

RESEARCH PAPER

Synthesis of Tetra-substituted Phenanthroimidazole Derivatives Using SBA-Pr-SO₃H

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ARTICLE INFO

Article History:

Received 24 June 2018

Accepted 03 September 2018

Published 01 October 2018

Keywords:

9,10-Phenanthraquinone

Phenanthro[9,10-*d*]imidazole

SBA-Pr-SO₃H

Tetrasubstituted imidazoles

ABSTRACT

SBA-15 (Santa Barbara Amorphous), as a hexagonal mesoporous silica with 6 nm pore diameter, was synthesized and then its internal surface was modified with (3-mercaptopropyl)trimethoxysilane following an oxidation process to gain SBA-Pr-SO₃H. The latter was then characterized; SEM image showed uniform particles about 700-900 nm and TEM image demonstrated the presence of parallel channels, which resemble the pores configuration of SBA-15. Additionally, the weight reduction in TGA curve in the temperature range of 200-600 °C (about 20% mass loss) established that the anchored propyl sulfonic acid groups onto the SBA-15 pores is about 1.2 mmol/g. This data was also confirmed by back-titration of SBA-Pr-SO₃H with standardized NaOH and HCl solutions. Afterwards, a one pot four-component reaction of 9,10-phenanthraquinone, aromatic aldehyde, aniline, and ammonium acetate was designed for the preparation of tetrasubstituted imidazoles (phenanthro[9,10-*d*]imidazole) derivatives in the presence of SBA-Pr-SO₃H as a mesoporous solid acid catalyst. Phenanthro[9,10-*d*]imidazole derivatives were produced by the use of this technique in short reaction times and good to high yields.

How to cite this article

Mohammadi Ziarani G, Tavaf E, Gholamzadeh P, Badiie A. Synthesis of Tetra-substituted Phenanthroimidazole Derivatives Using SBA-Pr-SO₃H. J Nanostruct, 2018; 8(4): 343-349. DOI: 10.22052/JNS.2018.04.003

INTRODUCTION

Phosphorescent organic light emitting diodes have attracted tremendous attention among the scientists due to their excessive potential in both solid-state lighting and flat-panel presentations. Some of phosphorescent organic compounds are blue organic emitters, although a few of them can be used in organic lighting emitting diodes (OLEDs) [1]. Tri- and tetra-substituted imidazoles [2], especially phenanthro[9,10-*d*]imidazole derivatives exhibit good electron injection and transport ability, high luminance intensity, therefore, they are good candidates for OLEDs [3]. Because of their industrial applications, introducing an economical synthetic procedure is essential for preparation of phenanthro[9,10-*d*]imidazole derivatives [4].

So far, a few methods were published for the synthesis of phenanthro[9,10-*d*]imidazole derivatives; Jawaharmal and coworkers performed the synthesis in glacial acetic acid as solvent within 3 h [5] while Mukhopadhyay used mercaptopropylsilica (MPS) used as a catalyst in a mixture of water and methanol [6]. In many publications, phenanthro[9,10-*d*]imidazole derivatives were prepared and used as the intermediate for the synthesis of modified electroluminescent materials or OLED [7-12].

After discovering the Santa Barbara Amorphous (SBA-15) by Zhao and coworkers [13], it was extensively used as a good substrate for loading drugs [14], organic compounds [15, 16], and metal cations [17]. The modified SBA-15 can be used in different fields of research such as catalysis [18-20], detection of heavy metals [21, 22], absorption

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[23-25], and so on. Sulfonic acid functionalized SBA-15 (SBA-Pr-SO₃H), as a heterogeneous solid acid catalyst, has been applied in several one-pot syntheses [26, 27]. Therefore, herein we want to report the role of SBA-Pr-SO₃H as a highly efficient heterogeneous acid catalyst in the efficient synthesis of phenanthro[9,10-*d*]imidazole derivatives *via* a one-pot four-component reaction.

EXPERIMENTAL SECTION

The chemicals employed in this work were obtained from Merck Company and used with no purifications. Infrared (IR) spectra were recorded on KBr disks using a FT-IR Bruker Tensor 27 instrument. Melting points of the products were measured by using the capillary tube method with an Electro thermal 9200 apparatus. The ¹H NMR and ¹³C NMR spectra were obtained by the use of a Bruker 250 MHz and 62.5 MHz, respectively, in either DMSO-*d*₆ or CDCl₃ solution. Mass spectra data were achieved by using a Network mass selective detector 5973 (Agilent) instrument.

Preparation of SBA-Pr-SO₃H

SBA-15 was prepared according to the previously published article [13]. Afterward, in order to functionalizing it, the calcined SBA-15 (2 g) and (3-mercaptopropyl)trimethoxysilane (10 mL) were treated together under reflux condition in dry toluene (20 mL) for 24 h. Subsequently, the mixture was filtered off and the obtained solid washed well with dichloromethane for 6 h using a soxhlet apparatus, and then dried under reduced pressure. The obtained crude SBA-Pr-SH was oxidized using excess amount of hydrogen peroxide solution in methanol (20 mL) in the presence of H₂SO₄ (1 drop) at room temperature overnight. The mixture was filtered and washed with water and acetone. The modified SBA-Pr-SO₃H was dried and characterized using TGA, XRD, SEM and TEM.

General procedure for the synthesis of phenanthro[9,10-*d*]imidazole derivatives 5(a-g)

A mixture of 9,10-phenanthraquinone (0.20 g, 1 mmol), aromatic aldehyde (1 mmol), aniline derivative (1.5 mmol), ammonium acetate (0.31 g, 4 mmol), and SBA-Pr-SO₃H (0.02 g) was stirred in refluxing acetic acid (3 mL) for about 5-20 min. After completion of the reaction which was traced by TLC technique, acetic acid was evaporated and the crude product was dissolved in ethanol

and DMF. Afterward, the solution was filtered for removal of the heterogeneous catalyst, and the filtrate was cooled to afford the pure product. The catalyst was washed subsequently with a diluted acidic solution, distilled water, and then acetone, and then dried under reduced pressure and reused for several times without significant loss of catalytic activity. The physical and spectroscopic data for the new compounds are given below.

4-(1-phenyl-1H-phenanthro[9,10-*d*]imidazol-2-yl)phenol (5e)

M.P. > 300 °C, FT-IR (KBr) ν (cm⁻¹): 3050, 2595, 1893, 1603, 1444, 1389, 1346, 1250, 1166, 1040, 835, 739, 615, 533, 428. ¹H NMR (250 MHz, DMSO-*d*₆) δ (ppm): 6.70 (d, 2H, J=8.2 Hz, ArH), 7.04 (d, 1H, J=8 Hz, ArH), 7.27-7.73 (m, 9H, ArH), 7.37 (d, 2H, J=8.2 Hz, ArH), 8.66 (d, 1H, J=7.7 Hz, ArH), 8.79-8.87 (m, 2H, ArH). ¹³C NMR (62.5 MHz, DMSO-*d*₆) δ (ppm): 115.4, 120.5, 121.4, 122.5, 122.9, 124, 124.8, 125.4, 126, 127, 127.1, 127.8, 128, 128.7, 129.6, 130.5, 130.7, 131.1, 136.8, 138.8, 151.5, 158.5. Mass m/z (%): 386 (M⁺, 40), 119 (53), 91 (43), 77 (80), 55 (87), 41 (100).

2-(3-nitrophenyl)-1-phenyl-1H-phenanthro[9,10-*d*]imidazole (5f)

M.P. 233-235 °C, IR (KBr) ν (cm⁻¹): 3049, 1809, 1671, 1587, 1521, 1451, 1380, 1336, 1150, 1079, 1035, 986, 913, 799, 755, 706, 527. ¹H NMR (250 MHz, CDCl₃) δ (ppm): 7.16-7.77 (m, 11H, ArH), 7.96 (d, 1H, J=7.7 Hz, ArH), 8.09 (d, 1H, J=7.7 Hz, ArH), 8.35 (s, 1H, ArH), 8.67 (d, 1H, J=8 Hz, ArH), 8.74 (d, 1H, J=8.5 Hz, ArH), 8.85 (d, 1H, J=8 Hz, ArH). ¹³C NMR (62.5 MHz, CDCl₃) δ (ppm): 120.9, 122.7, 122.8, 123.2, 123.7, 124.1, 125.3, 125.9, 126.4, 127, 127.4, 128.4, 128.9, 129.2, 129.6, 130.4, 130.6, 132.1, 134.7, 137.5, 138.2, 147.9, 148. Mass m/z (%): 415 (M⁺, 3), 324 (100), 265 (89), 237 (89), 192 (80), 43 (78).

2-(2,4-dimethoxyphenyl)-1-phenyl-1H-phenanthro[9,10-*d*]imidazole (5g)

M.P. 213-215 °C, IR (KBr) ν (cm⁻¹): 3010, 2941, 2832, 2588, 1945, 1612, 1579, 1527, 1461, 1378, 1295, 1202, 1158, 1115, 1031, 916, 833, 800, 750, 691, 622, 532, 426. ¹H NMR (250 MHz, CDCl₃) δ (ppm): 3.5 (s, 3H, CH₃), 3.77 (s, 3H, CH₃), 6.28 (s, 1H, ArH), 6.49 (d, 1H, J=8.2 Hz, ArH), 7.27 (s, 1H, ArH), 7.41-7.44 (m, 7H, ArH), 7.59-7.74 (m, 3H, ArH), 8.70 (d, 1H, J=8 Hz, ArH), 8.75 (d, 1H, J=8.5 Hz, ArH), 8.88 (d, 1H, J=7.7 Hz, ArH). ¹³C NMR

(62.5 MHz, CDCl₃) δ (ppm): 54.9, 55.3, 98.1, 104.4, 112.9, 120.9, 122.8, 123, 124, 124.7, 125.3, 126.1, 127.1, 127.4, 128.1, 128.6, 128.8, 128.9, 133.2, 137.4, 138.4, 150, 158.7, 162.1. Mass *m/z* (%): 430 (M⁺, 100), 399 (46), 353 (27), 267 (79), 190 (77), 163 (90), 77 (78).

RESULTS AND DISCUSSION

In this paper, a series of tetrasubstituted imidazoles named as phenanthro[9,10-*d*]imidazole were prepared through a one-pot four-component reaction of 9,10-phenanthraquinone **1**, aromatic aldehyde **2**, aniline **3**, and ammonium acetate **4** using SBA-Pr-SO₃H as a nanoporous solid acid catalyst (Fig. 1). At first, in order to modify the reaction conditions, different environments were tested including solvent-free system at ambient temperature and 120 °C, refluxing in water, ethanol and/or acetic acid as green solvents. As shown in Table 1, the best result was obtained under refluxing in acetic acid in the presence of SBA-Pr-SO₃H (0.02 g) with excellent yield of the

product. To investigate the effect of catalyst, this reaction was tested under modified conditions in the absence of SBA-Pr-SO₃H; the results showed that SBA-Pr-SO₃H can catalyze the reaction in a shorter time with high yield of the product. After this, to study the generality of this process, several aromatic aldehydes were applied for the synthesis of other phenanthro[9,10-*d*]imidazole derivatives as shown results in Table 2. Except compounds **5c** and **5g**, all the other products were obtained in high yields by varying the reaction time in the range of 5-20 min. The low efficiency of products **5c** and **5g** perhaps is owing to the side reaction which likely occurs in *p*-anisaldehyde stains [28] in which methoxy substituted benzaldehydes can form a triarylmethane dye in the presence of acetic acid under acidic media.

In order to understand the effect of SBA-Pr-SO₃H as catalyst in this reaction, the reaction conditions of present methodology were compared with some other published methods [5, 6, 29, 30] and summarized in Table 3 (Entries 2 and 3). As it is

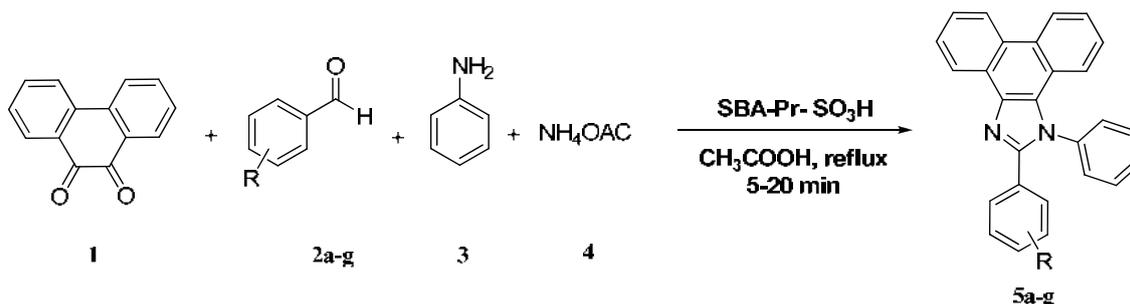


Fig. 1. Synthesis of phenanthro[9,10-*d*]imidazole derivatives **5a-g** in the presence of SBA-Pr-SO₃H.

Table 1. Optimizing the reaction conditions for the synthesis of **5a**

Entry	Catalyst	Solvent	Condition	Time (h)	Yield (%)
1	SBA-Pr-SO ₃ H	-	r.t.	24	trace
2	SBA-Pr-SO ₃ H	-	120 °C	1	80
3	SBA-Pr-SO ₃ H	H ₂ O	Reflux	>5	-
4	SBA-Pr-SO ₃ H	EtOH	Reflux	>5	70
5	SBA-Pr-SO ₃ H	CH ₃ COOH	Reflux	5 min	97
6	-	CH ₃ COOH	Reflux	0.5	93

*Reaction conditions: 9,10-Phenanthraquinone (1 mmol), 4-chlorobenzaldehyde (1 mmol), aniline (1.5 mmol), ammonium acetate (4 mmol), SBA-Pr-SO₃H (0.02 g).

Table 2: Synthesis of phenanthro[9,10-*d*]imidazole derivatives in the presence of SBA-Pr-SO₃H under reflux conditions in acetic acid.

Entry	R	Product	Time (min)	Yield (%)	mp (°C)	mp (Lit.)
1	4-Cl	5a	5	97	240-243	233 [9]
2	4-F	5b	20	80	193-195	-
3	4-OMe	5c	10	50	217-220	212-213 [9]
4	4-Me	5d	10	98	201-203	250-250.5 [9]
5	4-OH	5e	20	98	>300	New
6	3-NO ₂	5f	10	94	233-235	New
7	2,4-(OMe) ₂	5g	20	70	213-215	New

clear, the safe and green SBA-Pr-SO₃H catalyzed and accelerated the reaction more efficiently to give the higher yield of product within shorter reaction time.

The proposed mechanism is shown in Fig. 2. Firstly, the carbonyl group of aromatic aldehyde is protonated by the solid acid catalyst. Then, a nucleophilic attack of ammonia, produced by ammonium acetate, and then aniline to the activated carbonyl group of compound **6** gives intermediate **8**. Condensation of 9,10-phenanthraquinone **1** with intermediate **8** through an imination process produces compound **9** which is cyclized by addition of the second amine group. Finally, after a dehydration process, the desired product **5** is obtained.

Preparation and Characterization of SBA-Pr-SO₃H

SBA-Pr-SO₃H was prepared as mentioned in experimental section, and then, characterized.

The TGA curve of SBA-Pr-SO₃H is shown in Fig. 3. According to the weight reduction in the temperature range between 200-600 °C (about 20% mass loss), the amount of propyl sulfonic acid groups was calculated as 1.2 mmol/g. In addition, using back titration method, concentration of sulfonic acid functional groups onto the pores of SBA-Pr-SO₃H was estimated through addition of a very dilute standardized NaOH solution (0.1 M). The excess amount of NaOH was titrated with a standardized HCl. The obtained data showed that each grams of SBA-Pr-SO₃H contains 1.28 mmol sulfonic acid functional groups. Good agreement between both values obtained by back titration and TGA shows that the SO₃H groups were incorporated onto the internal surface of SBA-15.

The small angle powder XRD pattern of both SBA-15 and SBA-Pr-SO₃H (Fig. 4) display the three characteristic peaks at the 2θ (°) values of 1.00, 1.69 and 1.93 which are corresponded

Table 3: Comparing the reaction conditions in the synthesis of 2,3-dihydroquinazolin-4(1H)-one **4a**

Entry	Solvent	Catalyst	Conditions	Time (h)	Yield (%)	Year [ref.]
1	CH ₃ COOH	SBA-Pr-SO ₃ H	reflux	5-20 min	50-90	This work
2	CH ₃ COOH	-	reflux	3	63-79	2012 [5]
3	H ₂ O/MeOH	MPS*	Room temperature	3	84-92	2010 [6]
4	-	In(OTf) ₃	120 °C	0.5-1	75-94	2014 [29]
5	MeCN	L-proline	reflux	5	74-82	2013 [30]

* Mercaptopropylsilica

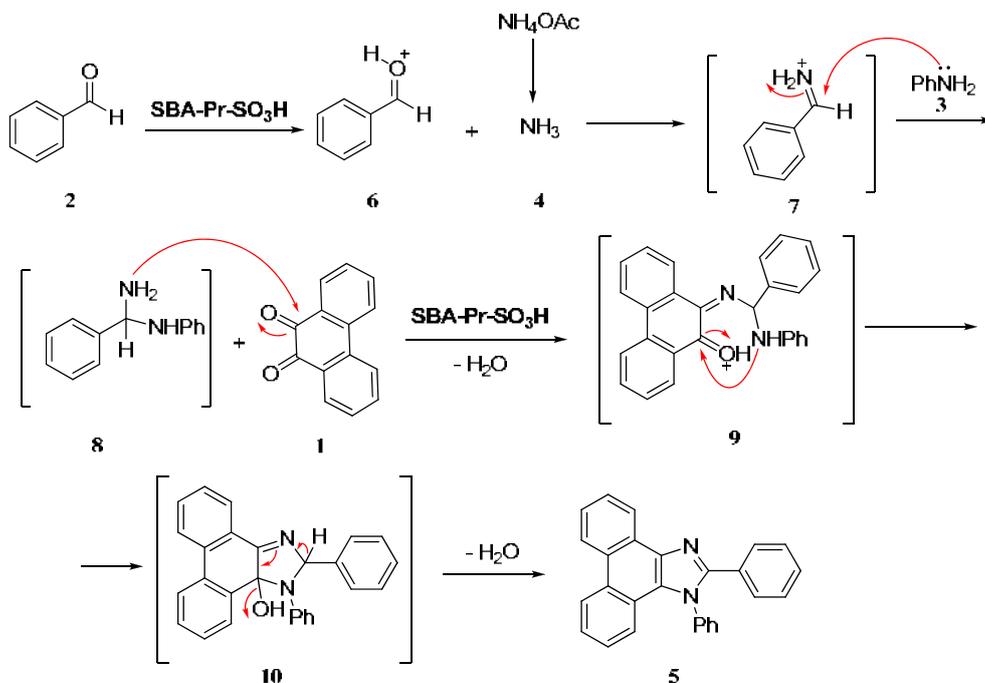


Fig. 2. The proposed mechanism for the synthesis of phenanthro[9,10-d]imidazole derivatives.

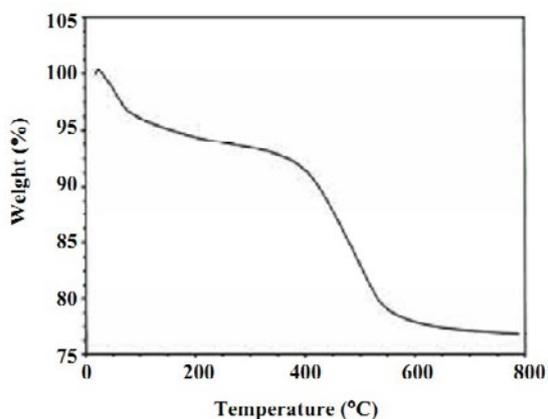


Fig. 3. TGA curve of SBA-Pr-SO₃H

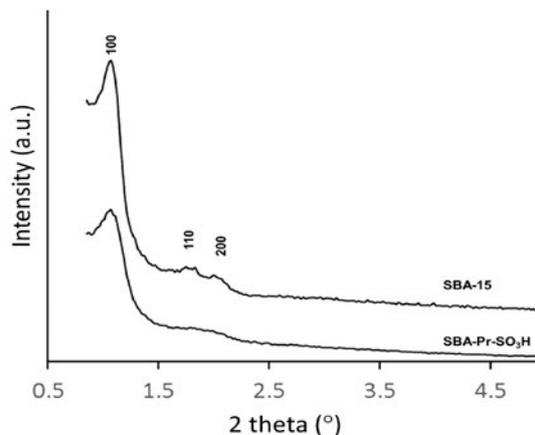


Fig. 4. The small angle powder XRD pattern of SBA-15 and SBA-Pr-SO₃H

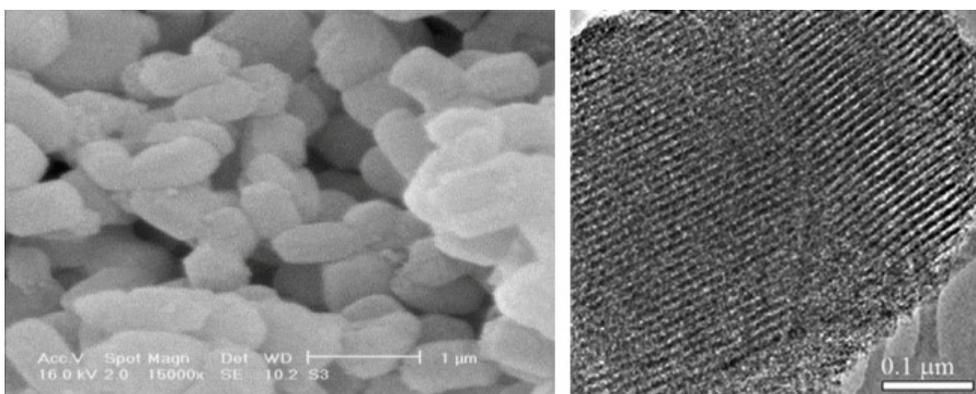


Fig. 5. SEM (Left) and TEM (Right) images of SBA-Pr-SO₃H

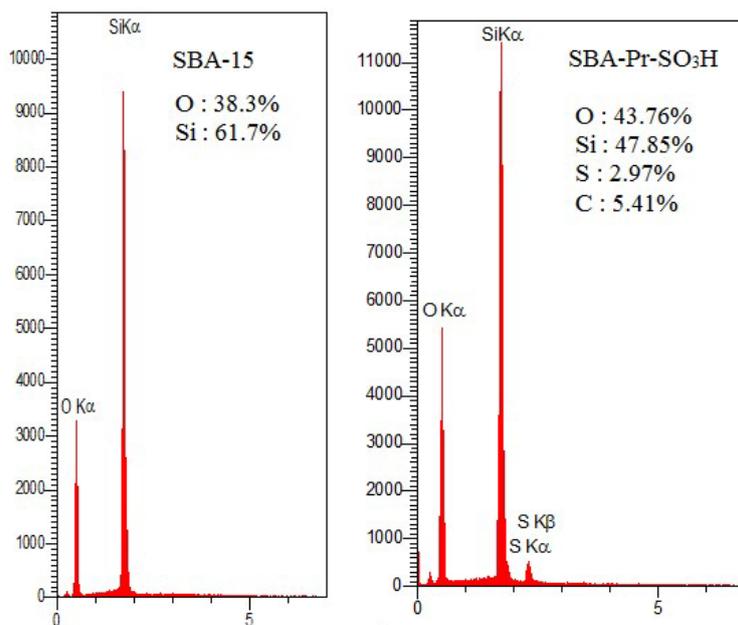


Fig. 6. EDS analysis of SBA-15 and SBA-Pr-SO₃H

to the 100 (strong), 110 (weak) and 200 (weak) reflections, respectively. Such pattern confirms the 2D-hexagonal structure of mesoporous compounds. Although, a considerable decrease is detected in the intensity of SBA-Pr-SO₃H which is due to grafting the propyl-SO₃H groups onto the pores of SBA-15.

SEM image of SBA-Pr-SO₃H (Fig. 5-Left) displays uniform particles about 700-900 nm. The same morphology was previously observed for SBA-15. It can be concluded that during the modification procedure, the morphology of SBA-15 framework was saved without any changes. Besides this, the TEM image (Fig. 5-Right) demonstrates the parallel channels, which resemble the pores configuration of SBA-15. This confirms that the pore of SBA-Pr-SO₃H was not collapsed during two steps modification.

CONCLUSION

In conclusion, in this research the importance of phenanthro[9,10-*d*]imidazole for OLED technology was shown. Then, SBA-Pr-SO₃H was used as a mesoporous solid acid catalyst in the synthesis of phenanthro[9,10-*d*]imidazole derivatives through a one pot four-component reaction of 9,10-phenanthraquinone, aromatic aldehydes, aniline derivatives and ammonium acetate in refluxing acetic acid. Short reaction time, good yield and easy isolation of the products are the advantages of this method and therefore, phenanthro[9,10-*d*]imidazole synthesis was improved by the use of an efficient catalyst.

ACKNOWLEDGEMENTS

We gratefully acknowledge the financial support from the Research Council of Alzahra University and the University of Tehran.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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