



Short communication

Ytterbium perfluorooctanesulfonate as an efficient and recoverable catalyst for the synthesis of trisubstituted imidazoles

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ABSTRACT

Synthesis of trisubstituted imidazoles was successfully accomplished using rare earth(III) perfluorooctanesulfonates ($\text{RE}(\text{OPf}_3)_3$), $\text{RE} = \text{Sc}, \text{Y}, \text{La-Lu}$ as catalysts in fluorous solvents. Ytterbium perfluorooctanesulfonates ($\text{Yb}(\text{OPf}_3)_3$) catalyze the high efficient synthesis of trisubstituted imidazoles in fluorous solvents. By simple separation, fluorous phase containing only catalyst can be reused several times.

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1. Introduction

The synthesis, reactions and biological properties of substituted imidazole constitute a significant part of modern heterocyclic chemistry. Multi-substituted imidazoles, an important class of pharmaceutical compounds, exhibit a wide spectrum of biological activity [1].

There are numerous classical methods for the synthesis of multi-substituted imidazoles [2]. In these methods, the one-pot reaction of diketones, aldehydes and ammonium acetate is a typical procedure which was widely researched. The typical procedure is condensed in the presence of H_3PO_4 [3], H_2SO_4 [4], HOAc [5], DMSO [6] as well as organocatalyst in HOAc [7]. However, all of these methods have problems, including drastic reaction conditions, low yields and severe side-reactions. Moreover, DMF and DMSO lead to complex isolation and recovery procedure. Several methods comprise the use of MW (microwave)/silica gel [8], MW/silica gel/H-Y [9], $\text{MW/Al}_2\text{O}_3$ [10], MW/ acetic acid [11], in DMF [12]. These methods promote the condensation efficiently. But, the use of high temperature, expensive instruments like microwave and corrosive reagents limiting these methods. Kidwai et al. [13] reported that I_2 can catalyze the typical procedure in good to excellent yield. However, this catalyst cannot

be reused. Wang et al. [14] reported that ytterbium triflate ($\text{Yb}(\text{OTf})_3$) can catalyze the condensation reaction successfully, the catalyst can be recovered conveniently and reused for at least three reaction cycles without any loss of activity.

There has been rapidly increasing interest in the design and synthesis of compounds that exhibit high affinities for “fluorous” phases since the technique of “fluorous biphasic system” (FBS) was described by Horváth and Rábai [15,16]. The technique of FBS, as a phase-separation and catalyst immobilization technique, has become one of the most important methods for facile catalyst separation from the reaction mixture and recycling of the catalyst [16]. In this catalytic system, the metal catalyst coordinated by perfluoroalkylated ligands can dissolve into the fluorous phase containing the product after the reaction. Recently, novel Lewis acids of lanthanide tris(perfluorooctanesulfonyl)methide $\{\text{Ln}[\text{C}(\text{SO}_2\text{R}_{\text{f8}})_3]_3$, $\text{R}_{\text{f8}} = (\text{CF}_2)_7\text{CF}_3$, $\text{Ln}(\text{CPf}_3)_3$ [17], lanthanide bis(-perfluorooctanesulfonyl)amide $\{\text{Ln}[\text{N}(\text{SO}_2\text{R}_{\text{f8}})_2]_3$, $\text{Ln}(\text{NPF}_2)_3$ [18] and lanthanide perfluorooctanesulfonate $[\text{Ln}(\text{OSO}_2\text{R}_{\text{f8}})_3]$, $\text{Ln}(\text{OPf}_3)_3$ [19,20] have been of special interest in that they have characteristic features such as low hygroscopicity, ease of handling, robustness for the reuse and high solubility in fluorous solvent.

During our studies to explore the utility of transition metal perfluorooctanesulfonates catalyzed reactions in fluorous solvents [20], we found that the one-pot condensation of aldehyde, benzil and NH_4OAc can proceed smoothly in the presence of ytterbium perfluorooctanesulfonate $[\text{Yb}(\text{OPf}_3)_3]$ in a FBS composed of HOAc and perfluorodecalin. Concurrently, we also found that this new

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process can be carried out successfully with no need of a stoichiometric amount Lewis base and the robustness of the catalytic system for reuse can be obtained by simple phase-separation.

2. Experimental

2.1. General

Chemicals used were obtained from commercial suppliers and used without further purifications. Melting points were determined on a Shimadzu DSC-50 thermal analyzer. IR spectra were recorded on a Bomem MB154S infrared analyzer. ^1H NMR and ^{19}F NMR spectra were recorded with a Bruker Advance RX500 spectrometer. Mass spectra were recorded on a Saturn 2000GC/MS instrument or an Agilent 1100LC/MS instrument. Inductively coupled plasma (ICP) spectra were measured on an Ultima2C apparatus. Elemental analyses were performed on a Yanagimoto MT3CHN recorder.

2.2. Typical procedure for preparation of $\text{RE}(\text{OPf})_3$

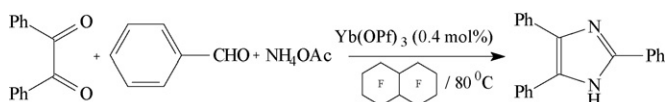
$\text{RE}(\text{OPf})_3$ was prepared according to the literatures [21]. Method A: the mixture of PFOH solution (aq) and $\text{YbCl}_3 \cdot 6\text{H}_2\text{O}$ solution (aq) was stirred at room temperature. Method B: the mixture of PFOH solution (aq) and Yb_2O_3 powder was stirred at boiling temperature. In both methods, the resulting gelatin-like solid was collected, washed with H_2O and dried at 150°C in vacuum to give a white solid, which does not have a clear melting point up to 500°C , but shrinks around 380°C and 450°C . IR (KBr) ν 1230 (CF_3), 1150 (CF_2), 1080 (SO_2), 1060 (SO_2), 740 (S–O) and 650 (C–S) cm^{-1} . ICP: Calcd for $\text{C}_{24}\text{O}_9\text{F}_{51}\text{SYb}$: Yb, 10.30%. Found: Yb, 9.88%. Anal. Calcd for $\text{C}_{24}\text{O}_9\text{F}_{51}\text{S}_3\text{Yb} \cdot \text{H}_2\text{O}$: C, 17.21%; H, 0.10%. Found: C, 17.03%; H, 0.18%.

2.3. Typical procedure for preparation of trisubstituted imidazoles in FBS

A mixture of $\text{Yb}(\text{OPf})_3$ (6.7 mg, 0.004 mmol), benzil (0.21 g, 1 mmol), benzaldehyde (0.212 g, 2 mmol) and NH_4OAc (0.77 g, 10 mmol) was added to HOAc (2 mL) and perfluorodecalin ($\text{C}_{10}\text{F}_{18}$, *cis* and *trans*-mixture, 1.5 mL). The mixture was stirred at 80°C for 6 h. After the reaction was finished, the reaction mixture was filtrated. The filtrate was cooled to room temperature, the fluoros layer on the bottom was separated for the next condensation, the solid obtained from the filtration and the combined organic phase was extracted with ether (10 mL \times 2), washed with NaHCO_3 solution aqueous (5% 10 mL), and water (10 mL), and then dried over Na_2SO_4 . The organic solvent was removed under reduced pressure and the residue was purified by SiO_2 gel column chromatography using CH_2Cl_2 :MeOH 99:1 as eluent to give a product 2,4,5-triphenylimidazole (0.287 g, 97%).

3. Results and discussion

Firstly, in the condensation, NH_4OAc was selected to be the ammonia source in the model reaction (Scheme 1). In a model reaction, in the presence of the catalyst, the mixture of benzaldehyde, benzil and NH_4OAc was stirred in HOAc and perfluorodecalin at 80°C . The corresponding product was obtained



Scheme 1. The model condensation reaction of benzaldehyde, benzil and NH_4OAc .

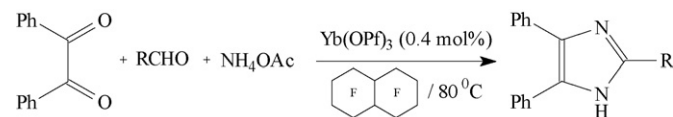
Table 1

Condensation reaction of benzaldehyde, benzil and NH_4OAc in different cosolvents^a

Entry	Cosolvent	Yield ^b (%)
1	HOAc	97
2	$\text{C}_2\text{H}_5\text{OH}$	91
3	Toluene	43
4	Dioxane	90
5	H_2O	3
6	$\text{CH}_3\text{COOC}_2\text{H}_5$	88

^a The reaction condition: benzaldehyde, 2 mmol; benzil, 1 mmol; NH_4OAc , 10 mmol; $\text{Yb}(\text{OPf})_3$, 0.004 mmol; solvent, 2 mL; perfluorodecalin ($\text{C}_{10}\text{F}_{18}$, *cis* and *trans*-mixture), 1.5 mL; 80°C ; 6 h.

^b Isolated yields.



Scheme 2. The condensations of various aldehydes with benzil and NH_4OAc .

in 97% yield. Then, the influences of cosolvents on catalytic property were investigated by using the model condensation of benzaldehyde, benzil and NH_4OAc . The results are shown in Table 1. It was found that among the solvents tested, HOAc proved to be the most efficient and was selected to be the reaction solvent for the subsequent investigation.

We next examined the catalytic activity of 10 $\text{RE}(\text{OPf})_3$ complexes in perfluorodecalin. It was found that $\text{Yb}(\text{OPf})_3$ was the most effective catalyst, which produced almost quantitative yields of imidazole in perfluorodecalin. Table 2 shows the results we investigated.

Then, we decided to use $\text{Yb}(\text{OPf})_3$ as a catalyst and perfluorodecalin as a fluoros solvent for condensation. The condensations of various aldehydes with benzil and NH_4OAc have been examined (Scheme 2). The results are summarized in Table 3. Aromatic aldehydes were efficient reagents and no side-reaction products were observed (Table 3, entries 1–10). Aromatic aldehydes with electron-donating groups gave higher yield of the corresponding imidazoles than those with electron-withdrawing groups. Aliphatic aldehydes were not efficient in such protocol, and gave rather lower yield than aromatic aldehydes (Table 3, entries 11–12). Steric effects did not influence the yield significantly, for example, in the reaction of *p*-chloro (Table 3, entry 6) and *o*-chloro (Table 3, entry 7) benzaldehyde, the corresponding condensation products were obtained in 83% and 80% yields, respectively. When the reaction was finished, the reaction mixture was filtrated and the filtrate was cooled to room temperature, the fluoros phase with $\text{RE}(\text{OPf})_3$ can separate from the organic layer and return to the

Table 2

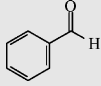
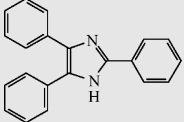
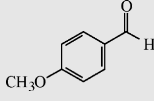
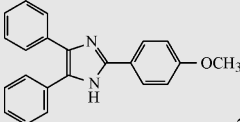
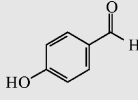
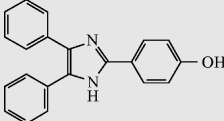
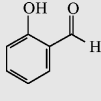
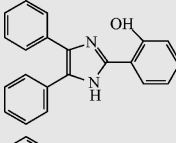
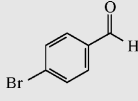
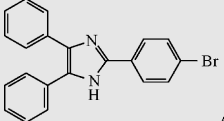
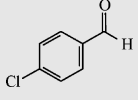
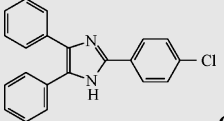
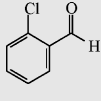
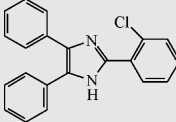
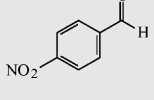
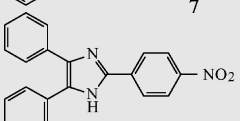
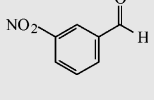
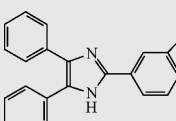
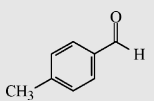
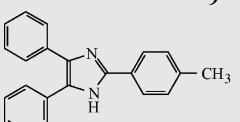
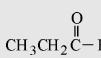
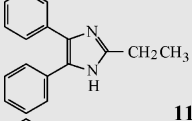
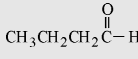
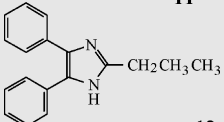
Effect of the catalysts on the condensation^a

Entry	Catalyst	Yield ^b (%)
1	$\text{La}(\text{OPf})_3$	80
2	$\text{Ce}(\text{OPf})_3$	91
3	$\text{Nd}(\text{OPf})_3$	91
4	$\text{Sm}(\text{OPf})_3$	90
5	$\text{Eu}(\text{OPf})_3$	82
6	$\text{Tb}(\text{OPf})_3$	85
7	$\text{Dy}(\text{OPf})_3$	88
8	$\text{Er}(\text{OPf})_3$	85
9	$\text{Yb}(\text{OPf})_3$	97
10	$\text{Lu}(\text{OPf})_3$	80

^a The reaction condition: benzaldehyde, 2 mmol; benzil, 1 mmol; NH_4OAc , 10 mmol; $\text{RE}(\text{OPf})_3$, 0.004 mmol; HOAc, 2 mL; perfluorodecalin ($\text{C}_{10}\text{F}_{18}$, *cis* and *trans*-mixture), 1.5 mL; 80°C ; 6 h.

^b Isolated yields.

Table 3
Condensation of aldehydes, benzil and NH_4OAc^a

Entry	Aldehyde	Product	Time (h)	Yield ^b (%)	mp (°C)
1		 1	6	97	272–273 (lit [14] >260)
2		 2	6	97	227–228 (lit [14] 227–228)
3		 3	6	90	235–237 (lit [22] 233)
4		 4	6	89	209–210 (lit [22] 205)
5		 5	6	85	244–246 (lit [14] 236–237)
6		 6	6	83	261–262 (lit [23] 259–261)
7		 7	6	80	188 (lit [22] 188)
8		 8	6	80	236–237 (lit [14] 241–242)
9		 9	6	75	>290 (lit [14] >260)
10		 10	6	93	227–228 (lit [23] 226–228)
11		 11	12	10	226–228 (lit [14] 223–224)
12		 12	12	31	256–258 (lit [14] 256–258)

^a The reaction condition: aldehyde, 2 mmol; benzil, 1 mmol; NH_4OAc , 10 mmol; $\text{Yb}(\text{OPf})_3$, 0.004 mmol; HOAc, 2 mL; perfluorodecalin ($\text{C}_{10}\text{F}_{18}$, *cis* and *trans*-mixture), 1.5 mL; 80 °C.

^b Isolated yields.

bottom layer. Based on GC-MS and ^{19}F NMR data, the distribution of $\text{Yb}(\text{OPf})_3$ found in organic layer was less than 1% and only trace of perfluorodecalin leached to organic phase can be detected.

We next sought to probe whether the catalyst could be recycled. The condensation of benzaldehyde, benzil and NH_4OAc under the conditions described in Table 2 with $\text{Yb}(\text{OPf})_3$ as catalysts was run for five consecutive cycles, furnishing the corresponding imidazole, with 97%, 96%, 95%, 94%, 94% isolated yields. The robustness of the catalyst for recycling using may be attributed that these rare-earth metal perflates are stable in aqueous conditions [24], and the metal catalyst coordinated by fluoros ponytail " C_8F_{17} " can dissolve into the fluoros phase. The separated fluoros phase containing only catalyst could be reused for the next condensation without any treatment, and this workup procedure of recycling was accomplished by simple phase-separation.

In conclusion, $\text{Yb}(\text{OPf})_3$ is demonstrated to be new and highly effective catalysts for preparation of trisubstituted imidazoles in fluoros biphasic system. By simple separation of the fluoros phase containing only catalyst, the reaction can be repeated many times. Further study on the application of FBS to other reactions, which can be promoted by such Lewis acids, is under way in this laboratory.

2,4,5-Triphenylimidazole (**1**): 97% yield; mp: 272–273 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.51–7.90 (m, 15H, Ar), 12.60 (brs, NH); MS (EI) m/z 296 (M^+).

2-(4-Methoxyphenyl)-4,5-diphenylimidazole (**2**): 97% yield; mp: 227–228 °C; ^1H NMR (500 MHz, CDCl_3) δ = 3.85 (s, 3H, OCH_3), 6.93–6.96 (d, 2H, Ar), 7.25–7.59 (m, 10H, Ar), 8.02–8.05 (d, 2H, Ar), 12.52 (brs, NH); MS (EI) m/z 326 (M^+).

2-(4-Hydroxyphenyl)-4,5-diphenylimidazole (**3**): 90% yield; mp: 235–237 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.00–7.90 (m, 14H, Ar), 9.50 (brs, NH); MS (EI) m/z 312 (M^+).

2-(2-Hydroxyphenyl)-4,5-diphenylimidazole (**4**): 89% yield; mp: 209–210 °C; ^1H NMR (500 MHz, CDCl_3) δ = 6.70–7.60 (m, 14H, Ar), 9.50 (brs, NH); MS (EI) m/z 312 (M^+).

2-(4-Bromophenyl)-4,5-diphenylimidazole (**5**): 85% yield; mp: 244–246 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.20 (d, 2H, Ar), 7.30–7.60 (m, 10H, Ar), 7.80 (d, 2H, Ar); MS (EI) m/z 375 (M^+).

2-(4-Chlorophenyl)-4,5-diphenylimidazole (**6**): 83% yield; mp: 261–262 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.30 (d, 2H, Ar), 7.40–7.70 (m, 10H, Ar), 7.90 (d, 2H, Ar), 12.50 (s, NH); MS (EI) m/z 330, 330 + 2 (M^+).

2-(2-Chlorophenyl)-4,5-diphenylimidazole (**7**): 80% yield; mp: 188 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.27–7.37 (m, 10H, Ar), 7.45–7.49 (dd, 1H, Ar), 7.57–7.59 (d, 2H, Ar), 8.02–8.05 (dd, 1H, Ar), 12.50 (brs, NH); MS (EI) m/z 330, 330 + 2 (M^+).

2-(4-Nitrophenyl)-4,5-diphenylimidazole (**8**): 80% yield; mp: 236–237 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.35–7.60 (m, 10H, Ar), 8.05–8.30 (m, 4H, Ar); MS (EI) m/z 341 (M^+).

2-(3-Nitrophenyl)-4,5-diphenylimidazole (**9**): 75% yield; mp: >290 °C; ^1H NMR (500 MHz, CDCl_3) δ = 7.55–8.95 (m, 14H, Ar), 13.10 (brs, NH); MS (EI) m/z 341 (M^+).

2-(4-Methylphenyl)-4,5-diphenylimidazole (**10**): 93% yield; mp: 227–228 °C; ^1H NMR (500 MHz, CDCl_3) δ = 2.90 (s, 3H, CH_3), 6.80 (d, 2H, Ar), 7.20–7.80 (m, 12H, Ar), 12.60 (brs, NH); MS (EI) m/z 310 (M^+).

2-Ethyl-4,5-diphenylimidazole (**11**): 10% yield; mp: 226–228 °C; ^1H NMR (500 MHz, CDCl_3) δ = 1.24 (t, 3H, CH_3), 2.59 (m,

2H, CH_2), 7.50–8.15 (m, 10H, Ar), 13.40 (brs, NH); MS (EI) m/z 248 (M^+).

2-Propyl-4,5-diphenylimidazole (**12**): 31% yield; mp: 256–258 °C; ^1H NMR (500 MHz, CDCl_3) δ = 0.96 (t, 3H, CH_3), 1.66 (m, 2H, CH_2), 2.55 (t, 2H, CH_2), 7.45–8.10 (m, 10H, Ar), 13.40 (brs, NH); MS (EI) m/z 262 (M^+).

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