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Green Synthesis of 2, 4, 5-triphenyl-1H-imidazole Derivatives by using Cellulose based Cerium (IV) Catalyst

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Abstract

We report simple, efficient and one pot multicomponent protocol for the synthesis of 2,4,5-triphenyl-1H-imidazole derivatives via reaction of various aromatic aldehyde and benzill in the presence of catalytic amount of cellulose based cerium (IV) catalyst. CMC-Ce^{IV} was prepared by metathesis strategy and characterized by FT-IR techniques. The catalyst was recovered and reused for fivseveral cycles without considerable loss of activity. The advantages of the protocol include rapid reactions with good yields and simple workup. The synthesized compounds were characterized by FT- IR technique.

Keywords: Imidazole derivatives, Multi-component reaction, Metathesis Reaction.

Introduction

Imidazole is an important core organic molecule. It is found in many naturally occurring compounds like vitamin B_{12}, histidine, histamine, pilocarpine alkaloids, and biotin.[1-3] It isalso showing good activity as herbicide, plant growth regulator, anti-epileptic, anticonvulsant, anti-inflammatory, analgesic, anticancer, etc.[4-8] Also, imidazoles are found as the main core molecule in drugs like Omeprazole, Pimobendan, Losarton, Olmesartan, Eprosartan, and Trifenagrel.[9]

Owing to their wide range of biological advancement, synthesis of title compounds are stillof intrigue. The available reported method for the synthesis of substituted imidazoles suffers from drawbacks such as the catalysts used for synthesis are either toxic or expensive and requires harsh reaction condition. Therefore, a need still exists for further development of an efficient, reusable, inexpensive and eco-friendly catalyst for the synthesis of substituted imidazoles. In organic synthesis, the product yield and reaction time are extremely important. The increase in reaction

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steps results in a decrease in final product yield and increase in total reaction time. Multicomponent reactions help to solve this problem. By novel developing multicomponent reaction strategies, synthesis of the desired product in the one-pot method is possible thereby increases the product yield and reducing reaction time required for the reaction.

The interest in metal NPs, attributable to their high surface area, incredible availability, high biocompatibility and low toxicity. In addition, the high catalytic activity of metallic NPs can be accounted due to its Lewis acid site.[10] Considering these facts, we have decided to synthesize 2,4,5 triaryl-imidazole derivatives of various substituted benzaldehydes and benzil efficiently using CMC-Ce^{IV} as a recoverable and reusable catalyst. For this transformation we used ethanol as green solvent and considering green chemistry approach.

Materials and Methods

All reagents used were of laboratory grade. Melting points were determined in open capillaries. Progress of reaction was monitored by silica gel-G coated TLC plates in n-hexane: ethyl acetate system (9:1).The spot was visualized by exposing dry plate in UV chamber.. IR spectra were recorded on Shimadzu FT-IR (Affinity Model) using KBr.

Preparation of Ce(IV)carboxymethylcellulose (CMC− Ce^{IV}) Catalyst[11]

The Ce(IV) carboxymethylcellulose (CMC− Ce^{IV}) catalyst was prepared by metathesis reaction of cerric ammonium nitrate and Na-CMC. The yellow solid was precipitated which was left to equilibrate in a solution for overnight. The resulting yellow solid was separated from the solution and washed thoroughly with distilled water. The wet CMC− Ce^{IV} was driedat 60^0C in the oven till constant weight.(**scheme 1**)

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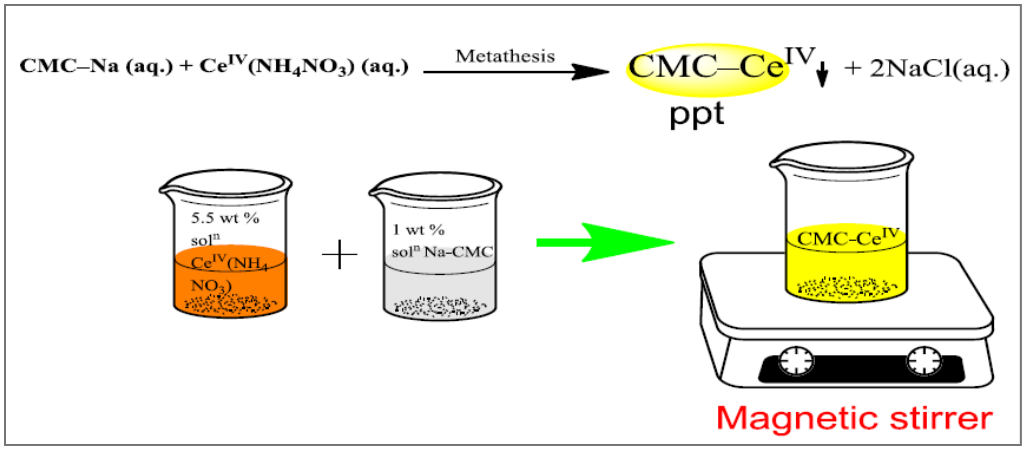

Scheme 1- Preparation of Ce(IV)carboxymethylcellulose (CMC− Ce^{IV}) Catalyst[11]

General procedure for synthesis of 2,4,5-triphenyl-1H-imidazole derivatives by using CMC− Ce^{IV} as catalyst:

In 150 ml round bottom flask, a mixture of benzaldehyde (10 mmol), benzil (10 mmol) and ammonium acetate (10mmol), as ammonia source, and CMC-Ce^{IV}(20mg) were stirred and refluxed in ethanol for appropriate time (Table 1). The progress of the reaction was monitored by TLC. After completion of the reaction, the precipitate thus obtained was wash with ethanol and then purified by recrystallization by ethanol to get corresponding pure product (**Scheme 2)**.

Results and Discussions

Optimized Reaction Conditions:

b) Effect of catalyst:

To optimize the reaction condition, we performed the model reaction of p-anisaldehyde with different amount of CMC− Ce^{IV} catalyst loaded as shown in **Table 1**.

Table 1: Optimized amount of catalyst loaded

Entry	Catalyst (mg)	Time (min)	Yield (%)
1	5	80	60
2	10	50	82
3	15	35	90
4	**20**	**15**	**96**
5	25	15	93

It was found that, the 20 mg catalyst is sufficient to push the reaction forward.

b) Effect of the Solvent

To investigate the role of solvent in model reaction of p-anisaldehyde was performed in different

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solvent like ethanol, water and another organic solvent, as shown in **Table 2**.

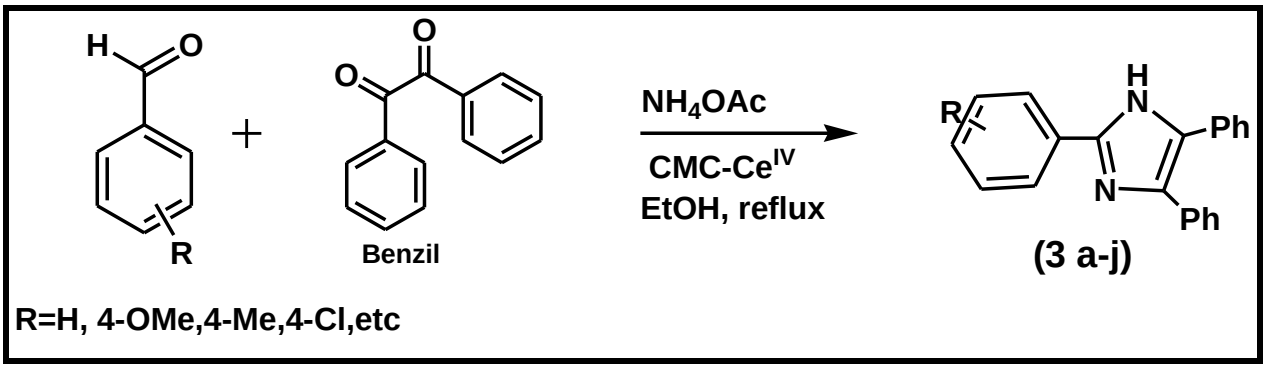
Table 2: Effect of solvent

Entry No.	Solvent	Time(Min)	Yield (%)
1.	Solvent Free	60	71
2.	**Ethanol**	**10**	**96**
3.	Water	20	40
4.	CH_2Cl_2	30	53
5.	n-Hexane	30	62
6.	Toluene	45	55

From the above table it clear in pure ethanol get high yield of product & minimum time required for the completion of reaction.

It was observed that, when we used pure ethanol as a solvent the yield of product increases up to 96% and time also reduced about 10 min. Imidazole formation was increases in ethanol, while the same reaction occurred slowly in water and another organic solvent.

After the study of above optimized reaction conditions were explored for the synthesis of series of 2,4,5-triphenyl-1H-imidazole derivatives(3 a-j) from various substituted benzaldehydes and benzil efficiently using CMC-Ce^{IV} catalyst as shown in **Scheme 1** and the results of (3a-d) derivatives are summarized in **Table 3.**



Scheme 1: Synthesis of 2,4,5-triphenyl-1H-imidazole derivatives

Table 3: Synthesis of 2,4,5-triphenyl-1H-imidazole derivatives (3a-d)

Sr. No.	Substituted benzaldehydes	Product	Time (min)	Yield (%)	Melting Point (^{0}C)

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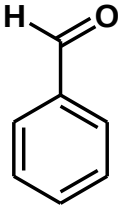
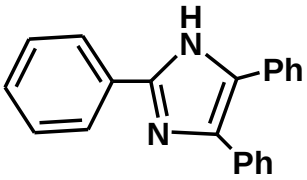
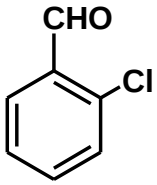
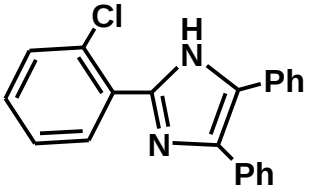
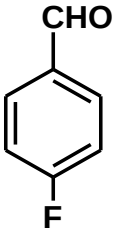
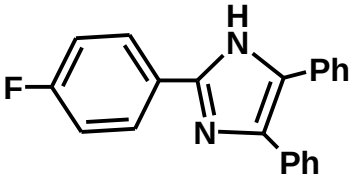
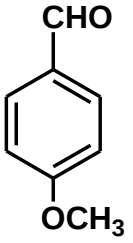
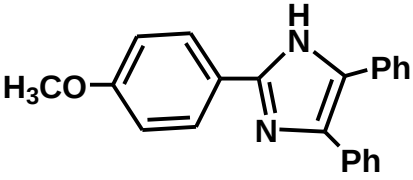
3a.			15	90	210
3b.			15	92	250
3c.			20	90	252
3d			10	96	212

Data

3a) Cream White solid, **IR (**cm^{-1}): 3340(NH); 1668(C=N), 1587(CN), 1320 (CN), 1510 (C=C aromatic), 3061(C=CH).

3b) Yellow solid, **IR** (cm^{-1}): 3319(NH); 1670(C=N), 1583(CN), 1311 (CN), 1520 (C=C aromatic), 3064(C=CH).

3c) Lemon Yellow solid, **IR** (cm^{-1}): 3322(NH); 1648(C=N), 1590(CN), 1313 (CN), 1534(C=C aromatic), 3066(C=CH).

3d) Brown solid, **IR** (cm^{-1}): 3340(NH); 1668(C=N), 1579(CN), 1315 (CN), 1519 (C=Caromatic), 3067(C=CH)

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Applications

These investigations involve use of green solvent method. The procedure offers advantages in terms of better yields, short reaction times, mild reaction conditions, and reusability of the catalyst. The low cost, and ready availability of catalyst, an environmentally benign procedure makes this methodology, a useful contribution to the existing procedures available for the synthesis of 2,4,5-triphenyl-1H-imidazole derivatives as a biologically and pharmaceutically relevant materials.

Conflict of Interest

There is no conflict of interest for given article

Conclusion

The CMC-Ce^{IV} NPs were prepared by the ion exchange reaction. The inclusion phenomenon of sodium carboxymethyl cellulose with cerric ammonium nitrate was successfully characterized by FT-IR techniques. We have developed a simple and efficient protocol for one-pot synthesis of of 2,4,5-triphenyl-1H-imidazole derivatives from various substituted benzaldehydes and benzil efficiently using CMC-Ce^{IV} as a catalyst. The high catalytic activity of CMC-Ce^{IV} was accounted due its Lewis acid sites. The advantages of procedure include simplicity of operation, good yields, wide substrate scope, no chromatographic separation technique, an easy recovery of the catalyst and recyclability of catalyst.

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