Catalysis Communications 145 (2020) 106097

Contents lists available at ScienceDirect

Catalysis Communications

journal homepage: www.elsevier.com/locate/catcom

Short communication

Fast cyclotrimerization of a wide range of isocyanates to isocyanurates over acid/base conjugates under bulk conditions

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ARTICLE INFO

Keywords:
Cyclotrimerization
Isocyanate
Isocyanurate
Acid/base conjugates

ABSTRACT

An array of organic bases DMAP (4-dimethylaminopyridine), DBU (1, 8-diazabicyclo [5.4.0] undec-7-ene), TBD (1, 5, 7-triazabicyclo [4.4.0] dec-5-ene), and their base/acid conjugate organocatalyst systems were evaluated in the trimerization of various isocyanates. The performance depended greatly on the combination of the catalyst systems, and the [HTBD][OAc] (acetic acid) catalyst systems were considerably the most active in contrast to the corresponding DMAP and DBU counterparts. The [HTBD][OAc] catalyst system was capable of providing isocyanurates from the cyclotrimerization of various isocyanate substrates in excellent yields in seconds even under bulk conditions. A bifunctional catalytic mechanism over [HTBD][OAc] was proposed.

1. Introduction

Isocyanurates produced by the trimerization of isocyanates can be used to enhance the chemical stability and physical mechanical properties of polyurethanes. Substituted aryl-functionalized isocyanurate is a useful activator for anionic polymerization of ε-caprolactams to Nylon-6. Additionally, it could substantially enhance the stability of polyurethane networks and coating materials with respect to thermal resistance, flame retardancy, chemical resistance, and film-forming characteristics [1]. It has been applied as a key component in chiral identification, microporous materials [2], coating materials [3], drug delivery, and as a crossing-linking agent for plastic. As a result, efficient and green isocyanurate synthesis is currently of commercial importance, especially in the production of rigid urethane-modified isocyanurate foams [2].

The common route for the preparation of isocyanurates is the cyclotrimerization of isocyanates. To promote the reaction, the catalyst systems including a broad range of compounds have been studied for many years. Metal catalysts have been reported such as organotin compounds, palladium(0) systems [4], hemilabile aluminumpyridyl-bis (iminophenolate) complex [5], as well as rare earth metal catalysts [6].

Recently, organocatalysis has proven to be powerful and attractive in organic synthesis. Compared to the organometallic catalysts, the main attribute of metal-free organocatalyst including Brønsted/Lewis base and/or acid was its versatility, high selectivity, reduced toxicity and cost, and ability to work under air- and water-tolerating reaction conditions. In the trimerization of isocyanates, Verkade et al. [1] developed a method for efficient trimerization of isocyanates using 0.0033 mol% $ZP(MeNCH_2CH_2)_3N$ (Z = lone pair, proazaphosphatrane) catalyst, which could be recycled six times without lost activity; however, the synthesis process was complicated and expensive. Giuglio et al. [7] proved that TDAE (tetrakis (dimethylamino) ethylene) had good activity under ambient conditions with low catalyst loading; however, the synthetic product had a strong odor in the final product [8]. High yields of isocyanurates could be obtained with a N-heterocyclic olefin catalyst loading as low as 0.005% [9].

N-heterocyclic carbene was also widely studied as an efficient organic catalyst. Duong et al. [10] found that isocyanurates could be obtained in excellent yields only with 0.1 mol% 1, 3-bis- (2, 6-diisopropylphenyl) -4, 5-dihydroimidazol-2-ylidene at room temperature. Li et al. [11] reported that amino-NHC (amine-linked N-heterocyclic carbene) was used in the trimerization of isocyanates with 10 mol% catalyst loading at room temperature for 3 h. However, this kind of catalyst system has the disadvantage of high toxicity and difficult separation.

In parallel, multi-component organocatalysts have proven useful in the generation of oligomers and polymers. Lewis pair was originally used to catalyze the hydrogenations of small molecular compounds [12] and to catalyze polymerization [13]. In the ring opening polymerization (ROP) of lactones, the trade-off between the selectivity and

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https://doi.org/10.1016/j.catcom.2020.106097
Received 9 March 2020; Received in revised form 22 June 2020; Accepted 22 June 2020

reactivity was solved by the multi-component system. The acid/base complexes could control the targeted molecular weights and narrow polydispersities by avoiding the transesterifiction [14,15]. Further, Lin and Waymouth found that the polymerization activities even span 6 orders of magnitude if the H-bond donors derived from ureas or thioureas combined with the basic cocatalysts [16]. Zhang and coworkers summarized the effect of acidity, basicity, and steric hindrance of Lewis pairs on polymerization of polar vinyl monomers [17]. At the same time, the strategies of the combined catalyst system were also effective in the isocyanurates trimerization. Among tetrabutylammonium halides, only the fluoride had catalytic activity for the reaction, while it was difficult to accurately control trimerization and generate dimerization products [18]. In the presence of potassium sulfate, tetrabutylammonium bromide also had the capability to trimerize isocyanates at high temperature in a solvent-free system, and effectively shortened the reaction time [19]. It has also been found that the addition of TBAPINO (tetrabutylammonium phthalimide-N-oxyl) and TEACB (tetraethylammonium 2- (carbamoyl)benzoate) has a great effect on the trimerization of isocyanates [20]. Besides the work on tetrabutylammonium halides, less attention has been focused on the multi-component organocatalysts.

Inspired by the above work by bifunctional catalyst, we report herein a synthesis of acid/base mixtures and their application to catalyze the trimerization of isocyanates. Among the successful organocatalysts, DMAP (4-dimethylaminopyridine), DBU (1, 8-diazabicyclo [5.4.0] undec-7-ene), and TBD (1, 5, 7-triazabicyclo [4.4.0] dec-5-ene) have been extensively studied. These base/acid conjugate organocatalyst systems from organobases and appropriate Brønsted acids were prepared and surveyed in trimerization of isocyanates. Under the optimal conditions, the reaction generality was evaluated and scale-up experiments was demonstrated.

2. Experimental section

2.1. Materials

1, 8-diazabicyclo [5.4.0] undec-7-ene, 1, 5, 7-triazabicyclo [4.4.0] non-5-ene, 4-dimethylamino-pyridine, and various types of isocyanates and other reagents were commercially available and used as received. All solvents were domestic analytical pure reagents and were not treated before use.

2.2. Experimental instruments and methods

Nuclear magnetic resonance apparatus: Bruke DRX 400, based on the chemical shift of TMS (tetramethylsilane), in ppm. High resolution mass spectrometry: Waters Micromass GCT Detector. Melting point instrument: XT3A micro melting point tester. Column chromatography silica gel and thin layer chromatography silica gel plate: Yantai Jiangyou Silicone Development Co., Ltd.

2.3. Synthesis of [HTBD][OAc] catalyst

1, 5, 7-Triazabicyclo [4.4.0] non-5-ene (500 mg, 3.59 mmol) was dissolved in 2 mL ethanol and stirred at 0 °C, and then acetic acid was slowly added (216 mg, 3.59 mmol). The mixture was stirred at room temperature for 1 h and concentrated under reduced pressure to remove all ethanol, and then transferred to a vacuum oven at 80 °C overnight. The catalyst [HTBD][OAc] (acetic acid) was a white crystalline solid after cooling (694.5 mg, 97% yield).

Other catalysts in the article can be readily prepared by neutralizing an organic base with carboxylic acid, ketone, and azole. [HDMAP][$PhCO_2$] (benzoic acid), [HDBU][OAc], and [HTBD][DioxoCy] (1, 3-cyclohexanedione) were synthesized according to the reported procedure [21]. [HTBD][TFE] (2, 2, 2-trifluoroethanol) and [HTBD][Benlm] (benzimidazole) [22], [HDBU][TFE] and [HDBU][$PhCO_2$] [23] were synthesized according to the reported procedures, respectively.

2.4. Cyclotrimerization of isocyanate over [HTBD][OAc] catalyst

In an oven-dried 10 mL round bottom flask, [HTBD][OAc] (10 mg, 0.5 mol%) and isocyanates (10 mmol) were added vigorously in air to form isocyanurates (**a-i**). For the liquid isocyanate, the mixture was vigorously stirred at room temperature, while the solid isocyanate was stirred at 50 °C for a few seconds. During this process, the reaction time was recorded with a stopwatch. The obtained white solid was cooled to room temperature and then ground into powder, which was stirred for 10 min in 4 mL cold ether. The solid was separated by vacuum filtration and washed with additional water to remove the catalyst. After filtration, recrystallization from ethanol gave the desired isocyanurate.

2.5. Scaled-up experiment

In an oven-dried 100 mL round bottom flask, [HTBD][OAc] (150 mg, 0.75 mmol) and p-tolyl isocyanate (20 g, 150 mmol) were added and the mixture was stirred vigorously at room temperature. During this process, the reaction time was recorded with a stopwatch. The solid was cooled to room temperature and ground into powder. A quantity of 40 mL of cold diethyl ether was added and stirred for 10 min. The resulting solid was separated by vacuum filtration and washed with additional water to remove the catalyst. After filtration, recrystallization from ethanol gave the desired p-tolyl isocyanurate (19.2 g, 96% yield).

3. Result and discussion

3.1. Catalyst screen in the isocyanate trimerization

Organocatalysts (Fig. 1) derived from DMAP, DBU, and TBD have been widely adopted in the polymerization of cyclic esters, including LA (L-lactide), VL (δ-valerolactone), and CL (ε-caprolactone) [24]. In this regard, their performances in the trimerization of p-tolyl isocyanate were surveyed in the absence of solvents.

In Table 1, the DMAP was almost inactive for the reaction, and only a small amount of cyclotrimerization products were generated at 70 °C for 24 h (Entry 1). For DBU, the yield was only 17% (Entry 2) after reaction for 1.5 h. Upon testing the TBD, the reaction mixture was solidified within seconds at room temperature. The basicity values in AN (acetonitrile) of DMAP, DBU, and TBD are 11.43, 24.34, and 26.03, respectively [25]. That is, the insufficiently basic DMAP with low pKa was unfavorable for the trimerization. The increased basicity had a beneficial effect on the activity. Unfortunately, the product of the reaction over TBD was found to be a mixture of dimers and trimers with the ratio of 1:1. Thus, the preliminary experiments indicated that the desired products were not obtained in an acceptable yield by the three single organocatalysts.

Consequently, to tune the reactivity and improve the thermal stability of organocatalysts, a versatile pairing of the organic superbasic proton acceptors with organic acids was explored [23]. Protic ionic liquids were synthesized via proton transferfrom acids to organic bases [26]. Organocatalysts based on ionic mixtures of acids and bases were successfully applied in the polymerization of trimethylene carbonate with MTBD (7-methyl-1, 5, 7-triazabicyclo [4.4.0] dec-5-ene)/TFA (trifluoromethanesulfonic acid) [27]. DMAP/HOTf (triflic acid) displayed an outstanding catalytic activity to produce poly (L-lactide) (PLLA) [28]. Such conjugates may be less active than the organocatalytic bases, but the polymerization appeared to be more controlled. In the presence of DBU/BA (benzoic acid), the dimerization of ethyleneglycol was proven effective [29]. TBD/MSA (methanesulfonic acid) was also demonstrated in PET (polyethylene terephthalate) depolymerization [30].

Nine conjugates with stoichiometry mixtures of the above bases and

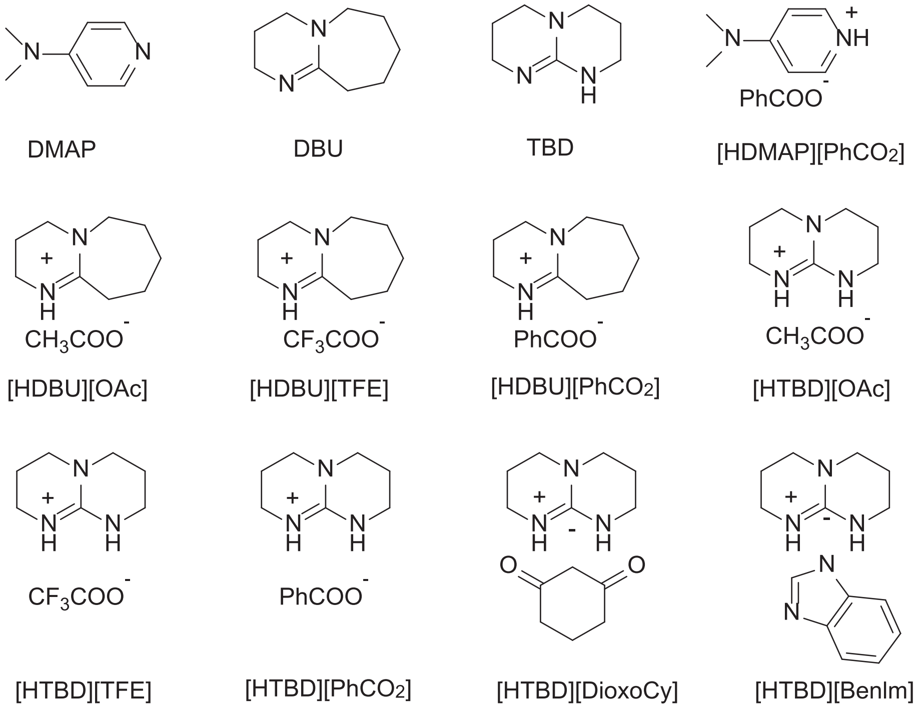
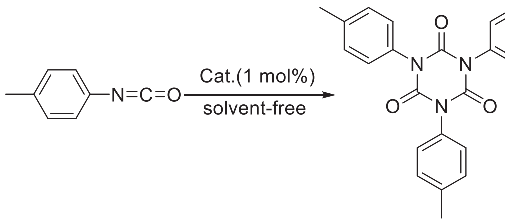


Fig. 1. Chemical structures of DAMP, DBU, TBD, and their derived salts.

Table 1
Catalytic activity of various organocatalysts and their derived salts on the cyclotrimerization of p-tolyl isocyanate.



Entry	Cat.	T (°C)	Time	Yield (%)[a,b,c]
1	DMAP	70	24 h	6
2	DBU	70	1.5 h	17
3	TBD	25	40 s	45
4	[HDMAP][$PhCO_2$]	70	5 h	13
5	[HDBU][OAc]	70	2 h	51
6	[HDBU][TFE]	70	3 h	41
7	[HDBU][$PhCO_2$]	70	2 h	53
8	[HTBD][OAc]	25	35 s	93
9	[HTBD][TFE]	50	21 min	90
10	[HTBD][$PhCO_2$]	25	2.5 min	91
11	[HTBD][DioxoCy]	50	7.5 min	86
12	[HTBD][BenIm]	50	6 min	91

[a] Reaction conditions: 10 mmol of p-tolyl isocyanate, 0.1 mmol Cat.
[b] Reactions were performed under air.
[c] Isolated yields (average of three runs).

acids in Fig. 1 were conveniently synthesized in high yield. The tested Brønsted acids are benzimidazole, CH_3COOH (acetic acid), PhCOOH (benzoic acid), DioxoCy (1,3-cyclohexanedione), and CF_3COOH (trifluoroacetic acid), and their pKa values in DMSO (dimethyl sulfoxide) of the proton donors are 16.4, 12.3, 11.0, 10.3, and 3.4, respectively [31].The pKa difference between the acids and the bases correlated with the stability of the mixtures [32]. The results of trimerization of p-tolyl isocyanate over these conjugates were presented in Table 1. The activity of [HDAMP][$PhCO_2$] improved from 6% to 13% with a shorter reaction time compared with the neutral DMAP. Similarly, the [HDBU][OAc], [HDBU][TFE] (2, 2, 2-trifluoroethanol), and [HDBU][$PhCO_2$] presented much higher catalytic activity than the original base catalyst.

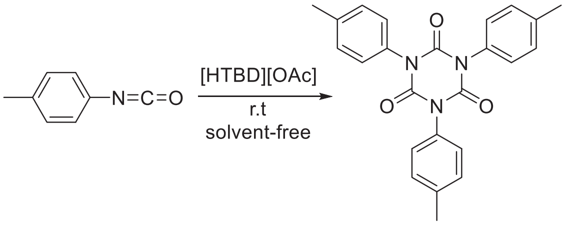
The selectivity increased significantly for the five TBD-derived conjugates. Varying acidity and structure of the conjugate acid could modulate the H-bond donating ability of the catalysts. The [HTBD][OAc] presented the highest yield in 35 s. While, the [HTBD][$PhCO_2$], or [HTBD][TFE] resulted in comparable yields of 90% varied the reaction time from 2.5 to 21 min, which was longer than the single TBD. [HTBD][DioxoCy] (1, 3-Cyclohexanedione) showed the lowest yield of 86% at higher temperature (Entry 11), while the yield could increase to 91% using [HTBD][BenIm] at 50 °C (Entry 12). Generally, the activity over the TBD-based conjugates increased with the increase of pKa, except [HTBD][DioxoCy] and [HTBD][BenIm] with large steric hindrance. [HTBD][OAc], with the weakest conjugate acid, was the most favorable of the trimerization of p-tolyl isocyanate.

3.2. Trimerization of isocyanate over [HTBD][OAc] catalyst

Based on the above analysis, [HTBD][OAc] was chosen in the next optimization experiments in terms of the catalyst loadings from 1.0% to 0.05%. In Table 2, the highest 95% yield was obtained (Entry 2) with 0.5 mol% vs. monomer in 52 s. With the decrease of [HTBD][OAc] loadings, the reaction time increased and the yield decreased significantly. The yield was only 84% over 0.05 mol% [HTBD][OAc] with the reaction time extended to 32 min. The reactions under air or nitrogen represented similar yields, indicating that the catalyst was effectively resistant to air and humidity. Meanwhile, the catalyst could be stored for several months without loss of performance.

The organocatalysts are often tolerant to a variety of solvents and even a solvent-free condition, which facilitates the separation of the product. Considering the solubility of [HTBD][OAc], as well as the solvent-dependent pKa values in various solvents in the reaction, seven solvents were examined and summarized in Table 3. Generally, the presence of solvent in the reaction led to a longer reaction time and decreased the yield regardless of the polarity of the solvent. The product yield could reach 95% within 52 s (Entry 1) at the solvent-free

Table 2
[HTBD][OAc] catalyst loadings on the trimerization of isocyanate.



Entry	Mol.(%)	Time	Yield (%)[a,b,d]
1	1.00	35 s	93
2	0.50	52 s	95
3	0.25	2.5 min	89
4	0.10	8 min	83
5	0.05	32 min	84
6[c]	0.50	57 s	95

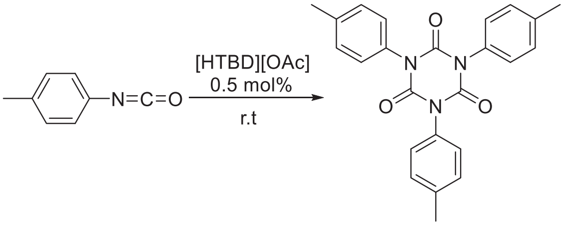
[a] Reaction conditions: 10 mmol of p-tolyl isocyanate.
[b] Reactions were performed under air.
[c] Reactions were performed under nitrogen.
[d] Isolated yields. (average of three runs).

condition, while only the diethyl ether realized the best yield 88% (Entry 3) among these solvents. Accordingly, the optimal condition for the trimerization reaction was 0.5 mol% [HTBD][OAc] in a bulk system, avoiding the use of toxic solvents.

3.3. Versatility of aryl isocyanates cyclotrimerization over [HTBD][OAc] catalyst

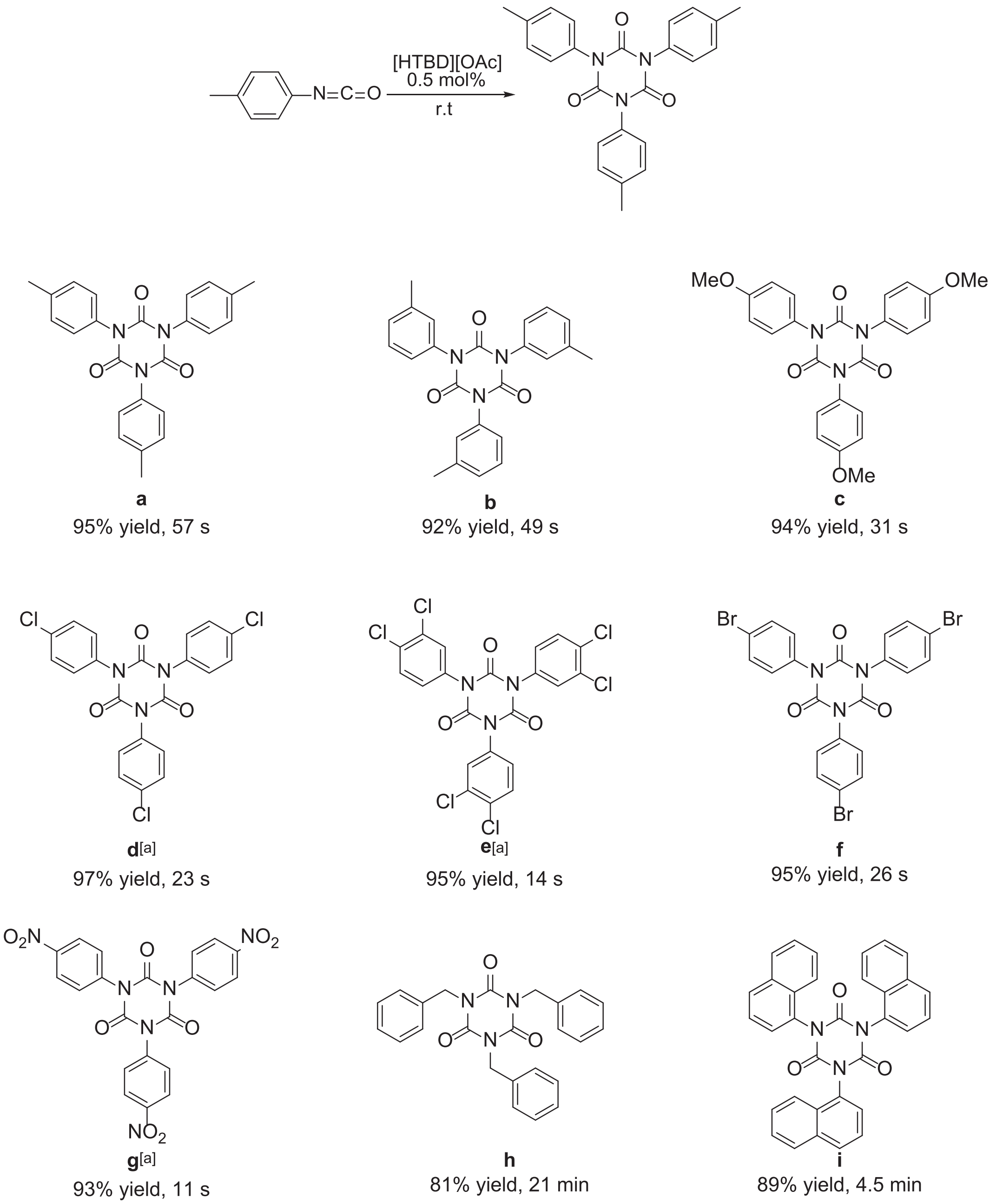
With the optimized conditions in hand, the scope and efficiency of the process were investigated. The results of a broad range of structurally diverse aryl isocyanates bearing electron-donating or electron-withdrawing substituents were summarized in Scheme 1. Aromatic isocyanates were well tolerated as substrates. For the substituents with methyl groups in the benzene rings, the isocyanurate with the methyl group at either para- or meta- position presented > 92% isolated yields within 1 min. The position of the substituents had little effect on the performance. Even the much stronger 4-methoxypheny moiety accelerated the reaction with 94% isolated yields in 31 s. The decrease [1,10] or even failure [33] over various catalysts in the reactivity due to the electron-dense substrates was not found in the case of [HTBD][OAc] bifunctional catalyst. For the aryl isocyanates containing various electron-withdrawing substituents, 4-bromophenyl isocyanate was immediately cyclotrimerized at room temperature within 26 s in a yield of 95% (**f**). As the 4-chlorophenyl isocyanate, 3,4-dichlorophenyl isocyanate, and 4-nitrophenyl isocyanate were solid at room temperature, the isocyanates melted at 50 °C and then trimerized with the addition of catalyst under vigorous stirring to ensure a homogeneous reaction condition. Neither the type nor the position of the substitutes of the isocyanates influenced the reaction, and the yields were 97%, 95%, and 93%, respectively (**d**, **e**, **g**). Furthermore, [HTBD][OAc] was also effective for sterically hindered isocyanates such as 1-naphthyl isocyanate, and the product (**i**) was in the yield of 89%. However, benzyl isocyanate took about 21 min to synthesize the expected product in the yield of 81% (**h**). Here, cyclohexyl isocyanate was chosen, but no

Table 3
Solvent effect in isocyanate trimerization.



Entry	Solvent	Time	Yield (%)[a,b,c,d]
1	None	52 s	95
2	THF	12 h	23
3	Et_2O	20 min	88
4	n-Hexane	18 min	79
5	CH_3CN	30 min	42
6	CH_2Cl_2	12 h	25
7	Toluene	12 h	27
8	EtOAc	12 h	31

[a] Reaction conditions: 10 mmol of p-tolyl isocyanate, 0.05 mmol [HTBD][OAc].
[b] Reactions were performed under air at room temperature.
[c] Reaction was performed with 1 mL of solvent.
[d] Isolated yields. (average of three runs).



Scheme 1. Various aryl isocyanates trimerization over [HTBD][OAc] catalyst.

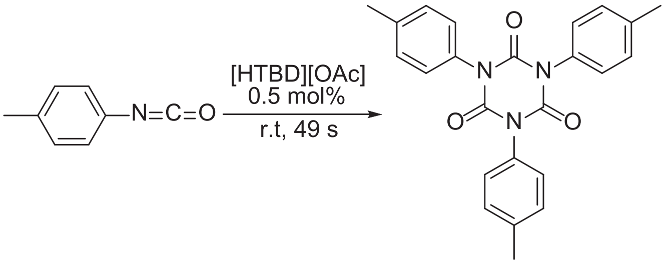
desired product was obtained in our catalytic system even after a long reaction time, indicating that the catalyst [HTBD][OAc] was not effective for catalytic alkyl isocyanate trimerization. The alkyl isocyanate trimerization was difficult to achieve, and few catalysts presented the capability [1]. The performance of cyclotrimerization of alkyl and aromatic isocyanates is greatly dependent on the catalysts. Aromatic isocyanurates were well synthesized by catalysts such as Pd (0) complexes [4], while *m*-terphenyl Mn(II) and Fe(II) complexes showed exclusive reactivity towards the cyclotrimerization of primary aliphatic isocyanates in toluene [34]. Furthermore, a few catalysts, such as hemilabile aluminum pyridyl-bis (iminophenolate) complex [5] and N-heterocyclic catalyst [9], were valid for both alkyl and aromatic isocyanates cyclotrimerization. Besides the electron-withdrawing and electron-donating effect of the substituent ligand on the nitrogen atoms, more recently, computational NOB (natural bond orbital) investigations showed that the resonance orbital interaction between the phenyl ring isocyanate molecule and the NCO moiety in aromatic isocyanates provided more significant resonance stabilization than that in the alkyl-substituted derivatives in the reaction [35].

[a] Reactions were performed under air at 50 °C.

3.4. Scaled-up production of isocyanate cyclotrimerization by [HTBD][OAc]

In order to test the potential application of the isocyanate trimerization catalyst over [HTBD][OAc], a scaled-up experiment was carried out (Scheme 2).

The reaction was scaled up under optimal conditions with 0.5 mol% amount of catalyst [HTBD][OAc]. With 20 g p-tolyl isocyanate, the separation yield was 96% within 49 s. Compared with the small scale reaction (Entry 1 in Table 3), the yield improved with the similar reaction time, showing that [HTBD][OAc] was also excellent at a level of



Scheme 2. Scaled-up production of isocyanate trimerization.

ten grams.

3.5. Catalytic mechanism of isocyanate cyclotrimerization over [HTBD][OAc]

It is common for isocyanate trimerization to proceed via nucleophilic attack on the isocyanate carbon. In combination with previous DFT studies on the catalytic trimerization of isocyanates catalysts [5,35], we hypothesized a potential synergistic H-bonding functional catalytic mechanism with repeated coordination–insertion of isocyanate. As shown in Scheme 3, the catalyst was able to activate the -NCO moiety that the oxygen atom with more electronegativity on the isocyanate functional group. Combined with an additional H-bonding in reaction of catalyst [HTBD][OAc] and isocyanate, a nucleophilic attack of the catalyst on the carbon was formed. The -NCO moiety then became sufficiently nucleophilic to attack another isocyanates. The catalyst [HTBD][OAc] could activate the isocyanate through its negatively charged oxygen atom (**I-II**), and the obtained anion cyclized with another activated isocyanate (**III-IV**). Finally, the cycle of isocyanate ring trimerization was completed to obtain the isocyanurate.

4. Conclusions

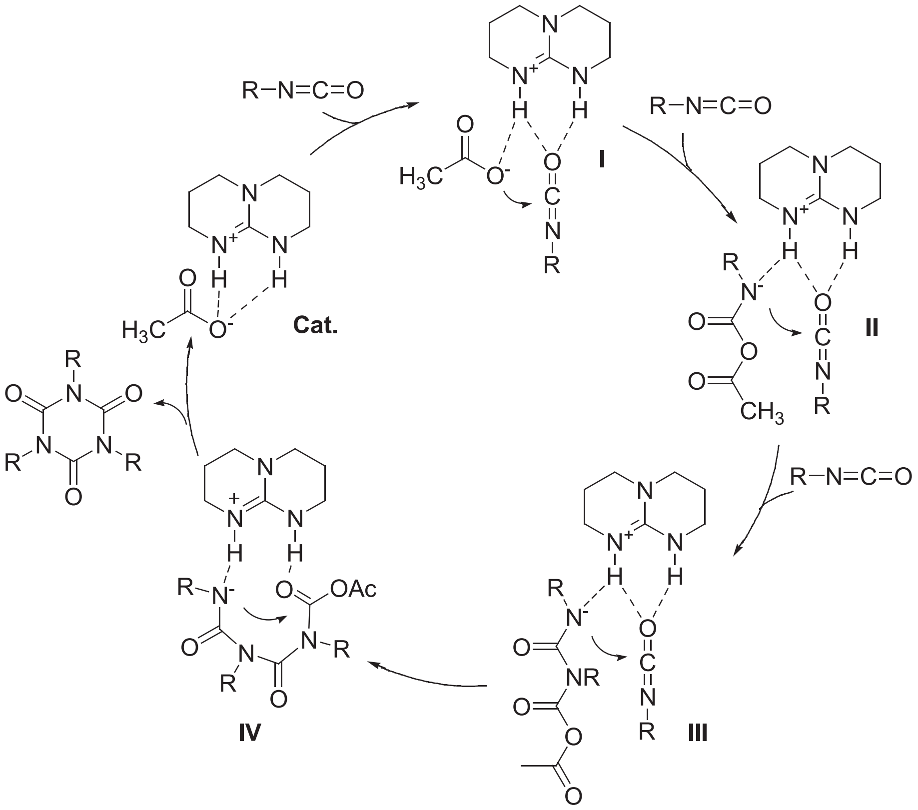
In summary, we have developed a new, mild, and efficient conjugates catalyst for the rapid and green synthesis of various aryl isocyanurates. The compacted counterparts of organocatalysis bases and the Brønsted acids, the amount of catalyst, and the solvent were tested in the trimerization reaction. The catalytic activity mainly depends on the basic strength of base conjugate, and the acid can tune the reaction performance. Under the optimal reaction conditions with 0.5 mol% [HTBD][OAc], the trimerization of aryl isocyanate can achieve an excellent yield within few seconds or minutes under the solvent-free condition at room temperature. This catalytic system is compatible for isocyanates with various substrates. A twenty-gram experiment on the trimerization of p-tolyl isocyanate demonstrated the scalability and potential applicability. A possible reaction mechanism of isocyanate trimerization was proposed based on the bifunctional in the conjugates. The study provided an eco-friendly synthetic method to access aryl isocyanurates.

Credit author statement

L. W. and W. L. contributed to materials synthesis, and catalytic evaluation; J. Y. and R. C. contributed to the conception, design and results interpretation; L. W. wrote the original draft. Finally, R. C. reviewed, edited, and submitted the manuscript in the final form. All authors contributed to the discussion, read and approved the final version of the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.



Scheme 3. Proposed catalytic cycle for the isocyanate trimerization over [HTBD][OAc] catalyst.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.catcom.2020.106097.

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