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A High Loading and Recyclable Pentaerythritol Supported Imidazolidin-4-one Catalyst for Enantioselective Diels–Alder Reactions

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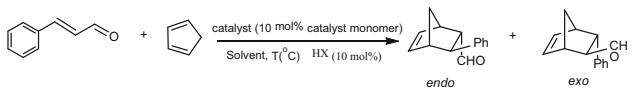
Abstract The synthesis of high loading and recyclable pentaerythritol supported imidazolidin-4-one catalyst I and its application in enantioselective Diels–Alder reactions of cyclopentadiene and α,β-unsaturated aldehydes with high performance were described. Especially noteworthy, the pentaerythritol supported imidazolidin-4-one with high loading capacity can be recovered by simple precipitation and filtration, and recycled for up to four runs without observing significant decrease in catalytic activity.

Graphical Abstract



Keywords High loading · Recyclable · Pentaerythritol · Imidazolidin-4-one · Diels–Alder reactions

1 Inroduction

During the past decades, much interest has been devoted to the development of efficient organocatalysts for asymmetric organic transformations [1–4]. Variety of organocatalysts have been explored and applied in different organic process, providing efficient access to valuable chiral compounds.

Among them, chiral imidazolidin-4-one, designed and developed by Macmillan's group [5–9], is one of the most efficient chiral organocatalysts for its high efficiency and selectivity in asymmetric reactions. However, its practical use in reactions is still hindered due to the need for high catalyst loading and difficulties in recovering the catalyst from the reaction mixture. One obvious way to overcome these problems is to immobilizing or attaching the catalyst unit on a recyclable support [10–31], such as PEG [11], dendrimer [12, 13], siliceous and polymer-coated mesocellular foam (MCF) [14], fluorous tag [15], poly(-methyl-hydrosiloxane) (PHMS) [16], ionic liquid [17–20].

In our ongoing research project on the development of recyclable catalyst, recently we have developed tetraarylphosphonium supported imidazolidin-4-one and applied it in asymmetric Diels–Alder reactions [28] and 1,3-dipolar cycloaddition [29]. Tetraarylphosphonium salt used as a support successfully overcomes the recycling issue, but its loading capacity and enantioselectivity still remains less satisfied.

Hence, we introduce pentaerythritol as a new support to immobilize chiral imidazolidin-4-one. Pentaerythritol is a well known, cheap, atoxic, widely used chemical raw materials, and it is considered to be ideal building blocks in

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many areas of materials chemistry [32–34] and in the synthesis of dendrimers [35–37]. Because of its low molecular weight and its special molecular structure, the loading capacity of pentaerythritol is higher than many other kind of supports such as traditional polymer support [10–12, 14, 16, 22], ionic liquid [17–20] and tetraarylphosphonium salt [28, 29]. Owing to its dendrimer-like structure, we believe that pentaerythritol used as a support not only can solve the problem of recovering which meet the requirement of modern green chemistry for sustainability, but also may enhance the catalytic performance like traditional dendrimer supports do, even without generation growing. In addition, chiral-functionalized unit immobilized on pentaerythritol for asymmetric organocatalysis is still absent in the literature.

Described in this paper is the high loading and recyclable pentaerythritol supported chiral imidazolidin-4-ones organocatalyst for the enantioselective Diels–Alder reactions, which is wished to run the catalyzed reaction under homogeneous conditions with excellent yields and high enantioselectivities and recover catalysts by the simple precipitation method.

2 Experimental

2.1 General

Reactions were monitored by TLC using precoated plates of silica gel HF_{254} (0.5 mm, Yantai, China). Column chromatography was performed with a silica gel column (200–300 mesh, Yantai, China). NMR spectra were recorded on WIPM 400 spectrometer (^1H at 400 MHz, ^{13}C at 100 MHz). IR spectra were recorded on an IR-spectrum one (PE) spectrometer. High-resolution mass spectra (HRMS) were recorded on Agilent 1260–6224 LC–MS TOF using ESI (electrospray ionization). Enantioselectivity were determined by HPLC (Dionex UltiMate 3000) analysis employing a Daicel ChiralCel OD-H, ChiralPak AS-H columns and Chiralcel OJ-RH. A UV detector (UVD-3000) was used for the peak detection.

2.2 General Procedure for Synthesis of Pentaerythritol Supported Chiral Imidazolidin-4-Ones I–II

To a solution of compound **3** (1.0 g, 1.3 mmol) or compound **4** (1.0 g, 0.87 mmol) in dry DMF (10 mL) was added imidazolidin-4-one **1** (1.6 g, 6.8 mmol) and Cs_2CO_3 (6.1 g, 19.5 mmol) at room temperature. After stirring at 80 °C for 30 h, the mixture was evaporated under vacuum, then the residue was dissolved in the minimum amount of CH_2Cl_2 (2 mL) and slowly poured into Et_2O (30 mL). The precipitate was filtered on Celite, washed with 10 mL of Et_2O and dried under vacuum to obtain **I** or **II** as a white solid. Catalyst **I** (1.12 g, 85 %); Catalyst **II** (1.0 g, 74 %).

2.3 Synthesis of Pentaerythritol Supported Chiral Imidazolidin-4-One III

Compound **5** (0.2 g, 0.85 mmol), compound **2** (1.85 g, 6.8 mmol) and dry THF (10 mL) were charged to a 25 mL three-neck round-bottomed flask equipped with a magnetic stirrer. The system was degassed and purged with nitrogen, then CuBr (24 mg, 0.17 mmol) and N,N,N′,N″,N‴-pentamethyldiethylenetriamine (PMDETA) (35 μL, 0.17 mmol) were added. The reaction mixture was stirred at room temperature for 20 h and dropped into dilute HCl (1 wt %, 20 mL). The resultant mixture was extracted with CH_2Cl_2 (20 mL × 3), dried over Na_2SO_4, and concentrated under vacuum to about 2 mL, and slowly poured into Et_2O (30 mL). The precipitate was filtered on Celite, washed with 10 mL of Et_2O and dried under vacuum to obtain catalyst **III** as a white solid (0.96 g, 86 %).

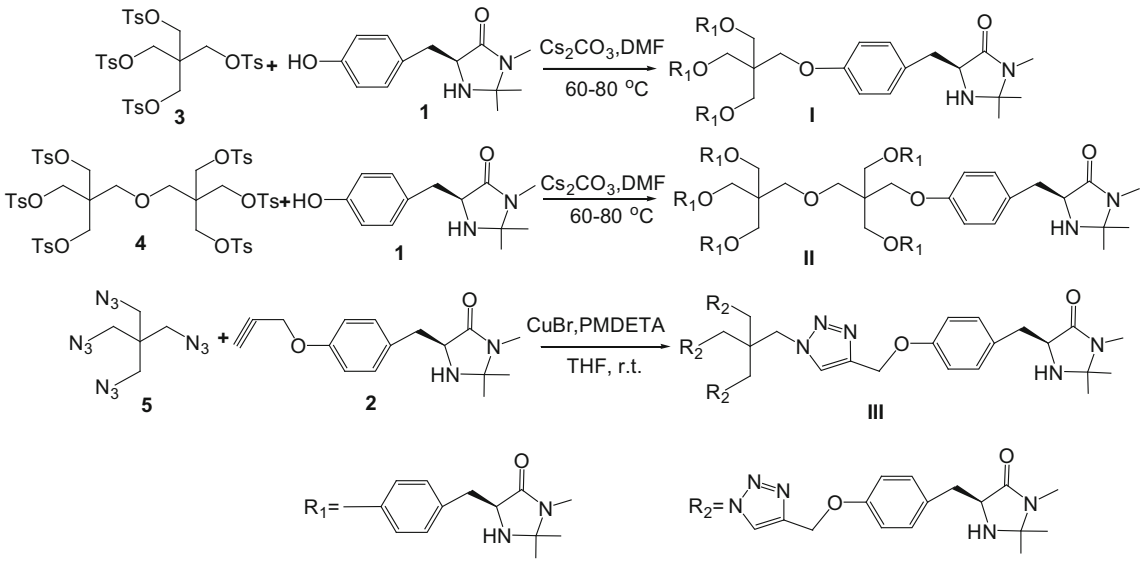
2.4 General Procedure for the Diels–Alder Reaction

To a solution of catalyst **I** (50 mg, 0.05 mmol, 0.2 mmol catalyst monomer) in CH_3CN/H_2O (1 mL, 95:5, v/v), 0.4 M $HClO_4$ (0.5 mL, 0.2 mmol) was added, and the mixture was stirred for 5 min at 25 °C. Freshly distilled cinnamaldehyde (0.13 mL, 1.0 mmol) and cyclopentadiene (0.33 mL, 4.0 mmol) were added respectively, and the resulting mixture was stirred at 25 °C for 12–48 h. $MgSO_4$ was then added, the mixture was filtered, and the organic solvent evaporated under vacuum. The residue was dissolved in the minimum amount of CH_2Cl_2 (2 mL) and then poured into Et_2O (30 mL). The precipitate was filtered, washed with Et_2O (10 mL), dried under vacuum and reused in further reactions as the recycled catalyst. The filtrate was concentrated under vacuum and purified by silica gel column chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product. The *endo*/*exo* ratio was determined by ^1H NMR spectroscopy of the crude mixture and the enantiomeric excess (ee) of the adduct was determined by HPLC on a chiral phase according to the established procedure [28]. All the products are known compounds and all the spectroscopic data matched those reported in the literature [5, 41].

3 Results and Discussion

3.1 Preparation of Catalysts I–III

The preparation of pentaerythritol supported chiral imidazolidin-4-ones **I–III** is shown in Scheme 1. Chiral



Scheme 1 Preparation of pentaerythritol supported chiral imidazolidin-4-ones **I–III**

imidazolidin-4-ones **1–2** [28, 40] and pentaerythritol derivatives **3–5** [38, 39] were prepared by the established procedure according to the corresponding literatures, respectively. Catalysts **I–II** can be obtained with good yield by the reaction between **3** or **4** with **1** in dry DMF in the presence of Cs_2CO_3. Then a CuAAC reaction between **5** with **2** was performed to give catalyst **III** in good yield. The loadings of pentaerythritol supported catalysts **I–III** are 4.00 mmol/g for catalyst **I**, 3.87 mmol/g for catalyst **II** and 3.02 mmol/g for catalyst **III**.

Catalysts **I–III** are soluble in polar solvents (CH_2Cl_2, CH_3CN, CH_3OH) and insoluble in less polar solvents (Et_2O, hexane, toluene), thus the pentaerythritol supported catalysts can be run the catalyzed reaction under homogeneous condition in polar solvent and readily isolated by precipitation from less polar solvents.

3.2 Catalytic Activities of Catalysts I–III in Diels–Alder Reactions

The catalytic capacity of the prepared pentaerythritol supported catalysts **I–III** was examined in the Diels–Alder reaction between cinnamaldehyde and cyclopentadiene. And in order to find an optimal conditions for the Diels–Alder reaction, we first explored the catalytic activities of the catalyst **I** (10 mol %, based on the amount of catalysis monomer) by using HCl (10 mol %) as reaction cocatalyst.

The solvent used in the reaction is often important, so we investigated the Diels–Alder reaction in different solvent system at 25 °C for 24 h (Table 1, entries 1–7). All of the mixed solvent system, the volume ratio of the solvent and water is 95/5. The use of CH_3NO_2/H_2O, CH_3CN/H_2O, $MeOH/H_2O$ and single H_2O gave the desired product in low to moderate yield and good enantioselectivity, while only trace amount of product was observed in the solvent system of THF/H_2O, CH_2Cl_2/H_2O and 1,4-dioxane/H_2O. Among the solvent system examined, CH_3CN/H_2O provided the best result (Entry 7, 73 % yield, 55/45 exo/endo, endo 99 % ee, exo 89 % ee). Therefore, CH_3CN/H_2O was chosen as the solvent to further optimize the reaction conditions by screening different acids as reaction co-catalysts.

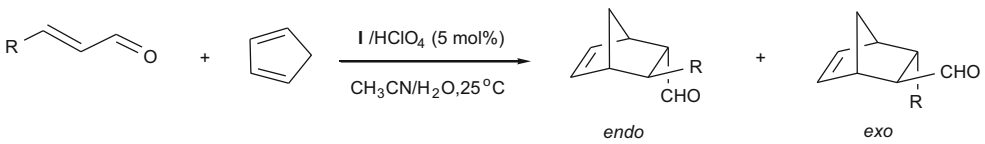
As illustrated in Table 1 (Entries 7–14), eight types of protic acids were screened as the reaction co-catalyst. The stereoselectivities are comparative with eight **I**/HX combinations, but the chemical yields are very different, with **I**/$HClO_4$ securing best chemical yield (Entry 14, 79 % yield). In the further exploration we chose $HClO_4$ as reaction co-catalysts.

The influence of the catalyst amount (based on the amount of catalysis monomer) in the Diels–Alder reaction was also examined (Table 1, entries 14–16). Decreasing the catalyst amount to 5 mol % preserved the stereoselectivity, but reduced the chemical yield (79 → 62 % yield). Increasing the catalyst amount to 20 mol %, the reaction preformed the best result (Entry 16, 89 % yield, 53/47 *exo*/*endo*, *endo* 99 % ee, *exo* 92 % ee).

Next, the effects of reaction temperature in the Diels–Alder reaction was investigated (Table 1, entries 16–21). Among the temperature examined, 25 °C provided the best yield and comparative stereoselectivity, and it is the best control temperature, so 25 °C is the best candidate for the Diels–Alder reaction.

Furthermore, the optimal reaction time of the Diels–Alder reaction under the condition of CH_3CN/H_2O as solvent, $HClO_4$ as cocatalyst, 25 °C and 5 mol % catalyst

Table 1 Optimization of Diels–Alder reactions between cinnamaldehyde and cyclopentadiene



Entry	Catalyst	Solvent [a]	HX	T(°C)	Time (h)	Yield (%)[b]	*exo*/*endo* [c]	ee (%)[d]	
								endo	*exo*
1	**I**	CH_2Cl_2/H_2O	HCl	25	24	trace	–	–	–
2	**I**	THF/H_2O	HCl	25	24	trace	–	–	–
3	**I**	1,4-dioxane/H_2O	HCl	25	24	trace	–	–	–
4	**I**	CH_3NO_2/H_2O	HCl	25	24	65	55:45	99	86
5	**I**	$MeOH/H_2O$	HCl	25	24	37	54:46	99	89
6	**I**	H_2O	HCl	25	24	36	55:45	99	87
7	**I**	CH_3CN/H_2O	HCl	25	24	73	55:45	99	
8	**I**	CH_3CN/H_2O	TFA	25	24	70	53:47	99	90
9	**I**	CH_3CN/H_2O	TfOH	25	24	64	55:45	97	92
10	**I**	CH_3CN/H_2O	p-TSA	25	24	40	58:42	99	93
11	**I**	CH_3CN/H_2O	Cl_3CCOOH	25	24	32	53:47	97	76
12	**I**	CH_3CN/H_2O	HBF_4	25	24	72	53:47	99	91
13	**I**	CH_3CN/H_2O	HPF_6	25	24	76	56:44	99	87
14	**I**	CH_3CN/H_2O	$HClO_4$	25	24	79	52:48	99	90
15	**I**	CH_3CN/H_2O [e]	$HClO_4$	25	24	62	54:46	98	91
16	**I**	CH_3CN/H_2O [f]	$HClO_4$	25	24	89	53:47	99	92
17	**I**	CH_3CN/H_2O	$HClO_4$	-20	24	40	52:48	99	95
18	**I**	CH_3CN/H_2O	$HClO_4$	0	24	63	52:48	99	95
19	**I**	CH_3CN/H_2O	$HClO_4$	10	24	75	54:46	99	94
20	**I**	CH_3CN/H_2O	$HClO_4$	15	24	78	52:48	99	92
21	**I**	CH_3CN/H_2O	$HClO_4$	20	24	80	52:48	99	92
22	**I**	CH_3CN/H_2O	$HClO_4$	25	12	52	56:44	99	92
23	**I**	CH_3CN/H_2O	$HClO_4$	25	48	90	54:46	99	90
24/*	**II**	CH_3CN/H_2O	$HClO_4$	25	24	78	54:46	99	90
25	**III**	CH_3CN/H_2O	$HClO_4$	25	24	81	54:46	99	90
26	**IV**	CH_3CN/H_2O	$HClO_4$	25	24	90	53:47	93	92
27	**I**	CH_3CN/H_2O [g]	$HClO_4$	25	24	85	52:48	99	91
28	**I**	CH_3CN/H_2O [h]	$HClO_4$	25	24	82	54:46	99	91
29	**I**	CH_3CN/H_2O [i]	$HClO_4$	25	24	75	52:48	99	91

[a] The volume ratio of the solvent and water in the mixed solvent system is 95/5. [b] Isolated yield of a mixture of *endo* and *exo* isomers. [c] *Exo*/*endo* ratios were determined by ^{1}H NMR analysis. [d] ee values were determined by HPLC of alcohol after reduction of the formyl group. [e] 5 mol % catalyst monomer. [f] 20 mol % catalyst monomer. [g] 2nd run. [h] 3rd run. [i] 4th run

was examined (Table 1, entry 16, and entries 22–23). Reducing reaction time to 12 h led to a decrease in the yield (89 → 52 % yield). Increasing the reaction time to 48 h, the yield was slightly increased to 90 %, while the stereoselectivity is nearly unaltered or lower, so 24 h is enough for the reaction.

With the optimized reaction conditions in hand, pentaerythritol supported chiral imidazolidin-4-ones **II** and **III** were evaluated for the Diels–Alder reaction to compare with the catalyst **I** (Table 1, entry 24–25 vs entry16). The enantioselectivities and chemical yields by the use of the catalysts **II** and **III** were lower than that using catalyst **I**. It seems that catalyst **II** with more branches has crowded surfaces, so the catalytically active imidazolidin-4-ones moieties are not freely accessible for enantioselective reactions, it being likely less available to the reaction substrate. Hence, the loss of enantioselectivity and chemical yield with catalyst **II** might be ascribed to steric

hindrance between the imidazolidin-4-ones moieties at the periphery. Catalyst **III** with a 1.2.3-triazole linker, which is designed as a spacer to increase spatial separation between catalyst moieties and as a block to increase the space shielding of the catalyst moieties, provides lower yield and enantioselectivity than catalyst **I**. These results demonstrate that the improvement in yield is ascribed to the different proximity of imidazolidin-4-ones moieties in the terminal units, while space shielding provided by 1.2.3-triazole has less impact on the enantioselectivity.

For further investigation of the impact of pentaerythritol structure on catalytic activity, the asymmetric Diels–Alder reaction of cinnamaldehyde and cyclopentadiene with methoxylated Macmillian catalyst **IV** (Fig. 1) under our optimized conditions was also examined (Table 1, entry 26). It was found that the chemical yield and the ee of *exo* was comparative, but pentaerythritol supported catalyst **I** exhibited a higher ee of *endo* than methoxylated Macmillan catalyst **IV**. We deduce branched structure of pentaerythritol had some effect on enantioselectivity but had little impact on reaction conversion.

To compare the catalytic activity of pentaerythritol supported catalyst **I** with our previously reported tetraarylphosphonium supported imidazolidin-4-one [28, 29], it was found that catalyst **I** exhibited the comparative yield and the higher enantioselectivity either for *endo* isomer or *exo* isomer. Especially noteworthy, catalyst **I** exhibited the better capacity for stimulating a slightly higher enantioselectivity for e*xo* isomer as compared to most of the supported imidazolidin-4-one catalysts [15–18, 21–23, 28, 30, 31]. A rationale of this finding may be found in the characteristics of the pentaerythritol employed.

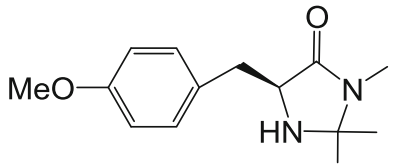


Fig. 1 Methoxylated Macmillan catalyst **IV**

3.3 Recycling and Reuse of Catalyst I

Catalyst recycling and reuse are important issues, so the recovery and reusability of catalyst **I** were investigated. After a reaction was completed, catalyst **I** which was collected by precipitation, filtration and dried under vacuum, was then used for the next reaction. Average recovery of the catalyst ranged from 75 to 85 %. The recovered catalysts were examined by ^{1}H NMR spectroscopy, which showed unchanged after each recovery. We have performed four recycling experiments, and the results are compiled in Table 1 (Entries 16, and 27–29). The data collected in Table 1 indicates that the stereoselectivity of the recovered catalyst remained almost intact for at least four reaction cycles, while the chemical efficiency of the recovered catalyst slowly eroded upon its iterative reuse.

3.4 Scope of Catalyst I

Experiments that probe the scope of Diels–Alder reactions catalyzed by pentaerythritol supported chiral imidazolidin-4-ones **I** are summarized in Table 2. Variation of the olefin substituent (R = Me, *n*-Pr, Entries 1 and 2) is possible with higher yield or enantioselectivity (> 92 % yield, *endo* > 92 % ee, *exo* > 90 % ee) and shorter reaction time. The reaction is also tolerant of aromatic groups on the dienophile component (Entries 3–5, > 82 % yield, *endo* > 92 % ee, *exo* > 90 % ee). Thus it can be seen that pentaerythritol supported chiral imidazolidin-4-one **I** is an efficient organocatalyst for Diels–Alder reactions.

4 Conclusions

In conclusion, a high loading and recyclable pentaerythritol supported chiral imidazolidin-4-one was developed through the simple synthesis route, and which was applied

Table 2 Scope of catalyst **I** in the Diels–Alder reactions

Entry	R	Time (h)	Yield (%)[a]	*exo*/*endo*[b]	*endo* ee (%)[c]	*exo* ee (%)[c]
1	Me	12	96	35:65	95	93
2	*n*-Pr	12	92	39:61	92	90
3	4-MeOPh	24	82	52:48	92	98
4	4-NO_2Ph	24	80	66:34	92	93
5	Ph	24	89	53:47	99	92

[a] Isolated yield of a mixture of *endo* and *exo* isomers. [b] *Exo*/*endo* ratios were determined by ^{1} H NMR analysis. [c] ee values in entries 1–2 were determined by HPLC after converted into the corresponding N,N-diphenylhydrazone, while ee values in entries 3–5 were determined by HPLC of alcohol after reduction of the formyl group

in enantioselective Diels–Alder reactions of cyclopentadiene and α,β-unsaturated aldehydes providing the desired products in good yields with excellent enantioselectivities. It is worth mentioning that the pentaerythritol supported imidazolidin-4-one catalyst, whose high loading capacity is comparable with the original Macmillan's catalyst, can be readily recovered from the reaction mixture by simple precipitation and filtration. The enantioselectivity of the recovered catalyst remained almost intact for at least four reaction cycles, while the chemical efficiency of the recovered catalyst slowly eroded upon its iterative reuse.

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