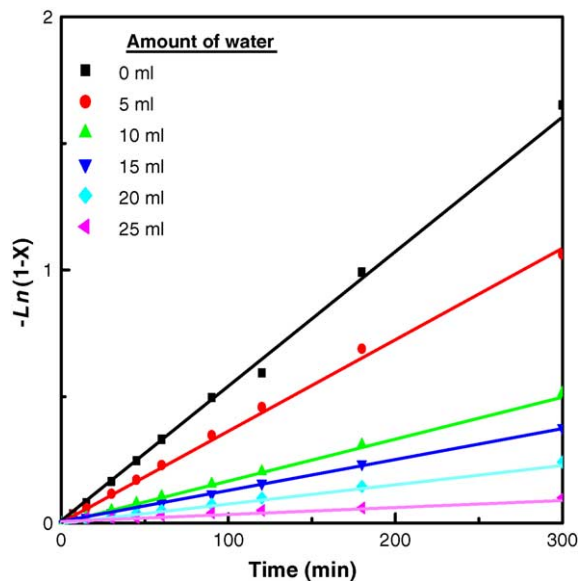

Fig. 7. A plot of $-\ln(1-X)$ of 1-bromobutane vs. time with different organic solvents; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of organic solvent, 800 rpm, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 70 °C.
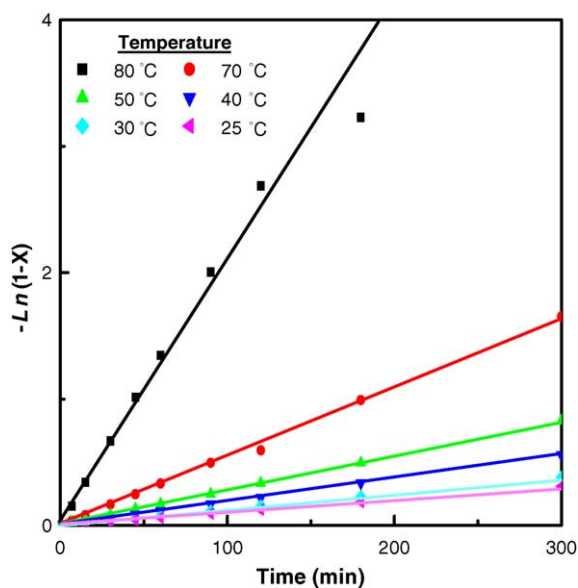
Fig. 9. A plot of $-\ln(1-X)$ of 1-bromobutane vs. time with different temperatures; 4 mmol of 1-bromobutane, 6 mmol of phthalimide potassium salt, 50 ml of acetonitrile, 0.7 mmol of tetrabutylammonium bromide, 0.5 g of toluene (internal standard), 800 rpm.

1.23×10^{-3}, 3.43×10^{-3} and 5.5×10^{-3} min^{-1} at 25, 30, 40, 50, 70 and 80 °C, respectively.

5. Conclusion

In this work, the reaction of potassium salt of phthalimide and 1-bromobutane to synthesize *N*-butylphthalimide was successfully carried out in a solid (PTK)–liquid (organic solution) two-phase medium under phase-transfer catalysis. The reaction is greatly enhanced by adding a small quantity of quaternary ammonium salt as a catalyst. Serious hydration occurs when water is added to the reaction solution, so the yield is decreased when water is added to the reaction solution. No hydration of potassium salt of phthalimide occurs when the reaction is carried out in the solid (PTK)–liquid (organic solution) two-phase solution. A pseudo first-order rate law is employed to describe the kinetic data. The conversion of 1-bromobutane (or the reaction rate) is increased with the increase in agitation speed, amount of TBAB catalyst, organic solvent of high dielectric constant and temperature. Nevertheless, the reaction rate (or the conversion) is decreased by increasing the amount of organic solvent. A quaternary ammonium salt of both appropriate hydrophilic and hydrophobic properties is recommended to obtain a high conversion of 1-bromobutane. The activation energy of the reaction system is 21.84 kcal/mol.

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