A simple synthesis of magnetic nanoparticles-supported 4-aminomethylbenzoic acid as a highly efficient and reusable catalyst for synthesis of 2-amino-4$H$-chromene derivatives

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Abstract A new 4-aminomethylbenzoic acid-functionalized Fe$_3$O$_4$ magnetic nanoparticles as a hybrid heterogeneous catalyst was synthesised and characterized by FT-IR, XRD, TGA, TEM, SEM and VSM techniques. The catalytic activity of this nanocatalyst was probed through one-pot synthesis of 2-amino-4$H$-chromene derivatives from three component reactions of various aldehydes, malononitrile and dimeredone. The chemically and thermally stable catalyst was easily recovered using an external magnet and reused for at least five successive runs without significant loss of its activity. The simplicity of the method, high yields of the products, mild reaction conditions and low reaction times are the other advantages of this procedure.

Keywords Nanoparticle · Heterogeneous catalyst · 4-Aminomethylbenzoic acid · 2-Amino-4$H$-chromene · Aldehydes · Dimeredone

Introduction

Synthesis of new magnetic nanoparticles-supported catalyst has attracted considerable attention due to their facile separation from the reaction mixture using an external magnet and providing improved reusability of the catalyst by sustaining their catalytic efficiency after several repeated reactions [1]. This type of catalyst support is now growing and finds a powerful position in catalytic reactions. The gap between homogeneous and heterogeneous catalysis is bridged by surface
functionalization of magnetic nanoparticles. These catalysts are widely used in biotechnology since they have good biocompatibility and biodegradability characteristics which could be important for functional organic materials grafted to Fe$_3$O$_4$ nanoparticles [2, 3]. The use of magnetic nanoparticles without surface functionalization as catalysts is not promising, since their surface is highly active and leads to the agglomeration of the catalyst particles; coating the catalyst surface with shell as prevents this deficiency [4–8]. It is important to note that organic catalyst-functionalized magnetic nanoparticles indicate not only a high degree of chemical and thermal stability but also high catalytic activity [9–11].

Compounds containing 2-amino-4$H$-chromene are found in a number of natural products such as tannins and polyphenols which are commonly found in a variety of fruits, vegetables, teas and red wines [12]. The interest in these compounds is increasing because of their reported benefit to health. In addition, the 2-amino-4$H$-chromene moiety is present in a variety of naturally occurring compounds that have anticoagulant, anticancer, anti-ancaphylactia, antibacterial and fungicidal activities [13–18].

In the present study, we present our results on the preparation and characterization of 4-aminomethylbenzoic acid (AMBA)-functionalized Fe$_3$O$_4$ magnetic nanoparticles (AMBA–Fe$_3$O$_4$) as an active and stable magnetically separable basic nanocatalyst and its catalytic application for the one-pot synthesis of 2-amino-4$H$-chromene derivatives from dimedone, various arylaldehydes and malononitrile in ethanol under mild conditions (Scheme 1).

![Scheme 1](image-url)
Experimental

Physical measurements

Melting points were measured on an Electrothermal 9100 apparatus. The X-ray powder diffraction (XRD) of the catalyst was carried out on a Philips PW 1830 X-ray diffractometer using a CuKα source (λ = 1.542 Å) in a range of Bragg’s angle (10°–80°) at room temperature. Scanning electron microscopy (SEM) analyses were taken using VEGA//TESCAN KYKY-EM 3200 microscope (acceleration voltage 26 kV). Transmission electron microscopy (TEM) experiments were conducted on a Philips EM 208 electron microscope. Thermogravimetric analysis (TGA) was recorded on a Stanton Red craft STA-780 (London, UK). NMR spectra were recorded on a Bruker DRX-400 AVANCE instrument (300 MHz for 1H, 75 MHz for 13C). The spectra were measured in DMSO-d6 as the solvent. FT-IR spectra were recorded on an FT-IR Bruker vector 22 spectrophotometer. Magnetic measurements were performed using a vibration sample magnetometer (VSM, MDK, and Model 7400) analysis.

General procedure

Preparation of 4-aminomethylbenzoic acid functionalized Fe₃O₄ nanoparticles

FeCl₃·6H₂O (2.43 g, 0.09 mol) and FeCl₂·4H₂O (0.89 g, 0.0045 mol) were dispersed in 100 mL distilled water by sonication until the salts dissolved completely. Then, 0.3 g of AMBA in 10 ml NH₄OH solution was added to the above mixture under constant nitrogen flow, and, as a result, a black suspension was formed. This suspension was refluxed at 100 °C for 12 h. AMBA–Fe₃O₄ nanoparticles were separated from the aqueous solution by a magnetic field, washed with distilled water four times and then dried in an oven overnight (Scheme 2).
General procedure for the synthesis of 2-amino-4H-chromene derivatives

To a 3 mL mixture of ethanol, aldehyde (1 mmol), malononitrile (1 mmol, 0.066 g), dimedone (1 mmol, 0.141 g) and AMBA–Fe₃O₄ magnetic nanoparticles (0.05 g) as a catalyst were added and the mixture was stirred at 60 °C for a specific time. When the reaction was complete (monitored by TLC), the solvent was evaporated. Then, the reaction mixture was dissolved in CH₂Cl₂ (15 mL), and subsequently AMBA–Fe₃O₄ was separated by a magnetic field and washed with dry CH₂Cl₂ three times and checked for its reusability. The solvent of the solution containing the product was evaporated, the solid residue was recrystallized using ethanol and the product obtained as a white powder. All of the desired products were characterized by comparison of their physical data with those reported in the literature.

Selected spectra of the products

2-Amino-4-phenyl-3-cyano-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b]pyran (Table 2, entry 1; see later): white solid; mp = 231–232 °C; IR (KBr): \( \nu_{\text{max}} = 3452 \text{ and } 3426 (\text{NH}_2), 2199 (\text{CN}), 1668 (\text{C}=\text{O}), 1222 (\text{C}–\text{O}) \text{ cm}^{-1} \); \(^1\)H NMR (300 MHz, DMSO-d₆): \( \delta = 0.99 (2s, 6\text{H, } 2\text{CH}_3), 2.09 \text{ and } 2.25 (d, 2\text{H, } J = 15.9 \text{ Hz, } 16.2 \text{ Hz CH}_2), 3.29 (d, 2\text{H, } J = 6.6 \text{ Hz CH}_2), 4.16 (s, 1\text{H, CH}), 7.00 (s, 2\text{H, NH}_2), 7.17 (\text{dd, 3H, } J = 9.0 \text{ Hz, } 3\text{CH}_{\text{arom}}), 7.27 (d, 2\text{H, } J = 9.0 \text{ Hz, } 2\text{CH}_{\text{arom}}) \text{ ppm}; \(^{13}\)C NMR (75 MHz, DMSO-d₆): \( \delta = 28.9, 32.3, 36.0, 50.4, 58.8, 113.2, 120.2, 127.0, 127.6, 128.1, 128.8, 145.2, 158.9, 162.9, 196.1 \text{ ppm.}

2-Amino-4-(2-chlorophenyl)-3-cyano-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b]pyran (Table 2, entry 8; see later): white solid; mp = 192–194 °C; IR (KBr): \( \nu_{\text{max}} = 3426 \text{ and } 3229 (\text{NH}_2), 2192 (\text{CN}), 1678 (\text{C}=\text{O}), 1220 (\text{C}–\text{O}) \text{ cm}^{-1} \); \(^1\)H NMR (300 MHz, DMSO-d₆): \( \delta = 1.04 (2s, 6\text{H, } 2\text{CH}_3), 2.07 \text{ and } 2.25 (\text{AB quartet, } 2\text{H, } J = 15.9 \text{ Hz, CH}_2), 2.46 (m, 2\text{H, CH}_2), 4.69 (s, 1\text{H, CH}), 7.00 (s, 2\text{H, NH}_2), 7.17 (d, 2\text{H, } J = 4.2 \text{ Hz, } 2\text{CH}_{\text{arom}}), 7.35 (d, 2\text{H, } J = 4.2 \text{ Hz, } 2\text{CH}_{\text{arom}}) \text{ ppm}; \(^{13}\)C NMR (75 MHz, DMSO-d₆): \( \delta = 27.3, 28.9, 32.2, 33.3, 40.0, 50.4, 27.3, 112.2, 119.7, 127.9, 128.6, 129.9, 130.4, 132.6, 142.0, 159.1, 163.6, 196.0 \text{ ppm.}

2-Amino-4-(4-methylphenyl)-3-cyano-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b]pyran (Table 2, entry 9; see later): white solid; mp = 214–215 °C; IR (KBr): \( \nu_{\text{max}} = 3420 \text{ and } 3282 (\text{NH}_2), 2195 (\text{CN}), 1679 (\text{C}=\text{O}), 1216 (\text{C}–\text{O}) \text{ cm}^{-1} \); \(^1\)H NMR (300 MHz, DMSO-d₆): \( \delta = 0.97 (2s, 6\text{H, } 2\text{CH}_3), 2.24 (3\text{H, CH}_3), 2.46 (d, 4\text{H, CH}_2), 4.12 (s, 1\text{H, CH}), 6.99 (m, 6\text{H, } 4\text{ CH}_{\text{arom}} \text{ and } \text{NH}_2); \(^{13}\)C NMR (75 MHz, DMSO-d₆) ppm: \( \delta = 21.0, 28.9, 32.4, 39.4, 39.7, 50.4, 58.9, 113.3, 120.2, 127.5, 129.3, 136.1, 142.3, 158.9, 162.7, 196.1 \text{ ppm.}

2-Amino-4-(4-hydroxy-3-methoxyphenyl)-3-cyano-7,7-dimethyl-5-oxo-4H-5,6,7,8-tetrahydrobenzo[b]pyran (Table 2, entry 12; see later): white solid; mp = 241–242 °C; IR (KBr): \( \nu_{\text{max}} = 3496 (\text{OH}), 3402 \text{ and } 3255 (\text{NH}_2), 2192 (\text{CN}), 1654 (\text{C}=\text{O}), 1208 (\text{C}–\text{O}) \text{ cm}^{-1} \); \(^1\)H NMR (300 MHz, DMSO-d₆): \( \delta = 1.03 (2s, 6\text{H, } 2\text{CH}_3), 2.09 \text{ and } 2.25 (\text{AB quartet, } 2\text{H, } J = 15.9 \text{ Hz, CH}_2), 2.49 (m, 2\text{H, CH}_2), 3.70 (s, 1\text{H, OCH}_3), 4.07 (s, 1\text{H, CH}), 6.51 (d, 1\text{H, } J = 6.9 \text{ Hz, } \text{CH}_{\text{arom}}), 6.66 (d, 2\text{H, } J = 10.8 \text{ Hz, } 2\text{CH}_{\text{arom}}), 6.90 (s, 2\text{H, } 2\text{CH}_{\text{arom}}), 8.80 (s, 1\text{H, OH}) \text{ ppm}; \(^{13}\)C NMR
(75 MHz, DMSO-d$_6$): $\delta = 21.1, 28.1, 30.3, 37.4, 39.8, 58.2, 59.9, 111.9, 113.5, 115.8, 119.9, 120.3, 136.3, 145.7, 147.7, 158.2, 162.6, 196.1$ ppm.

Results and discussion

Characterization of the prepared AMBA–Fe$_3$O$_4$ magnetic nanoparticles

X-ray diffraction (XRD) analysis

X-ray diffraction was used to identify phases of the synthesized AMBA-Fe$_3$O$_4$ magnetic nanoparticles. The result shown in Fig. 1 was fitted for the observed six peaks with the following Miller indices: (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) that identified them as Fe$_3$O$_4$ nanoparticles. The XRD peaks are broad indicating the small nanoparticles. Applying the Scherrer equation [19, 20], the calculated size was estimated to be about 15 nm which is in a good agreement with the TEM observations.

Fourier transform infrared (FT-IR) analysis

The peak around 3428 cm$^{-1}$ is referred to the N–H stretching vibration. The peaks around 2923 and 2854 cm$^{-1}$ are ascribed to the asymmetric and symmetric vibrations of C–H stretching. The adsorption peaks at 1632, 1398 and 1028 cm$^{-1}$ correspond to the asymmetric and symmetric stretching vibrations of the carboxylate group of the 4-aminomethylbenzoic acid moiety. The characteristic absorbing peak of Fe$_3$O$_4$ appeared at 584 cm$^{-1}$, which can be ascribed to the vibration of the Fe–O group. Therefore, the obtained data from FT-IR spectroscopy can confirm the existence of the magnetic nanoparticles and aromatic ligand moiety in the structure of the AMBA–Fe$_3$O$_4$ nanoparticles (Fig. 2).

![Fig. 1 XRD powder pattern of AMBA–Fe$_3$O$_4$ nanoparticles](image-url)
Thermogravimetric analysis (TGA)

Thermal gravimetric analysis (TGA) was implemented at the range of 25–900 °C under N₂ atmosphere in order to define the loading of organic groups coated on the Fe₃O₄ magnetic nanoparticles (Fig. 3). The TGA curve of the AMBA–Fe₃O₄ nanoparticles shows the mass loss of the organic functional group as it decomposes upon heating (volatile components disappeared before a temperature of about 100 °C were ignored). The curve shows a weight loss of about 9.37% between 100 °C and Fig. 3 TGA thermograms of AMBA–Fe₃O₄ nanoparticles
and 800 °C, resulting from the decomposition of the functional group grafting to the Fe₃O₄ surface.

**Morphological characteristic**

Surface morphology, particle shape, fundamental physical properties and size details of the prepared AMBA–Fe₃O₄ magnetic nanoparticles were investigated through SEM and TEM microscopies (Fig. 4a, b). As can be seen from the TEM and SEM images, the average particle size is estimated to be 15–30 nm while AMBA–Fe₃O₄ showed a sphere-like structure. As shown in Fig. 4b, a basically core–shell structure (dark-colored core for Fe₃O₄ magnetic nanoparticles and light-colored shell for AMBA) was obtained.

**Vibrating sample magnetometer (VSM)**

The VSM data for the Fe₃O₄ nanoparticles and 4-aminomethylbenzoic acid-functionalized Fe₃O₄ nanoparticles samples, recorded at room temperature, are shown in Fig. 5. The sigmoidal shape of the \( M(H) \) curves with nearly zero hysteresis confirms the superparamagnetic nature of these nanoparticles at room temperature. The saturation magnetization of the Fe₃O₄ nanoparticles was 68 emu/g, whereas that of 4-aminomethylbenzoic acid-functionalized Fe₃O₄ nanoparticles was reduced to 55 emu/g. The saturation magnetization of the AMBA–Fe₃O₄ nanoparticles clearly decreased because the diamagnetic contribution of the organic group resulted in a low mass fraction of the Fe₃O₄ nanoparticles.

**Catalytic activity of AMBA–Fe₃O₄ nanoparticles**

First, to find the optimization conditions, the reaction of 4-chlorobenzaldehyde, malononitrile and dimedone in the presence of the AMBA–Fe₃O₄ magnetic nanocatalyst was selected as a model reaction. The reaction was carried out with different amounts of nanocatalyst (0.02, 0.05 and 0.10 g) at different temperatures.
The obtained results in Table 1 show that an optimal condition was 20 mol% of AMBA–Fe$_3$O$_4$ magnetic nanocatalyst at 60 °C (Table 1, entry 8).

Next, various aldehydes were used in the reactions that led to the corresponding products in high to excellent yields (Table 2). As shown in Table 2, the reactions with arylaldehydes, including electron-donating groups or electron-withdrawing groups substituents, afforded the desired products in high to excellent yields. Natural aldehydes such as vanillin and cinnamaldehyde also gave the corresponding products in high yields. Isonicotincarboxaldehyde as a hetero-aromatic aldehyde

![Room-temperature magnetization curves of Fe$_3$O$_4$ nanoparticles and AMBA–Fe$_3$O$_4$ nanoparticles](image)

**Table 1** Optimization conditions for preparation of 2-amino-4-(4-chlorophenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4$H$-chromene-3-carbonitrile from 4-chlorobenzaldehyde, malononitrile and dimedone in the presence of different amounts of AMBA–Fe$_3$O$_4$ as magnetic nanocatalyst at different temperatures

| Entry | Amount of catalyst (g) | Temperature (°C) | Time (min) | Yield (%)$^a$ |
|-------|------------------------|------------------|------------|--------------|
| 1     | 0.02                   | 25               | 100        | –            |
| 2     | 0.05                   | 25               | 100        | Trace        |
| 3     | 0.10                   | 25               | 100        | 35           |
| 4     | 0.02                   | 40               | 75         | 49           |
| 5     | 0.05                   | 40               | 75         | 60           |
| 6     | 0.10                   | 40               | 75         | 74           |
| 7     | 0.02                   | 60               | 25         | 85           |
| 8     | 0.05                   | 60               | 20         | 93           |
| 9     | 0.10                   | 60               | 20         | 93           |

$^a$ Yields refer to isolated product
Table 2  One-pot synthesis of 2-amino-4H-chromene derivatives using AMBA–Fe$_3$O$_4$ as nanocatalyst

| Entry | Aldehyde | Product | Time (min) | Yield (%) | Melting point (°C) |
|-------|----------|---------|------------|-----------|-------------------|
|       |          |         |            |           | Found        | Reported [Refs.] |
| 1     | C$_6$H$_5$CHO | ![Image of product 1] | 21        | 92        | 231–232 | 228–231 [21] |
| 2     | 4-CNC$_6$H$_4$CHO | ![Image of product 2] | 21 | 94 | 226 | 225–226 [22] |
| 3     | 4-HOC$_6$H$_4$CHO | ![Image of product 3] | 22 | 91 | 215–217 | 212–214 [21] |
| 4     | 4-ClC$_6$H$_4$CHO | ![Image of product 4] | 20 | 93 | 209–210 | 209-211 [23] |
| 5     | 3-NO$_2$C$_6$H$_4$CHO | ![Image of product 5] | 20 | 92 | 180–182 | 181–182 [24] |
| 6     | 4-BrC$_6$H$_4$CHO | ![Image of product 6] | 23 | 92 | 201–203 | 199–201 [24] |
| 7     | 2-NO$_2$C$_6$H$_4$CHO | ![Image of product 7] | 20 | 92 | 180–182 | 181–184 [22] |
| 8     | 2-ClC$_6$H$_4$CHO | ![Image of product 8] | 21 | 93 | 192–194 | 194–195 [21] |
| 9     | 4-MeC$_6$H$_4$CHO | ![Image of product 9] | 22 | 91 | 214–215 | 210–213 [23] |
afforded a satisfactory result. However, butyraldehyde as an aliphatic aldehydes did not proceed further this reaction.

The mechanism of the formation of 2-amino-4H-chromene derivatives in the presence of AMBA–Fe₃O₄ as a catalyst via a three-component coupling strategy is proposed to begin with a Knoevenagel condensation between malononitrile and the aromatic aldehyde, followed by the Michael addition of dimedone to the Knoevenagel product, and finally an intramolecular ring closure (Scheme 3) [28].

We also investigated the recyclability of the AMBA–Fe₃O₄ as a magnetic nanocatalyst using the model reaction of benzaldehyde, malononitrile and dimedone (Table 2, entry 1). The results showed that AMBA–Fe₃O₄ is a stable catalyst in reaction media and can be reused five times without any significant loss of its activity (Fig. 6).

| Entry | Aldehyde            | Product | Time (min) | Yield (%) | Melting point (°C) |
|-------|---------------------|---------|------------|-----------|--------------------|
|       |                     |         |            |           | Found       | Reported [Refs.] |
| 10    | 1-Naphthaldehyde    | ![Image](image1.png) | 25         | 88        | 216–217     | 215–218 [24]   |
| 11    | Cinnamaldehyde      | ![Image](image2.png) | 25         | 85        | 180–181     | 182–184 [25]   |
| 12    | Vanilline           | ![Image](image3.png) | 23         | 91        | 241–242     | 138–240 [26]   |
| 13    | Isonicotinaldehyde  | ![Image](image4.png) | 21         | 90        | 210–212     | 214–216 [27]   |
| 14    | Butyraldehyde       | ![Image](image5.png) | 24 h       | 0         |             |                |

*Yields refer to isolated product*
To show the efficiency of this method, the results of the synthesis of 2-amino-4H-chromene derivatives by our method was compared with those reported in the literature. The results show that this nanocatalyst is very efficient with respect to the reaction times and yields (Table 3).

**Scheme 3** The suggested mechanism for the formation of the 2-amino-4H-chromene derivatives

**Fig. 6** The recycling of the AMBA–Fe$_3$O$_4$ nanoparticles as a catalyst

To show the efficiency of this method, the results of the synthesis of 2-amino-4H-chromene derivatives by our method was compared with those reported in the literature. The results show that this nanocatalyst is very efficient with respect to the reaction times and yields (Table 3).
Conclusions

We have developed a new 4-aminomethylbenzoic acid functionalized Fe$_3$O$_4$ magnetic nanoparticles as an active and reusable base hybrid heterogeneous catalyst. This basic nanocatalyst was characterized by different methods and its applicability for the preparation of 2-amino-4$^H$-chromene derivatives from arylaldehyde, malonitrile and dimesone was investigated. This heterogeneous catalyst is highly active and has a very high surface area, and its thermal stability was confirmed by different characteristic techniques. The recoverability and reusability of this inexpensive catalyst are other advantages of the protocol.

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