

Samarium(III) Triflate as an Efficient and Reusable Catalyst for the Facile Synthesis of 1,2,4,5-Tetrasubstituted Imidazoles

A. Gorepatil¹, P. Gorepatil², M. Gaikwad³, A. Ghumare¹, J. Mali¹, V. Ingle^{4*}

¹Department of Chemistry, S. G. R. G. Shinde College, Paranda, Dist-Osmanabad (MH), 413 502, India

²Department of Chemistry, Sangameshwar College, Solapur (MH), 413 001, India

³Department of Chemistry, B. P. C. A. S. Angar, Dist- Solapur (MH), 413 214, India

⁴Department of Chemistry, S. C. S. College, Omerga, Dist-Osmanabad (MH), 413 606, India

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Abstract

An efficient, simple, and green four-component reaction for synthesizing 1,2,4,5-tetrasubstituted imidazoles is developed. In present work exploring the use of Samarium triflate as a heterogeneous catalyst for the four-component reaction of aldehydes, aromatic amines, benzil, and ammonium acetate. The current approach offers many advantages, such as excellent yields, shortening of reaction time, reusability of catalyst, and use of mild reaction conditions.

Keywords: Tetrasubstituted imidazoles; Samarium triflate; Four component reaction; Recyclable catalyst; Green process; Heterogeneous Lewis acid.

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1. Introduction

Nitrogen-containing heterocycles have considerable significance in agrochemicals and pharmaceuticals [1,2]. Amongst N-containing heterocyclic compounds, imidazole derivatives have received more attention from researchers across the globe because of their vital roles in biochemical processes, pharmacological properties like antiallergic, analgesic, antifungal, anthelmintic, anti-tuberculosis, antiprotozoal and anti-inflammatory [3-7]. Many imidazole-based moieties have also been utilized as inhibitors of p38 MAP kinase, B-Raf kinase, glucagon receptors, plant growth regulators, corrosion inhibitors, antitumoral, and pesticides [8-10].

The aforesaid applicability of imidazole derivatives highlights its importance. This calls for researchers to develop simple and green methods for synthesizing poly-substituted imidazole derivatives. This can be achieved by finding an efficient, recyclable,

* Corresponding author: inglevilas71@yahoo.in

heterogeneous catalyst. To date, several methodologies have been explored for synthesizing substituted imidazole derivatives. This includes cross-coupling reactions, [3+2] cycloaddition reaction of nitroolefins and N-aryl benzamides, and multicomponent cyclocondensation synthesis in the presence of various catalysts [11-14].

However, the multicomponent cyclocondensation approach is hugely undertaken owing to its excellent reaction features; more selectivity, facile synthesis, low cost, lesser reaction time, and minimal byproducts. Synthesis of 1,2,4,5-tetrasubstituted imidazoles involves cyclocondensation of a 1,2-diketone or α -hydroxy ketone with several aldehydes, primary amine, and ammonium acetate in the presence of various catalysts. These include $\text{HBF}_4\text{-SiO}_2$ / $\text{BF}_3\text{-SiO}_2$ / $\text{P}_2\text{O}_5\text{-SiO}_2$ /Silica bonded PPSA [15-18], FePO_4 [19], $\text{NiFe}_2\text{O}_4\text{@SiO}_2\text{-H}_3\text{PMO}_{12}\text{O}_{40}$ [20], silica-supported sulfuric acid [21,22], phosphoric acid [23], silica-supported Wells–Dawson acid [24], silica chloride [25], heteropolyacid [26, 27], citrate trisulfonic acid [28], ionic liquids [29-32], SBA-15/2,2,2-trifluoroethanol [33], clay-supported titanium [34], L-Proline [35] and elemental sulfur [36]. There are also a few interesting strategies for the synthesis of 1,2,4,5-tetrasubstituted imidazoles by condensation of 1,2-diketone, aryl nitrile, and a primary amine. Balalai *et al.* employed microwave irradiation conditions [37]. Shivkumaret *et al.* discussed N-alkylation of trisubstituted imidazoles by hetero-Cope rearrangement [38], Bansal *et al.* explored the use of Sodium lauryl sulfate as a catalyst [39], Mohammadzadeh *et al.* used trifluoroacetic acid catalyst for the aforesaid synthesis [40]. Other essential strategies should also not be neglected [41-44]. Although these protocols are suitable for the synthesis of imidazole derivatives, they suffer from serious disadvantages like long reaction times, low selectivity towards a product, tedious work-up procedure, and expensive reagents or catalysts. In addition, large amounts of catalysts are required, leading to the generation of huge quantities of toxic waste.

In order to overcome aforesaid drawbacks, heterogeneous Lewis acids have been playing a vital role in organic synthesis due to their characteristics like ease of handling, increased reaction rate, higher selectivity, simple work-up, and most importantly, catalyst recoverability [45,46]. Our group has also been working on developing Lewis acid-based, greener approaches for synthesizing different types of heterocyclic compounds [47-49]. One of the popular catalyst metal triflates has widely been employed in cyclocondensation and other organic reactions [50-60]. The main reasons are again lying in its fantastic features such as water-tolerant nature, high selectivity, and its reusability. In this work, pioneering the use of samarium triflate [$\text{Sm}(\text{OTf})_3$] in the synthesis of 1,2,4,5-tetrasubstituted imidazoles in aqueous ethanol has been reported.

2. Experimental Section

2.1. Reagents and chemicals

All chemicals like benzil, ammonium acetate, all used aldehydes, aromatic amines, and reagents samarium triflate were obtained from Merck (India), and ethanol was obtained from Loba Chemie (India) and was used without further purification. ^1H NMR and ^{13}C

NMR spectra were recorded on 400 MHz & 100 MHz spectrometer instruments, respectively, using TMS as an internal standard and CDCl₃, DMSO-d₆ as a solvent. The progress of the reactions was monitored by TLC using silica gel 60 F254 pre-coated plates. Melting points were determined using an open capillary tube apparatus and were uncorrected.

2.2. General procedure for the synthesis of 1,2,4,5-trisubstituted imidazoles (3a-o)

A mixture of appropriate benzil (1 mmol), aldehyde (1 mmol), aryl amine (1 mmol), ammonium acetate (1 mmol), and samarium triflate (5 mol%) was stirred at 60-70 °C in ethanol (5 mL). Meanwhile, the progress of the reaction was monitored by TLC using an n-hexane and ethyl acetate (7:3) mixture. After completion of the reaction, the reaction mixture is cooled, and the catalyst is removed by filtration, washed with diethyl ether, and dried at 50 °C under vacuum for 1 h. The filtrate was added to ice cold to get a solid product. The solid product was then filtered by suction to give a crude product. The product was recrystallized in ethanol to give pure tetrasubstituted imidazoles. All the synthesized compounds are confirmed by their analytical data and compared with the literature data [15-18,29-32,58-60].

2.3. Spectral data of selected compounds

2.3.1. 1,2,4,5-Tetraphenyl-1H-imidazole (3a, Table 2 Entry 1)

White solid; Mp 220-222 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 8.04-7.97 (m, 6H), 7.46-7.28 (m, 14H). ¹³C NMR (100 MHz, DMSO-d₆): δ = 144.31, 139.15, 134.70, 131.14, 130.88, 129.83, 129.67, 128.38, 128.15, 128.09, 128.04, 126.55, 126.21, 121.88, 115.55, 114.92; MS (EI): m/z: 372.00 [M⁺].

2.3.2. 1-(4-chlorophenyl)-2,4,5-triphenyl-1H-imidazole(3b, Table 2 Entry 2)

White solid; Mp 242-244 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 8.21-8.19 (m, 4H), 7.57-7.11 (m, 15H). ¹³C NMR (100 MHz, CDCl₃): δ = 151.03, 138.42, 135.68, 133.26, 131.51, 131.25, 130.70, 129.35, 129.21, 128.91, 128.56, 127.96, 127.88, 127.51, 127.14, 126.36, 125.64, 121.58, 121.06; MS (EI): m/z: 406.00 [M⁺].

2.3.3. 2-(3-nitrophenyl)-1,4,5-triphenyl-1H-imidazole (3c, Table 2 Entry 3)

White solid; Mp 243-245 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.39-7.75 (m, 17H), 8.26 (m, 1H), 8.53 (t, 1H). ¹³C NMR (100 MHz, CDCl₃): δ = 150.10, 145.91, 136.61, 134.55, 131.59, 131.12, 130.63, 130.57, 129.78, 128.40, 128.27, 128.15, 128.12, 127.83, 126.34, 125.28; MS (EI): m/z: 417.00 [M⁺].

2.3.4. 4-(1-(4-chlorophenyl)-4,5-diphenyl-1H-imidazol-2-yl)phenol (3d, Table 2 Entry 4)

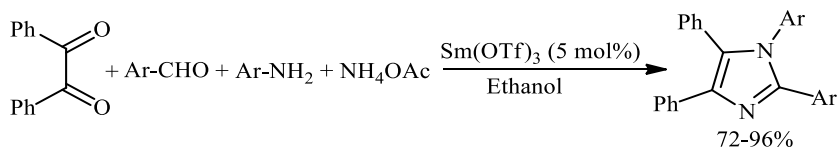
White solid; Mp 276-278 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.37 (d, J=8.5 Hz, 2H), 7.44 (d, J=8.5 Hz, 2H), 7.59-7.95 (m, 12H), 8.20 (d, J=7.4 Hz, 2H), 9.75 (s, 1H, Ar-OH). ¹³C NMR (100 MHz, CDCl₃): δ= 157.40, 145.09, 136.87, 134.39, 133.10, 131.79, 131.11, 130.44, 129.73, 129.71, 129.29, 128.43, 128.37, 128.26, 128.15, 127.50, 126.44, 126.32, 116.00; MS (EI): m/z: 422.00 [M⁺].

2.3.5. 2-(4-chlorophenyl)-1,4,5-triphenyl-1H-imidazole (3e, Table 2 Entry 5)

White solid; Mp 190-192 °C; ¹H NMR (400 MHz, DMSO-d₆): δ = 7.31-7.44 (m, 17H), 7.79 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): δ= 146, 133.50, 130.11, 129.75, 129.62, 128.90, 128.41, 128.15, 127.87, 127.78, 127.30, 127.09, 127.06, 126.84, 126.33, 125.49; MS (EI): m/z: 406.00 [M⁺].

3. Results and Discussion

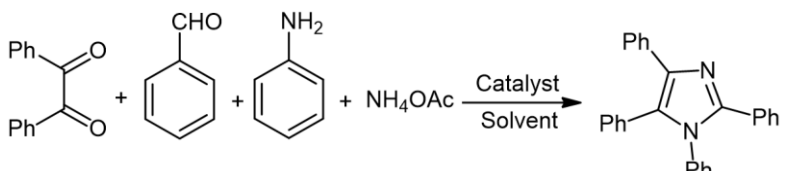
Owing to the aforementioned features of metal triflates, here reported the use of samarium triflate [Sm(OTf)₃] along-with greener solvent media for the facile, efficient four-component synthetic strategy for the synthesis of 1,2,4,5-tetrasubstituted imidazoles by engaging primary aromatic amines, aldehydes, benzil and ammonium acetate in the cyclocondensation (Scheme 1). To the best of our knowledge, this is the first attempt to utilize samarium triflate for the synthesis of tetra-substituted imidazole derivatives.



Scheme 1. Samarium triflate [Sm(OTf)₃] catalyzed the synthesis of tetrasubstituted imidazoles.

Initially, the samarium triflate catalyzed reaction between benzil, benzaldehyde, aniline, and ammonium acetate was selected as the model reaction for optimization under different concentrations of catalyst and different solvents summarized in Table 1. Compared with other catalysts and solvents, samarium triflate (5 mol%) was an efficient catalyst for completing reactions in ethanol at 60-70 °C with 95 % yield (Table 1, entry 7).

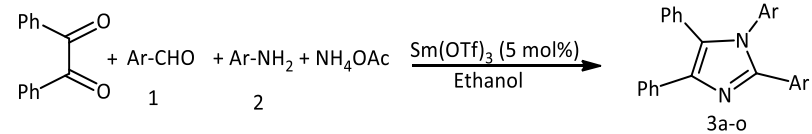
During the optimization, it was observed that using more samarium triflate (10 mol%) is not producing a satisfactory product. Also, many byproducts are formed with an increase in temperature >100 °C. The effect of the solvent is also noted, and it is established that polar solvents facilitate reactions more efficiently than non-polar ones.

Table 1. Optimization of reaction conditions for the synthesis of tetrasubstituted imidazoles^a.


Entry	Catalyst (mol%)	Solvent	Time(h)	Yield (%) ^b
1	-	EtOH	8	46
2	FeCl ₃ (5)	EtOH	4	65
3	ZnO (5)	EtOH	5	57
4	InCl ₃ .3H ₂ O (10)	EtOH	6	75
5	Sm(OTf) ₃ (5)	Dioxane	6	78
6	Sm(OTf) ₃ (5)	MeCN	5	65
7	Sm(OTf)₃ (5)	EtOH	2	95
8	Sm(OTf) ₃ (10)	EtOH	2	96
9	Sm(OTf) ₃ (5)	MeOH	3	85
10	Sm(OTf) ₃ (5)	Toluene	10	6

^aBenzil (1 mmol), benzaldehyde (1 mmol), aniline (1 mmol), ammonium acetate (1 mmol), stirring at 60-70 °C. ^bIsolated yield.

After optimization, this methodology was evaluated for four component reactions with various aromatic aldehydes and primary aromatic amines to produce a variety of 1,2,4,5- tetrasubstituted imidazole derivatives; it gives moderate to excellent yields under the same reaction conditions concerning various aldehydes and amines. The findings of reactions are tabulated in Table 2. Additionally, it has been noted that the aliphatic aldehydes gave a lower yield (Table 2, entry 15). In the case of aromatic aldehydes, electron-rich gave better yields in shorter reaction times when compared with electron-deficient ones (Table 2, entries 3, 11). However, lower yields are noted for the electron-deficient arylamines (Table 2, entries 2 and 7), like nitroaniline, and found that it gave a negligible amount of product with aldehydes under optimized reaction conditions.

Table 2. Sm(OTf)₃ Catalyzed synthesis of tetrasubstituted imidazole derivatives^a.


Entry	Ar-CHO (1)	Ar-NH ₂ (2)	Product (3)	Time h	Yield (%) ^b
1	C ₆ H ₅ -	C ₆ H ₅ -	3a	2	95
2	C ₆ H ₅ -	4-Cl-C ₆ H ₄ -	3b	2.5	88
3	3-NO ₂ -C ₆ H ₄ -	C ₆ H ₅ -	3c	3	90
4	4-OH-C ₆ H ₄ -	4-Cl-C ₆ H ₄ -	3d	2	96
5	4-Cl-C ₆ H ₄ -	C ₆ H ₅ -	3e	2	92
6	2-CH ₃ -C ₆ H ₄ -	C ₆ H ₅ -	3f	2.5	95

7	4-Cl-C ₆ H ₄ -	4-CH ₃ -C ₆ H ₄ -	3g	2.5	93
8	4-CH ₃ -C ₆ H ₄ -	C ₆ H ₅ -	3h	2	92
9	4-OH-C ₆ H ₄ -	C ₆ H ₅ -	3i	2.5	94
10	4-OH-C ₆ H ₄ -	4-CH ₃ -C ₆ H ₄ -	3j	2	95
11	4-NO ₂ -C ₆ H ₄ -	C ₆ H ₅ -	3k	3	86
12	4-Br-C ₆ H ₄ -	C ₆ H ₅ -	3l	3	89
13	4-CH ₃ O-C ₆ H ₄ -	C ₆ H ₅ -	3m	3	92
14	2-furyl	C ₆ H ₅ -	3n	2	90
15	CH ₃ -CH ₂ -	C ₆ H ₅ -	3o	4	72

^aStirring at 60-70 °C. ^bIsolated yield.

The recyclability of the catalyst is one of the important aspects of the methodology. To prove the recyclability of our catalyst, The catalyst was repeatedly reused when the reaction was completed for the synthesis of 1,2,4,5-tetraphenyl imidazole. As shown in Table 3, in the presence of 5 mol% of Sm(OTf)₃, the reaction gave excellent yields in three succeeding cycles without diminishing its activity. It's believed that samarium triflate is an environment-friendly catalyst and can be employed as an efficient catalyst for organic synthesis and to develop the new era of methodologies.

Table 3. Catalyst reusability study for the synthesis of 1,2,4,5-tetraphenyl imidazole^a.

Entry	Yield (%) ^b	Catalyst recovery (%)
1	96	94
2	92	90
3	86	85

^aReaction condition: Benzil (1 mmol), benzaldehyde (1 mmol), aniline (1 mmol), ammonium acetate (1 mmol), stirring at 60-70 °C. ^bIsolated yield.

4. Conclusion

The present work describes a simple, efficient, eco-friendly, and rapid methodology for the synthesis of 1,2,4,5-tetrasubstituted imidazoles in the presence of samarium triflate as a new and highly efficient heterogeneous catalyst under mild conditions. The key advantages of this technique are safety, little waste, facile separation, catalyst reusability, high product yields in short reaction time, non-corrosiveness, and environmental friendliness.

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