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Magnetic nickel ferrite nanoparticles as an efficient catalyst for the preparation of polyhydroquinoline derivatives under microwave irradiation in solvent-free conditions

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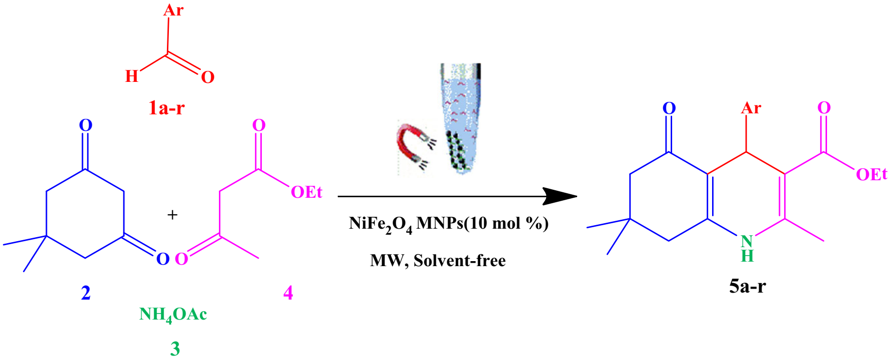
Abstract An easy and convenient method is reported for the synthesis of polyhydroquinoline derivatives in solvent-free conditions under microwave irradiation in the presence of magnetic nickel ferrite nanoparticles ($NiFe_2O_4$ MNPs) as a catalyst. Excellent yields (82–94 %), facile work-up, achieving high purity of products via simple recrystallization, and short reaction times (2–5 min) are some advantages of this procedure. It is worth noting, the catalyst is magnetically removed without significant loss of activity for reuse a few times.

Graphical Abstract



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Keywords Nickel ferrite · Magnetic nanoparticles · Polyhydroquinoline · Microwave irradiation · Solvent-free

Introduction

Polyhydroquinolines derivatives, 4-substituted 1,4-dihydropyridines (1,4-DHPs) compounds (Fig. 1), are an essential framework of a great category of biologically active heterocycles [1]. They are well-known compounds as an important class of drugs and analogues of NADH co-enzymes [2]. The therapeutic usages of 1,4-DHPs include significant biological activity [3, 4], various drugs such as nifedipine, nicardipine, benidipine, amolodipine, lacidipine, (Fig. 2) and other related derivatives for treatment of hypertension as an effective cardiovascular agent [5, 6], and having a variety of biological properties such as vasodilator, antitumour, antidiabetic, anti-atherosclerotic, bronchodilator, anti-oxidant, geroprotective and heptaprotective agents [7, 8].

Quinoline derivatives with 1,4-dihydropyridine scaffolding have pharmacological properties. Some compounds of this family are antimalarial, anti-asthmatic, anti-inflammatory, antibacterial, and tyrosine kinase inhibiting agents [9, 10]. Other researches indicate that 1,4-DHPs express different medical functions as neuroprotectants, having platelet anti-aggregatory activity, cerebral anti-ischemic agents, and chemosensitizers [11, 12]. Also, numerous synthetic methods by MCRs have been reported for the preparation of 1,4-dihydropyridine derivatives under different conditions [13–18].

In view of the importance of polyhydroquinolines derivatives, numerous and various methods by MCRs have been reported for synthesis of these compounds, which include the use of grinding [19], solar thermal energy [20], microwave irradiation by Ni nanoparticles [21], ZnO-Beta Zeolite [22], PEG 4000-bound acetoacetate [23], composite-A ($ZnFe_{0.2}Al_{1.8}O_4$) [24], and silica sulfuric acid [25]; and also using different catalysts such as L-proline [26], montmorillonite K-10 clay [27], molecular iodine [28], Scolecite ($CaAl_2Si_3O_{10}\cdot 3H_2O$) [29], ZnO nanoparticles [30], $HClO_4$–SiO_2 [31], nanocrystalline copper(II) oxide [32], ionic liquids [33], and metal triflates such as $Yb(OTf)_3$ [34]. In addition, these MCRs have been performed by use of BINOL-phosphoric acid [35], Carbon-based Solid acid (CBSA)

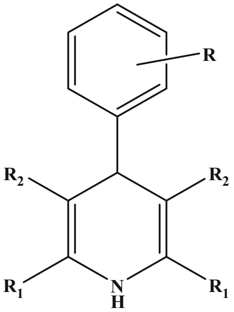
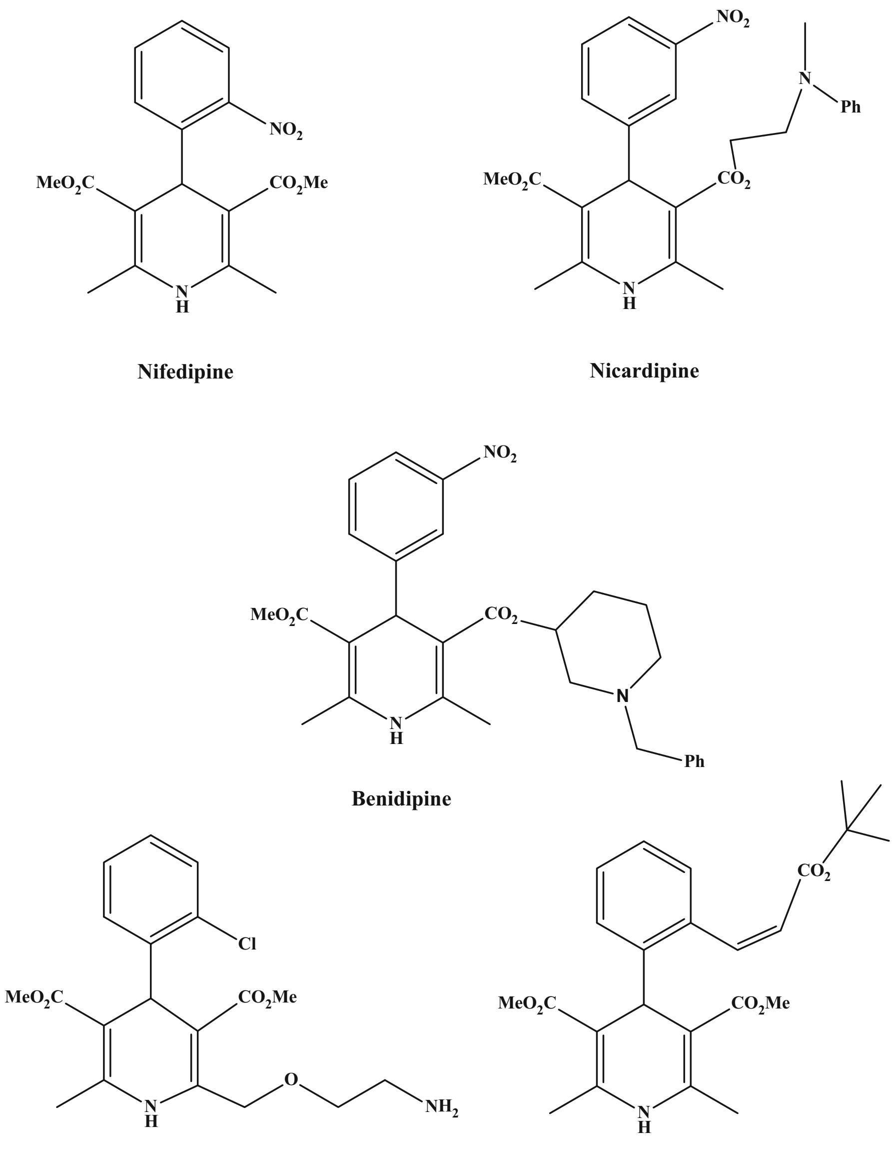


Fig. 1 General molecular structure of 1,4-DHPs



Nifedipine

Nicardipine

Benidipine

Amlodipine

Lacidipine

Fig. 2 Molecular structures of dihydropyridine drugs

[36], DBH or DCH [37], and SO_3H-functionalized ILs under ultrasound irradiation [38].

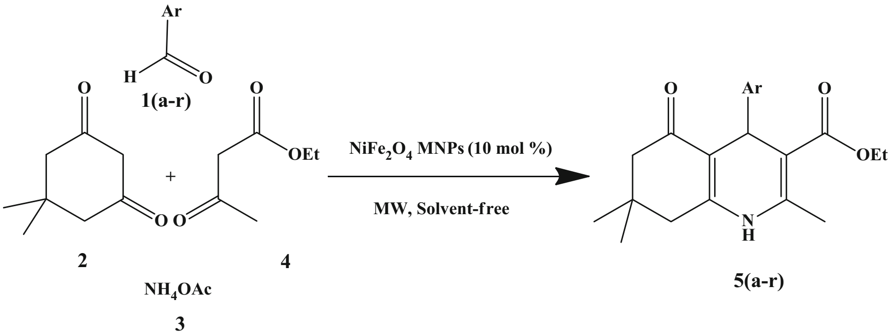
However, in past reports, the existence of some drawbacks such as long reaction times, harsh conditions, toxic organic solvents, high temperatures, high costs, low product yields, being expensive, having difficulty to reuse, and unecofriendly catalysts limit the use of these methods.

In recent years, magnetic nanoparticles (MNPs) have emerged as an attracting class of catalysts, because of the increasing need to develop low-cost, green, efficient, and reusable catalysts. Their nano-size property maximizes the contact between reactants and the catalyst, thus mimicking a heterogeneous catalyst. Use of magnetically separable catalysts is one of the most promising strategies. These systems combine the advantages of nanosized catalysts and magnetic properties, thus, providing the opportunity for quantitative recovery of the catalyst by the use of an external magnet, making it cost-effective and potentially applicable for industrial application [39, 40].

In this regard, iron-based materials, which benefit their terrestrial abundance and low toxicity, have been extensively exploited as catalysts in many organic transformations. Nickel ferrite MNPs are inexpensive, air-stable, magnetically separable, and economical catalysts [41–44]. There are very limited and there are less references about catalytic properties of nickel ferrite MNPs [45–47]. These spinel-type bimetal oxides, have shown great potential in the field of use for catalysis, although they have so far been less investigated.

In recent years, the organic reactions on solid supports in the absence of a solvent and in solvent-free conditions have received considerable attention of synthetic chemists because of their high efficiency, environmentally benign conditions, convenient workup procedures, enhanced selectivity, milder reaction conditions, much improved reaction rates and formation of cleaner products [48]. Aiming to use these advantages, we planned to develop a solvent-free protocol for synthesis of polyhydroquinoline derivatives in solvent-free conditions.

As part of our ongoing program to develop efficient and robust methods for the synthesis of heteroatom-containing organic compounds [49–52], we report an easy and convenient method for synthesis of polyhydroquinoline derivatives in solvent-free conditions under microwave irradiation in the presence of magnetic nickel ferrite nanoparticles ($NiFe_2O_4$ MNPs) as a catalyst. According to the best of our knowledge, there is no report on the use of $NiFe_2O_4$ MNPs as a magnetic



Scheme 1 Synthesis of polyhydroquinoline derivatives in the presence of $NiFe_2O_4$ MNPs

nanocatalyst. Therefore, this fact has caused us to apply the new catalyst for the synthesis of polyhydroquinoline derivatives with a facile and appropriate method under eco-friendly conditions, as shown in Scheme 1.

Experimental

Materials and methods

Starting materials were obtained from Merck, Fluka, and Aldrich and were used without further purification. The methods used to follow the reactions are TLC. The microwave-assisted procedures were carried out in a Milestone Microwave Oven operating at 1600 W. Melting points were measured on an Electrothermal 9100 apparatus (LABEQUIP LTD., Markham, Ontario, Canada) and are uncorrected. ^{1}H (DMSO-d$_6$) and ^{13}C NMR (DMSO-d$_6$) spectra were recorded on a Bruker DRX-250 Avance spectrometer at 250.13 and 62.90 MHz, respectively. IR spectra were measured on a Jasco 6300 FTIR spectrometer. Elemental analysis was performed using inductively coupled plasma optical emission spectroscopy (ICP-OES, Model: VISTA-PRO).

The structural properties of $NiFe_2O_4$ MNPs were analyzed by X-ray powder diffraction (XRD) with a X'Pert-PRO advanced diffractometer using Cu (Kα) radiation (wavelength: 1.5406 Å), operated at 40 kV and 40 mA at room temperature in the range of 2θ from 20° to 80°. The particle size and morphology of the surfaces of the sample were analyzed by a scanning electron microscopy) KYKY Co., China, Model: EM 3200). The disc was coated with gold in an ionization chamber. Transmission electron microscopy was studied using a Zeiss-EM10C TEM instrument with an accelerating voltage of 80 kV.

Preparation of catalyst ($NiFe_2O_4$ MNPs)

Nickel ferrite nanoparticles were prepared via the auto-combustion assisted sol–gel method of Fe(III) and Ni(II) ions (molar ratio 2:1) in ammonia solution [53, 54]. Briefly, $Fe(NO_3)_3 \cdot 9H_2O$ (2 mol), $Ni(NO_3)_2 \cdot 6H_2O$ (1 mol) and citric acid as chelating agent (3 mol) were dissolved in deionized water. The pH value was controlled at 7 by dropwise adding ammonia solution (28 %) to the reaction mixture under fixed stirring. Then, the solution was vaporized in a water bath (60 °C) to form a sticky gel. The temperature was increased to 80 °C in order to obtain a thick gel. The thick gel was held on a hot plate for auto combustion and the temperature was increased to ca. 200 °C. A large amount of gases such as CO_2, H_2O, and N_2 were released and auto combustion occurred, giving rise to a dark brown ferrite powder. The powder was washed with deionized water and acetone three times and separated by using a strong external magnet.

General experimental procedure for the synthesis of polyhydroquinoline derivatives

A mixture of aldehyde **1a**–**r** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), ethylacetoacetate **4** (1 mmol), and 23.4 mg of $NiFe_2O_4$ nanoparticles (10 mol%) were taken in a glass rod and irradiated in a microwave oven (500 W; when the reaction mixtures were placed under microwave irradiation, MW heating causes melting reactants that led to the partially liquefied reaction mixture and achieved fairly good mixing of the reactants with the catalyst). The progress of the reaction was checked by TLC (*n*-Hexane: EtOAc, 10:4). After completion, the resulting product was heated in ethanol. The catalyst was magnetically removed from the mixture and washed several times with ethanol for reuse. Then, the residue was poured into crushed ice and stirred for several minutes. The solid product was filtered and the pure product was obtained by recrystallization from hot ethanol–water.

Physical and spectral data of ethyl 4-(4-hydroxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,5,7,8-hexahydroquinoline-3-carboxylate (**5m**, Table 3, Entry 13): Yield: 94 %; M.P. 233–235 °C (Ref. [42] 232–234 °C). IR (KBr): 3488, 3277, 3073, 2958, 2930, 1683, 1491, 1590, 1480, 1194 cm^{-1}. ^1H NMR (DMSO-d_6, 250.13 MHz) δ 0.83 (3H, s, CH_3), 0.98 (3H, s, CH_3), 1.12 (3H, t, CH_3, $J = 6.5$ Hz), 1.91–2.42 (4H, m, 2 × CH_2), 2.38 (3H, s, CH_3), 3.94 (2H, q, CH_2, $J = 6.2$ Hz), 4.71 (1H, s, CH), 6.54 (2H, d, $J = 6.8$ Hz, Arom.), 6.91 (2H, d, $J = 7.0$ Hz, Arom.), 8.96 (1H, s, OH), 9.04 (1H, s, NH). ^{13}C NMR (DMSO-d_6, 62.90 MHz) δ 14.60, 18.68, 26.89, 29.61, 32.56, 35.21, 50.73, 59.39, 104.56, 110.76, 114.87, 128.77, 138.86, 144.82, 149.59, 155.67, 167.45, 194.76.

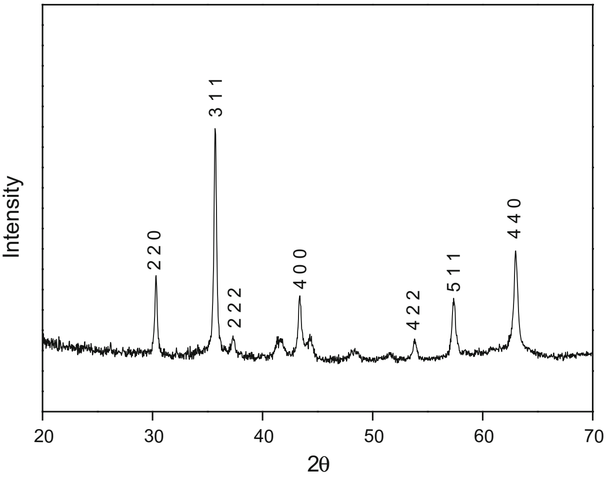


Fig. 3 XRD pattern of $NiFe_2O_4$ MNPs

Results and discussion

Catalyst characterization

Magnetic nickel ferrite nanoparticles were synthesized by the auto-combustion assisted sol–gel method [43, 44]. The structural properties of the $NiFe_2O_4$ MNPs were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), inductively coupled plasma (ICP), and IR. The X-ray diffraction (XRD) pattern of catalyst powder presented the average diameter of that was determined to be 59.2 nm by the Debye–Scherrer equation (Fig. 3). The diffraction peaks at 30.45°, 35.76°, 37.30°, 43.43°, 53.84°, 57.34°, and 63.01° corresponded with Miller indices observed in (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) planes, respectively. These results show the cubic spinel structure of nickel ferrite (JCPDS: 01-074-2081).

The grain size and morphology of the $NiFe_2O_4$ MNPs powder were determined by scanning electron microscopy (SEM) (Fig. 4). SEM images show the average size of the grains with spherical morphology is 59 nm and that has good agreement with the XRD pattern.

Also, the grain and size morphology of the $NiFe_2O_4$ MNPs were investigated by transmission electron microscopy (TEM) (Fig. 5). They had spherical morphology

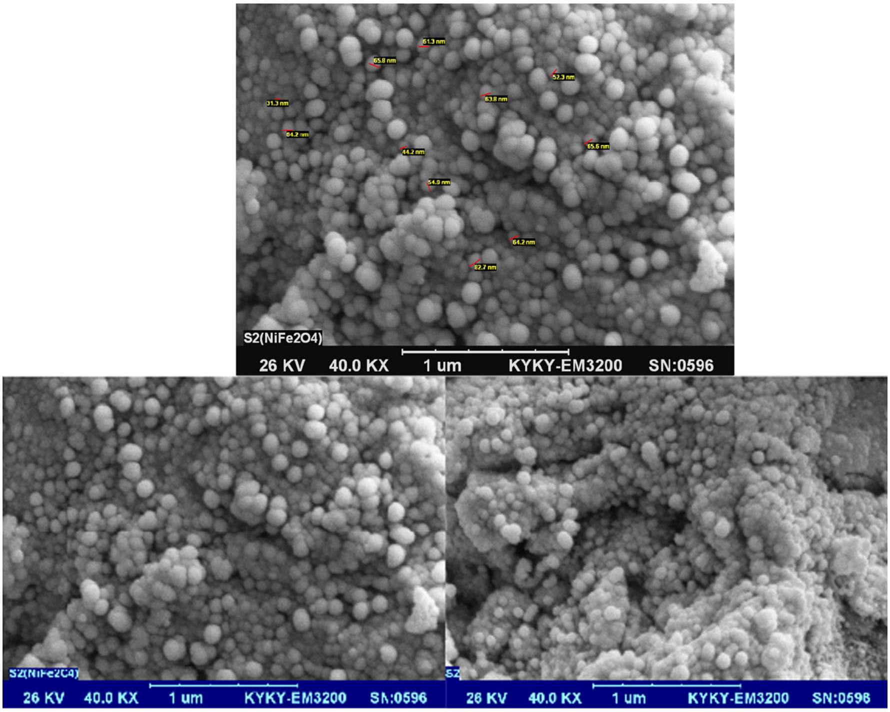


Fig. 4 SEM images of $NiFe_2O_4$ MNPs

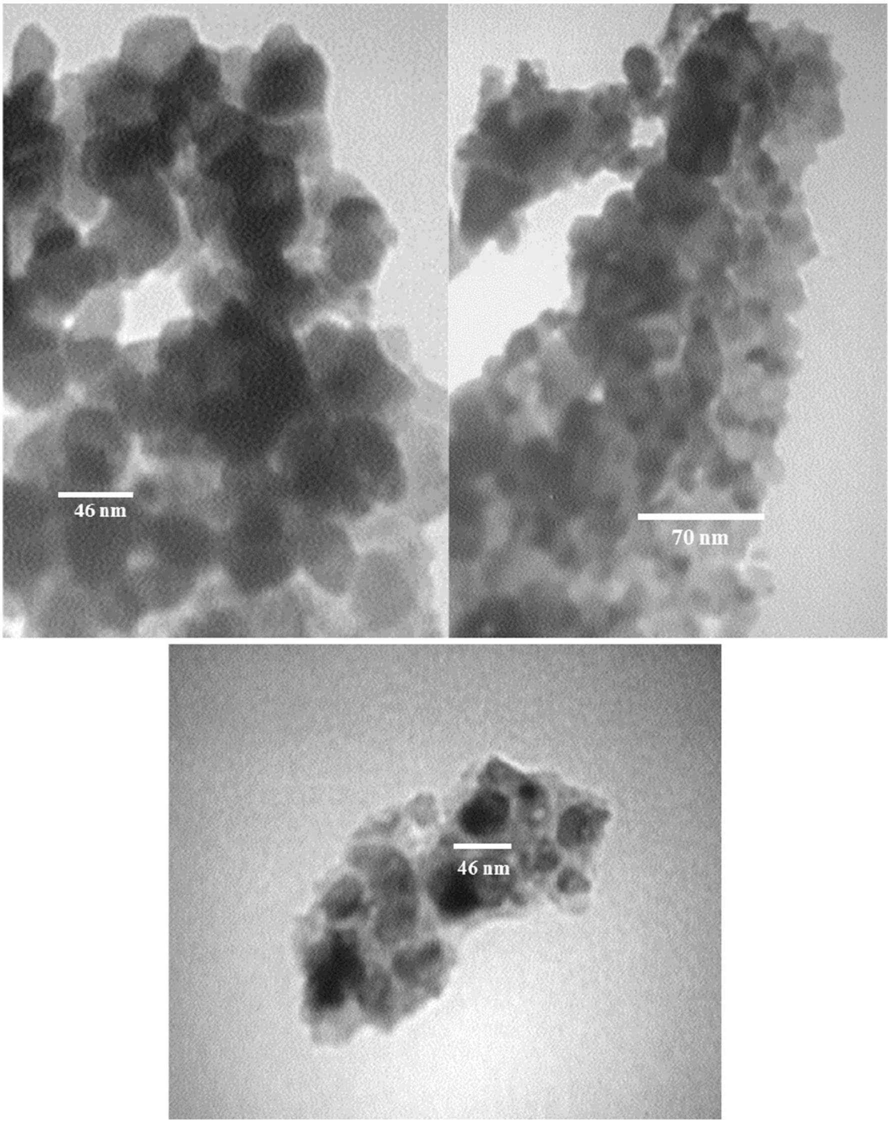


Fig. 5 TEM images of $NiFe_2O_4$ MNPs with magnification of 63KX

with a size distribution from 20 to 84 nm and a mean grain size of 46 nm. The presence of some larger particles could be attributed to aggregating (it may be a result of a long time (ca. 20 months) holding the $NiFe_2O_4$ MNPs in their pure form at the laboratory conditions) or overlapping of smaller particles.

Inductively coupled plasma (ICP) analysis of nickel ferrite presented the atomic ratio of Ni–Fe at about 0.53, which is close to $NiFe_2O_4$ and 24.8 % of its weight contains the nickel element.

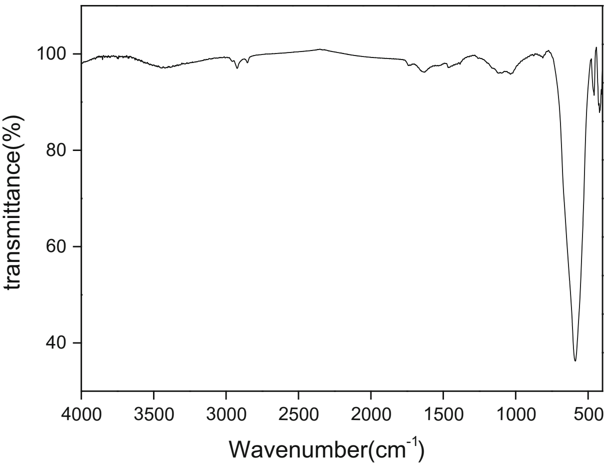


Fig. 6 FTIR spectrum of $NiFe_2O_4$ MNPs

Table 1 Effect of the catalyst amount on the synthesis of polyhydroquinoline derivatives

Entry	Catalyst (mol%)	Time (min)	Yield (%)[a]
1	None	20	75
2	5	12	85
3	10	4	94
4	15	4	92

Reactions were carried out with 4-hydroxybenzaldehyde **1m** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), and ethyl acetoacetate **4** (1 mmol), under microwave irradiation at 500 W in solvent-free conditions

[a] Isolated yield

Table 2 Effect of microwave power on the synthesis of polyhydroquinoline derivatives

Entry	Catalyst (mol%)	Microwave power (W)	Time (min)	Temperature (°C)	Yield (%)[a]
1	10	400	4	–	78
2	10	450	4	–	89
3	10	500	4	–	94
4	10	600	4	–	72
5	10	–	30	110	81
6	–	–	45	110	79

Reactions were carried out with 4-hydroxybenzaldehyde **1m** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), and ethyl acetoacetate **4** (1 mmol), with 23.4 mg of $NiFe_2O_4$ MNPs (10 mol%) in solvent-free conditions

[a] Isolated yield

Table 3 $NiFe_2O_4$ MNPs catalyzed the synthesis of polyhydroqunoline derivatives via a four-component Hantzsch condensation

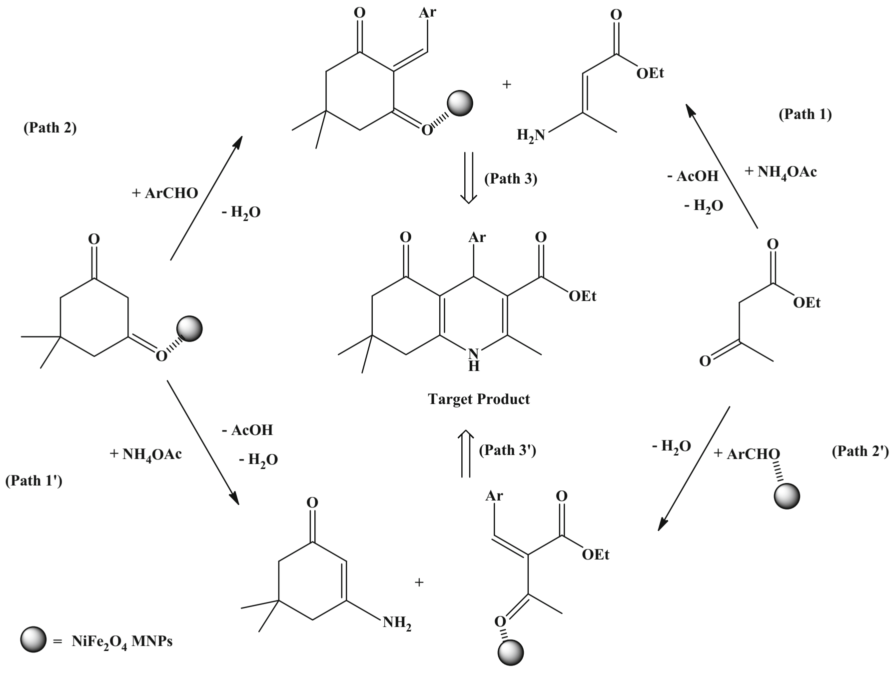
Entry	Ar	Product	Time (min)	Yield (%)[a]	M.P. (°C)	References
1	Ph	**5a**	3	90	201–204	202–204 [34]
2	4-NO_2–C_6H_4	**5b**	2	92	240–242	241–243 [26]
3	3-NO_2–C_6H_4	**5c**	3	90	176–178	176–179 [26]
4	2-NO_2–C_6H_4	**5d**	3	85	207–209	206–207 [37]
5	4-Cl–C_6H_4	**5e**	3	90	244–247	245–246 [30]
6	3-Cl–C_6H_4	**5f**	4	93	231–234	231–233 [30]
7	2-Cl–C_6H_4	**5g**	4	89	207–209	208–210 [30]
8	4-Br–C_6H_4	**5h**	3	85	254–256	253–255 [34]
9	3-Br–C_6H_4	**5i**	4	90	233–235	234–235 [38]
10	4-Me–C_6H_4	**5j**	4	87	259–261	260–261 [34]
11	4-MeO–C_6H_4	**5k**	4	82	256–258	257–259 [34]
12	4-MeS–C_6H_4	**5l**	4	91	239–241	241–243 [32]
13	4-OH–C_6H_4	**5m**	4	94	233–235	232–234 [42]
14	4-$(Me)_2CH$–C_6H_4	**5n**	4	87	182–184	181–183 [33]
15	4-$(Me)_2N$–C_6H_4	**5o**	4	87	228–231	229–231 [34]
16	2-Furyl	**5p**	3	87	247–249	246–248 [34]
17	2-Thienyl	**5q**	3	85	237–240	238–240 [37]
18	2-Naphthal	**5r**	5	90	198–200	197–199 [38]

Reaction conditions: aldehyde **1a–r** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), ethyl acetoacetate **4** (1 mmol), and 23.4 mg of catalyst (10 mol%) under microwave irradiation (500 W) in solvent-free conditions

[a] Isolated yield

Also, the FT-IR spectrum of the $NiFe_2O_4$ MNPs is demonstrated in Fig. 6. Two principle absorption bands in the range of 400–600 cm^{-1} are seen. The first band is around 469 cm^{-1} and the second one is around 580 cm^{-1}, attributed to the long bond length of oxygen metal ions in the octahedral sites and the shorter bond length of oxygen–metal ions in the tetrahedral sites in the spinel structure, respectively. A large number of OH groups are presented on the surface of the MNPs that show a broad absorption band at about 3439 cm^{-1} for the stretching mode of H_2O molecules.

The role of prepared catalyst in the synthesis of polyhydroquinoline derivatives was investigated under microwave irradiation in solvent-free conditions. We optimized the reaction conditions such as catalyst amount, microwave power, and reaction times. First, the efficiency and amount of the $NiFe_2O_4$ MNPs catalyst were investigated in a model reaction of 4-hydroxy benzaldehyde **1m** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), and ethylacetoacetate **4** (1 mmol) for the synthesis of compound **5m** (Table 3, entry 13). As shown in Table 1, the optimum yield of the product was obtained when 23.4 mg of catalyst (10 mol%) was used.



Scheme 2 Proposed mechanistic paths for the synthesis of polyhydroqunoline derivatives

Table 4 Comparison of the catalytic efficiency of $NiFe_2O_4$ MNPs with known catalysts

Entry	Catalyst	Solvent	Conditions	Time (min)	Yield (%)[a]	References
1	Nanocrystalline copper(II) oxide	–	140 °C	35	80	[32]
2	($ZnFe_{0.2}Al_{1.8}O_4$)	Water–HCl	MW	32	86	[24]
3	$Yb(OTf)_3$	EtOH	rt	480	90	[34]
4	Scolecite	EtOH	reflux	60	91	[29]
5	ZnO Nps	–	rt	20	86	[30]
6	$NiFe_2O_4$ MNPs	–	MW	4	94	This work

Reactions were carried out with 4-hydroxybenzaldehyde **1m** (1 mmol), dimedone **2** (1 mmol), ammonium acetate **3** (1.5 mmol), ethyl acetoacetate **4** (1 mmol), and 23.4 mg of $NiFe_2O_4$ MNPs (10 mol%)

[a] Isolated yield

The effect of microwave power inputs from 400 to 600 W on the synthesis of compound **5m** (Table 3, entry 13) as a model reaction was evaluated (Table 2). The reaction yield increased with the microwave power at 500 W in comparison to 400 and 450 W but decreased at 600 W. Also, we investigated this reaction in thermal conditions without microwave irradiation in the absence of catalyst and with

Table 5 Reusability and recovery of the catalyst

Run	Yield (%)[a]	Recovery of $NiFe_2O_4$ MNPs (%)
Fresh	94	97
First	94	96
Second	90	96
Third	93	95
Forth	91	93

[a] Isolated yield

catalyst in solvent-free conditions for the synthesis of compound **5m** (Table 3, entry 13). Ultimately, solvent-free conditions under microwave irradiation were preferred due to short reaction times and high yields.

Then, various aromatic aldehydes carrying electron-donating and electron-withdrawing groups on the aromatic ring in the *ortho*, *meta,* and *para* positions and heterocyclic aldehydes were evaluated. Yields of the all reactions were good to excellent. It was found that the aldehydes with electron-donating groups reacted longer than in comparison to aldehydes with electron-withdrawing groups (Table 3).

A proposed mechanism for the synthesis of polyhydroqunoline derivatives is shown in Scheme 2. A Knoevenagel reaction involves the evident coupling of aldehydes with active methylene compounds (dimedone and ethyl acetoacetate) in paths 2 and 2′. Also, in paths 3 and 3′ a Michael addition of intermediates gives the target products.

In addition, we compared the efficiency of catalytic activity of the $NiFe_2O_4$ MNPs with some known catalysts from the prior methods (Table 4). The result expresses that this work is superior to some of the previous methods in terms of magnetic separation of catalyst, reaction time, and yield.

Finally, the recyclability, reusability, and catalytic activity of the reaction catalyst were examined. After the magnetic separation of catalyst from the reaction mixture, the catalyst was washed with ethanol and dried to remove any remaining ethanol, and reused in the further reactions for several times. As shown in Table 5, $NiFe_2O_4$ MNPs could be reused at least five times without any inordinate reduction in of its activity.

Conclusions

To summarize, the reported method offers a new, simple, and efficient route for the synthesis of polyhydroquinolines derivatives via a four-component Hantzsch condensation without solvent under microwave irradiation using magnetic nickel ferrite nanoparticles as catalyst. Some important superiorities of this method are short reaction time, no use of hazardous solvents, easy work-up, high yields, and high purity. Furthermore, the use of the recyclable, reusable, inexpensive, and commercially available catalyst makes it a useful procedure in modern synthetic methodologies.

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