





An eco-friendly synthesis of polyhydroquinoline derivatives using MoO₃ promoted CeO₂-ZrO₂ solid heterogeneous catalyst

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Abstract

The molybdenum (8–20wt%) (MoO₃) promoted CeO₂-ZrO₂ (ceria-zirconia) solid solutions were prepared using precipitation and impregnation method, calcined at 500°C. The catalytic potency of synthesized materials were tested for the synthesis of polyhydroquinoline derivatives using a mixture of various aromatic aldehydes, dimedone, and ethyl acetoacetate and ammonium acetate in ethanol under reflux condition. Amazingly, it was observed that with increasing Mo wt% loading increases the excellent yield of product this may be due to acidic nature of MoO₃. Particularly, the 20wt% MoO₃ promoted CeO₂-ZrO₂ catalyst exhibited promising catalytic activity in terms of excellent yield of the products with short reaction time. The present methods significantly contributed as efficient and eco-friendly method for the synthesis of polyhydroquinoline derivatives.

Introduction

Molybdenum oxide (MoO₃) catalysts are very significant for industrial applications. The pure molybdenum oxide and in the form of supported molybdenum oxide is extensively used as a catalyst in various organic chemical transformations. The supported molybdenum oxide system is

active for great number of chemical reactions, such as hydrogenation of benzene [1] condensation of Anisole [2] partial oxidation of alcohols [3] dehydration [4] dehydrogenation [5] isomerization [6] cracking of hydrocarbons [7] Condensation [8]. The molybdenum oxide supported on SiO₂ can be used as a solid acid catalyst, with both the strong Bronsted and Lewis acidity [9], [10]. The catalytic potency of supported metal oxides governed by the degree of dispersion, structure and stability of supporting material. Thus, the catalytic activity and selectivity is associated to the type of active sites that depends on the type of supports, metal loading as well as on the synthesis technique. Usually, the supports are silica or alumina, but the considerable, interest is now devoted to other supports such as SnO₂ [11] ZrO₂ [12] TiO₂ [13] Nb₂O₅ [14] Ni/CeO₂-ZrO₂ [15].

Amongst the zirconia (Zr) based mixed oxides, the CeO₂-ZrO₂ composition has gained tremendous focus as an interesting catalytic material owing to its superior redox and oxygen storage/release properties [16]. The CeO₂-ZrO₂ mixed oxides is also widely used as a supporting material because due to their high thermal stability [17]. The combination of Zr cation with Ce cation alters the surface acid-base sites, where Zr⁴⁺ and Ce⁴⁺ ions acts as O²⁻ ions as Bronsted or Lewis base and Lewis acid sites. In addition to that, the cerium oxide and zirconium oxide have different lattice abilities the insertion of one oxide matrix into the other oxide matrix would be the origin of a generation of surface acidity as per Kungs Model [18]. Therefore, inspiring from these results and the potential catalytic applications of molybdenum oxide (MoO₃) we are reported the preparation of molybdenum (8–20wt%) MoO₃ promoted Ceria-Zirconia (CeO₂-ZrO₂) solid catalyst by precipitation and impregnation method and its application in the synthesis of polyhydroquinoline derivatives.

Polyhydroquinoline are important class of chemical compound possessing excellent biological and pharmacological activities such as calcium channel blockers [19] neurotropic [20] antitumor [21] antimicrobial [22] antiallergics [23] antibacterial [24] antioxidant agent [25] and many more. It is used as drug in the treatment of cardiovascular diseases [26] hypertension [27]. Because of excellent biological activities of these compounds, many synthetic strategies have been reported in the literature such as, Yb (OTF)₃ [28] Zinc Oxide [29] Ionic Liquid [30] TMSCI [31] CAN [1,32] Tin-dioxide [33] Bismuth Nitrate [34] Gadolinium Triflate [35] Carbon Nano Tubes[36] etc.

Many of these reported techniques was established for the synthesis of polyhydroquinoline derivatives, and have their own importance. However, many of these techniques are associated with some drawbacks, such as use of expensive reagents, dangerous solvents, longer reaction time and harsh reaction conditions. Thus, it is highly desirable to develop a more convenient, efficient and green route for the synthesis of polyhydroquinoline derivatives. Thus in turn, in continuation of our previous work [37], [38], [39] here we are reporting, the synthesis of polyhydroquinoline derivatives by using various aromatic aldehydes, dimedone, ethyl acetoacetate and ammonium acetate in the presence of MoO₃ promoted CeO₂-ZrO₂ as a catalyst in ethanol under reflux condition (Scheme 1).

Section snippets

Methods and materials

All chemicals and reagents were purchased from Aldrich and Rankem limited and used without further purification. The uncorrected m.p. of compounds was measured by using open capillary in paraffin bath. All chemicals were purchased from Aldrich and Rankem chemicals limited and used as such. ^1H NMR, (80MHz FT-NMR) spectrometer and CDCl_3 as a solvent used for spectral analysis and chemical shift recorded in units of δ (ppm) using tetramethylsilane (Me_4Si) as standard....

Preparation of pure $\text{CeO}_2\text{-ZrO}_2$ supports by simple co-precipitation method

The pure $\text{CeO}_2\text{-ZrO}_2$ (1:1)...

Results and discussions

We have previously reported the surface characterization of samples by XRD, FT-IR, and SEM and EDS techniques and its applications for synthesis of β -enaminones. [40] Here, we wish to describe another catalytic application of prepared materials for synthesis of polyhydroquinoline derivatives one of the biologically important class of chemical compounds, using MoO_3 loaded on $\text{CeO}_2\text{-ZrO}_2$ as solid heterogeneous catalyst....

Conclusions

The present work unveil an eco-friendly and efficient technique for the preparation of polyhydroquinoline derivatives. Among the 8, 10, 12, 15 and 20wt% MoO_3 promoted $\text{CeO}_2\text{-ZrO}_2$ catalyst; 20wt% MoO_3 promoted $\text{CeO}_2\text{-ZrO}_2$ catalyst possessed better catalytic potency for the condensation of numerous aromatic aldehydes, with electron donating and withdrawing groups. Catalytic potency of catalysts increased with molybdenum contents (8–20wt%) and the catalyst can be easily recovered and recycled. The...

CRedit authorship contribution statement

Sandip Rathod: Conceptualization, Investigation, Methodology and Writing - original draf. **Vinod Dhage:** Formal analysis, Writing - original draft. **Machhindra Lande:** Conceptualization, Supervision....

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal

relationships that could have appeared to influence the work reported in this paper....

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