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Original Article

Catalytic synthesis of Pyrano pyrimidine derivative using mixed transition metal tartrate complex: A step towards sustainable Chemistry

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Abstract

This research article studies the synthesis of pyrano[2,3-d] pyrimidine dione derivative by using Zn-doped Cobalt tartrate complex as a catalyst. The method used for the synthesis is simple, efficient and less time-consuming. Aldehydes, malononitrile, and barbituric acid are condensed using 1:1 ethanol as solvent and zinc-Doped cobalt Tartrate as catalyst. The catalyst used is economically favourable and reusable. So, the current method sheds new light on organic Chemistry, which can be further used in many disciplines like pharmacy and microbiology.

Keywords: *Pyrano [2,3-d] pyrimidine Dione, zinc-doped cobalt Tartrate*

Introduction

In recent years, significant attention has been directed toward developing new methods for synthesizing heterocyclic compounds, given their potential applications in the pharmaceutical and agricultural sectors. Among these, pyrano[2,3-d] pyrimidine dione derivatives have garnered particular interest due to their wide range of biological activities, including antimalarial, antihypertensive, cardiotonic, vasodilatory, bronchodilatory, antiallergic, herbicidal, antitumor, analgesic, and fungicidal properties[1-3].

This reaction typically proceeds rapidly due to its intermolecular characteristics, clean reaction conditions, and excellent atom economy[4-8]. Conducting the reaction under solvent-free conditions introduces an environmentally friendly approach that conserves both energy and resources. Such reactions represent a key aspect of green chemistry and are of considerable interest from both synthetic and economic perspectives. Compared to conventional reactions carried out in organic solvents, solvent-free processes offer several advantages, including reduced solvent waste and, in many cases, enhanced reaction rates.Multicomponent reactions (MCRs)[9–12] are processes in which three or more starting materials react together in a single step to form a product, with each component contributing to the final structure. In MCRs, the product is constructed through a sequence of elementary reactions occurring in a cascade manner, often without the need for intermediate isolation or purification steps. With this strategy in mind, we synthesized pyrano[2,3-d] pyrimidine dione derivatives via a tandem Knoevenagel–Michael cyclocondensation reaction involving malononitrile, aldehyde, and barbituric acid. The reaction was catalyzed by cobalt-doped iron tartrate, a water-soluble, recoverable, and highly efficient catalyst.

Experimentation

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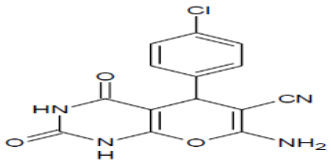
During the experimental phase, a range of analytical techniques was employed to characterize and confirm the structure of the synthesized pyrano[2,3-d] pyrimidine dione derivatives.

(Fourier Transform Infrared) Spectra was acquired using a Spectrum 400 spectrophotometer, with potassium bromide (KBr) employed as the medium. To further analyze the compound, NMR (H1 Nuclear Magnetic Resonance) spectra was recorded using a Bruker AVANCE NEO 500 MHz high-resolution NMR spectrometer. The sample was prepared in dimethyl sulfoxide (DMSO), with tetramethylsilane (TMS) added as an internal reference standard to ensure precision and consistency in the spectral data.All chemicals used in the synthesis were commercially available and were subjected to thorough purification using standard methods to ensure maximum purity before use in the experimental procedures.

Procedure for the Synthesis of Pyrano[2,3-d] pyrimidine dione

The synthesis of pyrano[2,3-d] pyrimidine dione was performed through a carefully optimized procedure. A precisely measured mixture consisting of barbituric acid, an aldehyde, malononitrile, and cobalt-doped zinc tartrate was combined in 10 mL of water solution. The reaction mixture was subjected to reflux at 75°C using an oil bath setup, enabling the multicomponent reaction to proceed efficiently.

The cobalt-doped zinc tartrate served as an effective catalyst, facilitating the tandem reaction and promoting the formation of the target heterocyclic compound. Upon completion of the reaction, the mixture was cooled, and the resulting crude product was purified by recrystallization using ethanol. This purification step was essential in obtaining the pyrano[2,3-d] pyrimidine dione in high purity, ensuring the structural integrity of the final product.



7-amino 5-(4-chloro phenyl)-2,4-dioxo-1,3,4, 5-tetrahydro 2*H*-pyrano[2,3-d] pyrimidine-6-carbonitrile

Analytical Data

1H-NMR (CDCl3,200 MHz) _ (ppm): 7.15–7.95 (m, 4H),
6.05 (s, 1H), 11.25 (s, 1H) 11.45 (s, 1H), 4.75 (s, 1H)
IR (KBr): 3362, 3390, 3259, 3187, 2149, 1717, 1637, 1519,
1440, 1347, 1278, 1191, 1096, 963, 868, 774 cm−1

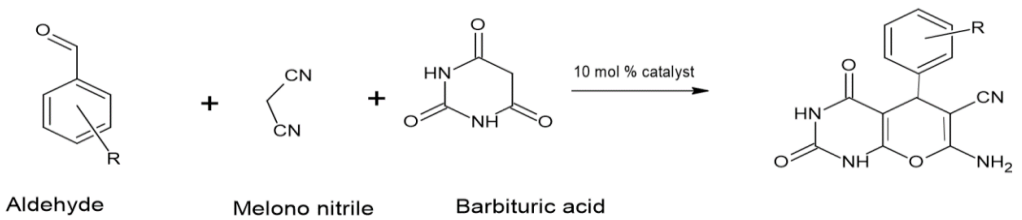
Results And Discussion

To obtain optimum efficiency with a catalyst, the quantity of 10 mole percent of zinc-doped cobalt tartrate at varying temperatures from 50 °C to 80 °C was used. Table 1 elucidates the subtle complexities of the reaction conditions to obtain suitable solvent.

Table 1: Selection of a proper solvent

Catalyst (mol%)	Solvent	Temperature (°C)	Time (Min.)	Yield (%)
10	H2O	75	40	88
10	Ethanol/H2O	75	40	80

1 mmol of each of barbituric acid, 4-chloro benzaldehyde, malononitrile,10 mol %Zn doped cobalt catalyst, then water is added and kept for reflux for about 40 min. to carry out the reaction.



Scheme: Synthesis of 7-amino 5-(4-chloro phenyl)-2,4-dioxo-1,3,4, 5-tetrahydro 2*H*-pyrano[2,3-d]pyrimidine-6-carbonitrile

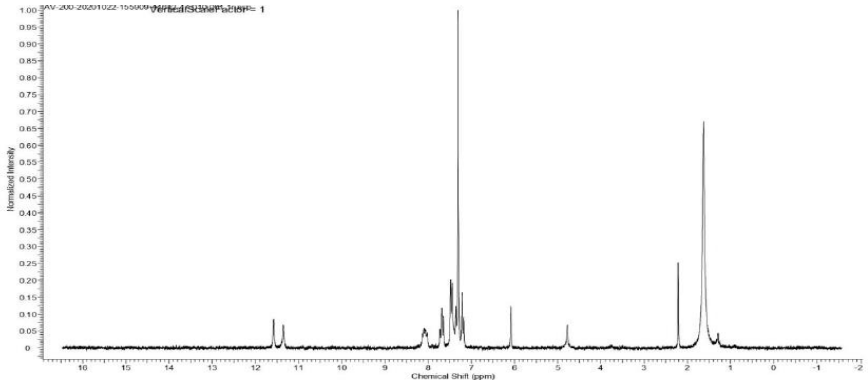


Figure 1. H1 NMR spectra of 7-amino 5-(4-chloro phenyl)-2,4-dioxo-1,3,4, tetrahydro 2*H*-pyrano[2,3-d] pyrimidine-6-carbonitrile

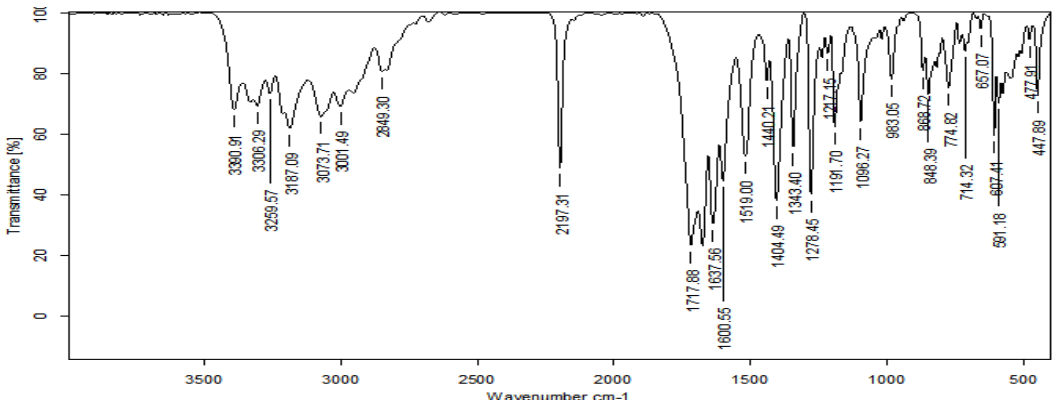


Figure 2. IR spectra of 7-amino 5-(4-chloro phenyl)-2,4-dioxo-1,3,4, 5-tetrahydro 2*H*-pyrano[2,3-d] pyrimidine-6-carbonitrile

Conclusion

We have demonstrated a green, efficient, and solvent-free method,environmental friendly method to synthesize pyrano[2,3-d] pyrimidine dione derivative by the tandem Knoevenagel-Michael cyclocondensation reaction. Further analysis is still required to study more properties of the catalyst as well as the product.

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Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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