Javad Safaei-Ghomi*, Reza Aghagoli and Hossein Shahbazi-Alavi

Synthesis of hexahydro-4-phenylquinoline-3-carbonitriles using Fe_3O_4@SiO_2-SO_3H nanoparticles as a superior and retrievable heterogeneous catalyst under ultrasonic irradiations

https://doi.org/10.1515/znb-2017-0200
Received November 10, 2017; accepted February 25, 2018

Abstract: An efficient synthesis of hexahydro-4-phenylquinoline-3-carbonitriles is described by the four-component condensation reaction of cyclohexanone, ammonium acetate, malononitrile, and aromatic aldehydes using Fe_3O_4@SiO_2-SO_3H nanoparticles as a superior and retrievable heterogeneous catalyst under ultrasonic irradiations. The reusability of the catalyst and little catalyst loading, excellent yields, short reaction times, using the sonochemical procedure as a green process and an alternative energy source are some benefits of this method.

Keywords: nanocatalyst; nano-Fe_3O_4; one-pot reaction; quinoline; ultrasonic conditions.

1 Introduction

Quinolines possess many biological activities including anti-mycobacterial [1], analgesic [2], anti-inflammatory [3], anti-cancer [4], anti-tuberculosis [5], and anti-obesity [6]. These activities make quinolines attractive targets in organic synthesis. A number of procedures were developed to improve the synthesis of quinolines in the presence of catalysts such as $SnCl_2$/$ZnCl_2$ [7], $InCl_3$ [8], $Y(OTf)_3$ [9], 1-butylimidazolium tetrafluoroborate [10], cellulose sulfuric acid [11], and I_2 [12]. Despite the availability of these procedures, there remains a need for a capable and retrievable catalyst with high catalytic activity for the preparation of quinolines. Ideally, using green and environmental catalysts, which can be easily recovered at the end of the reaction, has gained remarkable consideration in recent years [13–17]. Recently, much attention has been paid to one-pot reactions with a nanocatalyst under ultrasonic irradiation [18, 19]. The ultrasound approach decreases the reaction times by generating the activation energy in microsurroundings [20, 21]. Several nanocatalysts were utilized for the preparation of organic compounds under ultrasonic conditions [22, 23]. Magnetic materials have appeared as a proper group of heterogeneous catalysts owing to their diverse applications in synthesis and catalysis [24, 25]. To overcome the separation drawbacks of the catalysts, nanomagnetics have emerged as recoverable and retrievable catalysts. The surface of magnetic nanoparticles (MNPs) can be functionalized simply through convenient surface modifications to enable the loading of a diversity of required functionalities [26, 27]. Herein we reported the use of Fe_3O_4@SiO_2-SO_3H MNPs as an efficient catalyst for the preparation of hexahydro-4-phenylquinoline-3-carbonitrile by the four-component condensation reaction of cyclohexanone, ammonium acetate, malononitrile, and aromatic aldehyde under ultrasonic irradiation (Scheme 1).

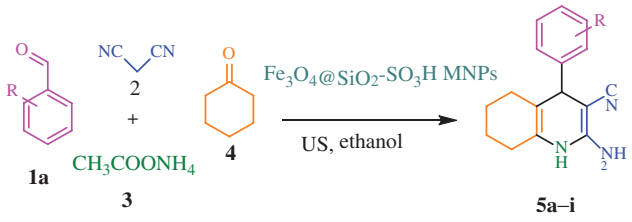
2 Results and discussion

The morphology and particle size of Fe_3O_4, Fe_3O_4@SiO_2, and Fe_3O_4@SiO_2-SO_3H nanoparticles were investigated using scanning electron microscopy (SEM). It is observed that the average size of Fe_3O_4@SiO_2-SO_3H MNPs is about 30–40 nm (Fig. S1; see Supporting Information available online).

The XRD patterns of Fe_3O_4 and Fe_3O_4@SiO_2-SO_3H are shown in Fig. S2 (Supporting Information). The characteristic peaks in the both spectra are in agreement with the standard XRD pattern of iron oxide (cubic phase). A broad peak in 2θ range of 19°C–27°C is related to the silica shell coated on Fe_3O_4 MNPs. Meanwhile, the nanocatalyst has been characterized by vibrating sample magnetometer (VSM) (Fig. S3), FT-IR (Fig. S4), energy-dispersive X-ray spectroscopy (EDS) (Fig. S5), and thermogravimetric analyzer (TGA) (Fig. S6) spectra (Supporting Information).

Initially, in order to optimize the reaction conditions, the model reaction was preceded by the four-component condensation reaction of cyclohexanone, ammonium acetate, malononitrile, and 4-chloro-benzaldehyde under different conditions. These reactions were carried out in

***Corresponding author: Javad Safaei-Ghomi,** Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, P.O. Box 87317-51167, Kashan, I.R. Iran, Phone: +98-31-55912385, Fax: +98-31-55912397, E-mail: safaei@kashanu.ac.ir
Reza Aghagoli and Hossein Shahbazi-Alavi: Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, P.O. Box 87317-51167, Kashan, I.R. Iran



Scheme 1: Synthesis of hexahydro-4-phenylquinoline-3-carbonitrile using Fe_3O_4@SiO_2-SO_3H MNPs.

the presence of various catalysts, such as $(NH_4)_2Ce(NO_3)_6$, SiO_2 nanoparticles (NPs), $BF_3 \cdot SiO_2$, ZrO_2 NPs, Fe_3O_4, and Fe_3O_4@SiO_2-SO_3H MNPs. The best results were obtained in ethanol under ultrasonic conditions (40 W) and found that the reaction gave satisfying results in the presence of 8 mg Fe_3O_4@SiO_2-SO_3H MNPs. When the reaction was carried out under reflux conditions, it gave low yields of products and took longer reaction times, while the same reaction was carried out under ultrasonic irradiation to give good yields of products in short reaction times. In further studies on the catalyst loading, we realized that the yield of compound **5a** remained almost the same when 10 mg of Fe_3O_4@SiO_2-SO_3H MNPs was utilized (Table 1). The use of lower catalyst loading (6 mg) afforded **3a** in 89% yield.

With these hopeful results in hand, we turned to investigate the scope of the reaction using various aromatic aldehydes as substrates under optimized reaction conditions. The results show the present catalytic method is extensible to a wide diversity of substrates to create a variety-oriented library of quinolines (Table 2).

After completion of the reaction, the magnetic nanocatalyst is easily separated from the product by an external magnetic field. The Fe_3O_4@SiO_2-SO_3H MNPs

Table 1: Optimization of reaction conditions using different catalysts (cyclohexanone, 2 mmol; ammonium acetate, 3 mmol; malononitrile, 2 mmol; and 4-chloro-benzaldehyde, 2 mmol).

Entry	Solvent	Catalyst (quantity)	Time (min)	Yield (%)[a]
1	–	–	300	Trace
2	CH_3CN (reflux)	$(NH_4)_2Ce(NO_3)_6$ (8 mg)	200	29
3	$CHCl_3$ (reflux)	$BF_3 \cdot SiO_2$ (15 mg)	90	45
4	EtOH (reflux)	SiO_2 NPs (15 mg)	90	32
5	EtOH (reflux)	ZrO_2 NPs (15 mg)	80	40
6	EtOH (reflux)	Fe_3O_4 MNPs (20 mg)	80	32
7	$CHCl_3$ (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (12 mg)	60	48
8	DMF (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (12 mg)	40	60
9	CH_3CN (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (12 mg)	40	68
10	EtOH (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (10 mg)	40	80
11	EtOH (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (12 mg)	40	84
12	EtOH (reflux)	Fe_3O_4@SiO_2-SO_3H MNPs (14 mg)	40	84
13	EtOH (US)[b]	Fe_3O_4@SiO_2-SO_3H MNPs (6 mg)	10	89
14	EtOH (US)	Fe_3O_4@SiO_2-SO_3H MNPs (8 mg)	10	94
15	EtOH (US)	Fe_3O_4@SiO_2-SO_3H MNPs (10 mg)	10	94

[a]Isolated yield. [b]Ultrasonic irradiation (40 W).

Table 2: Preparation of quinolines using Fe_3O_4@SiO_2-SO_3H MNPs under ultrasonic irradiation.

Entry	Aldehyde	Product	Time (min)	Yield (%)[a]	M. p. (°C)	M. p. (°C) [Ref.]
1	4-Cl-C_6H_4	**4a**	10	94	252–254	252–253 [28]
2	4-Me-C_6H_4	**4b**	15	89	230–232	–
3	4-OMe-C_6H_4	**4c**	15	85	238–240	–
4	4-Br-C_6H_4	**4d**	10	95	262–264	–
5	4-F-C_6H_4	**4e**	10	96	240–242	–
6	4-NO_2-C_6H_4	**4f**	10	96	276–278	–
7	3-OMe-C_6H_4	**4g**	15	88	222–224	–
8	3-Me-C_6H_4	**4h**	15	90	212–214	–
9	C_6H_5	**4i**	10	92	225–227	225–227 [28]

[a]Isolated yields.

was washed four times with ethanol and dried at room temperature for 10 h. The reusability of the Fe_3O_4@SiO_2-SO_3H MNPs catalyst was examined and it was found that product yields decreased to a small extent on each reuse (run 1, 94%; run 2, 94%; run 3, 93%; run 4, 93%; run 5, 92%, run 6, 92%).

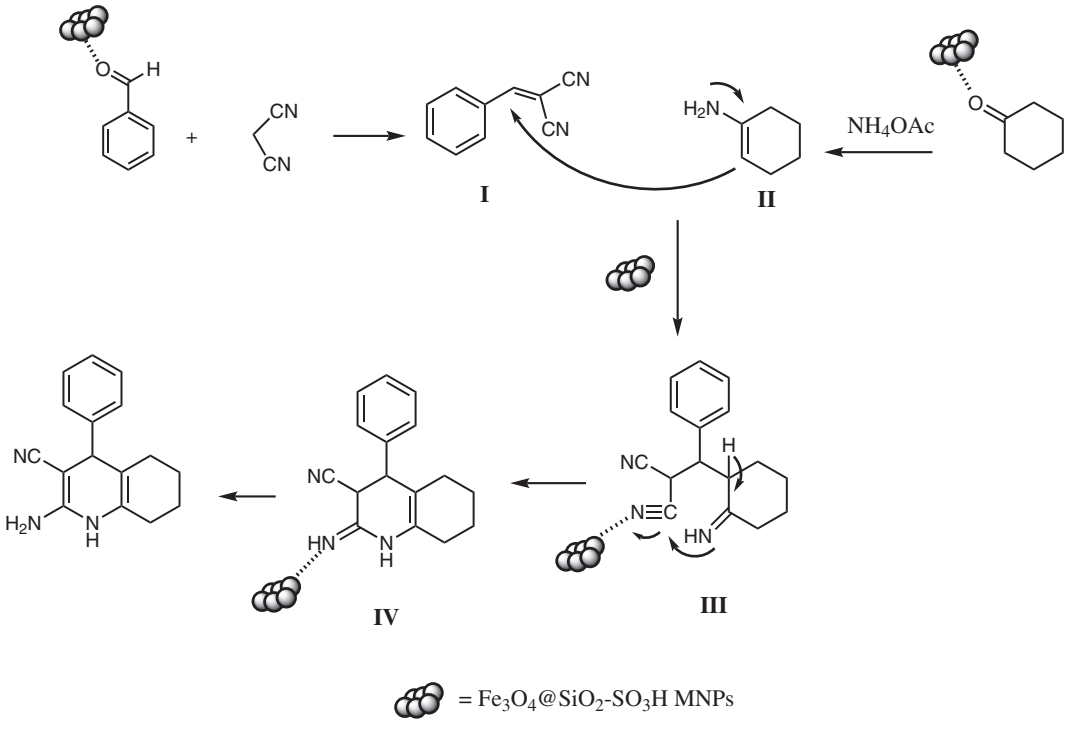
A probable mechanism for the synthesis of quinolines using Fe_3O_4@SiO_2-SO_3H MNPs is shown in Scheme 2. At first, we assumed that the reaction occurs via a Knoevenagel condensation between benzaldehydes and malononitrile, forming the intermediate **I** on the active sites of nanocatalyst. Then, the Michael addition of cyclohex-1-enamine **II** with intermediate **I** affords the intermediate **III** that undergoes cyclization to the title product. This proposed mechanism is also supported by literature examples [28, 29]. In this mechanism nano-Fe_3O_4@SiO_2-SO_3H acts as a highly efficient and green catalyst activating the C=O, C≡N groups for better reaction with nucleophiles.

3 Conclusions

In conclusion, we have developed a straightforward and efficient method for the synthesis of hexahydro-4-phenylquinoline-3-carbonitriles using Fe_3O_4@SiO_2-SO_3H MNPs as high performance catalyst under ultrasonic conditions. The advantages offered by this method include the use of a superior catalyst, recoverability of the catalyst, little catalyst loading, low reaction times, simple procedure, high atom economy, and excellent yields.

4 Experimental section

^{1}H NMR and ^{13}C NMR spectra were recorded using a Bruker Avance-400 MHz spectrometer using $CDCl_3$ as a solvent. The elemental analyses (C, H, N) were obtained using a Carlo ERBA Model EA 1108 analyzer. Fourier transform infrared spectra were recorded using a WQF-510, spectrometer 550 Nicolet. The EDS measurements were performed using a SAMX analyzer. Powder X-ray diffraction measurements were carried out using a Philips diffractometer of X'pert Company with monochromatized CuKα radiation ($\lambda = 1.54056$ nm). A TGAQ5 was used to study the thermal properties of the compounds under an inert N_2 atmosphere at 20 mL min^{-1} and at a heating rate of 10°C min^{-1}. The SEM images were taken by MIRA3-TESCAN. The magnetic properties of nanoparticles were measured using a VSM (Meghnatis Daghigh Kavir Co.; Kashan Kavir) at 300 K in Kashan University, Iran.



Scheme 2: Probable mechanism for the formation of hexahydro-4-phenylquinoline-3-carbonitrile.

4.1 Preparation of Fe_3O_4 nanoparticles

Fe_3O_4 nanoparticles were synthesized by co-precipitation method. $FeCl_3 \cdot 6H_2O$ (11.68 g) and $FeCl_2 \cdot 4H_2O$ (4.30 g) were dissolved in 200 mL deionized water, then 15 mL of aqueous NH_3 (25%) was added to the solution dropwise under N_2 atmosphere and vigorous stirring at 70°C–75°C. The magnetic nanoparticles were separated from solution by using an external magnet and washed twice with deionized water.

4.2 Preparation of Fe_3O_4@SiO_2 nanoparticles

Some 1 g of Fe_3O_4 MNPs was dispersed in 20 mL ethanol in ultrasonic bath and sonicated for 30 min at room temperature. Then, 6 mL of aqueous NH_3 (25%) and 2 mL of tetraethyl orthosilicate were added to the solution. The resulting solution was stirred at 35°C–40°C for 24 h. The Fe_3O_4@SiO_2 MNPs were separated from solution by using an external magnet and washed with ethanol (3×15 mL) and dried at room temperature.

4.3 Preparation of Fe_3O_4@SiO_2-SO_3H nanoparticles

First, 1 g of Fe_3O_4@SiO_2 was dispersed in dry CH_2Cl_2 (16 mL) and sonicated for 10 min. Then, chlorosulfonic acid (0.8 mL in dry CH_2Cl_2) was added dropwise to a cooled (ice bath) solution of Fe_3O_4@SiO_2, during a period of 30 min under vigorous stirring. The mixture was stirred for 60 min, while the residual HCl was removed by suction. The resulting MNPs were separated by using a magnet, washed several times with dried CH_2Cl_2 and methanol before being dried under vacuum at 60°C. The number of H^+ sites of Fe_3O_4@SiO_2-SO_3H MNPs was determined by pH-ISE conductivity titration (Denver Instrument Model 270) and found to be 1.69 H^+ sites per 1 g of solid acid at 25°C.

4.4 General procedure for the preparation of hexahydro-4-phenylquinoline-3-carbonitrile

A mixture of aldehydes (2 mmol), cyclohexanone (2 mmol), ammonium acetate (3 mmol), malononitrile (2 mmol), and 8 mg of Fe_3O_4@SiO_2-SO_3H MNPs in EtOH (15 mL) was sonicated at 40 W power. After completion of the reaction, the nanocatalyst was removed by an external magnet and reused. Then, the solid product was collected by filtration and recrystallized from ethanol to afford the pure product.

5 Spectral data of products

5.1 2-Amino-4-(4-chlorophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5a): [28]

White solid, yield: 94%; m. p. 252°C–253°C. – IR (KBr): ν = 3419, 3342, 3250, 2943, 2866, 2211, 1644, 1492 cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.58–1.75 (m, 4H, $2CH_2$), 2.20 (m, 2H, CH_2), 2.71 (m, 2H, CH_2), 4.59 (s, 2H, NH_2), 5.93 (s, 1H, CH), 7.33–7.55 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.22, 23.54, 24.96, 28.75, 37.20, 80.43, 109.53, 116.32, 128.65, 129.48, 132.87, 135.90, 140.54, 160.42. – Analysis for $C_{16}H_{16}ClN_3$: calcd. C 67.25, H 5.64, N 14.70; found C 67.20, H 5.58, N 14.64.

5.2 2-Amino-4-(4-methylphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5b)

White solid, yield: 89%; m. p. 230°C–232°C. – IR (KBr): ν = 3422, 3336, 3250, 2947, 2865, 2212, 1646, 1495 cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.50–1.62 (m, 4H, $2CH_2$), 2.15 (m, 2H, CH_2), 2.32 (s, 3H, CH_3), 2.60 (m, 2H, CH_2), 4.67 (s, 2H, NH_2), 5.98 (s, 1H, CH), 7.20–7.59 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.03, 21.19, 23.50, 24.85, 28.64, 37.15, 79.98, 108.50, 116.18, 128.54, 129.23, 133.07, 135.07, 140.05, 160.32. – Analysis for $C_{17}H_{19}N_3$: calcd. C 76.95, H 7.22, N 15.84; found: C 76.85, H 7.14, N 15.80.

5.3 2-Amino-4-(4-methoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5c)

White solid, yield: 85%; m. p. 238°C–240°C. – IR (KBr): ν = 3425, 3338, 3252, 2940, 2863, 2214, 1648, 1499cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.52–1.67 (m, 4H, $2CH_2$), 2.12 (m, 2H, CH_2), 2.43 (m, 2H, CH_2), 3.65 (s, 3H, OCH_3), 4.52 (s, 2H, NH_2), 5.64 (s, 1H, CH), 7.12–7.43 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$) = δ (ppm): 21.17, 23.43, 24.80, 27.55, 37.15, 54.32, 79.93, 108.43, 116.13, 128.58, 128.93, 132.09, 134.12, 155.12, 160.42. – Analysis for $C_{17}H_{19}N_3O$: calcd. C 72.57, H 6.81, N 14.94; found: C 72.50, H 6.75, N 14.87.

5.4 2-Amino-4-(4-bromophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5d)

White solid, yield: 95%; m. p. 262°C–264°C. – IR (KBr): ν = 3423, 3334, 3246, 2940, 2865, 2210, 1643, 1494 cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.67–1.87 (m, 4H, $2CH_2$), 2.18 (m, 2H, CH_2), 2.52 (m, 2H, CH_2), 4.68 (s, 2H, NH_2), 6.02 (s, 1H, CH), 7.58–7.88 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 20.98, 23.62, 24.74, 28.70, 37.25, 80.05, 108.59, 116.36, 128.69, 129.54, 132.72, 135.95, 141.54, 160.35. – Analysis for $C_{16}H_{16}BrN_3$: calcd. C 58.19, H 4.88, N 12.72; found: C 58.12, H 4.84, N 12.65.

5.5 2-Amino-4-(4-fluorophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5e)

White solid, yield: 96%; m. p. 240°C–24°C. – IR (KBr): ν = 3420, 3338, 3246, 2948, 2859, 2218, 1640, 1495 cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.65–1.85 (m, 4H, $2CH_2$), 2.28 (m, 2H, CH_2), 2.59 (m, 2H, CH_2), 4.69 (s, 2H, NH_2), 6.03 (s, 1H, CH), 7.44–7.65 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.02, 23.53, 24.72, 28.62, 37.22, 80.02, 108.55, 116.32, 128.60, 129.58, 132.74, 138.51, 157.42, 160.20. – Analysis for $C_{16}H_{16}FN_3$: calcd. C 71.36, H 5.99, N 15.60; found: C 71.31, H 5.92, N 15.55.

5.6 2-Amino-4-(4-nitrophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5f)

White solid, yield: 96%; m. p. 276°C–278°C. – IR (KBr): ν = 3422, 3335, 3241, 2940, 2854, 2214, 1643, 1494 cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.55–1.88 (m, 4H, $2CH_2$), 2.22 (m, 2H, CH_2), 2.54 (m, 2H, CH_2), 4.88 (s, 2H, NH_2), 6.12 (s, 1H, CH), 7.65–8.12 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.15, 23.80, 24.90, 28.88, 37.15, 80.08, 109.52, 117.36, 129.05, 129.88, 132.79, 145.75, 147.59, 160.39. – Analysis for $C_{16}H_{16}N_4O_2$: calcd. C 64.85, H 5.44, N 18.91; found: C 64.79, H 5.39, N 18.85.

5.7 2-Amino-4-(3-methoxyphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5g)

White solid, yield: 88%; m. p. 22°C–224°C. – IR (KBr): ν = 3412, 3326, 3250, 2943, 2855, 2218, 1649, 1496cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.58–1.69 (m, 4H, $2CH_2$), 2.14 (m, 2H, CH_2), 2.49 (m, 2H, CH_2), 3.75 (s, 3H, OCH_3), 4.62 (s, 2H, NH_2), 5.68 (s, 1H, CH), 7.10–7.55 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.19, 23.41, 24.89, 27.59, 37.10, 54.31, 79.85, 108.33, 116.23, 128.62, 128.84, 132.12, 134.18, 155.19, 160.23. – Analysis for $C_{17}H_{19}N_3O$: calcd. C 72.57, H 6.81, N 14.94; found: C 72.52, H 6.76, N 14.89.

5.8 2-Amino-4-(3-methylphenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (5h)

White solid, yield: 90%; m. p. 212°C–214°C. – IR (KBr): ν = 3414, 3330, 3246, 2943, 2860, 2218, 1632, 1486cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.55–1.68 (m, 4H, $2CH_2$), 2.08 (m, 2H, CH_2), 2.29 (s, 3H, CH_3), 2.48 (m, 2H, CH_2), 4.74 (s, 2H, NH_2), 5.85 (s, 1H, CH), 7.23–7.64 (m, 5H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 20.94, 21.32, 23.64, 24.83, 28.60, 37.19, 79.85, 109.55, 116.28, 128.57, 129.28, 133.09, 134.07, 141.02, 160.12. – Analysis for $C_{17}H_{19}N_3$: calcd. C 76.95, H 7.22, N 15.84; found: C 76.88, H 7.17, N 15.78.

5.9 2-Amino-1,4,5,6,7,8-hexahydro-4-phenylquinoline-3-carbonitrile (5i): Ref. [28]

White solid, yield: 92%; m.p. 225°C–227°C – IR (KBr): ν = 3418, 3340, 3252, 2944, 2868, 2212, 1643, 1495cm^{-1}. – ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.50–1.78 (m, 4H, $2CH_2$), 2.14 (m, 2H, CH_2), 2.50 (m, 2H, CH_2), 4.65 (s, 2H, NH_2), 5.91 (s, 1H, CH), 7.44–7.68 (m, 6H, Ar–H, NH). – ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 21.25, 23.44, 24.85, 28.70, 37.34, 80.40, 109.58, 116.30, 128.54, 129.58, 132.80, 135.85, 141.52, 160.28. – Analysis for $C_{16}H_{17}N_3$: calcd. C 76.46, H 6.82, N 16.72; Found: C 76.42, H 6.75, N 16.65.

6 Supporting Information

Further information on synthesis and characterization of the MNPs as well as copies of the NMR spectra of the products are given in the Supporting Information available online (https://doi.org/10.1515/znb-2017-0200).

Acknowledgement: The authors are grateful to the University of Kashan for supporting this work.

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Supplemental Material: The online version of this article offers supplementary material (https://doi.org/10.1515/znb-2017-0200).