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Solvent-free synthesis of dihydropyrimidinone derivatives catalyzed by $NiCoFe_2O_4$ nanoparticles under thermal condition

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Original Research

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Abstract:

This research offered the preparation of nickel cobalt ferrite nanoparticles ($NiCoFe_2O_4$ NPs) as a nanocatalyst and applied this catalyst successfully for the synthesis of 1,4-dihydropyrimidinone derivatives under solvent-free and conventional heating. We could recover this catalyst five times and reuse it in the reaction. In addition, this catalyst was characterized by using Fourier transform infrared (FT-IR), X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and vibrating sample magnetometer (VSM) techniques. Also, the purification of organic products was done by crystallization and identified by melting point, FT-IR, and hydrogen nuclear magnetic resonance (^{1}H NMR) analyses.

Keywords: Aromatic aldehydes; Nanocatalyst; Thermal condition; Solvent-free; 1,4-Dihydropyrimidinone

1. Introduction

Multicomponent reactions (MCRs) are generally defined as reactions in which three or more starting materials react to form a product, where basically all or most of the atoms contribute to the newly formed product [1]. Their usefulness can be rationalized by multiple advantages of MCRs over traditional multistep sequential assembly of target compounds. In MCRs, a molecule is assembled in one convergent chemical step in one pot by simply mixing the corresponding starting materials, which is different from traditional ways of synthesizing a target molecule over multiple sequential steps. At the same time, considerably complex molecules can be assembled by MCRs. This has considerable advantages as it saves precious time and drastically reduces effort [1–4].

Dihydropyrimidinones have garnered significant interest from the scientific community due to their extensive pharmacological actions and inherent biological characteristics [5]. These substances are known for their diverse therapeutic effects, such as antitumor, antibacterial, antiviral, and anti-inflammatory capabilities. There are several methods for the synthesis of dihydropyrimidinones [6]. The first report on the Biginelli reaction is still an important reaction in 1893 for the synthesis of these heterocyclic compounds [7]. Steel and co-workers prepared dihydropyrimidinones using $BF_3.OEt_2$ [8]. Peng and co-workers synthesized dihydropyrimidinones using ionic liquid [9]. Bus and co-workers also prepared dihydropyrimidinones with para-toluene sulfonic acid (*P*-TSA) [10]. The other catalysts that have been applied for the synthesis of the dihydropyrimidinones are such as; TsIL [11], $Zr(H_2PO_4)$ [12], CTAB CH_3 [13], $H_6GeW_{10}V_2O_{40}.22H_2O$ [14], MCM-41-APS-PMDA-$NHSO_3H$ [15], $Sn(NO_2)_3.6H_2O$ [16], [H-NMP][CH_3SO_3]- [17], $AlCl_3.6H_2O$ [18], $ClCH_2COOH$ [19], ZnO/ BMI.BF_6 [20], $ZnCl_2$ [21], Granite [22], oxalic acid [23], γ-Al_2O_3/ BF_3/ Fe_3O_4 [24], lanthanum oxide [25], Copper(II) sulfamate [26], In(OTf) [27], $InBr_3$ [28], ruthenium (III) chloride [29] and lactic acid [30].

In recent years, magnetic nanoparticles have attracted the

attention of many researchers due to some physical and chemical properties. One of the characteristics of these nanoparticles is their high ability to absorb organic or inorganic compounds. These compounds, due to their large surface area compared to their bulk form, have significant features in the fields of catalysis [31–34].

In this research, we hope to report a simple, rapid, and highly effective method for the synthesis of 1, 4-dihyropyrimidinones through condensation of aldehyde, urea or thiourea and ethyl acetoacetate using $NiCoFe_2O_4$ NPs as nanocatalyst at conventional heating under solvent-free condition. The high efficiency of this reaction and the purity of the obtained products in a relatively short reaction time, high yield products, mild conditions, and the ability to recycle the catalyst repeatedly are among the advantages of this protocol.

2. Experimental section

2.1 Apparatus

All chemicals and reagents were purchased from Fluka and Merck Chemical Companies in high purity and utilized as received without further purification. The purity determination of the starting materials and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company). The infrared (IR) spectra were recorded using KBr pellets on both a Perkin Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. The proton nuclear magnetic resonance (^{1}H NMR) spectra were acquired in DMSO-d_6 on a Bruker DRX-400 spectrometer, using tetramethylsilane (TMS) as the internal standard. The crystalline structures of nanoparticles were analyzed via X-ray powder diffraction (XRD) on a Philips Xpert diffractometer using Cu-Kα radiation ($\lambda = 0.154056$ nm), scanning at 20°/min over a range of 10° to 100° (2θ). The elemental composition of the nanoparticles was determined by energy-dispersive X-ray (EDX) spectroscopy on a Zeiss Pl GMa vp instrument. Magnetic measurements of the nanoparticles were conducted at 300 K using a Quantum Design PPMS-9T vibrating sample magnetometer (VSM) at the University of Kashan, Iran. The morphology of the nanoparticles was examined by scanning electron microscopy (SEM) on a KYKYEM-3200 microscope. Melting points were measured using a Yanagimoto micro melting point apparatus and were reported without correction.

2.2 General procedure for preparation of $NiCoFe_2O_4$ nanoparticles

In accordance with the literature [35], a solution of 100 mL HCl 1.2 M (4.37 mg), 0.27 mg (1 mmol) of $FeCl_3.6H_2O$, 3.24 mg (0.025 mmol) of $NiCl_2$, and 3.24 mg (0.025 mmol) of $CoCl_2$ were added. This mixture was subjected to ultrasonic irradiation for 30 minutes. Subsequently, 150 mL of NaOH 1.25 M (7.5 g) was introduced, followed by about 5 minutes of exposure to nitrogen gas. The reaction mixture was then heated to 80 °C for 2 hours. Afterward, the resulting black precipitate was repeatedly washed with water and dried in an oven at 80 °C.

2.3 General procedure for synthesis of 1,4-dihydropyrimidinones

In the synthetic process, the aromatic aldehyde (1 mmol) and 0.13 mg (1 mmol) ethyl acetoacetate, along with 1.5 mmol of the urea, were combined with 0.04 g of $NiCoFe_2O_4$ nanoparticles as catalyst. The mixture was then agitated at 110 °C in a solvent-free environment for the necessary duration. The advancement of the reaction was monitored using thin-layer chromatography (TLC). After the reaction concluded, the mixture was allowed to return to ambient temperature and then diluted with water. Following filtration, the resultant substances were refined through recrystallization using ethanol. The purified compounds were then identified and characterized by their spectroscopic profiles, including the IR ^{1}H NMR data, and their melting points were verified against those of known standards in the literature [36–42].

Selected spectral data for dihydropyrimidinone derivatives

Ethoxycarbonyl-4-(4-phenyl)-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4a) White solid; Yield: 95%, MF: $C_{14}H_{16}N_2O_3$ MW: 260.29 mp: 202 – 204 °C, mp (Lit. [35]: 201 – 203 °C); IR (KBr) ν (cm^{-1}): 3243, 3114, 2977, 1723, 1711, 1646, 1421, 1222, 1091. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 1.08 (t, J = 13.2 Hz, 3H), 2.24 (s, 3H), 3.99 (q, 2H, CH_2), 5.14 (s, 1H), 7.28 – 7.30 (m, 5H, ArH), 7.73 (1H, NH), 9.19 (s, 1H, NH).

Ethoxycarbonyl-4-[(4-methyl) phenyl]-6-methyl-3,4-dihydropyrimidine-2(1H)-one (4b) Yellow solid, Yield: 94%, MF: $C_{15}H_{18}N_2O_3$ MW: 274.13 mp: 211 – 213 °C, mp (Lit. [36]: 215 – 216 °C); IR (KBr) ν (cm^{-1}): 3260, 2371, 1736, 1485, 1219, 1079. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 1.08 (t, J = 7.2 Hz, 3H, CH_3), 2.22 (s, 3H, CH_3), 2.24 (s, 3H, CH_3), 3.95 (q, 2H, CH_2), 5.08 (s, 1H, CH), 7.10 (q, 4H, ArH), 7.68 (s, 1H, NH), 9.15 (s, 1H, NH).

3. Results and discussion

3.1 Preparation and characterization of catalyst

The $NiCoFe_2O_4$ nanoparticles (NPs) were synthesized following a method outlined in reported literature [43], involving the reaction of $FeCl_3.6H_2O$, $NiCl_2$, and $CoCl_2$, with NaOH and HCl under the N_2 atmosphere as depicted in Fig. 1.

The prepared nanocatalyst underwent various analytical evaluations including FT-IR, XRD, SEM, EDX, and VSM. The FT-IR spectrum of $NiCoFe_2O_4$, presented in Fig. 2, is typically shown the O-H bond's stretching vibration associated with the hydroxyl groups in the range of 3200 – 3600 cm^{-1}, with the bending vibration of OH observed at 3358 cm^{-1}. Additionally, the faint peaks indicative of the metal-oxygen bonds, Fe-O and Co-O, were identified as broad bands around 669 and 580 cm^{-1}.

The XRD patterns are illustrated in Fig. 3 and aligned with the standard $NiCoFe_2O_4$ XRD profile with an average crystallite size of 34 nm calculated by using Scherrer's equation. The SEM analysis, shown in Fig. 4, is revealed the morphology of the $NiCoFe_2O_4$ NPs to be predominantly spherical-like particles with an average size of approximately 35 nm.

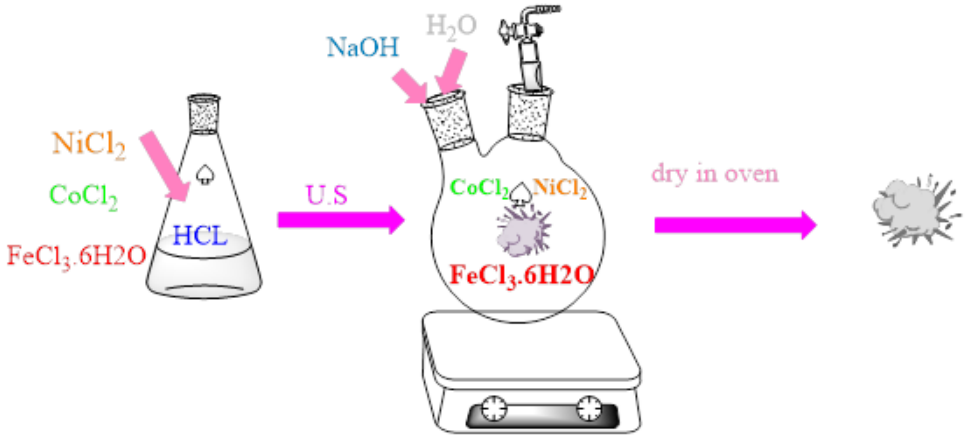


Figure 1. Preparation of $NiCoFe_2O_4$ catalyst.

Also, the EDX analysis confirmed the catalyst's composition, as displayed in Fig. 5. According to this Figure, the presence of peaks of iron, oxygen, cobalt, and nickel atoms is confirmed the existence of these elements in the nanocatalyst. Also, the percentage of these elements in nanocatalyst is determined the iron 61.8%, oxygen 29.6%, cobalt 8.5%, and nickel 0.1%.

The VSM results, depicted in Fig. 6, demonstrated an increase in magnetization post-oven treatment, confirming the magnetic properties of the $NiCoFe_2O_4$ NPs.

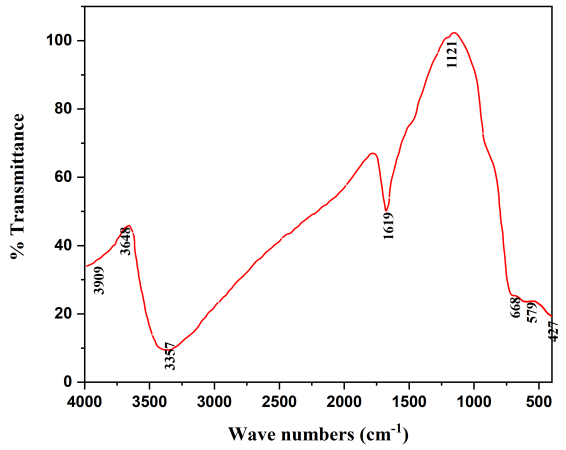


Figure 2. The FT-IR spectrum of $NiCoFe_2O_4$ catalyst.

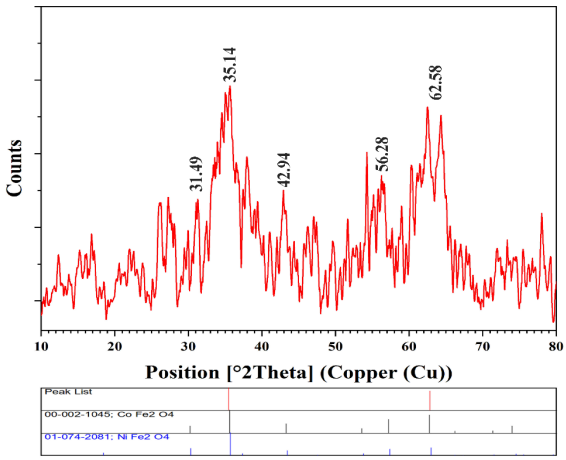


Figure 3. The XRD patterns of $NiCoFe_2O_4$ catalyst.

Investigation of catalytic activity

Optimization of the catalyst amount

In a typical experimental procedure, the condensation reaction occurred between 1 mmol ethyl acetoacetate, 1 mmol p-nitrobenzaldehyde, and 1.5 mmol urea in the presence of different amounts of catalyst and heated at 110 °C under solvent-free conditions as a model reaction (Scheme 1). Initially, the reaction was carried out in the absence of the catalyst, but no product was obtained. The highest product yield was obtained by using 0.04 g of catalyst (Table 1,

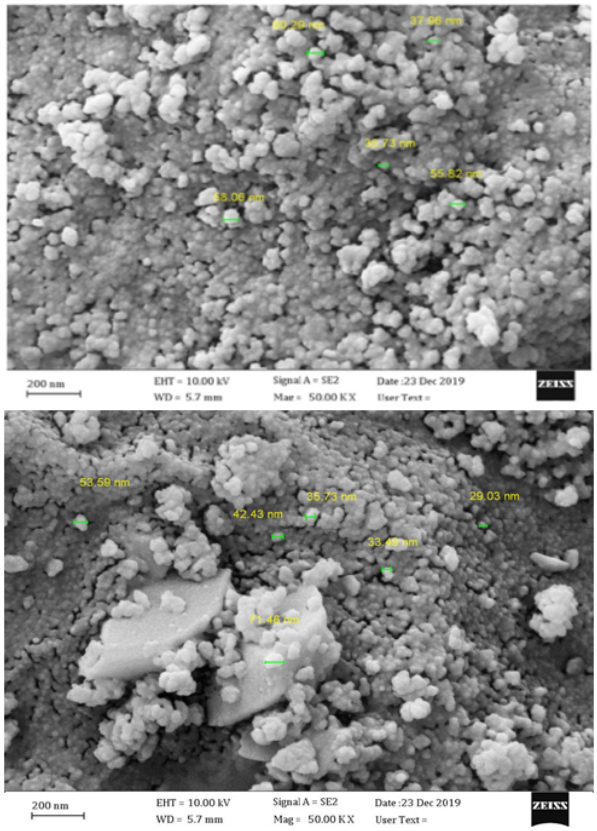


Figure 4. The SEM images of $NiCoFe_2O_4$ catalyst.

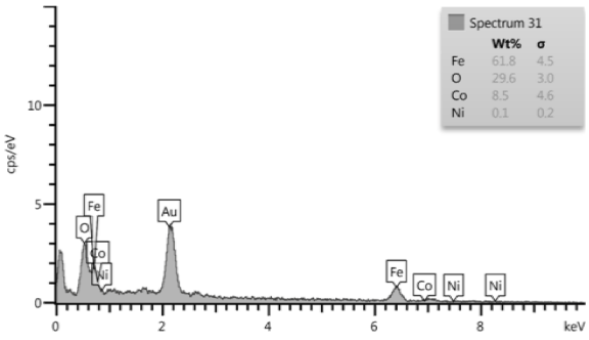


Figure 5. Electron diffraction X-ray (EDX) analysis of $NiCoFe_2O_4$ NPs.

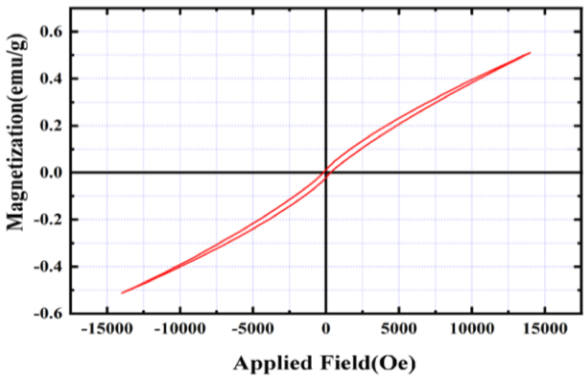
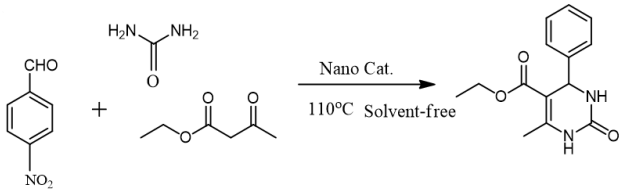


Figure 6. The vibration of magnetization (VSM) for $NiCoFe_2O_4$ NPs.

entry 4). Using a lower amount of the catalyst resulted in a lower yield, while a higher amount of the catalyst did not affect the reaction yield.

Subsequently, the impact of different temperatures on the reaction was examined (refer to Table 2). It was observed that conducting the reaction at 110 °C resulted in the synthesis of the product with the highest yield and in the shortest amount of time compared to the other temperatures tested (see Table 2, entry 6).



Scheme 1. Synthesis of dihydropyrimidinone under solvent-free condition.

Based on the optimized reaction conditions, a variety of the aryl aldehydes were reacted with ethyl acetoacetate and urea for the synthesis of dihydropyrimidinone derivatives (Table 3). The products from aromatic aldehydes that having electron withdrawing group obtained in the best yield, while the aldehydes having electron donating group obtained the reduced yield.

Therefore, the different derivatives of dihydropyrimidinones were synthesized at the temperature of 110 °C in the yields about 83 – 98% and the reaction times about 7 – 14 min. The structure of the obtained products was confirmed by FT-IR and ^{1}H NMR analyses. According the Table 3, the withdrawing group such as NO_2 group have the highest efficiency and the lowest time (Table 3, 4f).

A comparison of the present work with the previously reported works in literature was investigated. For this purpose, the reaction of aldehyde (1 mmol), ethylacetoacetate (1 mmol), and urea (1.5 mmol) as a model reaction

Table 1. Effect of different amounts of catalyst on the synthesis of dihydropyrimidinone under solvent-free condition.

Entry	Catalyst amount (g)	Time (min)	Yield (%)[b]
1	-	100	-
2	0.02	30	50
3	0.03	15	60
4	0.04	4	98
5	0.05	4	98
6	0.06	4	98

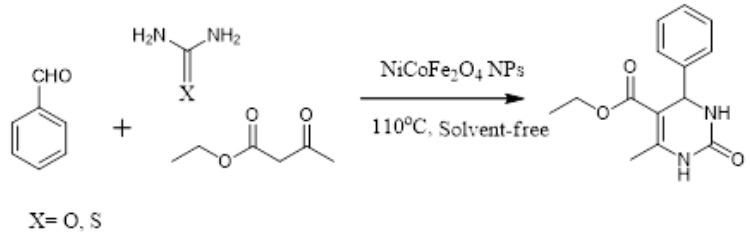
a) The reaction conditions: ethyl acetoacetate (1 mmol), 4-nitrobenzaldehyde (1 mmol) and urea (1.5 mmol) using different amounts of catalyst under solvent-free condition.
b) Isolated yield.

Table 2. Influence of the various temperatures on the reaction for the synthesis of dihydropyrimidinone,[a].

Entry	Time (min)	Temp. (°C)	Yield (%)[b]
1	25	65	60
2	25	70	65
3	20	80	70
4	15	90	75
5	10	100	83
6	4	110	98
7	4	115	98

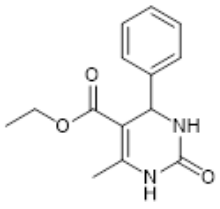
a) General reaction conditions: ethyl acetoacetate (1 mmol), 4-nitrobenzaldehyde (1 mmol) and urea (1.5 mmol) with 0.04 g catalyst under solvent-free condition.
b) Isolated yield.

Table 3. Synthesis of different dihydropyrimidinones catalyzed by $NiCoFe_2O_4$ NPs under solvent-free condition.

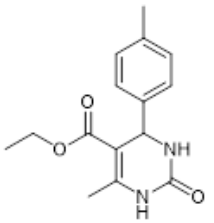


X= O, S

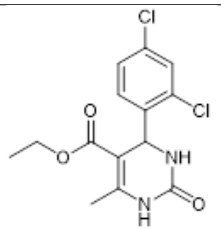
4a-l, **Yield (%)**, **Time (min)**



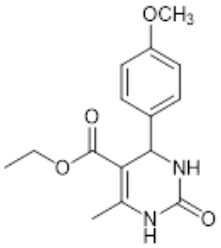
4a: R = -H, X = O, 93%, 6 min



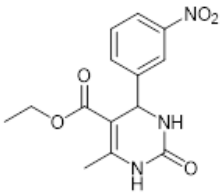
4b: R = 4-Me, X = O, 93%, 6 min



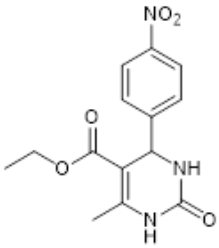
4c: R = 2,4-Cl, X = O, 87%, 7 min



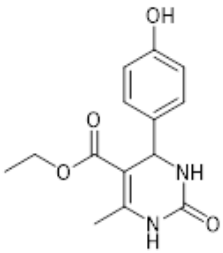
4d: -OMe, X = O, 89%, 8 min



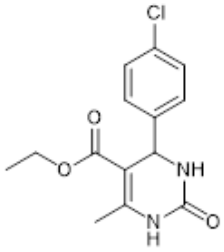
4e: -3NO_2, X = O, 96%, 5 min



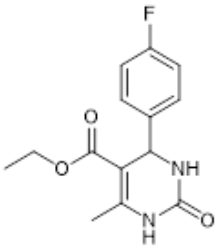
4f: -4NO_2, X = O, 98%, 4 min



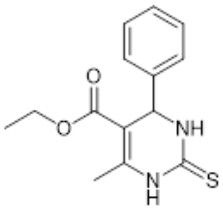
4g: -4OH, X = O, 90%, 7 min



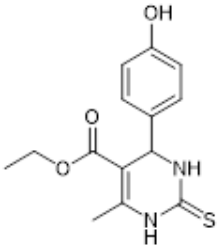
4h: -4Cl, X = O, 90%, 6 min



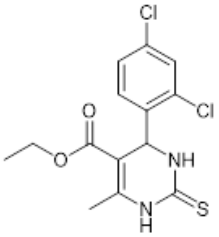
4i: -4F, X = O, 94%, 6 min



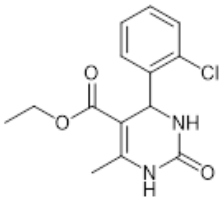
4j: R = -H, X = S, 85%, 7 min



4k: R = -OH, X = S, 83%, 9 min



4l: -2,4-Cl, X = S, 85%, 7 min



4m: -2Cl, X = O, 92%, 9 min

[a] Reaction condition: Aldehyde (1 mmol), ethylacetoacetate (1 mmol), urea (1.5 mmol), $NiCoFe_2O_4$ NPs (0.04 g), 110 °C, solvent-free.
[b] Isolated yield.

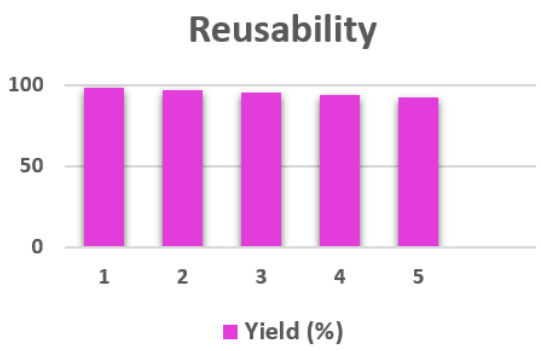


Figure 7. Reusability of catalyst for synthesis of dihydropyrimidinones.

were carried out, and the results are summarized in Table 4. The study discovered that the current approach using $NiCoFe_2O_4$ NPs as a catalyst has a simple synthetic method that is effective, applicable, and comparable to many catalytic systems for the synthesis of dihydropyrimidinones (entry 5 vs entries 1–4 in Table 4).

Reusability of the catalyst

In order to assess the catalyst's reusability, the $NiCoFe_2O_4$ catalyst was magnetically decoupled from the reaction mixture upon the model reaction's completion. The isolated catalyst underwent multiple washes with ethanol and acetone, followed by drying at 50 °C for 8 hours. It was then redeployed for subsequent reactions. This process demonstrated that the catalyst maintained its efficacy for a minimum of five cycles, with no significant loss in catalytic activity, as illustrated in Fig. 7.

Fig. 8 (a) shows the XRD pattern of the recovered catalyst after five runs. No change has been observed in the structure of the catalyst after five runs of recovery and reuse indicating the strength and stability of nanoparticles. Also, the VSM of the recovered catalyst (Fig. 8 (b)) was provided after undergoing five cycles. The spectrum of the recovered catalyst was not different from the original sample, so it can be proved that the catalyst remains unchanged in the molecular structure after five runs of recovery.

3.2 Proposed reaction mechanism

The plausible reaction mechanism for the synthesis of dihydropyrimidinones is shown in Scheme 2. At first, the oxygen atom of the aldehyde carbonyl group is activated in the presence of a nanocatalyst as a Lewis acid, and the

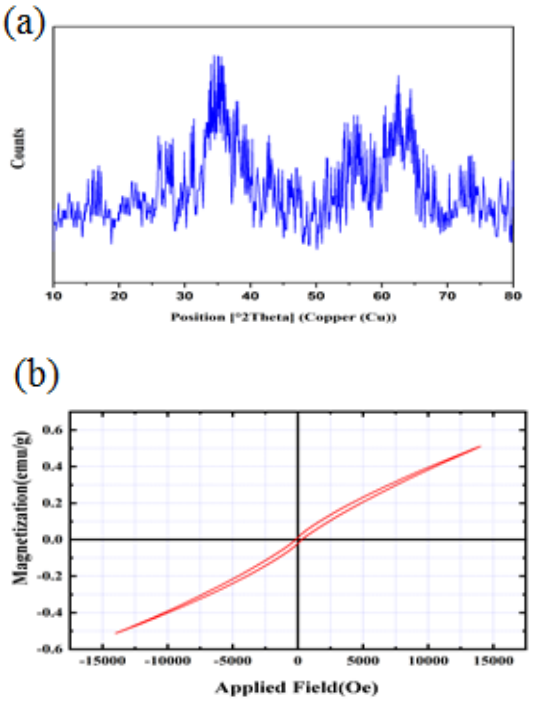


Figure 8. The XRD pattern (a) and VSM diagram (b) of the $NiCoFe_2O_4$ catalyst after five runs.

nucleophilic attack of urea to aldehyde takes place to obtain intermediate (I). In the next step, by eliminating the water, the intermediate (II) is formed. Then, the intermediate (II) reacts with ethyl acetoacetate, and the intermediate (III) is produced. Finally, dihydropyrimidinone is obtained as a target product through cyclization, which removes the second water molecule.

4. Conclusion

In this research, we have presented a simple, effective, and available protocol for the synthesis of dihydropyrimidinones under solvent-free conditions. Then, the nano nickel cobalt ferrite was used as a good catalyst with high activity. It was obtained as a pure product with high yields and short reaction times without any exposure and toxic solvent. The catalyst was easily separated by an external magnet and was reusable for up to five cycles. This method tends to have noteworthy advantages such as solvent-free condition, highly stable, much short reaction time, and inexpensive and echo-friendly catalyst. On the other hand, the workup and the purification of the products are simple.

Table 4. Comparison of the present work with other previously reported works.

Entry	Condition	Time (min)	Yield (%)	Ref.
1	Nano ZnO, 5 mol%, H_2O, 120 – 140 °C	720	67	[44]
2	$[Al(H_2O)_6]BF_4$, MeCN	1200	84	[45]
3	Al-MCM-41, 80 °C, Solvent-free	15	84	[46]
4	MCM-41-APS-PMDA-NHSO3H, 80 °C	50	80	[47]
5	$NiCoFe_2O_4$, 110 °C, Solvent-free	5	98	This work

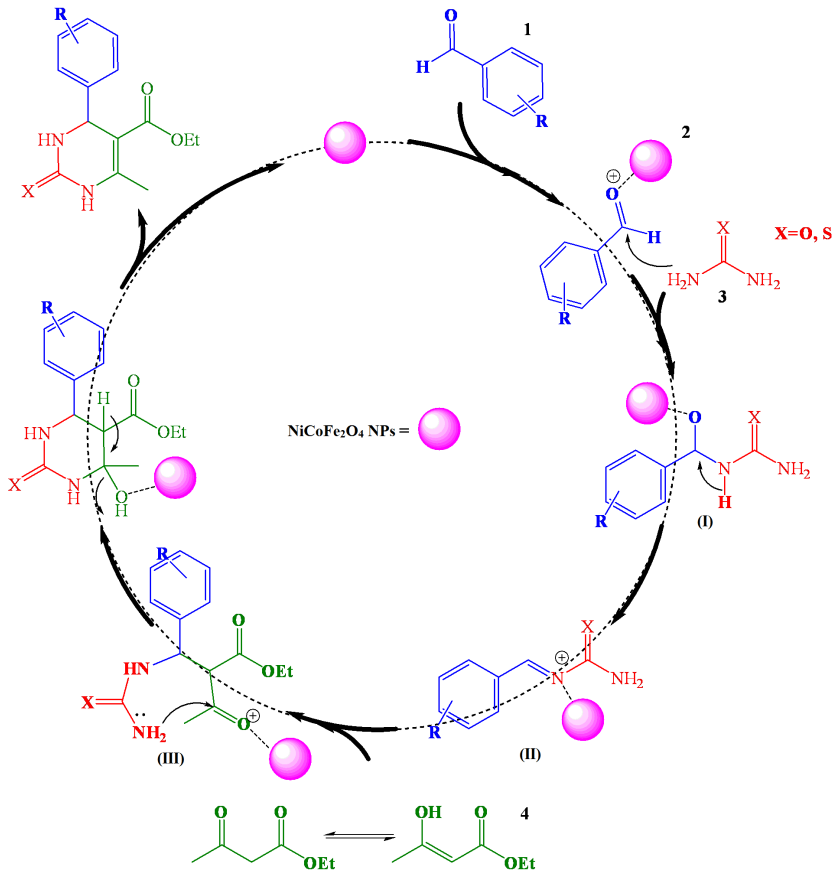


Figure 9. Reaction mechanism of dihydropyrimidinones preparation.

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Authors contributions

Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Conflict of interests

The author 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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