

One-pot three-component synthesis of tetrahydrobenzo[b]pyrans in the presence of $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles under microwave irradiation in solvent-free conditions

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ABSTRACT

$\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles using Arabic gel (AG) as a reducing and stabilizing agent was prepared by the sol-method. The catalyst identification was performed using Fourier transform infrared spectroscopy (FT-IR), powder X-ray diffraction (PXRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM). The magnetic analysis showed that the $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ had a ferromagnetic behavior with a saturation magnetization of 31.22 emu/g at room temperature. Then, $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles were applied as a green, convenient, effective and reusable catalyst for the one-pot three-component synthesis of tetrahydrobenzo[b]pyrans in good to excellent yields. It should be noted, the nanocatalyst is separated by a magnet was used several times and it was as efficient as ever.

Keywords: Ferrite, Magnetic nanoparticles, Tetrahydrobenzo[b]pyran derivatives, Microwave irradiation, Solvent-free.

1. Introduction

Recently, tetrahydrobenzo[b]pyran derivatives have been attracting increasing interest due to their wide range of pharmacological and biological properties [1]. The substituted tetrahydrobenzo[b]pyran have been known as effective heterocyclic compounds with interesting activities as, e.g., anti-antitumor [2], anti-HIV [3], anti-oxidant [4], anti-microbial and anti-fungal [5], anti-coagulant [6], anti-inflammatory [7] and diuretic [8]. Meanwhile, some drugs have tetrahydrobenzo[b]pyran skeleton for treatment of Alzheimer's, Parkinson's, and Down's syndrome disease [9]. Moreover, heterocyclic compounds with this skeleton mostly applied in the field of biodegradable agrochemicals, cosmetic, and pigments industries [10, 11]. Uvafzlelin [12], Erysenegalensein C [13], and Conrauinone A [14] are natural compounds with the tetrahydrobenzo[b]pyran moiety. Vitamin E [4] and Cromakalim are drugs that include this moiety [15].

Because of the importance of these compounds, various and numerous methods have been reported by multicomponent reactions (MCRs) for the synthesis of tetrahydrobenzo[b]pyrans in the presence of a variety of homogeneous and heterogeneous catalysts in different conditions such as under microwave [16-22] and ultrasound irradiation [23-25], conventional heating [17, 26-40], and room temperature [41-47].

In the past decades, using of various nanoparticles as green nanocatalysts in MCRs has caused chemists to try synthesizing different types of heterocyclic compounds. The nanoparticles are produced by various chemical methods, which have disadvantages such as the use of toxic solvents, inadequate particle size, impurity of nanoparticles, solubility instability, low efficiency, and the need for advanced production equipment [48-53]. For this reason, researchers are more inclined to biopsynthesize nanoparticles that have minimal environmental hazards and have a simple production method. In recent years, many living organisms, such as bacteria, fungi, algae, plants, plant extracts and metabolites, have been used to synthesize nanoparticles [54-56]. In this work, a new method is reported for the synthesis of

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tetrahydrobenzo[b]pyran derivatives in the presence of $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles as a green nanocatalyst under microwave irradiation in solvent-free conditions (Scheme 1).

2. Experimental

2.1. Material and methods

All pure compounds were purchased from Sigma-Aldrich (Germany), Fluka (Switzerland), Merck (Germany), and Daijung (Darmstadt, Korea). The Arabic gum (AG) was prepared from a medicinal plant store. The FT-IR spectra were recorded on a Jasco 6300 FT-IR spectrometer by dry KBr. The XRD pattern was studied by a X'Pert-Pro advanced diffractometer using Cu ($K\alpha$) radiation (wavelength: 1.5406 Å). The morphology and grain size of the sample was tested by FESEM and EDX on the Tescan Mira3 device. Also, the magnetic behavior of the sample was measured using a vibrating sample magnetometer at room temperature (VSM, Meghnatis Kavir Kashan Co., Kashan, Iran).

The progress of the reactions was checked by TLC. The milestone microwave oven was used in all experiments. Melting points of the products were recorded on an Electrothermal 9100 device. ^1H and ^{13}C NMR spectra (CDCl_3) were run on a Bruker 250 DRX Avance instrument at 250.13 and 62.90 MHz, respectively.

2.2. The synthesis of $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles by Arabic gum

At first, 0.3 g of the Arabic gel was solved in 40 ml of distilled water and stirred for 2 h at 70 °C to obtain a clear solution. Subsequently, 0.5 mmol of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 0.5 mmol of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and 2 mmol $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were added to the AG solution and was then stirred for 10 h at 80 °C to achieve a brown color dry gel. The gel obtained was annealed at 600 °C in the air for 4h to form the nickel-copper ferrite magnetic nanoparticles.

2.3. General method for the synthesis of tetrahydrobenzo[b]pyrans

A mix of aldehyde **1a-m** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol), and 10 mol%

(24 mg) of $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs was poured in a glass rod and exposed to microwave radiation (400 W). The progress of the reaction was carried out by TLC (petroleum ether: EtOAc, 8:2). After the reaction is over, the resulting product was heated in ethanol. The nanocatalyst was separated from the mixture with an external magnet and washed three times with ethanol for reuse. Then, the residual solution containing the product was placed in crushed ice. The filtered solid product was recrystallized in hot ethanol as a pure product.

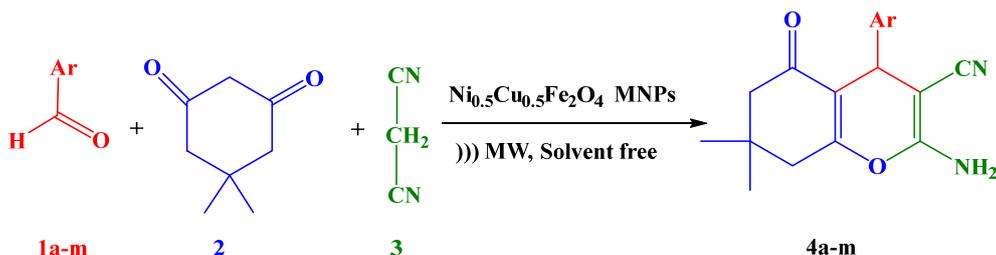
3. Results and Discussion

3.1. Characterization of $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles

First, the FT-IR spectrum of the $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs was investigated. As shown in Fig. 1, a broad absorption band above 3400 cm^{-1} is the stretching mode of H_2O molecules. Also, two principle absorption bands are observed in the range of 400-600 cm^{-1} . Two absorption bands are observed at 602 and 476 cm^{-1} , which demonstrate the longer and shorter bond length of oxygen metal ions in the octahedral and tetrahedral sites in the spinel structure, respectively [57, 58]. The broad absorption peaks in the regions of 1636 cm^{-1} and 3425 cm^{-1} are related to the flexural and tensile vibrations of the hydroxyl (OH) groups [59].

Fig. 2 shows the X-ray diffraction (XRD) pattern of magnetic ferrite nanoparticles $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$. The results of X-ray diffraction (XRD) analysis confirm the formation of pure cubic crystal phase. All peak diffraction characteristic (XRD) patterns of the magnetic $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ magnetic nanoparticles correspond to the plates specified in the standard card (JCPDS Card no. 77-0010) for ferrite with pure cubic phase. The presence of strong, sharp peaks in the crystalline pattern above the synthesized nanoparticles. The crystallite size was calculated using the Scherrer equation of about 24 nm [52, 60].

Fig. 3 shows the SEM image of and particle size distribution of synthesized $\text{Ni}_{0.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs after calcining at 600 °C for 4 hours.



Scheme 1. Synthesis of tetrahydrobenzo[b]pyran derivatives

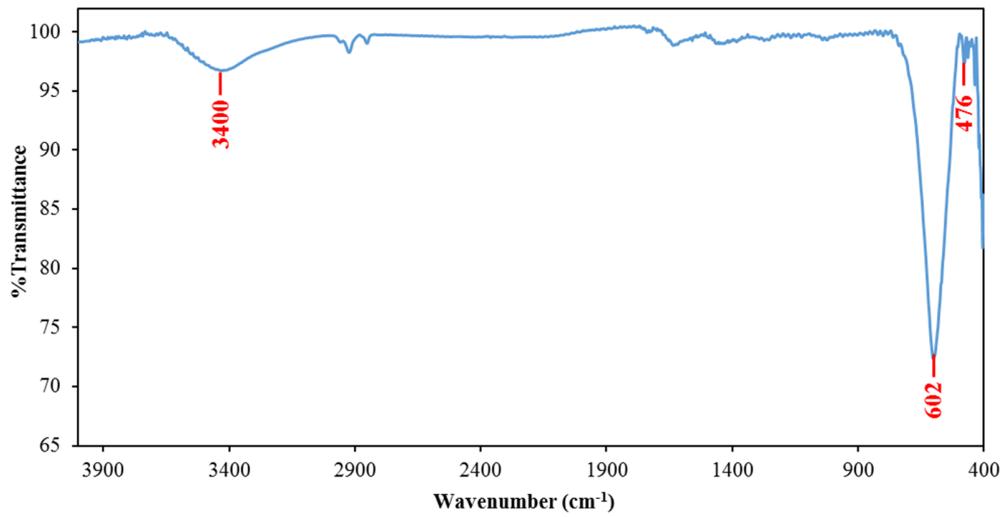


Fig. 1. FT-IR spectrum of Ni_{0.5}Cu_{0.5}Fe₂O₄ MNPs.

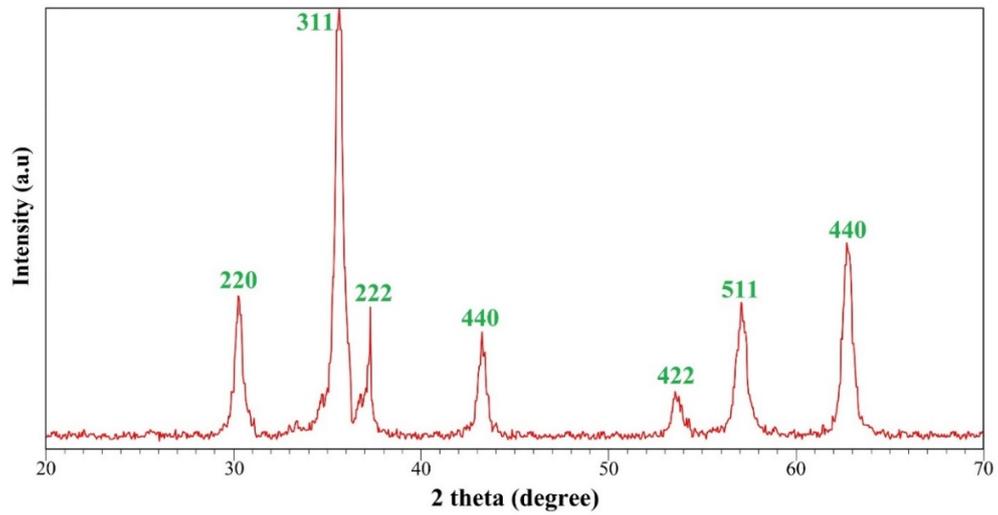


Fig. 2. XRD pattern of synthesized Ni_{0.5}Cu_{0.5}Fe₂O₄ MNPs.

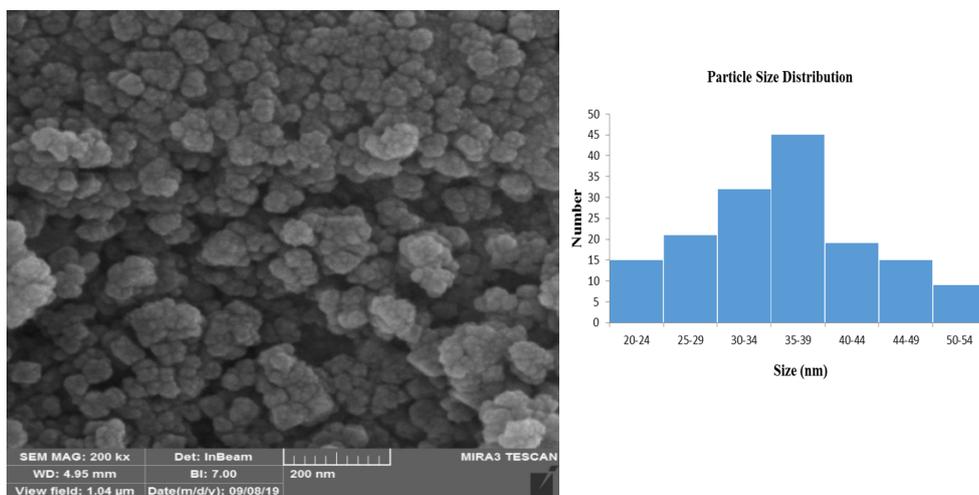


Fig. 3. SEM image and particle size distribution of synthesized Ni_{0.5}Cu_{0.5}Fe₂O₄ MNPs.

The SEM image shows a uniform distribution of nanoparticles with spherical morphology and the size of the particles was estimated between 35-40 nm. EDX analysis was performed to investigate the presence of elements present in the compound. Fig. 4 shows the presence of iron, nickel, oxygen and copper elements in the composition. The weight and atomic percentages are given in Table 1.

The magnetic hysteresis loop of $\text{Ni}_{10.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs was displayed in Figure 5 that measured by Vibrating Sample Magnetometer (VSM). Magnetization curve exhibits ferromagnetic properties with remanence magnetization (M_r) of 6.75 emu/g and the coercivity (H_c) of 94.32 Oe. As can be observed in Fig. 5, the specific saturation magnetization value was determined to be 31.22 emu/g for the sample.

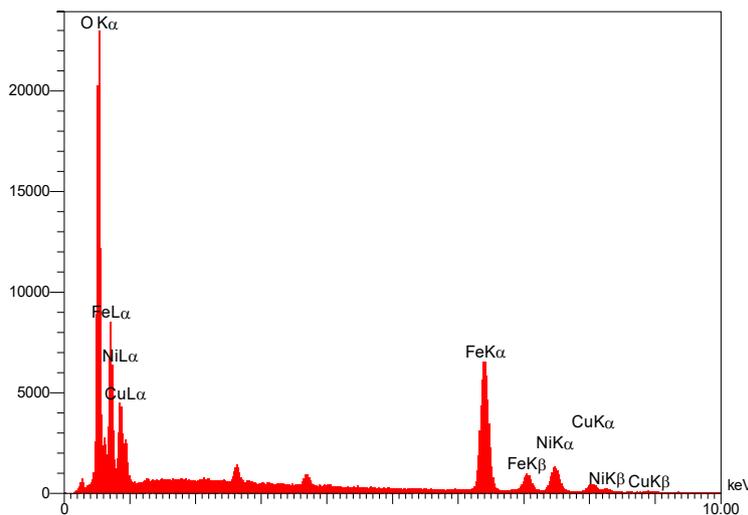


Fig. 4. EDX analysis of synthesized $\text{Ni}_{10.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs.

Table 1. Weight percent of the elements measured by the EDX analysis.

Elements	Ni	Cu	Fe	O	Total
Weight (%)	7.82	2.17	38.64	51.37	100
Atomic (%)	3.27	0.84	17.00	78.89	100

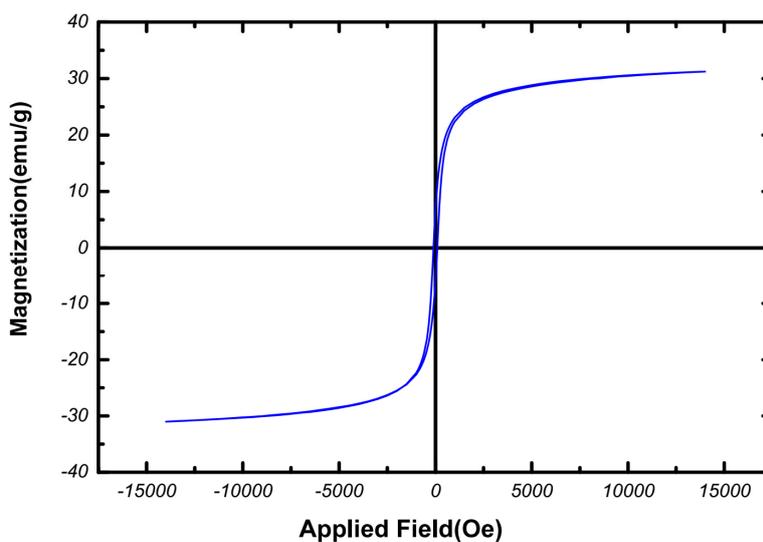


Fig. 5. VSM curve of $\text{Ni}_{10.5}\text{Cu}_{0.5}\text{Fe}_2\text{O}_4$ MNPs.

3.2. Synthesis of tetrahydrobenzo [b]pyrans in the presence of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ magnetic nanoparticles

The role of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ MNPs in the synthesis of target products was checked. The microwave power, catalyst amount and adequacy and reaction times were optimized. At first, the influence of microwave irradiation on the synthesis of compound **4a** (Table 4, entry 1) as a model reaction was investigated (Table 2). When the power of microwave irradiation was raised at 200, 300 and 400, the reaction yield was increased, but the yield was not changed at 500 W.

In the following, the amount and adequacy of the $Ni_{0.5}Cu_{0.5}Fe_2O_4$ MNPs as a catalyst were checked in a sample reaction for the synthesis of compound **4a** (Table 4, entry 1). As shown in Table 3, when the catalyst was not used, the efficiency of the reaction was minimized. By using a catalyst and increasing its amount, the adequacy of the reaction increased. Eventually, the use of 10 mol% of catalyst resulted in high yield of compound **4d** (Table 3).

As shown in Table 4, different aromatic aldehydes with electron-withdrawing and electron-donating groups on the aromatic ring were investigated in the *ortho*, *meta*, and *para* positions. The efficiency of all products was were good to excellent.

Moreover, the efficiency of the $Ni_{0.5}Cu_{0.5}Fe_2O_4$ MNPs

with some literature methods was compared (Table 5). The comparison results show that this research has several advantages premier to some of the literature methods such as use of reusable magnetic nanocatalyst, convenient reaction time and good to excellent yield.

Eventually, collected magnetic nanoparticles from previous reactions were washed by ethanol and used for many times. As shown in Table 6, the efficiency of the recycled catalyst was investigated in five reactions.

4. Conclusions

In this work, a simple, efficient, cheap, reusable, environment-friendly and green method for synthesis of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ MNPs as a nanocatalyst was reported in the presence of Arabic gum as a natural and non-toxic template and stabilizing agent. Furthermore, one-pot three-component synthesis of tetrahydrobenzo[b]pyran derivatives was investigated under microwave irradiation in solvent-free conditions in excellent yields. The present procedure has many advantages such as use of nanocatalyst, operational simplicity, easy work-up of products, in solvent-free conditions, short reaction time and high yields.

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Table 2. The influence of microwave irradiation on the synthesis of tetrahydrobenzo[b]pyrans.^a

Entry	Catalyst (mol%)	Microwave power (W)	Time (min.)	Yield(%) ^b
1	10	200	5	49
2	10	300	5	84
3	10	400	5	92
4	10	500	5	92

^aReactions conditions: benzaldehyde **1a** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol) under different microwave irradiations.

^bIsolated yield.

Table 3. Effect of the catalyst amount on the synthesis of tetrahydrobenzo[b]pyrans.^a

Entry	Catalyst (mol%)	Microwave power (W)	Time (min.)	Yield (%) ^b
1	None	400	5	26
2	5	400	5	78
3	10	400	5	92
4	15	400	5	91

^aReactions conditions: benzaldehyde **1a** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol) and various mol% of catalyst.

^bIsolated yield.

Table 4. Synthesis of tetrahydrobenzo [b]pyrans in the presence of Ni_{0.5}Cu_{0.5}Fe₂O₄ MNPs.^a

Entry	Ar	Product	Time (min)	Yield (%) ^b	m.p. (°C)		Ref.
					Found	Reported	
1	Ph	4a	5	92	225-227	225-226	[35]
2	2-Cl-C ₆ H ₄	4b	6	92	209-211	209-211	[36]
3	3-Cl-C ₆ H ₄	4c	6	93	223-225	222-224	[37]
4	4-Cl-C ₆ H ₄	4d	5	95	202-203	202-203	[45]
5	2,4-(Cl) ₂ -C ₆ H ₃	4e	7	97	181-183	180-182	[46]
6	2-NO ₂ -C ₆ H ₄	4f	6	91	229-231	227-230	[38]
7	3-NO ₂ -C ₆ H ₄	4g	6	90	201-203	198-201	[35]
8	4-NO ₂ -C ₆ H ₄	4h	6	89	175-177	175-176	[45]
9	4-Br-C ₆ H ₄	4i	7	92	212-214	207-209	[46]
10	4-Me-C ₆ H ₄	4j	8	87	210-212	209-211	[45]
11	4-OH-C ₆ H ₄	4k	8	85	205-207	206-208	[38]
12	4-OMe-C ₆ H ₄	4l	8	82	199-201	197-199	[45]
13	4-CN	4m	5	96	227-230	225-228	[46]

^aReaction conditions: aldehyde **1a-m** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol) and 10 mol% of catalyst under the optimum conditions.

^bIsolated yields.

Table 5 Comparison of synthesis of tetrahydrobenzo[b]pyrans in the presence of Ni_{0.5}Cu_{0.5}Fe₂O₄ magnetic nanoparticles with some literature methods.^a

Entry	Catalyst	Solvent	Conditions	Time (min.)	Yield (%) ^b	Ref.
1	NaBr	-	MW-70 °C	10	95	[16]
2	LiBr	-	MW (500W)	8	84	[17]
3	NH ₄ Cl	EtOH	MW (140W)	2	85	[19]
4	Without Catalyst	-	MW-80 °C	7	88	[21]
5	[Et ₃ NH][HSO ₄]	-	MW (140W)	5	88	[22]
6	Ni _{0.5} Cu _{0.5} Fe ₂ O ₄ MNPs	-	MW(400W)	5	92	This work

^aReaction conditions: benzaldehyde **1a** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol) and 10 mol% of catalyst.

^bIsolated yield.

Table 6. Reusability of the Ni_{0.5}Cu_{0.5}Fe₂O₄ MNPs.^a

Run	Yield (%) ^b	Reusability of Ni _{0.5} Cu _{0.5} Fe ₂ O ₄ MNPs (%)
1	92	99
2	92	99
3	91	98
4	90	97
5	90	95

^aReaction conditions: benzaldehyde **1a** (1 mmol), dimedone **2** (1 mmol), malononitrile **3** (1 mmol) and 10 mol% of catalyst under reaction conditions.

^bIsolated yields.

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