FULL PAPER



DABCO-modified super-paramagnetic nanoparticles as an efficient and water-compatible catalyst for the synthesis of pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione derivatives under mild reaction conditions

Zahra Heydari¹ | Saeed Bahadorikhalili² | Parviz Rashidi Ranjbar¹ | Mohammad Mahdavi³

¹School of Chemistry, College of Science, University of Tehran, P. O. Box 14155-6455 Tehran, Iran

²Iranian Space Research Center, Institute of Mechanics, Shiraz, Iran

³Endocrinology and Metabolism Research Center, Endocrinology and Metabolism Clinical Sciences Institute, Tehran University of Medical Science, Tehran 14176, Iran

Correspondence

Parviz Rashidi Ranjbar, School of Chemistry, College of Science, University of Tehran, P. O. Box 14155-6455, Tehran, Iran.

Email: prrashidi@khayam.ut.ac.ir

Mohammad Mahdavi, Endocrinology and Metabolism Research Center, Endocrinology and Metabolism Clinical Sciences Institute, Tehran University of Medical Science, Tehran, 14176, Iran. Email: momahdavi@sina.tums.ac.ir In this paper, a novel catalyst is introduced based on the immobilization of 1,4diazabicyclo[2.2.2] octane (DABCO) on super-paramagnetic iron oxide nanoparticles. For the synthesis of the catalyst, silica-magnetic iron oxide core-shell nanoparticles were first functionalized by (3-chloropropyl)trimethoxysilane. The functionalized silica-magnetic iron oxide core-shell nanoparticles then reacted with DABCO to form the final catalyst. The catalyst was characterized by scanning electron microscopy, vibrating-sample magnetometer, thermogravimetric analysis, X-ray photoelectron spectroscopy and Fourier-transform infrared spectroscopy. This catalyst was used for the synthesis of pyrano[3,2*c*:5,6-*c*']dichromene-6,8-dione derivatives. Several starting materials were used, and all of them led to the desired products in high isolated yields (19 examples). The catalyst also showed very good reusability in the reaction.

KEYWORDS

DABCO, magnetic nanoparticles, nanocatalyst, pyrano[3,2-c:5,6-c']dichromene-6,8-dione

1 | INTRODUCTION

Catalysts are an important part of many reactions, due to their unique properties. These agents cause better reaction performance in lower temperature and milder reaction conditions to give products in higher isolated yields.^[1] For better productivity of the catalysts, it is critical to recover them. To give the reusable properties to a catalyst, a successful approach is to immobilize them on a solid support.^[2–4] Among different supports, magnetic nanoparticles (MNPs) are of high interest due to their thermal and chemical stability, good functionalization possibility, facile preparation and simple separation from the reaction medium by an external magnet. Super-paramagnetic iron oxide nanoparticles (SPIONs) are of the most favorable magnetic nanoparticles. Apart from all the desired properties of the MNPs, magnetic iron oxide nanoparticles are not expensive and can be synthesized from cheap reagents by experimentally simple co-precipitation in basic conditions.^[5] The low-cost, simple and efficient synthesis of SPIONs has made them an appropriate choice for being used in a wide range of applications, especially as support for catalysts in organic transformations.^[6–11] Regarding the importance of water as a green, low-cost, available and environmentally friendly solvent, various efforts have been focused on the functionalization of the SPIONs, such that make them more water-compatible in the reactions.^[12–16] In this regard, modified SPIONs have been used as support for the immobilization of both metallic and non-metallic catalysts.^[17–20]

Pyranochromene is a very important skeleton, which can be found in various structures. Different derivatives of this structure are shown to have several biological activities.^[21-24] Due to the wide applications and various activities of pyranochromene derivatives, several efforts have been focused on the synthesis of these compounds. The most efficient synthetic routes contain the reaction of 4-hydroxycoumarine with hydrobenzoin^[25] or aldederivatives.^[26-28] hvde The reaction of 4hydroxycoumarine with mandelic acid in acidic conditions was also shown to lead to the pyranochromene derivatives.^[29] Regarding the importance of pyrano[3,2c:5,6-c']dichromene-6,8-dione derivatives in various biological applications, the synthesis of this class of compounds is of high interest.^[25,30]

1,4-Diazabicyclo[2.2.2] octane (DABCO) is a very common base, which is used as a catalyst in various reactions.^[31–35] Continuing our previous efforts in the immobilization of the catalysts on nanoparticles for the synthesis of reusable catalysts on one hand,^[36–43] and the synthesis of biologically active organic compounds on the other hand,^[44–52] in this report a novel catalyst is introduced for the synthesis of pyrano[3,2-*c*:5,6-*c*'] dichromene-6,8-dione derivatives. The catalyst is prepared based on the immobilization of DABCO on magnetic nanoparticles (DABCO-SPION). The reaction scheme is presented in Scheme 1.

2 | RESULTS AND DISCUSSION

Novel methods for green synthesis of biologically active chemicals have been of high interest in our group. Regarding the benefits of nanocatalysts, in this paper we



SCHEME 1 The preparation of pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-diones in the presence of DABCO-SPION catalyst

develop a novel catalyst based on the functionalization of DABCO on magnetic nanoparticles. The catalyst is used for efficient and simple synthesis of pyrano[3,2c:5,6-c']dichromene-6,8-dione derivatives. For the synthesis of DABCO-SPION catalyst, magnetic iron oxide nanoparticles encapsulated by silica were used as support for the immobilization of DABCO as the active catalytic agent. Encapsulation of SPIONs with a silica shell is to make them more stable, and to supply more reactive hydroxyl groups on the surface of the NPs. The active DABCO was covalently attached to the SPIONs using (3-chloropropyl)trimethoxysilane. Finally, the counter ion was replaced by BF_4^- . The synthetic steps for the synthesis of DABCO-SPION catalyst are presented in Scheme 2.

The catalyst was characterized by several methods. Fourier-transform infrared (FT-IR) spectroscopy was used for studying the covalent attachment of DABCO to SPION@SiO₂. The FT-IR spectrum is presented in Figure 1. The FT-IR spectrum SPION@SiO₂ is shown in Figure 1a, and that of DABCO-SPION can be seen in Figure 1b. In Figure 1a, two bands at 3430 and 1246 cm⁻¹ are correlated to the stretching and bending vibrations of hydroxyl groups of SPION@SiO₂. In addition, vibrations at 570 cm⁻¹ are correlated to Fe-O bonds. Si-O vibrations can be seen as an intense bond at 1106 cm⁻¹. After the modification of SPION@SiO₂ by DABCO, additional bonds are presented.

Structure, particle size and the morphology of DABCO-SPION catalyst is very important in its role as catalyst in the reaction. The structure and the particle size of the nanoparticles were studied using scanning electron microscopy (SEM). SEM images can be seen in Figure 2. In this figure, the images of DABCO-SPION catalyst can be observed in two different magnifications. The morphology of the catalyst can clearly be observed in the SEM image to be spherical. The particles showed narrow dispersity with particles size between 20 and 25 nm.

The magnetic behavior of the catalyst is a significant feature, especially in its recovery. The magnetic catalyst can be simply isolated from the reaction mixture by an external magnet, to be used in the next reaction. Therefore, the magnetic behavior of SPION@SiO₂ and



SCHEME 2 The preparation steps of DABCO-SPION catalyst



FIGURE 1 Infrared (IR) spectrum of (a) 1,4-diazabicyclo[2.2.2] octane (DABCO), (b) super-paramagnetic iron oxide nanoparticle (SPION) $@SiO_2$ and (c) DABCO-SPION catalyst



FIGURE 2 Scanning electron microscopy (SEM) images of 1,4diazabicyclo[2.2.2] octane (DABCO)-super-paramagnetic iron oxide nanoparticle (SPION) catalyst in different magnifications

DABCO-SPION catalysts were studied using the vibrating-sample magnetometer (VSM) method. The VSM results can be seen in Figure 3. The super-paramagnetic behavior of SPION@SiO₂ and DABCO-SPION was clearly observed in the VSM results.^[53] The S-type magnetization hysteresis loop of SPION@SiO₂ and DABCO-SPION catalyst proves the super-paramagnetic characters of these nanoparticles. In DABCO-SPION catalyst the magnetization magnitude is decreased in comparison with SPION@SiO₂, but it still shows the magnetic behavior in an external magnetic field.

For studying the thermal stability of the catalyst, thermogravimetric (TG) analysis was used. The weight loss in TG analysis was utilized to determine the amount of loaded DABCO on the surface of SPION@SiO₂ NPs. The TG analysis (TGA) curves can be seen in Figure 4. By studying the TGA curves, it can be seen that no significant weight loss has been observed in temperatures lower than 300°C. By this observation it can be concluded that DABCO-SPION catalyst is thermally stable up to 300°C. The TG curve of DABCO-SPION shows a sharp decrease in weight and was observed at 300–700°C. The amount of DABCO on the surface of the catalyst was measured by acid–base titration. The titration results showed that the amount of the attached DABCO in the catalyst is 1.9 g mol⁻¹.

For exact characterization of the catalyst, the X-ray photoelectron spectroscopy (XPS) method was applied (Figure 5). The XPS results proved the formation of



FIGURE 3 Vibrating-sample magnetometer (VSM) diagram of (a) super-paramagnetic iron oxide nanoparticle (SPION)@SiO₂ and (b) 1,4-diazabicyclo[2.2.2] octane (DABCO)-SPION catalyst

DABCO ionic liquids on the surface of the catalyst. The existence of Fe (2p), O (1s), Si (2p), Si (2s) and O (2s) signals in the spectrum confirms the presence of both iron oxide and silica in the structure of the SPION $@SiO_2$ core-shell. In addition, N (1s) and C (1s) signals confirm



FIGURE 4 Thermogravimetric analysis (TGA) superparamagnetic iron oxide nanoparticle (SPION)@SiO₂ of 1,4diazabicyclo[2.2.2] octane (DABCO)-SPION catalyst



FIGURE 5 X-ray photoelectron spectroscopy (XPS) spectrum of 1,4-diazabicyclo[2.2.2] octane (DABCO)-super-paramagnetic iron oxide nanoparticle (SPION) catalyst

the presence of DABCO in the structure of the catalyst. The presence of BF_4^- counter ion can also be confirmed by observing the F (1 s) and B (1 s) signals. We note that N (1 s) spectra shows two peaks with their binding energies of 399.7 and 401.9 eV, which belong to amine and ammonium groups, respectively.^[54]

The aim of this paper was the synthesis of pyrano[3,2c:5,6-c']dichromene-6,8-dione derivatives using DABCO-SPION catalyst. Therefore, the activity of the catalyst in the synthesis of pyrano[3,2-c:5,6-c']dichromene-6,8diones was studied. For this purpose, the optimal conditions were chosen by changing different variables and studying the effect of each variable on the reaction performance. The reaction was performed under different conditions, and the best one was selected as the optimal conditions. To do this, a series of bases including NaOH, KOH, K₂CO₃, pyridine, NEt₃ and non-immobilized DABCO were used as catalyst, and their effect was observed on the isolated yields of the products. It can be seen that the desired products have been obtained when non-immobilized DABCO has been applied as catalyst in very lower yields. Moreover, other bases have given low isolated yields. We note that blank runs without any amount of catalyst did not lead to the desired product. This proves that the presence of the catalyst is necessary for the reaction performance. The effects of different bases on the reaction time and the isolated yield are presented in Table 1.

Then the effect of the amount of catalyst on the reaction was investigated, and different quantities of catalyst ranging from 1 to 50 mol% were tested. The best results were observed when 10 mol% of the catalyst has been applied, while using more quantities of the catalyst did not improve the reaction performance. We note that in the absence of the catalyst, no product was formed, which proves that the presence of the catalyst is critical for the reaction performance.

TABLE 1 Effect of different bases on the reaction time and the isolated yield^a

Entry	Base	Time (h)	Yield (%)
1	No base	6	0
2	DIPEA	4	62
3	КОН	3	58
4	K ₂ CO ₃	6	67
5	Pyridine	4	75
6	NEt ₃	4	71
7	NaOH	4	66
8	Pyridine	4	71
9	DABCO	2	64
10	DABCO-SPION	1.5	84
11	DABCO-SPION ^b	1.5	71

^aReaction conditions: benzaldehyde (1 mmol), 4-hydroxycumarine (2 mmol) in EtOH/H₂O (10 ml, 50% v/v) in the presence of different bases as catalyst (10 mol%) at 80°C.

^bDABCO-SPION with Cl-counter ion.

TABLE 2 Effect of different solvents on the reaction time and the yield^a

Entry	Solvent	Time (h)	Isolated yield (%)
1	H ₂ O	4	41
2	DMF	2	56
3	DMSO	2	62
4	EtOH/H ₂ O ^b	1.5	83
5	EtOH	3	48
6	CH_2Cl_2	4	55
7	CH ₃ CN	4	41

^aReaction conditions: benzaldehyde (1 mmol), 4-hydroxycumarine (2 mmol) in the presence of catalyst (10 mol%) at 80°C.

^b50:50 (v/v) mixture.

Entry

1

2

3

4

5

TABLE 3 Synthesis of pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione derivatives in the presence of DABCO-SPION catalyst^a





(Continues)





(Continues)

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TABLE 3 (Continued)



^aReaction conditions: benzaldehyde (1 mmol), 4-hydroxycumarine (2 mmol) in $EtOH/H_2O$ (10 ml, 50% v/v) in the presence of catalyst (10 mol%) at 80°C.

Then, the effect of different solvents on the performance of the reaction was studied. For studying the effect of different solvents on the reaction performance, various protic and aprotic solvents with different polarities were used as solvent, and the isolated yields of the products were compared. The results are presented in Table 2. It can be seen in Table 2 that the synthesis of pyrano[3,2c:5,6-c']dichromene-6,8-dione derivatives was performed in different solvents. But a 50:50 (v/v) mixture of H₂O/ EtOH is the best solvent, and the highest isolated yield is obtained in this green mixture as solvent. The effect of temperature on the reaction performance is very important. Performing the reaction under different temperature conditions proved 80°C to be the best temperature for yielding the desired product.

Using the optimized conditions, the efficiency of the DABCO-SPION catalyst in the synthesis of pyrano[3,2*c*:5,6-*c*']dichromene-6,8-dione derivatives was studied. As shown in Table 3, DABCO-SPION successfully catalyzed the reaction to give the desired products. The corresponding pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione derivatives were obtained in good isolated yields for various examined substrates at 80°C in the presence of DABCO-SPION catalyst. As illustrated, a wide range of starting materials can be efficiently produced with this catalytic system. All substrates possessing electron-rich as well as electronpoor substituents underwent intramolecular cyclization catalyzed by DABCO-SPION catalyst.

A proposed mechanism is suggested for DABCO-SPION-catalyzed synthesis of pyrano[3,2-*c*:5,6-*c*'] dichromene-6,8-dione derivatives. The suggested mechanism can be seen in Scheme 3. In the suggested mechanism, the catalyst first activated the hydroxyl group of the coumarin. The activated coumarin reacts with aldehyde and forms an intermediate that again reacts with another activated coumarin. The product of this step



SCHEME 3 Suggested mechanism for DABCO-SPION-catalyzed synthesis of pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione derivatives

undergoes an intermolecular reaction, which is activated by DABCO-SPION catalyst to form the desired pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione.

A very important advantage of the current catalyst is its reusability. The separation of the catalyst from the reaction mixture is simply possible by a magnet, due to the magnetic nature of the catalyst. The recycling of DABCO-SPION catalyst was tested in 10 sequential reactions for the preparation reaction of pyrano[3,2-*c*:5,6-*c*']dichromene-6,8-dione. The results are presented in Figure 6. It is obvious in Figure 6 that no significant loss in catalyst activity can be observed after the 10th run.

For better studying of the stability of the catalyst, the morphology of the recovered DABCO-SPION catalyst was analyzed by SEM microscopy. The SEM image of the recovered catalyst after the 5th run is presented in Figure 7. It can be seen in Figure 7 that the morphology and the structure of the recovered catalyst has not significantly changed after being used in the reaction. This



FIGURE 6 Reusability of 1,4-diazabicyclo[2.2.2] octane (DABCO)-super-paramagnetic iron oxide nanoparticle (SPION) catalyst after 10 runs



FIGURE 7 Scanning electron microscopy (SEM) image of the recovered 1,4-diazabicyclo[2.2.2] octane (DABCO)-super-paramagnetic iron oxide nanoparticle (SPION) catalyst after the 5th run

observation proves the stability of the catalyst in the reaction conditions.

3 | CONCLUSIONS

In summary, we have introduced DABCO-catalyzed synthesis of pyrano[3,2-c:5,6-c']dichromene-6,8-dione derivatives. To do this, DABCO was successfully immobilized on the surface of SPION@SiO₂ nanoparticles. The catalyst was characterized by various methods. After the characterization of the catalyst, it was used for the synthesis of pyrano[3,2-c:5,6-c']dichromene-6,8-dione derivatives were synthesized in the presence of DABCO-SPION catalyst from a wide range of starting materials. Various starting materials were used and, in all cases, the desired products were obtained in good isolated yields. The catalyst also showed very good reusability in the reaction.

4 | EXPERIMENTAL

4.1 | Preparation of DABCO-SPION catalyst

Super-paramagnetic iron oxide nanoparticles were prepared according to our previously reported paper.^[35] SPIONs were synthesized by co-precipitation of Fe₂O₃ and FeO in basic conditions. SPIONs were then coated by tetraethyl orthosilicate according to a previously reported method to form SPION@SiO₂^[35] SPION@SiO₂ nanoparticles were functionalized by (3-chloropropyl) trimethoxysilane. For this purpose, 10 mmol of (3chloropropyl)trimethoxysilane in 30 ml of EtOH was added drop-wise to a vigorous stirring solution of 25 mg of Fe₃O₄@SiO₂ nanoparticles in 30 ml EtOH/H₂O (1:2) and HCl (pH 4). After vigorous stirring for 24 h, the product was isolated by an external magnet and washed using ethanol $(3 \times 10 \text{ ml})$. The product was dried at 100° C in vacuum for 12 h to obtain (3-chloropropyl) trimethoxysilane modified magnetic nanoparticles.

Then, DABCO (0.4 g, 5 mmol) was poured into a mixture of (3-chloropropyl)trimethoxysilane modified magnetic nanoparticles (2 g) in 50 ml toluene, and the mixture was stirred at 90°C overnight. Then the solid residue was magnetically separated, washed with toluene and Et₂O, and dried in a vacuum oven at 50°C for 24 h. The product was added to a solution containing NaBF₄ (0.55 g, 5 mmol) in dry acetonitrile (15 ml) and stirred at room temperature for 72 h. The product was magnetically isolated from the reaction mixture, washed and dried under vacuum to give the catalyst.

4.2 | General procedure for the preparation of pyrano[3,2-c:5,6-c'] dichromene-6,8-dione

Benzaldehyde (1 mmol) and 4-hydroxycumarine (2 mmol) were added to a mixture of ethanol and water (10 ml, 50% v/v) in a round flask equipped with condenser, heater and magnetic stirrer. Then, DABCO-SPION catalyst (10 mol%) was added to the reaction mixture. The reaction mixture was stirred at 80°C for 90 min. The reaction performance was controlled by thin-layer chromatography. After the reaction was completed, the solvent was eliminated using a rotary evaporator and the product was recrystallized in ethanol to obtain pure product. The products were characterized by ¹H NMR, ¹³C NMR, FT-IR and mass spectroscopy.

ORCID

Mohammad Mahdavi http://orcid.org/0000-0002-8007-2543

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SUPPORTING INFORMATION

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