International Journal of Scientific Research in Science and Technology
Available online at : www.ijsrst.com
OPEN ACCESS
Print ISSN: 2395-6011 | Online ISSN: 2395-602X
doi : https://doi.org/10.32628/IJSRST173876

$MnFe_2O_4$ Nanoparticle as a New and Magnetically Separable Nanocatalyst for Solvent-Free Synthesis of dihydropyrano[2,3-c] Pyrazole Derivatives

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ARTICLEINFO

Article History:
Accepted: 22 Jan 2024
Published : 29 Jan 2024

Publication Issue :
Volume 11, Issue 1
January-February-2024
Page Number :
318-326

ABSTRACT

This study focused on synthesising $MnFe_2O_4$ (Manganese Ferrite) nanoparticles using the sol-gel method. We then applied these nanoparticles as a catalyst for synthesising dihydropyrano[2,3-c]pyrazole derivatives at a specific temperature. The significant advantage of this process is that it requires only short reaction times and no solvents. Additionally, the crude pyranopyrazole derivatives can be purified through a simple recrystallization process. The catalyst is reusable, magnetically separable and maintains its activity even after five uses. The chemical integrity of the catalyst was confirmed through FT-IR, ^{1}H NMR, and ^{13}C NMR techniques.

Keywords: Solvent-free synthesis, Magnetically separable, Manganese Ferrite, Pyranopyrazole, Nanocatalyst

I. INTRODUCTION

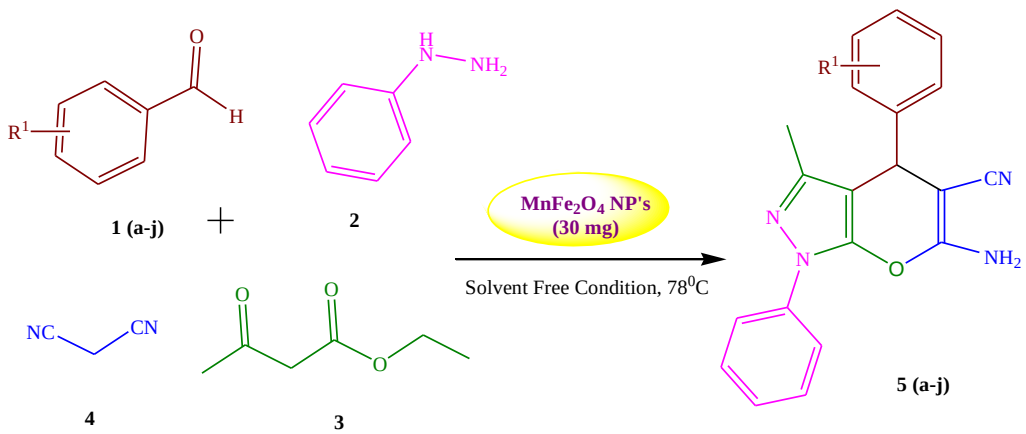
Multicomponent reactions (MCRs) are considered one of the best ways to synthesize heterocyclic compounds using simple and easily available materials. MCRs are highly efficient as they reduce the number of synthesis steps required to form complex compounds, which makes them time-saving and environmentally friendly. Therefore, there is a growing interest in designing new MCRs that follow the principles of green chemistry [1, 2]. Pyranopyrazole, which could be synthesized through a four-component reaction, is one of the most important types of oxygen–nitrogen heterocyclic compounds with lots of biological, pharmaceutical, and agrochemical applications. For instance, antimicrobial, antiplatelet, anti-inflammatory, antitumor, antibacterial, insecticidal, fungicidal, antitubercular agent, herbicidal, and inhibitor of human chk1 kinase, as well as UV absorber are some of the important activities of pyranopyrazole and its derivatives (Fig. 1). Because of the great importance and numerous utilizations of

pyranopyrazole and its derivatives, modifying the procedure for its synthesis are at the top of scientific research [3, 4]. Using heterogeneous nanocatalysts to synthesize pyranopyrazole has attracted great importance among previous methods on account of high efficiency.

Manganese and ferric oxide are commonly used metal oxides due to their practical advantages such as being readily available, low cost, less toxic, and easy to synthesize. In recent times, binary metal oxide photocatalysts have gained significant attention for their efficient catalytic activity and stability in removing organic pollutants from the air and sewage [5-6]. Spinel structures with the formula AB_2O_4 are well-known materials for their ability to incorporate various ions into their crystal structure. In the $MnFe_2O_4$ structure, Mn^{2+} and Fe^{3+} are distributed in tetrahedral and octahedral sites respectively, which is a typical spinel structure. The corresponding general formula is written as $A_{tet}B_{2oct}O_4$ [7]. The high activity of this spinel compound is due to the high amounts of Fe^{3+} species and compositional homogeneity. Magnetic nanoparticles possess unique physicochemical properties and have attracted the attention of various researchers [8-10]. Spinel ferrite compounds show great potential as green heterogeneous catalysts in various organic functional group transformations and as catalytic supports [11-14].

Pyrano[2,3-c]pyrazoles are a type of heterocycles that have great potential for use in the pharmaceutical field. They exhibit a wide range of biological activities such as anti-inflammatory [15], anticancer [16, 17], inhibition of human Chk1 kinase [18], antimicrobial [19], and also as biodegradable agrochemicals [20]. It has been reported that pyrazoles play a vital role as essential synthetic intermediates [21]. One of the methods used for their production involves the base-catalyzed cyclization of 4-aryliden-5-pyrazolone [22]. However, new approaches have been developed for the synthesis of pyrazoles, including processes based on β-cyclodextrin[23], cinchona alkaloid derivatives[24], $Sc(OTf)_3$ [25], triethylamine [26], water or ethanol media [27, 28], imidazole [29], CuO/ZrO_2 [30] and triethyl benzyl ammonium chloride [31].

As part of our ongoing research into the development and use of nanoparticles and their application [32-35], we have decided to use these nanomaterials as a nanocatalyst in organic transformation. Hence, We used one of the new nanocatalysts for the synthesis of dihydropyrano[2,3-c]pyrazole derivatives under solvent-free conditions at specific temperature conditions. (**Scheme-1**).



Scheme-1: Schematic representation of Manganese ferrite catalysed synthesis of dihydropyrano[2,3-c]pyrazole derivatives

II. METHODS AND MATERIAL

A) Materials:

All chemicals were purchased from Sigma-Aldrich and SD-Fine Chemical in Mumbai, India, and used without purification. All the aqueous solutions were prepared in distilled water.

B) Characterisation Methods:

Fourier transform infrared (FT-IR) spectra of dihydropyrano[2,3-c]pyrazole derivatives were recorded on the Shimadzu IR spectrophotometer. ^{1}H and ^{13}C NMR spectroscopy performed by Bruker Advance Neo 500 MHz NMR Spectrophotometer, SAIF, Chandigarh.

C) Synthesis of magnetic separable $MnFe_2O_4$ nanocatalyst:

The sol-gel method was employed to synthesize nanoparticles of manganese ferric oxide ($MnFe_2O_4$). The procedure involved dissolving 0.25 M of manganese chloride and 0.25 M of ferric chloride separately in 100 ml of distilled water. The solubility of manganese chloride and ferric chloride in water was improved by adding 0.1 N hydrochloric acid. Two solutions were mixed together with constant stirring. After the solutions were mixed, 1 gram of citric acid solution was added as a surfactant. The pH of the solution was then adjusted to be 8-8.5 (basic) using 3-3.5 ml of NH_4OH solution. The mixture formed a precipitate which was stirred constantly for an hour at room temperature. The mixture was then filtered using Whatman filter paper No. 42 and washed with deionized water 2-3 times. Finally, the obtained product was dried in an oven at 100°C for 2 hours. After drying, the material was calcinated for 3 hours at 500°C. The final product was a black precipitate, which was used as a magnificent for the synthesis of dihydropyrano[2,3-c]pyrazole derivatives.

D) Synthesis of dihydropyrano[2,3-c]pyrazole derivatives:

Magnetic separable $MnFe_2O_4$ nanocatalyst as catalyst (30 mg) was added and mixed to a mixture of aromatic aldehyde (1 mmol), malononitrile (1 mmol), ethyl acetoacetate (1 mmol) and phenylhydrazine (1 mmol) under solvent-free conditions at 78°C temperature for the suitable time. After completion of the reaction, which was identified by TLC (n-hexane/ethyl acetate: 8:2), the magnetically separable nanocatalyst was separated by external magnetic material and washed with aqueous ethanol. After confirmation of product formation, recrystallised from ethanol which resulted in precipitation of the desired dihydropyrano[2,3-c]pyrazole derivatives. All of the obtained product(s) are known and were identified by comparison of their physical data with those reported in the literature (see Table 1). Some of the synthesized compounds were characterized by IR and NMR data. Given below are spectral data for some selected pyranopyrazole derivatives.

E) Spectral data analysis for compounds:

6-Amino-3-methyl-4-(4-nitrophenyl)-1-phenyl-1,4 dihydropyrano[2,3-c]pyrazole-5-carbonitrile ***(5c)****:*

^{1}H NMR (500 MHz, DMSO-d6): δ = 8.21 (d, J = 8.8 Hz, 2H), 7.72 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 8.8 Hz, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.21 (s, NH2), 7.16 (t, J = 6.4, 1H), 4.91 (s, 1H), 2.35 (s, 3H) ppm. **^{13}C NMR (500 MHz, DMSO-d6):** δ = 168.85, 159.62, 150.28, 145.80, 144.33, 142.49, 133.49, 128.80, 125.87, 123.91, 120.49, 97.43, 67.69, 33.13, 13.90 ppm. **FT-IR (KBr):** $\bar{\nu}$ = 3227, 2921, 2193, 1685, 1566, 1506, 1415, 1347, 1267, 1105, 847 cm^{-1}.

III.RESULTS AND DISCUSSION

The study investigated the impact of $MnFe_2O_4$ magnetic nanoparticle catalyst with different concentrations (10-30 mg) on the one pot four-component reaction of substituted benzaldehyde, phenylhydrazine with Malononitrile and ethyl acetoacetate under various conditions (**Table 1**). The

results clearly indicate that the best yield of 6-amino-3-methyl-1, 4-diphenyl-1, 4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (5a) was achieved by carrying out the reaction in the presence of 10 mol% of $MnFe_2O_4$ magnetic nanoparticle without solvent. (**Table 1, run no. 8**). The yield increased as the catalyst load was raised from 10 to 30 mg, while with the use of larger amounts of the catalyst (35 mol %), the yield of the product remained the same.

Table 1:

Synthesis of dihydropyrano[2,3-c]pyrazole (**5a**) using different conditions with different concentrations of Manganese ferrite nanocatalyst at 78°C temperature.

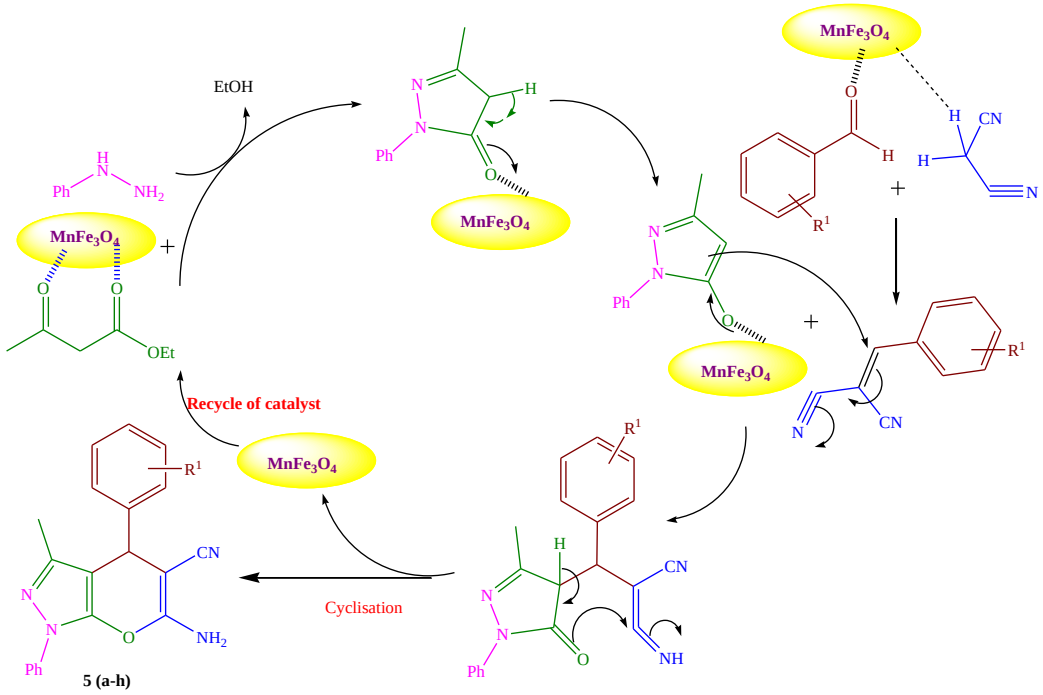
Sr. No.	Solvents	Catalyst (mg)	Time (min)	Yield (5a)
1.	DCM	10	68	58
2.	CH_3CN	10	74	64
3.	THF	10	46	67
4.	H_2O	10	49	70
5.	EtOH	10	40	74
6.	Solvent-free	10	37	80
7.	Solvent-free	20	30	90
8.	Solvent-free	30	22	98
9.	Solvent-free	40	21	98

After optimizing the reaction conditions, we aimed to investigate the efficiency and scope of a procedure that resulted in the production of numerous dihydropyrano[2,3-c]pyrazole derivatives. This was achieved through a one-pot four-component condensation reaction involving substituted benzaldehyde, phenylhydrazine, Malononitrile, and ethyl acetoacetate. The reaction was catalysed by a small amount of $MnFe_2O_4$ magnetic nanoparticle catalyst and carried out under solvent-free conditions. The results are displayed in **Table 2**. The impact of substituents on the aromatic ring was evaluated in terms of yields under these reaction conditions. Both types of aromatic aldehydes, including those with electron-releasing and electron-withdrawing substituents on their aromatic ring, produced the desired products in high to excellent yields in a short reaction time. The reaction time of aromatic aldehydes with electron-withdrawing groups was faster than those with electron-donating groups. Moreover, the recyclability and reusability of the catalyst were also considered in the condensation of benzaldehyde, phenylhydrazine, Malononitrile, and ethyl acetoacetate. Similarly, the magnetic nanoparticle catalyst was separated and reused for an alternative reaction.

TABLE 2:

Synthesis of numerous dihydropyrano[2,3-c]pyrazole derivatives using 30 mg of manganese ferrite nanocatalyst with solvent-free reaction conditions at 78°c temperature.

Sr. No.	Aldehyde	Product (5a-5h)	Time (min)	Yield (%)	Melting Points (°C)	
					Found	Reported
1.	Benzaldehyde	5a	21	98	170-173	(169-171) [36]
2.	4-Methoxybenzaldehyde	5b	23	91	173-175	(174-176) [37]
3.	4-Nitrobenzaldehyde	5c	14	95	196-198	(197-198) [38]
4.	2-Chlorobenzaldehyde	5d	12	98	138-140	(140-142) [36]
5.	4-Chlorobenzaldehyde	5e	17	95	173-176	(175-178) [36]
6.	4-Hydroxybenzaldehyde	5f	28	89	211-214	(213-215) [39]
7.	4-Methylbenzaldehyde	5g	39	85	168-170	(170-171) [38]
8.	4-Bromobenzaldehyde	5h	18	93	181-185	182–184 [40]



Scheme 2: The probable mechanism for the synthesis of dihydropyrano-[2,3-c]-pyrazole derivatives using the magnetically separable $MnFe_2O_4$ catalyst

In addition to performing a one-pot four-component reaction of substituted benzaldehyde, phenylhydrazine with Malononitrile and ethyl acetoacetate, we isolated the magnetic nanoparticle catalyst using magnetic separating material upon completion of the reaction and studied the feasibility of reusing and recycling the catalyst for several runs. The results, illustrated in **Table 3**, demonstrate that the magnetically separable $MnFe_2O_4$ catalyst could be recycled and reused for up to five runs without significant loss of its catalytic activity. The results are summarized in **Table 3**.

TABLE 3:

Reusability of the magnetically separable $MnFe_2O_4$ catalyst for the Synthesis of dihydropyrano[2,3-c]pyrazole derivatives

Run of typical reaction **(5a)**	1	2	3	4	5
Isolated % of yields (%)	98	97	97	95	94

IV. CONCLUSION

In conclusion, firstly, we have synthesised $MnFe_2O_4$ magnetic nanoparticles using the sol-gel method via the eco-friendly method. A new and efficient protocol for the solvent-free one-pot four-component synthesis of dihydropyrano[2,3-c]pyrazole derivatives at 78°C in the presence of the magnetically separable $MnFe_2O_4$ catalyst. Its catalytic investigation was done in the organic transformation of the synthesis of one-pot four-component dihydropyrano[2,3-c]pyrazole derivatives under solvent-free conditions at 78°C. One of the noteworthy advantages of this study is the easy product isolation, cleaner reaction profile, eco-friendliness, high yield, short reaction time, and recycling and reusability of the $MnFe_2O_4$ magnetic nanocatalyst.

V. ACKNOWLEDGEMENT

We thank the Principal, Shri Shivaji College of Arts, Commerce and Science, Akola, for providing the Research Laboratory facility and the Sophisticated

Analytical Instrumentation Facility (SAIF) and CIL, Chandigarh, for their characterisation facility.

VI. CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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Cite this article as :

Santosh B. Gaikwad, Kishore Puri, "MnFe2O4 nanoparticle as a new and magnetically separable nanocatalyst for solvent-free synthesis of dihydropyrano [2,3-c]pyrazole derivatives", International Journal of Scientific Research in Science and Technology (IJSRST), Online ISSN : 2395-602X, Print ISSN : 2395-6011, Volume 11 Issue 1, pp. 318-326, January-February 2024. Available at doi : https://doi.org/10.32628/IJSRST173876

Journal URL : https://ijsrst.com/IJSRST173876