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Efficient and Recyclable Ce(salophen)/HY Catalyst for the Green Synthesis of Oxazoline Derivatives

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Abstract: A hybrid zeolite-based catalyst, Ce(salophen)/HY (CSDY), was prepared using a ship-in-a-bottle approach and examined in the cyclization of ethanolamine with different nitriles for the formation of oxazoline derivatives. The presence of cerium Lewis acidic sites, together with the confinement effect of the HY zeolite, led to satisfactory catalytic activity and stability under relatively mild conditions. The optimum results were achieved at 140 °C with a catalyst loading of 0.15–0.20 mmol and reaction times ranging from 30 to 60 min. Both aromatic and aliphatic nitriles participated efficiently in the reaction. As expected, nitriles bearing electron-withdrawing substituents reacted faster than those containing electron-donating groups. In this series, 4-hydroxybenzonitrile afforded the lowest yield (35%), whereas the highest yield (95%) was obtained from 3-chlorobenzonitrile. Moreover, the catalyst could be easily recovered and reused for five consecutive cycles, while maintaining its activity with negligible cerium leaching. Overall, Ce(salophen)/HY can be regarded as a stable and recyclable heterogeneous catalyst for the synthesis of oxazoline heterocycles.

Keywords: Ce(salophen)/HY, Oxazoline, Heterogeneous catalysis, Reusability, Ship-in-a-bottle method.

Introduction

Oxazolines are A class of five-membered heterocycles including nitrogen and/or oxygen in the ring structure of the compound and have the formula C_3H_5NO. Oxazolines are known as crucial intermediates in organic synthesis and are used extensively [1]. In general, oxazolines were prepared by cyclodehydrating β-hydroxyamides or from amino alcohols, and under acid-catalyzing conditions. Due to their unique characteristic of coordinating with transition metals, oxazolines have become some of the most common chiral ligands used in asymmetric catalysis, particularly in hydrogenation and the formation of carbon–carbon bonds [2,3]. Oxazolines are structurally stable compounds and have tunable steric and electronic properties, making them highly valuable in pharmaceuticals, catalysis, and advanced materials. One of the main challenges in synthesizing oxazolines is achieving efficient cyclization while maintaining selectivity. Consequently, heterogeneous catalysis has emerged as a controlled and environmentally friendly strategy for chemical reactions [4-8]. Zeolites, with their structured microporous frameworks and adjustable Brønsted/Lewis acidity, provide a stable scaffold for facilitating chemical transformations. Their precisely defined pore dimensions help stabilize reaction intermediates and promote the selective formation of five-membered rings, often surpassing some of the homogeneous acids. Zeolites such as H-Beta, H-ZSM-5, and HY have demonstrated robust effectiveness in activating hydroxyl or carbonyl groups during ring closure reactions [9- 11].

Recent research has underlined the advantages of Lewis acid-modified zeolites, particularly those exchanged with zinc (Zn) and copper (Cu), for the mild and selective transformation of amino alcohols and β-hydroxyamides into oxazolines [12-15]. Moreover, studies on organic functionalized zeolites, which are modified with sulfonic acids, amines, or Schiff base ligands, have led to the development of hybrid catalysts [16,17]. These catalysts effectively combine the confinement effect of zeolites with adjustable organic coordination environments. Schiff base moieties are particularly attractive because they can stabilize transition metals such as nickel (Ni(II)) or copper (Cu(II)), resulting in highly stable and selective bifunctional catalysts [18, 19]. **Schiff base-functionalized zeolites combine the selectivity of homogeneous complexes with the recyclability of**

heterogeneous supports [20]. These advances demonstrate a growing trend toward more environmentally friendly and sustainable catalytic processes. Zeolite-based catalysts are easy to recover and reuse, minimize the need for hazardous solvents, and allow them to function effectively at moderate temperatures. These advantages contribute to lower costs, cleaner reaction pathways, and a reduced environmental impact in the industrial synthesis of oxazolines.

In the industrial synthesis of oxazolines, these advantages lead to lower costs, cleaner reaction pathways, and reduced environmental impact. Therefore, Schiff base-modified zeolites emerge as robust candidates for scalable applications. Significant progress has been made in the study of metal-exchanged zeolites and Schiff base catalysts. However, earlier research has predominantly treated these two strategies as separate approaches. Studies by Kumari and Moosavifar et al. demonstrated that Schiff base groups can effectively immobilize metal centers within zeolites [21]. Nevertheless, most of these systems have primarily been tested for oxidation or coupling reactions, rather than for the formation of heterocycles. Research on zeolites loaded with Zn, Cu, or Co for the synthesis of oxazolines has shown promising results [18,22]; however, these systems still encounter challenges related to selectivity, control of acid sites, and catalyst durability. This knowledge gap indicates that integrating the structural confinement and acidity of zeolites with the coordination flexibility of Schiff base ligands could offer a more effective strategy for optimizing reaction pathways during cyclization. Recent advances highlight the growing utility of cerium-based Lewis acid systems as efficient and sustainable catalysts for organic transformations **[23].** Therefore, this study focuses on Schiff base-modified zeolite catalysts, examining how their chemical environment, acidity, and metal distribution affect the formation of oxazoline derivatives. This research aims to design greener, more selective, and reusable solid catalysts by clarifying the links between catalyst structure and reactivity for modern heterocyclic synthesis.

Materials and Methods

Chemicals reagents

All reagents used in this study were of analytical grade and purchased from Sigma-Aldrich, Merck, Fluka, and Mojallali Co. They were used as received without further purification. The HY zeolite (delaminated Y zeolite) was prepared by post-synthetic treatment with ethylenediaminetetraacetic acid (EDTA), following the procedure described in our previous work [24]. In this process, careful control of the EDTA concentration, contact time, and addition rate is essential to avoid framework amorphization [25]. The H_2salophen ligand was synthesized according to the standard procedure reported previously [26].

Preparation of cerium salophen-encapsulated into dealuminated Y zeolite

The Ce(salophen)/HY catalyst was prepared following our previously reported methodology [26]. First, cerium-exchanged zeolite (CeY) was obtained by the ion-exchange technique. The Ce(salophen) complex was then incorporated inside the supercages of dealuminated Y zeolite through a “ship-in-a-bottle” method. In a typical preparation, 1 g of CeY was mixed with 3 g of H_2salophen and heated in an oil bath at 383–413 K for 18 hours. Under these conditions, the ligand melts and diffuses into the zeolite cavities, where it reacts in situ with the cerium ions to form the encapsulated complex.

At the end of the reaction, the product was purified by Soxhlet extraction with ethanol to remove unreacted ligand and any surface-deposited metal complexes. The resulting solid was dried at 353 K and then treated with a 0.01 M NaCl solution for 5 hours to eliminate residual uncoordinated cerium ions from the zeolite channels. The final catalyst was filtered, washed thoroughly with hot distilled water, and dried at 393 K for 8 hours.

General Procedure for the Synthesis of Oxazoline Derivatives

In a typical experiment, ethanolamine (1 mmol) and the selected aromatic or aliphatic nitrile (4 mmol) were placed in a 25 mL round-bottom flask fitted with a reflux condenser. Ce(salophen)/HY catalyst (0.15–0.20 g) and ethanol (10 mL) were added, and the mixture was stirred under reflux at 140 °C. Reaction progress was monitored by TLC. Once complete, the catalyst was recovered by filtration, washed with ethanol, and reused in subsequent runs without any notable loss of activity. All products were characterized by FT-IR and NMR spectroscopy.

Results and Discussions

Synthesis of Oxazoline Derivatives

The Ce(salophen) complex encapsulated within dealuminated Y zeolite (CSDY) was synthesized and characterized using powder X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), and field-emission scanning electron microscopy (FESEM).

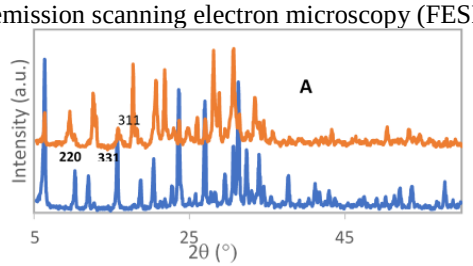

Fig. 1. XRD pattern of A) NaY zeolite, B) CSDY

Fig. 1 shows the XRD patterns of NaY zeolite and the CSDY catalyst. After encapsulation of the Ce(salophen) complex within the zeolite framework, the main diffraction peaks remain essentially unchanged, although a slight decrease in their intensities is observed. This result indicates that the crystalline structure of the zeolite is preserved during the incorporation of the metal complex. The reflections at 2θ corresponding to the (220), (311), and (331) planes are known to be sensitive to the distribution of cations within the zeolite cavities. After the introduction of cerium species, the relative intensity order of these peaks changes from 331 > 220 > 311 to 331 > 311 > 220. Such variation suggests a redistribution of cations within the zeolite pores, which can be attributed to the partial replacement of Na^+ ions by Ce^{3+} species during the encapsulation process [21].

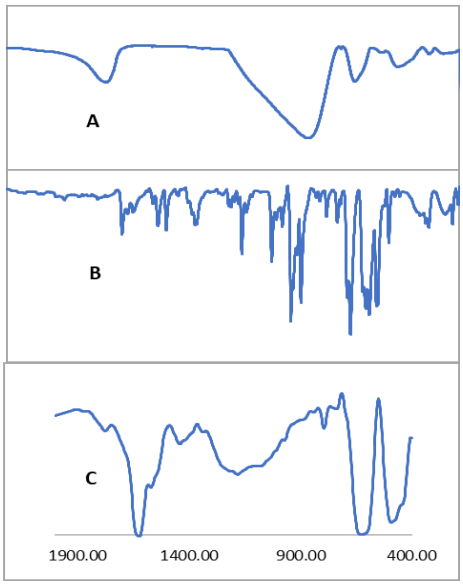

Fig. 2. FT-IR spectra of A) NaY zeolite, B) Cesalophen, and C) CSDY

The FT-IR spectra of NaY zeolite, the free Ce(salophen) complex, and the CSDY catalyst are presented in Fig. 2. The broad absorption bands observed in the 450–1200 cm^{-1} region correspond to the characteristic lattice vibrations of the zeolite framework. The strong band centered at approximately 1050 cm^{-1} is assigned to the asymmetric stretching vibrations of the $(Si/Al)O_4$ tetrahedral units.

In the spectrum of the encapsulated catalyst, several characteristic bands of the Ce(salophen) complex are partially masked by the intense framework vibrations of the zeolite host. Nevertheless, the free ligand shows distinct stretching vibrations associated with C–O, C–N, and aromatic ring modes in the 1100–1700 cm^{-1} region [30]. Upon coordination of the imine nitrogen atoms to the cerium center, these bands exhibit a slight blue shift, which can be attributed to the electronic changes resulting from metal–ligand interaction.

Additionally, bands in the 500–1000 cm^{-1} range are assigned to metal–ligand stretching vibrations and serve as a fingerprint for the presence of the encapsulated complex.

The FESEM micrograph of CSDY (Fig. 3) reveals well-defined cubo-octahedral crystallites, characteristic of the faujasite-type zeolite morphology. Importantly, the preservation of these uniform crystal shapes confirms that the Ce(salophen) complex has been successfully incorporated within the supercages of the zeolite, rather than deposited on the external surface.

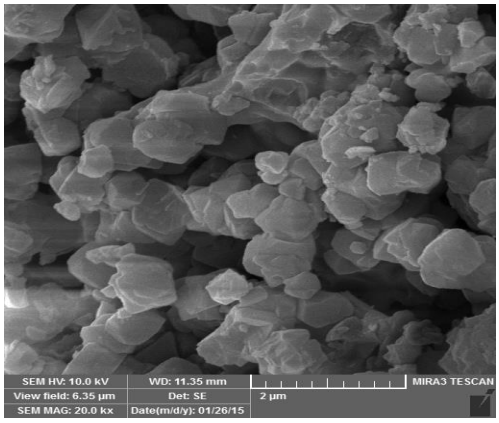

Fig. 3. FESEM micrograph of CSDY

After characterization of the catalyst, the catalytic activity of this system was studied in the synthesis of the oxazoline as heterogeneous catalyst. In this case, (Ce(salophen)/HY) integrates two functional domains: The Lewis acidity of Ce^{3+} centers and the confinement effect of HY's microporous channels. The confinement effect within zeolitic nanocavities modulates both the geometry and reactivity of encapsulated metal complexes **[27].** This combination promotes the electrophilic activation of nitriles and stabilizes reaction intermediates during the cyclodehydration with ethanolamine to form oxazoline derivatives. Preliminary screening at 140 °C showed that both aromatic and aliphatic nitriles underwent cyclization smoothly, achieving yields ≥ 90 % and short reaction times when compared to traditional homogeneous systems.

Effect of Temperature on the reaction yield

Temperature has a direct impact on conversion rates and

Table 1. Effect of temperature and time on yield (Ce(salophen)/HY, 0.15 mmol catalyst, 4:1 amine: nitrile ratio (model substrate: 4-Chlorobenzonitrile)

Tem (°C)	Time (min)	Yield (%)
100	100	82
120	60	90
140	50	95

overall yield. At temperatures below 120 °C, reactions take more than 90 minutes to achieve over 90% yield. However, at the optimal temperature of 140 °C, most substrates complete their reactions within 30 to 60 minutes (Table 1).

Effect of Catalyst Loading

The catalyst amount had a significant influence on catalytic activity. Yields increased noticeably between 0.05 mmol and 0.15 mmol; after that, the conversion remained constant. Slight reduction over 0.25 mmol occasionally led to reduced conversion due to pore saturation (Table 2) and (Fig.4).

Table 2. Effect of catalyst loading on oxazoline yield (140 °C, 60 min, 4:1 amine:nitrile), (model substrate: 4 chlorobenzonitrile)

Catalyst amount	Yield (%)	Time (min)
No catalyst	40	30
0.03	100	48
0.05	100	58
0.07	100	65
0.10	60	70
0.15	50	90
0.20	54	93
0.25	50	94
0.3	52	90

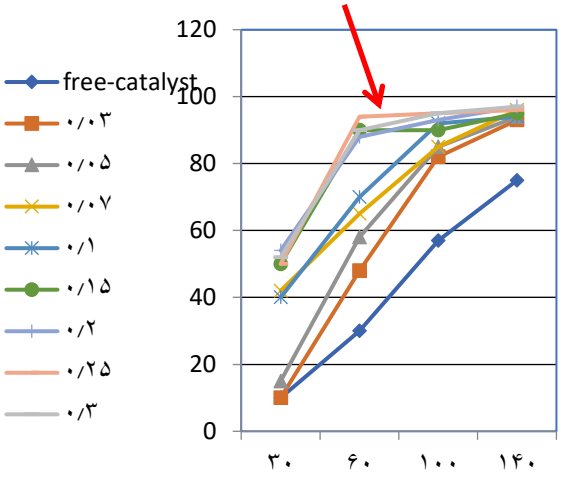

Fig. 4. Effect of catalyst loading on oxazoline yield

Under optimized reaction conditions, various nitriles including different electronic and steric properties were investigated in the synthesis of oxazolines (Table 3). Aromatic nitriles such as 4-chlorobenzonitrile and benzonitrile showed significantly higher yields (90–95 %) within short reaction times, whereas aliphatic nitriles,

Table 3. Synthesis of oxazoline of different nitril under various times under optimum conditions[a]

Chemical Name	Chemical Formula	Time (min)	Yield (%)
Fumaronitrile	$C_4H_2N_2$	10	65
		20	90
4-Hydroxy benzonitrile	C_7H_4NO	180	20
		240	30
		300	35
3-chlorobenzonitrile	C_7H_4NCl	30	70
		40	85
		60	95
Cyanophenyl	C_7H_4N	90	65
		120	80
		150	95
Butyronitrile	C_4H_7N	10	70
		20	85
		30	95
Cynamonitrile	C_9H_7N	120	55
		180	72
		210	85
4-chlorobenzonitrile	C_7H_4NCl	30	50
		40	75
		60	90

[a] tem:140 °C, catalyst amount: 0.15 mmol catalyst, 4:1 amine: nitrile ratio

including butyronitrile or fumaronitrile, required prolonged time (up to 180–210 min) with lower conversions (70–85 %) [28]. This contrast primarily arises from the electronic polarization of the –C≡N group in aromatic substrates, where electron-withdrawing substituents increase the electrophilicity of nitrile carbon, facilitating nucleophilic attack by ethanolamine. The higher reactivity of electron-withdrawing substituted benzonitriles is consistent with Hammett σ values (σ-p > 0), reflecting enhanced electrophilicity at the nitrile carbon [29].

Additionally, the planar geometry of aryl nitriles reduces steric hindrance around the reactive center, enabling faster ring closure. In contrast, alkyl nitriles possess less polar C≡N bonds and more flexible substituent chains, which diminish activation by Lewis-acidic Ce^{3+} sites and hinder effective cyclization. Lewis acid sites coordinate to the nitrogen lone pair of the nitrile group, increasing the electrophilicity of the carbon center and facilitating nucleophilic attack by ethanolamine [30].

These effects explain the superior performance of Ce(salophen)/HY toward aromatic substrates and demonstrate its balanced Lewis/Brønsted acidity necessary for efficient oxazoline formation.

Spectral Data of Synthesized Oxazoline Derivatives

The FT-IR and NMR spectral data of the synthesized oxazoline derivatives, along with their corresponding references, are presented below [4, 31, 32].

2-(4-Hydroxyphenyl)-4,5-dihydrooxazole ($C_9H_9NO_2$), 163.17 g/mol– IR: 3320–3360 (O–H), 1660 (C=N), 1105 (C–O–C), 1510–1600 (Ar). ^{1}H NMR: 4.08 (t, 2H), 4.50 (t, 2H), 6.85 (d, 2H), 7.55 (d, 2H), 9.5 (s, OH).

2-(3-Chlorophenyl)-4,5-dihydrooxazole (C_9H_8ClNO), 181.62 g/mol– IR: 1660 (C=N), 1100 (C-O), 750 (C–Cl). ^{1}H NMR: 4.10 (t, 2H), 4.52 (t, 2H), 7.15–7.45 (m, 2H), 7.8-7.9 (m, 2H).

2-(4-Chlorophenyl)-4,5-dihydrooxazole (C_9H_8ClNO), 181.62 g/mol – IR: 1655 (C=N), 1100 (C-O), 1590 (C=C), 820 (C-Cl). ^{1}H NMR: 4.08 (t, 2H), 4.45 (t, 2H), 7.30 (d, 2H), 7.65 (d, 2H).

2-Phenyl-4,5-dihydrooxazole (C_9H_8NO)–147.17 IR: 1660 (C=N), 1100 (C-O), 1595 (C=C). ^{1}H NMR: 4.10 (t, 2H), 4.55 (t, 2H), 7.20–7.45 (m, 3H), 7.8-7.9 (m, 2H).

2-Propyl-4,5-dihydrooxazole ($C_6H_{11}NO$), 113.16– IR: 1660(C=N), 1110 (C-O), 2950–2870 (C-H). ^{1}H NMR: 0.90 (t, 3H), 1.40 (sextet, 2H), 2.2 (t, 2H) 3.8 (t, 2H), 4.10 (t, 2H).

2-Styryl-4,5-dihydrooxazole ($C_{11}H_{11}NO$) – IR: 1660 (C=N), 1100 (C-O), 1600–1620 (C=C). ^{1}H NMR: 4.10 (t, 2H), 4.55 (t, 2H), 6.45 (d), 6.70 (d), 7.20–7.40 (m).

Bis(oxazolin-2-yl) ethylene ($C_8H_{10}N2O2$), 166.18 – IR: 1660×2 (C=N), 1100×2 (C-O), 1620. ^{1}H NMR: 4.10 (t, 4H), 4.50 (t, 4H), 6.٧5 (s, 2H).

Reusabity of the catalyst

The reusability of the Ce(salophen)/HY catalyst was examined in the synthesis of oxazoline derivatives under the optimized reaction conditions. After each run, the solid catalyst was easily recovered by simple filtration, thoroughly washed with ethanol, and dried at 120 °C and reused in a fresh reaction cycle. The results demonstrated excellent durability, with only a slight decrease in catalytic activity after five consecutive cycles (yields remained above 84 %) (Fig. 5). This can be attributed to partial blockage of the active sites by reaction residues rather than structural degradation of the catalyst. These findings confirm that Ce(salophen)/HY acts as a robust and recyclable heterogeneous catalyst, offering both high activity and stability, making it an efficient system for oxazoline synthesis reactions.

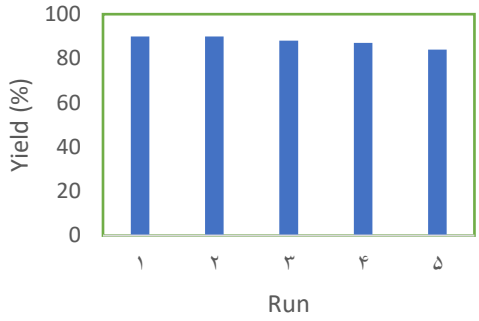

Fig. 5. The reusability of the catalyst under optimum conditions (model substrate: 4 chlorobenzonitrile, tem:140 °C, catalyst amount:0.15 mmol catalyst, time: 60 min)

A plausible reaction pathway for the formation of oxazoline derivatives is proposed based on the catalytic properties of the CSDY material. It seems the cerium centers coordinated with the salophen ligand act as Lewis-acidic sites that activate the nitrile group through coordination, thereby increasing the electrophilicity of the nitrile carbon. Subsequently, nucleophilic attack of the amino alcohol on the activated nitrile leads to the formation of an amidine-type intermediate. Intramolecular cyclization followed by proton transfer then affords the corresponding oxazoline ring. The confined environment of the HY zeolite and the presence of both Lewis and Brønsted acidic sites are expected to facilitate the activation of the nitrile group and promote the cyclization process under the applied reaction conditions.

Conclusions

In this paper, we reported the synthesis of Ce(salophen)/HY catalyst through the “ship-in-a-bottle” approach and used in the cyclization of ethanolamine with various nitriles to form oxazoline derivatives under relatively mild conditions. The optimized parameters were 140 °C, 0.15–0.20 mmol catalyst loading, and 4:1 amine: nitrile ratio, provided high yields across a rang

substrate. Moreover, the catalyst showed higher activity toward aromatic nitriles compared to aliphatic ones, mainly due to the enhanced electrophilicity of the C ≡ N bond in aryl systems and reducing the steric hinderance of their planar structures. Furthermore, the catalyst activity is preserved over several consecutive recycling runs, confirming its potential as an efficient, sustainable, and reusable heterogeneous catalyst for scalable oxazoline synthesis.

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