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FULL PAPER

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A novel magnetic nanocatalyst Fe_3O_4@PEG–Ni for the green synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

Nader Noroozi Pesyan | **Aria Danandeh Asl** | **Shadi Namdar**

Department of Organic Chemistry, Faculty of Chemistry, Urmia University, Urmia, 57159, Iran

Correspondence
Nader Noroozi Pesyan, Department of Organic Chemistry, Faculty of Chemistry, Urmia University, 57159, Urmia, Iran.
Email: nnp403@gmail.com; n.noroozi@urmia.ac.ir

Ni-PEG (polyethylene glycol) complex supported on magnetic nanoparticle was created by grafting. The catalytic activity of Fe_3O_4@PEG–Ni was explored through one-pot green synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones and used as an efficient and recoverable nanocatalyst. FT-IR, XRD, EDS, BET, TGA, VSM and SEM techniques were employed to specify the nanocatalyst. This heterogeneous nanocatalyst demonstrated acceptable recyclability and could be reused several times with no considerable loss of its catalytic activity.

KEYWORDS

2,3-dihydroquinazolin-4(1*H*)-ones, magnetic nanoparticle, nano catalyst, polyethylene glycol

1 | INTRODUCTION

The superficial functionalization of magnetic nanoparticles can be used as a well-organized system between homogeneous and heterogeneous catalysis. Magnetite (Fe_3O_4) is a well-described material that is employed to obtain stable and magnetically recyclable heterogeneous catalysts for some homogeneous catalytically active metals like Cu, Ni and Co.[1]

Magnetic nanoparticles have attracted attention because of their broad applications in catalysis and synthesis.[2,3] The surface of magnetic nanoparticles (MNPs) could be utilized to enable the loading of the range of desirable functionalities.[4] Recently, nanocomposites of organic materials and inorganic nanoparticles have drawn interest because of their applications in rapid and high-capacity optical and magnetic information storage media.[5–12] Nanoparticles have received much attention in the last two decades owing to their fascinating properties related to their size.[13] In scientific research, magnetite nanoparticles have been studied comprehensively, because their synthesis is easy and well known.[14]

In addition, magnetic nanocatalysts are studied for the synthesis of various organic compounds[15–22] and the degradation of organic pollutant dyes.[23–25] The magnetic properties make them useful to facilitate the separation of the nanocatalyst from the reaction mixture using an external magnet.[26]

Usually, 2,3-dihydroquinazolin-4(1*H*)-ones are prepared using the reductive cyclization of aldehydes or ketones with 2-aminobenzamide in the presence of acid catalysts.[27,28] These heterocyclic compounds are also synthesized by various metal nanocatalysts.[29–31] 2,3-Dihydroquinazolin-4(1*H*)-one derivatives exist that included six-membered heterocyclic ring molecules that have been reported to retain a wide range of biological properties, such as antihistaminic, pharmaceutical, antibiotic, analgesic, antitumor and antibacterial activities.[27,32,33] However, most of these processes suffer from longer reaction time, tedious workup, harsh reaction conditions, unsatisfactory yields, the use of large quantities of environmentally toxic catalysts. Thus, the development of simple, efficient, high-yielding and environmentally friendly approaches using new catalysts for the synthesis of these compounds is an important task.

Nickel complexion has been complexed with PEG functionalized nanostructured Fe_3O_4 and applied as an excellent nanocatalyst for the green synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones by cyclocondensation of 2-aminobenzamide with aromatic aldehydes under mild reaction conditions in ethylene glycol as a green solvent.

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2 | RESULTS AND DISCUSSION

This paper describes the synthesis and characterization of a new recoverable magnetic nanocatalyst and its use for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. A versatile and concise route for the synthesis of the new magnetic nanoparticle is outlined in Scheme 1. Polyethylene glycol was used as a widely available and green compound as a ligand for capturing Ni ions.

2.1 | Catalyst characterization

FT-IR, X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), Brunauer–Emmett–Teller(BET), TGA and scanning electron microscopy (SEM) techniques were used to characterize the structure of Fe_3O_4@PEG–Ni magnetic nanocatalyst. Figure 1 shows the FT-IR spectra obtained for MNPs, MNP–$(CH_2)_3Cl$ and MNP–$(CH_2)_3$PEG–Ni, in curves a, b and c, respectively. In the FT-IR spectrum of the nanocatalyst the bands appeared at 3397, 2956 and 2924 cm^{-1} related to stretching vibrations of OH and CH aliphatic absorption bands, respectively. The bands appearing at 1626, 997–1383, 576 and 450 cm^{-1} correspond to the symmetric and asymmetric stretching vibrations of Si–O, Fe–O and Ni–O bond vibrations in the catalyst, respectively.

Figure 2 shows morphological changes in MNPs and MNP–$(CH_2)_3$PEG–Ni by SEM images. These images confirm that the catalyst was formed in uniform nanometer-sized particles. In addition, it can be concluded that the morphology of MNP–$(CH_2)_3$PEG–Ni (Figure 2b) is the same as the particle form of MNP–$(CH_2)_3$PEG (Figure 2a).

The EDX data indicate the existence of Fe, Si, C, O, Cl and Ni in MNP–$(CH_2)_3$PEG–Ni (Figure 3a). The EDS results of the MNP–$(CH_2)_3$PEG–Ni are provided in Figure 3b. As can be seen from curve a, Fe, Si, C, O and Ni elements with mass percentages 39.36, 1.74, 14.23, 42.50 and 0.45% were respectively detected in MNP–$(CH_2)_3$PEG–Ni.

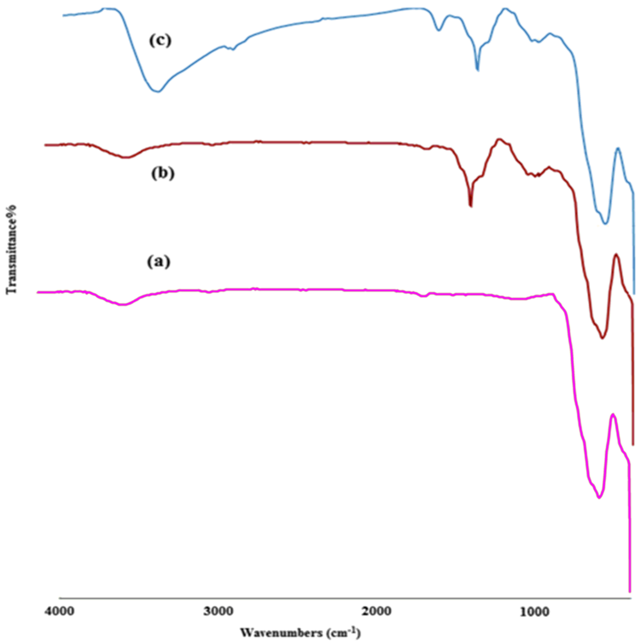
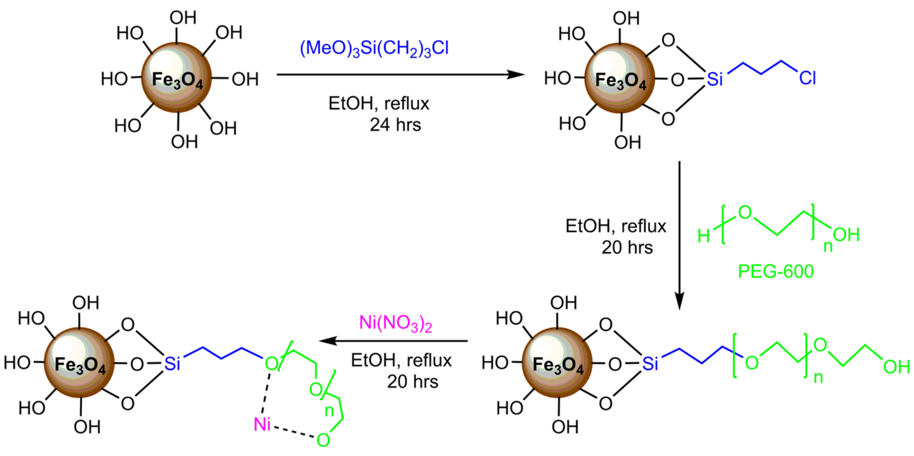


FIGURE 1 FT-IR spectra of: (a) MNPs; (b) MNP–$(CH_2)_3Cl$; and (c) MNP–$(CH_2)_3$PEG–Ni

Figure 4 shows the dark-brown colored powder of the sample and SEM–EDS mapping of MNP–$(CH_2)_3$PEG–Ni (distribution of C, O, Si, Cl, Fe, Ni elements). Elemental mapping is one of the best tools to analyze a single spot to give a spectrum that provides information about the presence of elements and can show the quantity of each element as a map. In the present nanocatalyst, SEM–EDS mapping assigned each element and confirmed the formation of the nanocatalyst (Figure 4).

Figure 5 shows the measurement of the magnetic characteristics of MNP–$(CH_2)_3$PEG–Ni, which was done by vibrating sample magnetometry (VSM) at room temperature. The value of magnetization saturation of MNP–$(CH_2)_3$PEG–Ni was nearly 40 emu g^{-1}. The saturation



SCHEME 1 Synthesis of Fe_3O_4@PEG–Ni magnetic nanocatalyst

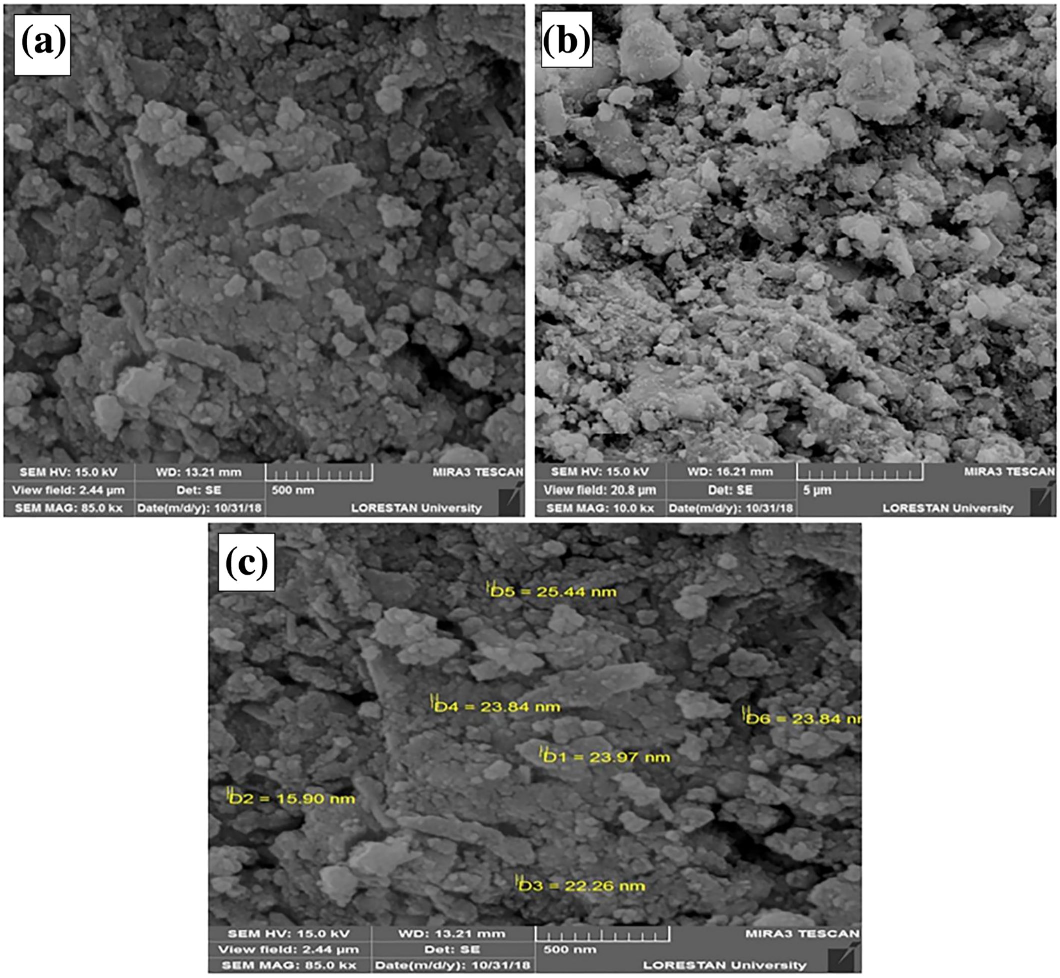
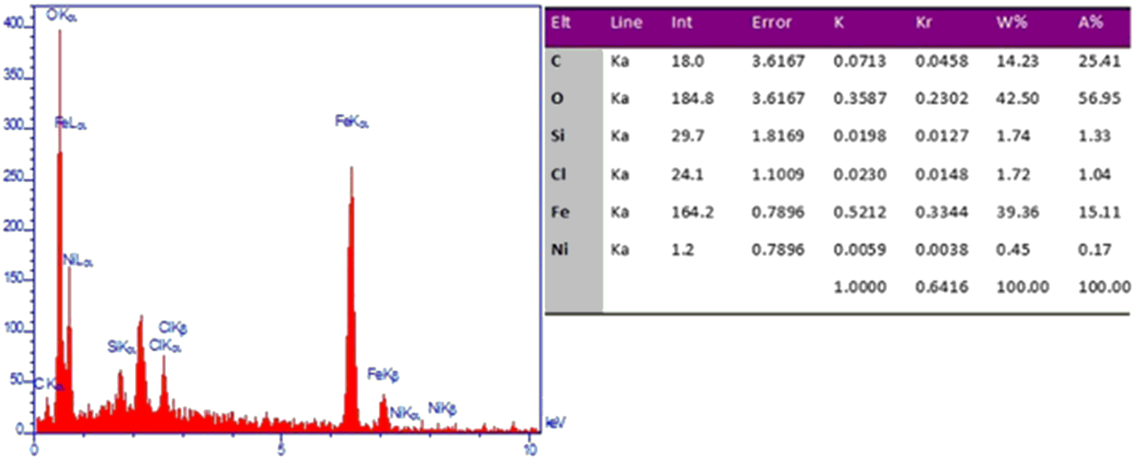


FIGURE 2 Scanning electron microscopy (SEM) images of MNP–$(CH_2)_3$PEG–Ni



Elt	Line	Int	Error	K	Kr	W%	A%
C	Ka	18.0	3.6167	0.0713	0.0458	14.23	25.41
O	Ka	184.8	3.6167	0.3587	0.2302	42.50	56.95
Si	Ka	29.7	1.8169	0.0198	0.0127	1.74	1.33
Cl	Ka	24.1	1.1009	0.0230	0.0148	1.72	1.04
Fe	Ka	164.2	0.7896	0.5212	0.3344	39.36	15.11
Ni	Ka	1.2	0.7896	0.0059	0.0038	0.45	0.17
				1.0000	0.6416	100.00	100.00

FIGURE 3 EDS of MNP–$(CH_2)_3$PEG–Ni

magnetization of the Fe_3O_4@SiO_2-DMG–Ni (II) catalyst was about 35 emu g^{-1}.[34] The amount was decreased compared with the saturation magnetization of the Fe_3O_4 MNPs (about 60 emu g^{-1})[35] owing to the anchoring of the SiO_2 and Ni–PEG complex onto the surface of the Fe_3O_4 MNPs. In comparison, the saturation magnetization of MNP–$(CH_2)_3$PEG–Ni was more than the reported MNP–DMG–Ni (II) nanocatalyst.

The XRD patterns of magnetic nanocatalyst showed five distinct peaks at $2\theta = 2, 11, 51, 58$ and $86°$,

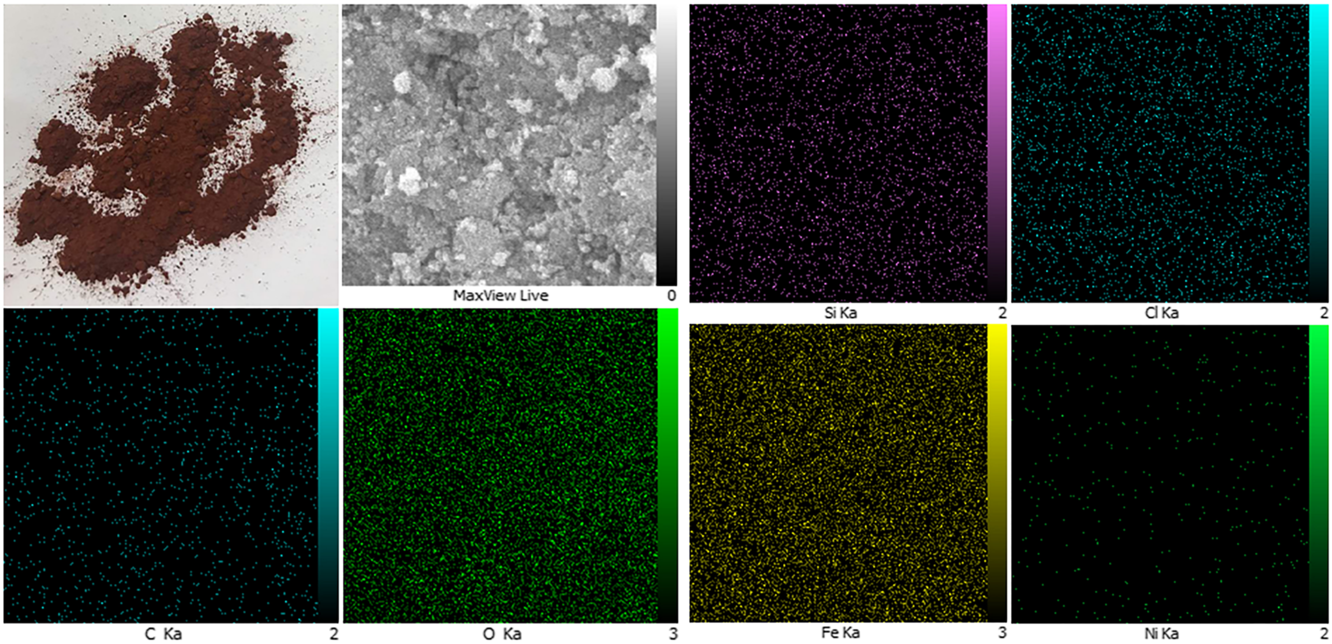


FIGURE 4 The dark-brown colored powder and SEM–EDS mapping of MNP–$(CH_2)_3$PEG–Ni (distribution of C, O, Si, Cl, Fe and Ni elements)

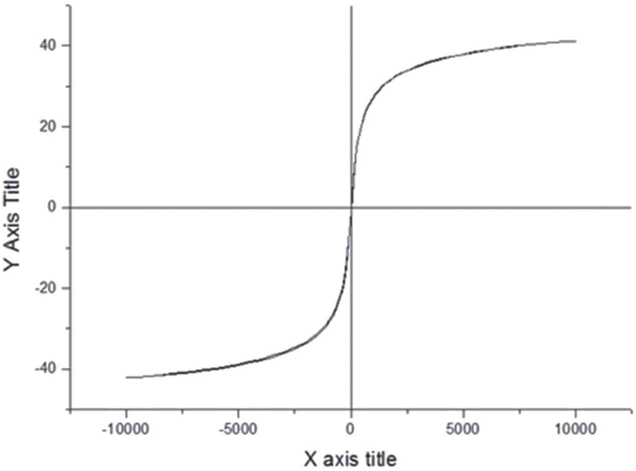


FIGURE 5 Vibrating sample magnetometry (VSM) analysis of the MNP–$(CH_2)_3$PEG–Ni

respectively. Two broad and single diffraction peaks at $2\theta = 2°$ and 11° observed in XRD patterns of MNP–$(CH_2)_3$PEG–Ni confirmed the presence and preservation of the mesoporous framework of the nano-structures. Two peaks at $2\theta = 51$ and 58° confirmed the existence of Fe_3O_4 nanoparticles (Figure 6).

The thermogravimetric analysis - differential scanning calorimetry (TGA-DSC) analysis of MNP–$(CH_2)_3$PEG–Ni is shown in Figure 7. TGA/DSC analysis of MNP–$(CH_2)_3$PEG–Ni showed weight loss owing to decomposition upon heating. The 10.13% weight loss below 350°C was attributed removal of solvent molecules and PEG. The weight loss between 350 and 800°C was associated with the decomposition of organic functional groups located inside the framework. The TGA diagrams confirmed the formation of Ni complex.

Figure 8 shows the nitrogen adsorption and desorption isotherms of the MNP–$(CH_2)_3$PEG–Ni nanocatalyst. The hole properties of the made microspheres were specified by nitrogen absorption–desorption. The specific plane area was computed using the BET technique for the synthesized MNP–$(CH_2)_3$PEG–Ni nanocatalyst and it was 38.487 $m^2.g^{-1}$. The hole volume was 8.8427 $cm^3.g^{-1}$ (Figure 8). The corresponding hole size distribution of the nanocatalyst was specified as 10.859 nm, which was done using the Barrett–Joyner–Halenda technique (for more information see the Supplementary Material). These results indicated that the MNP–$(CH_2)_3$PEG–Ni nanocatalyst was obtained as mesoporous type ($2 < D_v < 50$ nm, D_v is the particle diameter of the volume distribution).

2.2 | Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones by catalytic amounts of Fe_3O_4 @ PEG–Ni

After provision and characterization of the magnetic nanocatalyst structure, we concentrated our attention on the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones (**3a–l**) in the reaction of various aldehydes (**1a–l**) with 2-aminobenzamide (**2**). In finding a simple, proficient and eco-friendly method for the synthesis of **3a–l** in the presence of Fe_3O_4@PEG–Ni as an efficient, stable and

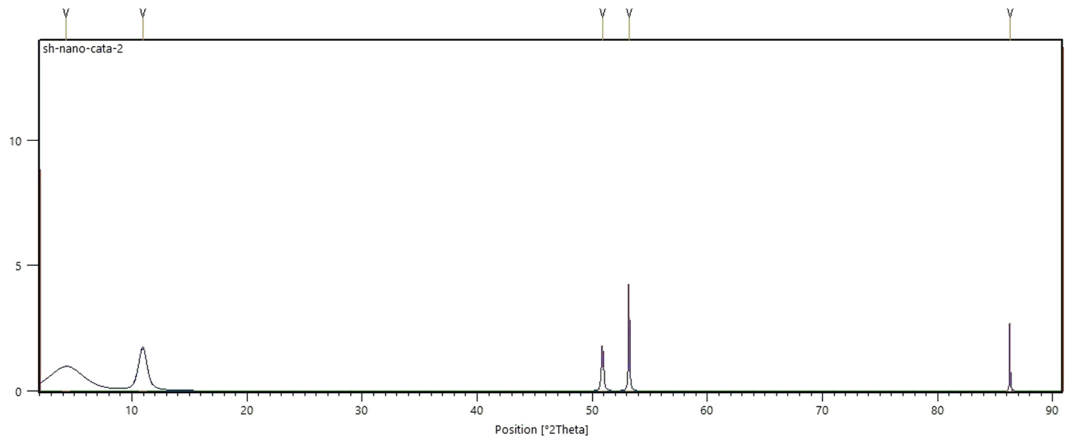
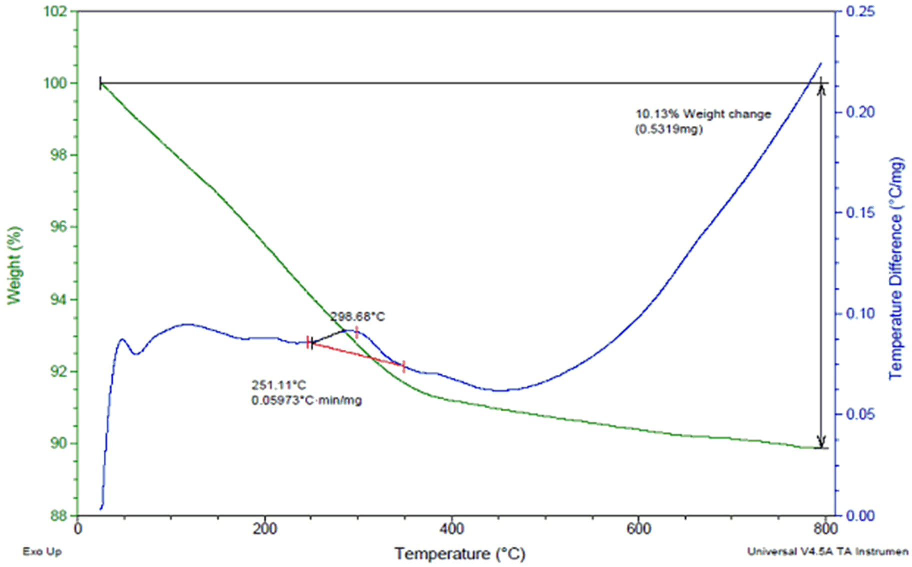


FIGURE 6 XRD patterns of MNP–$(CH_2)_3$PEG–Ni

FIGURE 7 TGA–DSC analysis of MNP–$(CH_2)_3$PEG–Ni



recyclable nanocatalyst, the reaction of 2-aminobenzamide **2** and benzaldehyde **1a** was studied to create optimal conditions. Optimization of the catalyst for the synthesis of the preferred **3** in the presence of different catalytic amount of Fe_3O_4@PEG–Ni is shown in Table 1. The best results were obtained with benzaldehyde **1a** (1.0 mmol) and 2-aminobenzamide **2** (1.0 mmol) in the presence of Fe_3O_4@PEG–Ni (5.0 mg) and Ethylene glycol (EG) (5.0 ml) at room temperature (Table 1, entry 3). The use of lower and higher amounts of catalyst (3.0 and 8.0 mg) did not affect the outcome (Table 2, entries 2 and 4, respectively). The reaction of various aldehydes **1** with 2-aminobenzamide **2** was done only after successfully synthesizing **3**. The results are reported in Table 2. In this table, it can be established that a broad range of aldehydes can react with 2-aminobenzamide, and as a result, compounds **3** are obtained. We performed the reaction of terphthalaldehyde as a dialdehyde under the same reaction and the product 2,2′-(1,4-phenylene) *bis*[2,3-dihydroquinazolin-4(1*H*)-one], **3l**, was obtained in excellent yield (Table 2, entry 12).

The productivity and capability of MNPs@PEG–Ni system in synthesis of quinazolines were highlighted by comparison of the gained result with other reported systems (Table 3). As shown in this table, MNPs@PEG–Ni is the best catalyst for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives in green solvent under gentle reaction conditions. This catalyst has important characteristics such as biocompatibility, low cost, commercial availability and also chemically stable materials. A plausible reaction mechanism for the formation of 2,3-dihydroquinazolin-4(1*H*)-ones is shown in Scheme 2.

2.3 | Recyclability of the catalyst

Reuse of catalysts is an important advantage for commercial applications. To investigate this issue, catalyst

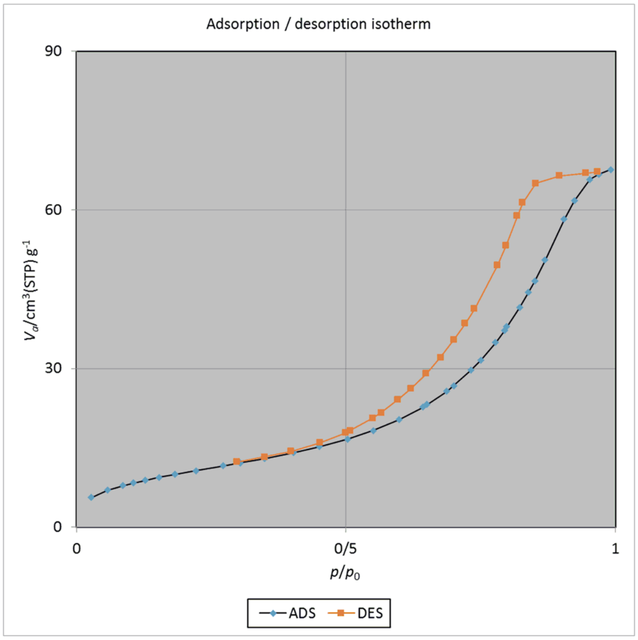


FIGURE 8 The nitrogen adsorption and desorption isotherm of the MNP–$(CH_2)_3$PEG–Ni

recovery was investigated for the one-pot cyclization reaction between benzaldehyde **1a** and 2-aminobenzamide **2** under optimized reaction conditions in ethylene glycol (Table 1, entry 3). After completion of the reaction, the catalyst was removed quickly and easily from the reaction mixture and washed several times with water and ethanol. After drying the catalyst, we used it directly for the next run. The catalyst was found to be reusable at least six times, without causing catalytic cleaning to be detectable or significantly altered (Figure 9). Reproducible catalytic activity and remarkably low leaching of MNPs with PEG were seen.

2.4 | Hot filtration

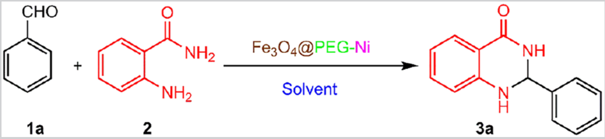
In testing for the leaching of nickel in the reaction mixture and the heterogeneity of the catalyst, hot filtration was performed for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones with 2-aminobenzamide and various derivatives of aldehydes. Also in this test, we obtained a 35% yield of product in the first 20 min of the reaction. Then, the catalyst was separated and the filtrate permitted to react further. After this hot filtration, it was concluded that no further reaction was seen. The yield of the reaction at this stage was 35%, confirming that the leaching of nickel was insignificant (Figure 10).

3 | EXPERIMENTAL

3.1 | Material and methods

All solvents, substrates and reagents were purchased from Merck and Aldrich Chemical Companies in high purity and used without further purification. FT-IR spectrometer measurements were recorded on Nexus 670 apparatus using KBr pellets (Urmia University, Urmia, Iran). Nanostructures were characterized using XRD measurements collected using X'pertpro with wavelength 1.54 Å while some diffraction patterns were entered in the 2θ range (10–80°) (Lorestan University,

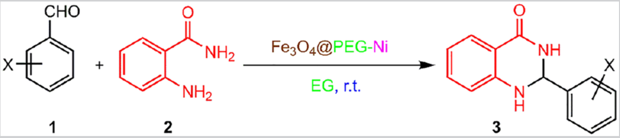
TABLE 1 Effects of different parameters on the synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (**3a**)

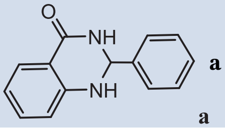
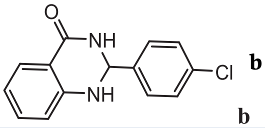
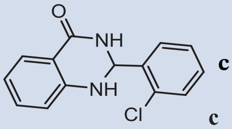
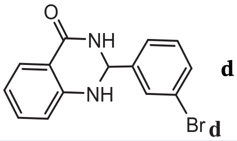
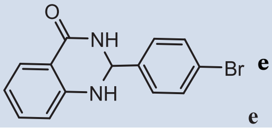
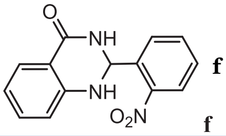
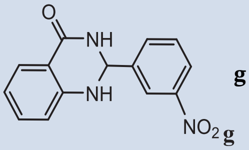
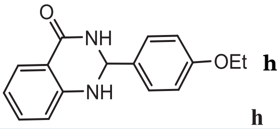
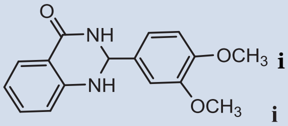
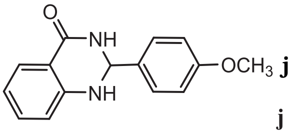


Entry	Catalyst (mg)	Solvent	Temperature (°C)	Time (min)	Yield (%)
1	0	PEG	rt	200	Trace
2	3	EG	rt	55	78
3	5	EG	rt	27	93
4	8	EG	rt	25	88
5	5	EG	60	20	89
6	5	EtOH	70	30	85
7	5	EtOAC	70	90	82
8	5	CH_2Cl_2	30	90	80

Reaction conditions: aldehyde (1.0 mmol), 2-aminobenzamide (1.0 mmol) with different solvents.

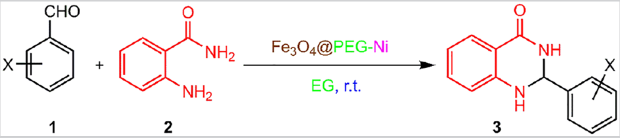
TABLE 2 Fe_3O_4 catalyzing the one-pot synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones (**3a–l**) in EG

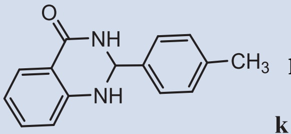
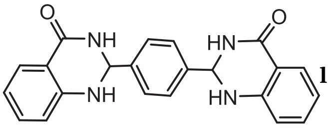


Entry	Product (3)	Time (min)	Yield (%)	M.p. (°C)^a [reference]
1	 **a**	20	94	224–226[27,29,30]
2	 **b**	20	90	201–203[29,30,36]
3	 **c**	7	78	229–231[29,30,37]
4	 **d**	10	92	186–188[26,29,30]
5	 **e**	10	95	196–198[27,29,30]
6	 **f**	5	95	191–193[29,30,37]
7	 **g**	5	95	179–181[29,30,37]
8	 **h**	45	80	167–169[26,29,30]
9	 **i**	30	93	208–210[26,29,30]
10	 **j**	35	83	188–190[27,29,30]

(Continues)

TABLE 2 (Continued)



Entry	Product (3)	Time (min)	Yield (%)	M.p. (°C)[a] [reference]
11	 **k**	25	95	224–225[27,29,30]
12	 **l**	40	93	241–242[26,29,30]

[a]The products were assigned by comparing their spectral and physical data with reliable samples and the literature.

TABLE 3 Comparison of the performance of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one with magnetic nanoparticles (MNPs)@PEG–Ni and other reported systems

Entry	Catalyst	Temperature (°C)	Time (min)	Yield (%)	Reference
1	MNPs@PEG–Ni	rt	45	95	_
2	$NaHSO_4$	70	30	94	[38]
3	Sc $(OTf)_3$	70	25	92	[39]
4	PPA-SiO_2	70	90	91	[40]
5	TFE	78	90	90	[41]
6	Ga $(OTf)_3$	70	55	87	[42]
7	TBAB	100	90	82	[43]

TBAB, Tetra-*n*-butylammonium bromide.

Khorramabad, Iran). The Barrett–Joyner–Halenda method was utilized and the pore volume and pore measure distribution were deduced from the desorption profiles of the isotherms. The BET technique was used to measure the specific surface area (Beam Gostar Taban lab, Tehran, Iran). TGA curves were entered using a Shimadzu DTG-60 instrument (Beam Gostar Taban lab, Tehran, Iran). Also, the magnetic properties of nanoparticles were measured using a VSM (PPMS-9 T) at 300 K (University of Mohaghegh Ardabili, Ardabil, Iran).

3.2 | Synthesis of Fe_3O_4 nanoparticles (MNPs)

One of the best ways to separate nanoparticles from a solution is by magnetizing the particles. Magnetic particles were synthesized by the following reaction:

Different solutions of ferrous chloride hexahydrate ($FeCl_3.6H_2O$) and ferric chloride tetra hydrate ($FeCl_2 \cdot 4H_2O$) were prepared with a molar value of Fe^{3+}/Fe^{2+}. In the typical method, 1.2 g of $FeCl_2 \cdot 4H_2O$ and 2.4 g of $FeCl_3 \cdot 6H_2O$ were dissolved in 40 ml of deionized water. Then, 10 ml of 25% ammonium hydroxide (NH_4OH) solution was slowly added to the above mixture and it was vigorously stirred (60 min, 700 rpm, 70°C). The result of the reaction was a black precipitate that was subsequently isolated with the aid of an external magnet. The separated nanoparticles were rinsed with ethanol and water and finally dried in a vacuum 50°C for 20 h.

3.3 | Preparation of MNP–$(CH_2)_3Cl$

The MNP powder (1.3 g) with of 3-chloropropyltrimethoxysilane (1.5 g) was added to

SCHEME 2 Proposed mechanism for synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones in the presence of MNP–$(CH_2)_3$PEG–Ni

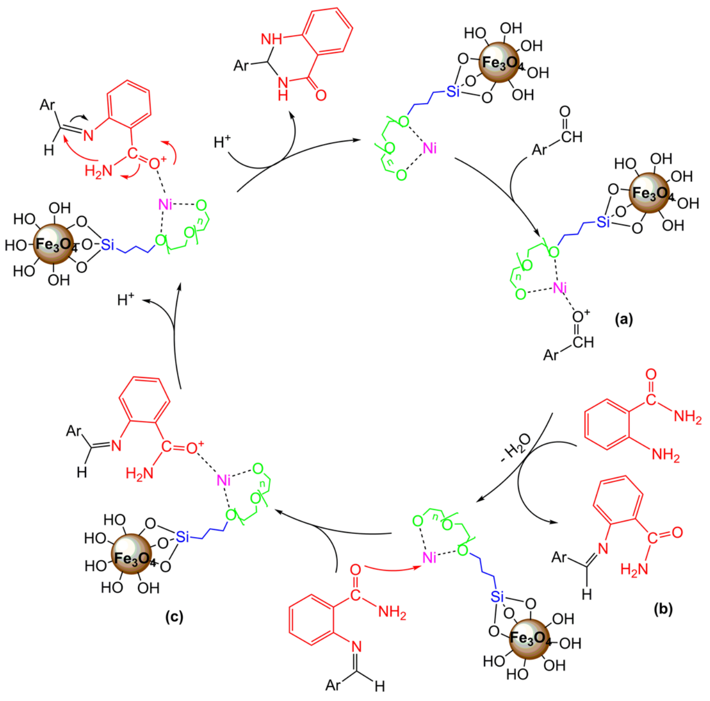
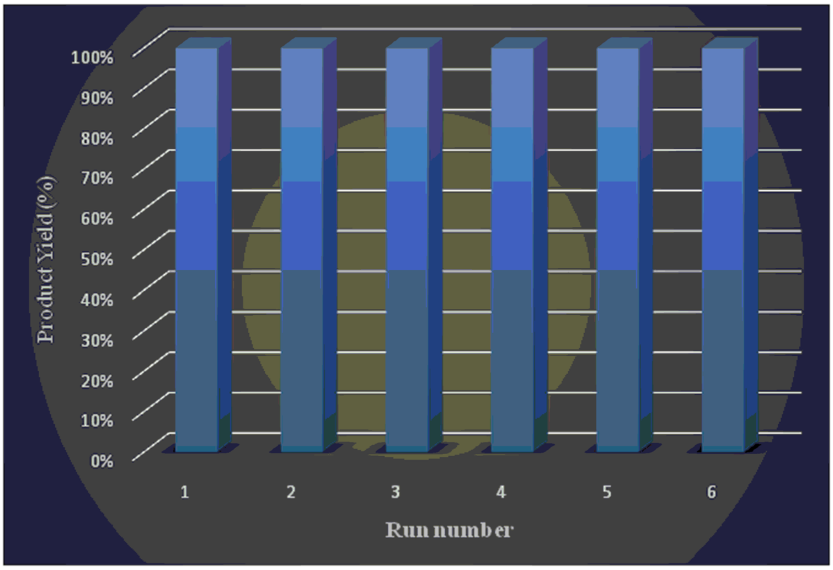


FIGURE 9 Recycling of MNP–$(CH_2)_3$PEG–Ni in the construction of 2,3-dihydroquinazolin-4(1*H*)-ones



ethanol (25 ml) and the combination was stirred under reflux for 24 h. Then the resulting solid was washed with EtOH and then, as the last step, the solid was dried under vacuum in order to obtain MNP–$(CH_2)_3$–Cl.

3.4 | Preparation of support PEG

In a 50 ml round-bottom flask a mixture of MNP–$(CH_2)_3$–Cl (1 g), PEG-600 (1 g) and EtOH (35 ml) under reflux was stirred for 20 h. Then the product was washed repeatedly with EtOH. Finally, it was dried for 12 h in a vacuum oven at 60°C.

3.5 | Preparation support PEG–Ni

The functionalized support PEG (1.6 g) was blended with Ni $(NO_3)^{\cdot}6H_2O$ (0.8 g) in EtOH (30 ml) and stirred under reflux for 20 h. The resulting solid was collected using an external magnet. Then, in order to remove any unanchored metal ions, it was washed repeatedly with EtOH

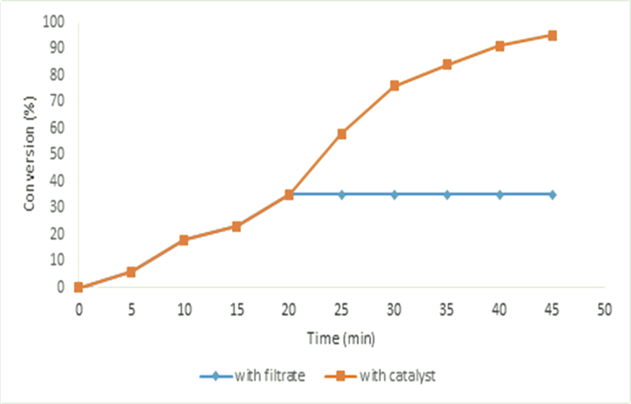


FIGURE 10 Hot filtration test for MNP–$(CH_2)_3$PEG–Ni

and dried under vacuum at 60°C for 15 h. Finally, the catalyst obtained was designated as Fe_3O_4@PEG–Ni.

3.6 | General procedure for the preparation of 2,3-dihydroquinazolin-4(1*H*)-ones

Firstly, to a one-necked flask were added aldehydes (1 mmol), 2-aminobenzamide (1 mmol), nanocatalyst (5 mg) and ethylene glycol (5 ml). The mixture was stirred at room temperature for a suitable reaction time (Tables 1 and 2). Then the reaction's progress was checked using thin layer chromatography (TLC). At the end of the reaction, the catalyst was separated using an external magnet and washed with ethanol and water, and vacuum dried until used for the next reaction. The reaction blend was extracted with H_2O and EtOAc. Finally, the product was recrystallized from ethanol for additional purification, obtaining 2,3-dihydroquinazolin-4(1*H*)-ones in good yields. Then, the data reaction products **3** were characterized by ^{1}H, ^{13}C NMR and FT-IR spectroscopy (See Supplementary Material). Details of the method of synthesis of the nanocatalyst are shown in Scheme 1.

4 | CONCLUSION

In summary, the Fe_3O_4@PEG–Ni nanocomposite was synthesized by means of grafting. The catalytic activity of Fe_3O_4@PEG–Ni was explored through one-pot green synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. This catalyst had a high conversion value and could be easily recovered and reused many times with no considerable loss of its catalytic activity. Briefly, the proposed catalyst was useful as well as nontoxic and adsorbent for the synthesis of these series of compounds economically and with more selectivity.

ORCID

Nader Noroozi Pesyan https://orcid.org/0000-0002-4257-434X

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rt, Room temperature.

ORCID

Nader Noroozi Pesyan https://orcid.org/0000-0002-4257-434X

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