Metaboric acid-catalyzed synthesis of cyclic carbonates from epoxides and CO₂

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Abstract

Carbon dioxide stands out as the most prevalent greenhouse gas on Earth, presenting itself as not only abundant but also as a resource that is inexpensive, and renewable. Consequently, extensive research has been conducted on the conversion of CO_2, with particular emphasis on the cycloaddition reaction involving CO_2 and epoxides, resulting in the formation of cyclic carbonates. In this study, we report a metaboric acid/tetrabutylammonium bromide system for catalyzing the cycloaddition reaction. Employing HBO_2 (1.25 mol %) and tetrabutylammonium bromide (3 mol %) as catalysts, the cycloaddition reaction between 4-vinylcyclohexene oxide and CO_2 exhibited exceptional efficiency, yielding a 96% yield in 5 hours at 140 °C under 14 bar pressure in solvent-free conditions. Notably, this catalytic system exhibited commendable efficacy in facilitating cycloaddition reactions with various mono- and disubstituted epoxides.
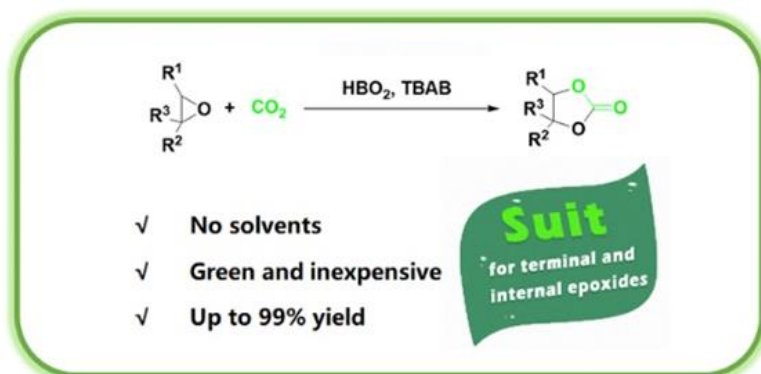
Introduction

Since the onset of the industrial revolution, the consumption of fossil energy has resulted in a substantial emission of CO_2 into the atmosphere. This has given rise to a myriad of issues, including climate anomalies, elevated sea levels, and the melting of glaciers, all of which exert a detrimental impact on the environment.[1,2] The greenhouse effect, a consequence of this prolific CO_2 emission, has garnered considerable attention from the scientific community. Consequently, there is a burgeoning interest in exploring methods to judiciously harness and utilize CO_2. Recognized as an integral component of the carbon cycle, CO_2 boasts extraordinary abundance. Through chemical conversion of CO_2, it is not only feasible to capture and utilize CO_2 but also to derive valuable chemical products. This renders chemical conversion an exemplary avenue for the efficient transformation and utilization of CO_2, addressing both environmental concerns and yielding valuable products.

The synthesis of cyclic carbonates through the cycloaddition of CO_2 with epoxides represents a prominent area of research in the field of CO_2 conversion. This reaction stands out for its ability to accomplish the one-step synthesis of cyclic carbonates, achieving a 100% atom efficiency. In recent decades, a plethora of catalysts has emerged, with metal complexes and ionic liquids being the most extensively investigated. Metal complexes, encompassing metals such as Al,[3-6] Co,[7-10] Fe,[11-14] Zn[15-19] and Ni[20-22] have demonstrated robust catalytic activity under mild conditions. However, the synthesis of relatively expensive metal complexes poses challenges. On the other hand, ionic liquid catalysts,[23-30] while generally exhibiting weaker catalytic activity compared to metal complexes, offer advantages such as environmental friendliness, straightforward synthesis, and good stability, making them a subject of extensive research. In our quest for a catalyst that aligns with criteria of environmental friendliness, cost-effectiveness, and superior catalytic performance, our attention has turned to boron catalysts.

Boron, a plentiful element in the Earth's crust with substantial development potential, has been thoroughly explored. Xue et al.[31] introduced a BPO_4/KI binary system, showcasing efficient catalysis for the conversion of various epoxides (93%-99% yield) at 110 °C and 40 bar. Lu et al.[32] innovatively demonstrated that B_2O_3 can serve as a heterogeneous catalyst for the cycloaddition reaction of epoxides and CO_2. Utilizing ball-milled B_2O_3 (0.5 mol %) and TBAB (1 mol %), they achieved a separation yield of 89% with PO (propylene oxide) at 100 °C, 20 bar, and 2 hours. Wu et al.[33] contributed a groundbreaking dual-functional organic boron catalyst, yielding PC (propylene carbonate) with a 99% yield at room temperature and 20 bar for 16 hours, demonstrating excellent catalytic performance across 14 epoxy substrates. Krishnan et al.[34] introduced boron-doped graphitic carbon nitride catalysts, featuring both acidic (boron) and basic (nitrogen) sites, exhibiting excellent recyclability, and stability under atmospheric pressure at 100 °C for 60 hours. Various epoxy substrates achieved conversion rates exceeding 80%. Liu et al.[35] established an H_3BO_3/TBAB binary catalytic system for epoxidized soybean oil fatty acid methyl esters, boasting advantages of low cost, non-toxicity, and high efficiency. Operating at 100 °C and 10 bar for 10 hours, a yield of 92.3% was reported. Liu et al.[36] further screened 14 boron-based compounds for the cycloaddition reaction of 1,2-epoxybutane and CO_2, identifying 12 of them with certain catalytic activity. The tetrahydroxydiboron catalyst (9 mol %) and TBAI (13.5 mol %) achieved a 99% yield at room temperature and atmospheric pressure for 24 hours. The tetrahydroxydiboron catalyst also displayed notable catalytic activity across various epoxy substrates with different functional groups. Presently, while boron-based catalysts demonstrate commendable catalytic activity for mono-substituted epoxides, research on challenging-to-catalyze internal epoxides remains limited.

Results and Discussion

In this study, we employ commercially available HBO_2 and TBAB as a dual catalytic system to facilitate the cycloaddition reaction of the internal epoxide 4-vinylcyclohexene oxide (VCHO, with a diastereoisomeric ratio of approximately 1:1) with carbon dioxide. Owing to significant steric hindrance, the cycloaddition reaction of VCHO with carbon dioxide is constrained, rendering it a particularly challenging substrate.[5,17,37]

    Catalysts play a pivotal role in the cycloaddition reaction between carbon dioxide and epoxides. Initially, a range of inorganic boracic species was screened. Employing TBAB as a co-catalyst, the influence of various inorganic boracic species on the reaction was investigated under solvent-free conditions at 100 °C, 10 bar and 6 hours (Table 1). Notably, H_3BO_3 and B_2O_3 exhibited positive catalytic activity (Table 1, entries 1, 2), aligning with findings from prior studies.[32,35] Conversely, sodium borate and ammonium borate displayed subpar catalytic activity (Table 1, entries 3, 4), while zinc borate and HBO_2 demonstrated robust catalytic effects (Table 1, entries 5, 6), achieving a VCHO conversion rate exceeding 80% and a cyclic carbonate yield surpassing 60%. Given the commendable catalytic performance, cost-effectiveness, and environmental friendliness of HBO_2, it emerged as the optimal inorganic boracic catalyst for further investigation.

Table 1. Optimization of catalyst systems for the cycloaddition reactions[a]
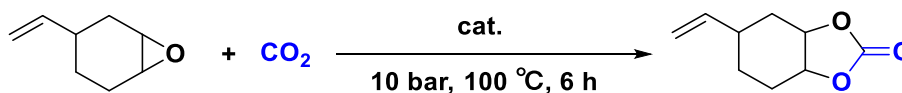
Entry	Cat.	Co-cat.[b]	Conv.(%)[c]	Yield(%)[c]	TON[d]
1	H_3BO_3	TBAB	66	44	9.0
2	B_2O_3	TBAB	93	42	8.4
3	$Na_2B_4O_7 \cdot 10H_2O$	TBAB	61	18	3.6
4	$2ZnO \cdot 3B_2O_3 \cdot 3.5H_2O$	TBAB	81	61	12.0
5	HBO_2	TBAB	84	66	13.2
6	$NH_4HB_4O_7 \cdot 3H_2O$	TBAB	47	30	6.0
7	HBO_2	-	90	-	-
8	HBO_2	TBPB	85	46	9.0
9	HBO_2	TBAI	37	14	3.0
10	HBO_2	TEAB	36	6	-
11	HBO_2	KBr	29	-	-
12	HBO_2	KI	38	-	-
13	-	TBAB	83	18	-

[a]Reaction conditions: 20 mmol of VCHO, 5 mol % of catalyst, 3 mol % of co-catalyst, reaction time 6 h, CO_2 pressure 10 bar, reaction temperature 100 °C.
[b]TBAB = Tetrabutylammonium bromide, TBPB = Tetrabutylphosphonium bromide, TBAI = Tetrabutylammonium iodide, TEAB = Tetraethylammonium bromide.
[c]The yield and conversion rate were determined by ^1H NMR with dimethyl terephthalate as an internal standard.
[d]Turnover number (TON) = mol of production /mol of catalyst.

The choice of co-catalyst significantly influences the cycloaddition reaction. A comprehensive screening of commonly used quaternary ammonium salts, potassium salts, and phosphorus salt was conducted for the cycloaddition reaction. Notably, using TBPB as a co-catalyst resulted in an 85% conversion rate for VCHO (Table 1, entry 8), comparable to the effectiveness of TBAB catalysis. However, the cyclic carbonate yield with TBPB was only 46%, and its cost is approximately five times that of TBAB. While TBAI and TBAB are well-established catalysts for the cycloaddition of CO_2 and epoxides, within the HBO_2 catalytic system, employing TBAI as a co-catalyst yielded only a 37% conversion rate with a 14% yield (Table 1, entry 9). TEAB displayed poor catalytic efficacy, exhibiting a VCHO conversion rate of only 36% and a yield of merely 6% (Table 1, entry 10). This may be attributed to the relatively short carbon chain of TEAB, leading to differing solubility compared to other co-catalysts, resulting in diminished catalytic performance. Additionally, experiments were conducted to assess the catalytic performance of KBr and KI. ^{1}H NMR analysis revealed low conversion rate of VCHO, and virtually no cyclic carbonate product was generated (Table 1, entries 11, 12). Control experiments were also performed (Table 1, entries 7, 13) with HBO_2 alone as the catalyst for the cycloaddition reaction, ^{1}H NMR indicated polymer formation. The use of TBAB alone exhibited some catalytic effect, although significantly lower than the synergistic catalytic effect of HBO_2/TBAB. Following the exploration of the catalytic systems mentioned above, it was determined that the HBO_2/TBAB catalytic system exhibited the most favourable catalytic effect. Under the specified reaction conditions (100 °C, 10 bar, 6 hours), it achieved a cyclic carbonate yield of 66% and a VCHO conversion rate of 84%. However, complete VCHO conversion was not achieved under these conditions, and the selectivity for cyclic carbonate production required improvement. Consequently, further optimization of the reaction conditions, including catalyst dosage, temperature, pressure, and reaction time, was undertaken (Table 2).

As the proportion of HBO_2 increases from 0.625 mol % to 2.5 mol %, both the conversion rate of VCHO and the yield of cyclic carbonate exhibited continuous improvements. Upon further escalation of HBO_2 to 5 mol %, the VCHO conversion rate continues to rise, while the yield of cyclic carbonate remains relatively stable. At the HBO_2 dosage of 1.25 mol %, the cyclic carbonate yield reaches 61%, accompanied by a TON value of 48.6. Consequently, 1.25 mol % of HBO_2 was selected for further exploration under alternative conditions (Table 2, entries 1-4). With an extension of the reaction time, the conversion rate of VCHO steadily increases, and the yield of cyclic carbonate exhibits continuous growth until the 6th hour, stabilizing at around 61% from the 6th to the 12th hour (Table 2, entries 2, 5-8). The influence of temperature on the reaction is apparent, with the VCHO conversion rate peaking at 140 °C, reaching 99%, and the yield at 86%. However, elevating the temperature to 160 °C results in complete VCHO conversion, but with a decreased yield of cyclic carbonate. This shift may be attributed to the higher temperature favoring polycarbonate formation (Table 2, entries 2, 9-11). It can be inferred that increasing the TBAB dosage enhances the VCHO conversion rate, but excessive TBAB (0.8 mmol) leads to a reduction in cyclic carbonate yield (Table 2, entries 10, 12-14). Pressure significantly impacts the cycloaddition reaction, with higher pressure favoring increased CO_2 concentration, thereby promoting the reaction. As pressure rises, the cyclic carbonate yield continuously increases, reaching 95% at 14 bar (Table 2, entries 10, 15, 16, 18). Entries 17, 18, and 19 in Table 2delve deeper into the effect of reaction time under the conditions of 1.25 mol % HBO_2, 3 mol % TBAB, 140 °C, and 14 bar CO_2 pressure. Remarkably, an over 99% conversion rate and a 96% yield were achieved within 5 hours of reaction time (Table 2, entry 17).

Table 2. Optimization of reaction conditions for the cycloaddition reactions[a]

Entry	Cat. (mol %)	Co-cat. (mol %)	T (°C)	P (bar)	t (h)	Conv. (%)[b]	Yield (%)[b]	TON[c]
1	HBO_2 (0.625 mol %)	TBAB (3 mol %)	100	10	6	63	47	94.8
2	HBO_2 (1.25 mol %)	TBAB (3 mol %)	100	10	6	77	61	48.6
3	HBO_2 (2.5 mol %)	TBAB (3 mol %)	100	10	6	79	66	26.4
4	HBO_2 (5 mol %)	TBAB (3 mol %)	100	10	6	84	66	13.2
5	HBO_2 (1.25 mol %)	TBAB (3 mol %)	100	10	4	54	32	25.2
6	HBO_2 (1.25 mol %)	TBAB (3 mol %)	100	10	8	73	61	48.8
7	HBO_2 (1.25 mol %)	TBAB (3 mol %)	100	10	10	74	60	48.0
8	HBO_2 (1.25 mol %)	TBAB (3 mol %)	100	10	12	76	62	49.2
9	HBO_2 (1.25 mol %)	TBAB (3 mol %)	120	10	6	85	68	54.6
10	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	10	6	>99	86	69.0
11	HBO_2 (1.25 mol %)	TBAB (3 mol %)	160	10	6	>99	83	67.2
12	HBO_2 (1.25 mol %)	TBAB (1 mol %)	140	10	6	76	59	47.4
13	HBO_2 (1.25 mol %)	TBAB (2 mol %)	140	10	6	94	82	65.4
14	HBO_2 (1.25 mol %)	TBAB (4 mol %)	140	10	6	>99	73	58.2
15	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	8	6	>99	83	66.0
16	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	6	6	>99	78	62.4
17	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	14	5	>99	96	77.0
18	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	14	6	>99	95	76.2
19	HBO_2 (1.25 mol %)	TBAB (3 mol %)	140	14	10	>99	92	74.0

[a]Reaction conditions: 20 mmol of VCHO, HBO_2, TBAB, CO_2 pressure, neat.
[b]The yield and conversion were determined by ^{1}H NMR with dimethyl terephthalate as an internal standard.
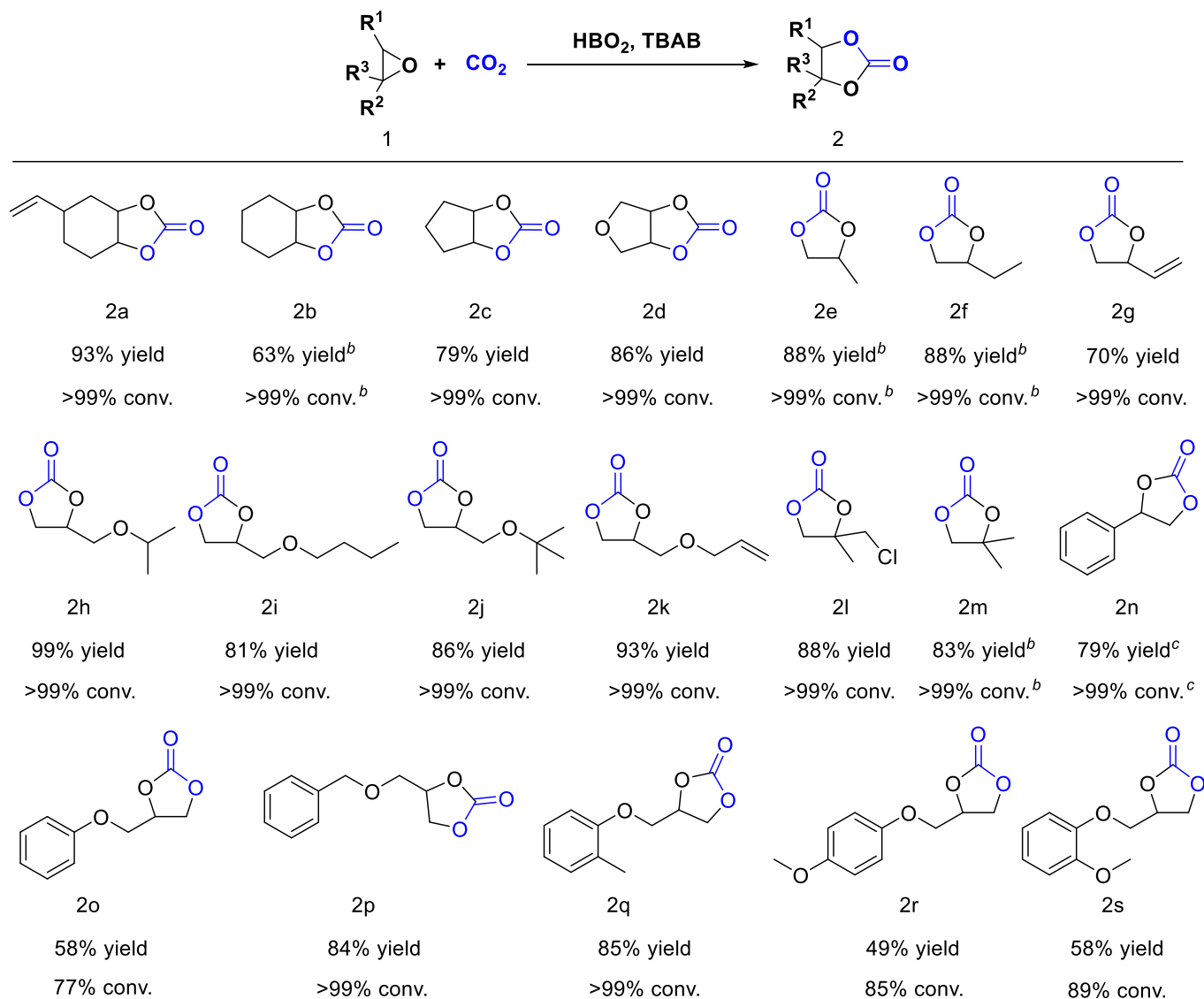[c]Turnover number (TON) = mol of production /mol of catalyst.

To confirm the broad applicability of HBO_2/TBAB in the cycloaddition reaction involving epoxy compounds and CO_2, a diverse array of epoxy compounds featuring distinct functional groups was investigated under the optimized conditions (Table 3).

For internal epoxides **1b**-**1d**, HBO_2/TBAB demonstrated good catalytic efficacy. Under optimized conditions, all internal epoxides achieved complete conversion, with a yield of 79% for **2c** and 86% for **2d**. However, compound **2b** exhibited a lower yield of 50% under the optimal conditions. Through ^{1}H NMR characterization, it was observed that cyclohexene oxide had completely converted, indicating poor selectivity under these conditions. Consequently, we reduced the catalyst dosage and observed a gratifying increase in the yield of **2b** to 63%. Similar substrates, such as **1e**, **1f**, and **1m**, which also exhibited poor selectivity, demonstrated significantly increased yields when the catalyst dosage was halved. Reactions of terminal epoxides without a benzene ring were investigated. The system displayed robust catalytic ability for terminal epoxides. Substrates **1h** and **1k** exhibited excellent yields, surpassing 90% and other terminal compounds (except for **1g**) achieved yields above 80%. Terminal epoxides were nearly completely converted under the given conditions, and for simple epoxides, lower catalyst dosage is used to enhance yields (**1e** and **1f**). Six

substrates (**1n-1s**) with benzene rings were also tested, and under the given conditions, the epoxides **1p** and **1q** achieved complete conversion, and gave corresponding cyclic carbonate in 84%, and 85% yield, respectively. **1n** exhibited a yield of 79% at 10 bar but epoxides **1o**, **1r**, and **1s** did not undergo complete conversion under these conditions. Overall, the system demonstrated robust catalytic effects for both terminal and internal epoxides under low catalyst dosage.

Table 3. Synthesis of cyclic carbonates from epoxides and CO_2[a]


2a	2b	2c	2d	2e	2f	2g
93% yield	63% yield[b]	79% yield	86% yield	88% yield[b]	88% yield[b]	70% yield
>99% conv.	>99% conv.[b]	>99% conv.	>99% conv.	>99% conv.[b]	>99% conv.[b]	>99% conv.
2h	2i	2j	2k	2l	2m	2n
99% yield	81% yield	86% yield	93% yield	88% yield	83% yield[b]	79% yield[c]
>99% conv.	>99% conv.	>99% conv.	>99% conv.	>99% conv.	>99% conv.[b]	>99% conv.[c]
2o	2p	2q	2r	2s		
58% yield	84% yield	85% yield	49% yield	58% yield		
77% conv.	>99% conv.	>99% conv.	85% conv.	89% conv.		

[a]Unless otherwise specified, reaction conditions: HBO_2 (1.25 mol %), TBAB (3 mol %), 140 °C, 14 bar, 5 h. [b]HBO_2 (0.625 mol %), TBAB (1.5 mol %), [c]10 bar. The yield is the isolated yield after silica gel column chromatography.

Based on previous reports,[36] a possible mechanism for the cycloaddition reaction of CO_2 and epoxides catalyzed by HBO_2 is proposed (Scheme 1). Boric acid reacts with CO_2 to form a complex and boron act as a Lewis acid to form a B-O coordination bond with the oxygen atom on the epoxy ring (step 1). This step activates the epoxy ring, making it easier for the next step. TBAB nucleophilic attack at the epoxy oxide causes ring opening (step 2), resulting in the formation of a metaborate intermediate. Finally, after nucleophilic attack

and rearrangement to carboxyl and further substitution of bromine, the intermediate undergoes cyclization (step 3 and step 4) to generate cyclic carbonate products and release HBO_2.
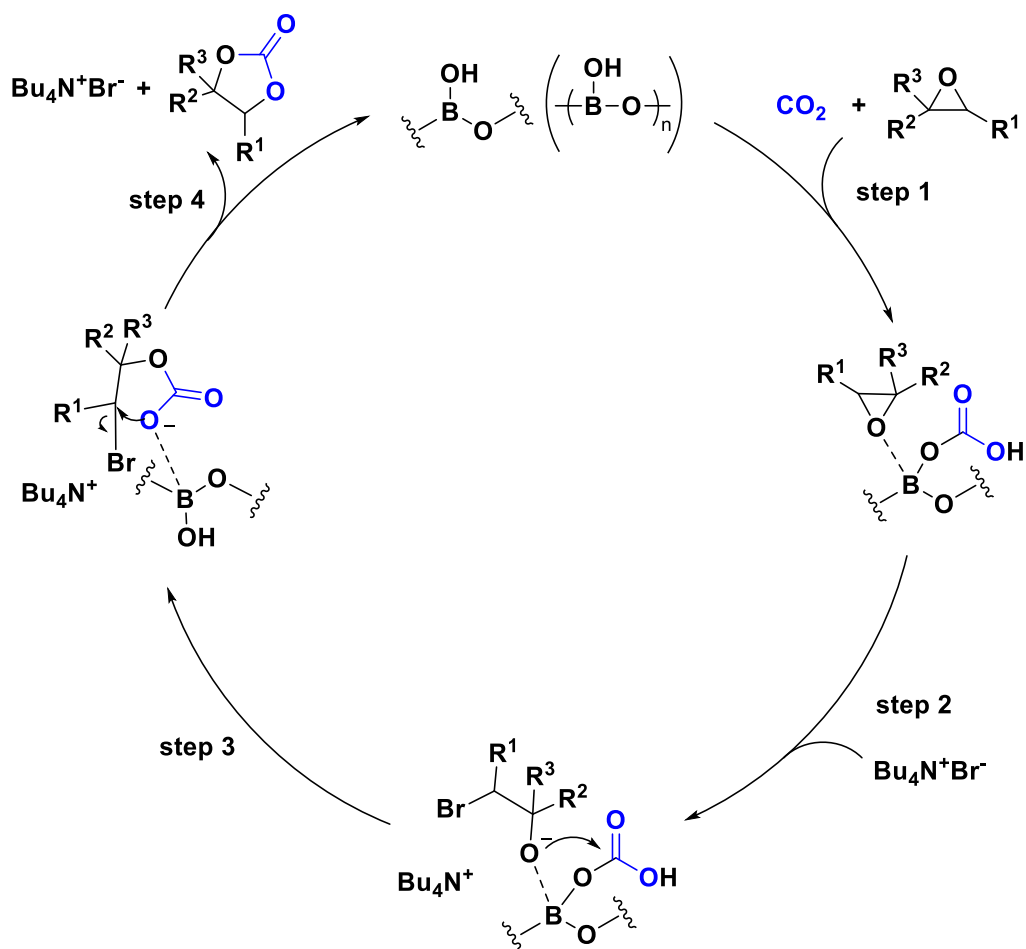
Scheme 1. Proposed mechanisms of HBO_2/TBAB-catalyzed cycloaddition reaction of CO_2 and epoxides.

Conclusions

In conclusion, the binary catalytic system of HBO_2/TBAB developed effects efficient catalysis of cycloaddition of epoxides and carbon dioxide. This catalytic system is cost-effective, environmentally friendly, and highly active. It can catalyze the addition to various terminal epoxides, as well as sterically hindered internal epoxides, with low catalyst dosage of HBO_2 (1.25 mol %) and TBAB (3 mol %). This catalytic system exhibits good to excellent catalytic performance for epoxides with different functional groups. For epoxides with low reactivity, this study provides a novel efficient catalytic system.

Experimental Section

General. Unless otherwise specified, all reagents and solvents are purchased from suppliers and used directly without further purification. Column chromatography was performed on silica gel (200-300 mesh) using

petroleum ether/ethyl acetate as eluent. All ^{1}H NMR (400 MHz or 500MHz), ^{13}C NMR (101 MHz or 126 MHz) were recorded on Bruker AVANCE II-400 or Bruker AVANCE III-500 spectrometers (in CDCl$_3$ with TMS as internal standard). Melting points were recorded on a Novel X-4 spectrometer. Infrared spectroscopies were recorded on Nicolet 6700 Fourier transform infrared spectrometer. Mass spectroscopies were recorded on the LTQ Orbitrap XL mass spectrometer.

General synthesis procedure. The reaction was carried out in a 25 ml high-pressure reactor. VCHO (20 mmol), HBO$_2$ (0.25 mmol), and TBAB (0.6 mmol) were added to the reactor at room temperature. The reactor was purged with argon gas three times, followed by two purges with carbon dioxide. Then, the reactor was pressurized to 14 bar and heated in an oil bath at 140 °C for 5 hours. After the completion of the reaction, the reactor was cooled to room temperature followed by gradual release of the pressure inside the reactor. The cyclic carbonate product was separated and purified by silica gel column chromatography. The structure of the product was determined by ^{1}H NMR, ^{13}C NMR, IR and MS spectroscopy.

The cyclic carbonate **2a** is known compound, and the characterization data is consistent with previous report.

5-Vinylhexahydrobenzo[d][1,3]dioxol-2-one (2a).[37] The ratio of two diastereoisomers is approximately 1:1, 93% yield, colorless oil, ^{1}H NMR (500 MHz, CDCl$_3$) δ 5.73 – 5.56 (m, 2H), 5.02 – 4.82 (m, 4H), 4.76 – 4.52 (m, 4H), 2.30 – 1.99 (m, 5H), 1.96 – 1.91 (m, 1H), 1.76 – 1.46 (m, 5H), 1.24-1.15 (m, 2H), 1.12– 1.04 (m, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 155.1, 155.1, 141.2, 141.1, 114.0, 113.7, 76.0, 75.7, 75.6, 75.1, 36.1, 33.8, 33.4, 31.4, 26.6, 25.6, 25.5, 24.9. MS (ESI) calculated for C$_9$H$_{13}$O$_3$ [M+H]$^+$: 169.09; found: 168.96. IR (KBr, cm^{-1}) v = 3081, 2943, 2867, 1796, 1641, 1357, 1191, 1147, 1032, 918, 782, 732.

Acknowledgements

Supplementary Material

Full experimental detail, ^{1}H and ^{13}C NMR spectra. This material can be found via the "Supplementary information" section of this article's webpage.

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