Synthesis of 8-Aryl-7*H*-acenaphtho[1,2-*d*]imidazoles Using Fe₃O₄ NPs@GO@C₄H₈SO₃H as a Green and Recyclable Magnetic Nanocatalyst

F. Hasanzadeh^a and F. K. Behbahani^{a,*}

^a Department of Chemistry, Karaj Branch, Islamic Azad University, Karaj, 314/85313 Iran ^{*}e-mail: farahnazkargar@yahoo.com

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Abstract—A general procedure has been developed for the rapid synthesis of 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazoles in high yields using $Fe_3O_4NPs@GO@C_4H_8SO_3H$ as a green and recyclable magnetic nanocatalyst under reflux conditions. The nanocatalyst was prepared and characterized by FT-IR, XRD, and EDX data. A variety of aromatic aldehydes underwent condensation with NH₄OAc and acenaphthenequinone to give 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazole derivatives. The use of magnetic nanoparticles, easy separation of the catalyst with an external magnet, high yields, and short reaction times are the main advantages of this catalytic method.

Keywords: magnetic nanocatalyst, acenaphthoquinone, imidazole, Fe₃O₄ NPs@GO@C₄H₈SO₃H

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INTRODUCTION

Imidazoles as an important class of heterocyclic compounds reveal various biological activities such as herbicidal, antiallergic, analgesic, antidepressant, antitubercular, anticancer, anti-inflammatory, antifungal, and antiviral [1–9]. Imidazole ring is present in histidine [10] and the related hormone histamine [11], and some of imidazole derivatives are employed in electronic and optoelectronic devices [12]. Tetrasubstituted imidazoles have been synthesized by the four-component cyclocondensation of a 1,2-diketone, aldehyde, primary amine, and ammonium acetate using various Lewis or protic acids as catalysts, namely BF₃-SiO₂ [13], silica supported NaHSO₄ [14], HPA-EtOH [15], L-proline [16], $K_5CoW_{12}O_{40} \cdot 3H_2O$ [17], and HY zeolite–Cu(NO₃)₂ [19]. reactions between a 1,2-diketone, nitrile, and primary amine under microwave irradiation [20] and between 1,3-oxazolium-5-olates and *N*-(arylmethylidene)benzenesulfonamides [21] have also been reported. Multicomponent reactions (MCRs) are a very powerful tool in organic and medicinal chemistry for the preparation of bulky products in a one-pot fashion from simple starting materials. A combination of magnetic nanocatalysts and multicomponent reactions could provide efficient and sustainable procedures for green syntheses [22]. Moreover, to the best of our knowledge, Fe_3O_4 NPs@GO@. C₄H₈SO₃H as a new reusable heterogeneous nanocatalyst has not been utilized previously for one-pot preparation of 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazoles. Therefore, in continuation of our studies on the synthesis of heterocyclic compounds [23–27], we examined a wide variety of substituted benzaldehydes as starting materials to establish the catalytic importance of Fe_3O_4 NPs@GO@C₄H₈SO₃H for one-pot multicomponent





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synthesis of 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazoles via condensation with acenaphthenequinone and ammonium acetate (Scheme 1).

RESULTS AND DISCUSSION

Initially, graphene oxide (GO) was synthesized from graphite according to procedure described in [29], and Fe₃O₄ NPs@GO and Fe₃O₄ NPs@GO@ \cdot C₄H₈SO₃H were prepared as reported in [30]. The synthesis of Fe₃O₄ NPs@GO@C₄H₈SO₃H, the growth of Fe₃O₄ nanoparticles, and the loading of C₄H₈SO₃H onto graphene oxide are illustrated by Scheme 2.

To confirm the structure of the nanocatalyst, its FT-IR spectrum was measured (Fig. 1). The FT-IR spectrum of graphene oxide exhibits absorption bands for the functionalities containing oxygen atoms such as OH group (\sim 3435 cm⁻¹), carboxylic C=O group (\sim 1719 cm⁻¹), and C=C (\sim 1585 cm⁻¹) and C=O bonds

(~1385 cm⁻¹) (Fig. 1a). The presence of Fe–O band (~564.13 cm⁻¹) in the FT-IR spectrum of Fe₃O₄ NPs@· GO confirms that Fe₃O₄ NPs are located on the GO structure (Fig. 1b). Figure 1c shows new bands at 2919 and 1029 cm⁻¹ which were assigned to stretching vibrations of Csp^3 –H and C–S bonds, respectively, indicating that butane-1,4-sultone was loaded on Fe₃O₄ NPs@GO@C₄H₈SO₃H.

The XRD pattern of the prepared Fe_3O_4 NPs@·GO@C₄H₈SO₃H composite is shown in Fig. 2. The peaks appearing at 30.22, 35.62, 43.62, 53.92, 57.32, and 62.77° confirmed loading of Fe₃O₄ NPs on GO. These peaks are due to the cubic spinel crystal planes of Fe₃O₄. The presence of butane-1,4-sultone groups on the surface of Fe₃O₄NPs@GO was further confirmed by energy dispersive spectroscopy (EDS) (Fig. 3). According to the EDS data, the prepared catalyst contains 30.14% of carbon, 33.56% of oxygen; 1.74% of sulfur, and 35.69% of iron.







Fig. 1. FT-IR spectra of (a) graphene oxide (GO), (b) GO@ Fe_3O_4 NPs, and (c) GO@ Fe_3O_4 NPs@C₄H₈SO₃H.









To investigate the substituent effect, a wide variety of aromatic aldehydes containing electron-withdrawing or electron-donating groups were utilized. Aromatic aldehydes with both electron-donating and electronwithdrawing groups reacted under the selected conditions in short reaction times (Table 2). The yields were good to excellent without formation of any byproducts such as oxidation products of aldehydes, etc., which are normally formed under strongly acidic conditions. In each case, the reaction profile was clean, and this one-pot three-component procedure revealed some advantages over existing methods. The other features of this new method are simple isolation and purification of the products, easy separation of the catalyst by an external magnet and its repeated use.

To show the merits of this procedure in comparison with the previously reported protocols, we compared the synthesis of 8-phenyl-7*H*-acenaphtho[1,2-*d*]imidazole (**4f**) under different conditions (Table 3). The advantages of our procedure are the use of Fe₃O₄ NPs@GO@C₄H₈SO₃H as heterogeneous, reusable, and magnetically separable catalyst, excellent yield, short reaction time, and mild reaction condition, which are very important for chemical industry.

A plausible mechanism for the formation of 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazoles from aldehydes, acenaphthenequinone, and ammonium acetate is given in Scheme 3.

EXPERIMENTAL

The melting points were measured in capillary tubes with an Electrothermal 9200 apparatus. The IR spectra were recorded on a Perkin Elmer FT-IR spectrometer between 4000 and 400 cm⁻¹. The ¹H NMR spectra

Amount of the catalyst, g	Solvent	Temperature, °C	Reaction time, min	Yield, %
0.05	EtOH	Reflux	7	84
0.08	EtOH	Reflux	7	87
0.09	EtOH	Reflux	9	94
0.09	EtOH	70	5	94
0.1	THF	75	8	95
0.1	CHCl ₃	80	10	80
0.1	EtOH	75	8	90
0.1	EtOH	Reflux	5	98
0.12	EtOH	Reflux	8	95
0.15	EtOH	Reflux	8	85
0.17	EtOH	Reflux	10	84
0.2	EtOH	Reflux	12	85

Table 1. The effects of the catalyst amount, temperature, and solvent in the synthesis of 8-phenyl-7*H*-acenaphto[1,2-*d*]-imidazole (**4f**) in the presence of Fe₃O₄ NPs@GO@C₄H₈SO₃H^a

^a Benzaldehyde (1 mmol), acenaphthenequinone (1 mmol), ammonium acetate (2 mmol) and Fe₃O₄ NPs@GO@C₄H₈SO₃H in a solvent (10 mL)

Table 2. Synthesis of 8-aryl-7*H*-acenaphto[1,2-*d*]imidazoles 4a–4j using Fe₃O₄ NPs@GO@C₄H₈SO₃H

Compound no.	R	Reaction time, min	Yield, %
4 a	3-NO ₂	15	82
4b	3-ОН	7	96
4c	4-Me	5	98
4d	4-Br	8	92
4 e	4-F	10	94
4f	Н	5	98
4g	2-ОН	9	94
4h	2-OH-3-OMe	8	96
4i	2-OMe	12	94
4j	2,6-Cl ₂	10	95

Table 3. Comparison of different reaction conditions for the synthesis of 4f

Catalyst	Reaction time, min	Yield, %	Solvent	Reference
Nano-SnO ₂	150	86	EtOH	[28]
Nano-SiO ₂	600	40	EtOH	[28]
Nano-ZrO ₂	360	50	EtOH	[28]
Nano-ZnO	240	60	EtOH	[28]
Nano-Fe ₂ O ₃	240	65	EtOH	[28]
NH ₃ /NH ₄ Cl	180	80	EtOH	[30]
Fe ₃ O ₄ NPs@GO@C ₄ H ₈ SO ₃ H	5	98	EtOH	This work

were obtained on a Bruker DRX- 300 NMR instrument operating at 300 MHz. Analytical TLC of all reactions was performed on precoated silica gel plates (silica gel 60 F-254 on aluminum, Merck). Elemental analyses (EDS) and X-ray diffraction (XRD) analysis of the catalyst were carried out by a PW1840-Philips instrument with Cu K_{α} radiation.

Synthesis of 8-aryl-7*H*-acenaphtho[1,2-*d*]imidazoles 4a-4j using Fe₃O₄ NPs@GO@C₄H₈SO₃H (general procedure). A mixture of aldehyde 3a-3j





(1 mmol), acenaphthenequinone (1 mmol), ammonium acetate (2 mmol), and Fe_3O_4 NPs@GO@C₄H₈SO₃H (0.1 g) in EtOH (10 mL) was refluxed for a time indicated in Table 2. The progress of the reaction was monitored by TLC. After completion of the reaction, the catalyst was separated by an external magnet, the solvent was evaporated, and the residue was recrystallized from aqueous ethanol.

8-(3-Nitrophenyl)-7*H***-acenaphtho[1,2-***d***]imidazole (4a). Yield 82%, orange solid, mp 227–230°C [30]. IR spectrum (KBr), v, cm⁻¹: 3433 (N–H), 3049 (C–H_{arom}), 1683 (C=N), 1525 (C=C), 1500 (NO₂), 1345 (C–N), 766 (\deltaC–H, out-of-plane). ¹H NMR spectrum (DMSO-***d***₆), \delta, ppm: 7.63–7.5 m (7H, H_{arom}), 8.2 d (1H), 8.4 d (1H), 8.8 s (1H).**

3-(7H-Acenaphtho[1,2-*d***]imidazol-8-yl)phenol (4b). Yield 96%, brown solid, mp 330–332°C [28]. IR spectrum (KBr), v, cm⁻¹: 3392 (N–H, O–H), 3055 (C–H_{arom}), 1678 (C=N), 1590 (\deltaN–H), 1480 (C=C), 1277 (C–N), 1227 (C–O), 770 (\deltaC–H, out-of-plane). ¹H NMR spectrum (CDCl₃), \delta, ppm: 8.88 d.d (1H,** *J* **= 7.6, 1.2 Hz), 8.77 d.d (1H,** *J* **= 7.6, 1.2 Hz), 8.43 d (1H,** *J* **= 6.8 Hz), 8.33 d.d (1H,** *J* **= 8, 0.8 Hz), 8.04– 8.10 m (2H), 7.83–7.89 m (3H), 7.79 s (1H), 7.63– 7.70 m (2H).**

8-(4-Methylphenyl)-7*H***-acenaphtho[1,2-***d***]imidazole (4c). Yield 98%, orange solid, mp. 230–232°C [30]. IR spectrum (KBr), ν, cm⁻¹: 3436 (N–H), 3041 (C–H_{arom}), 1687 (C=N), 1608 (δN–H), 1595 (C=C),** 1384 (C–CH₃), 1108 (C–N), 775 (δC-H, out-of-plane). ¹H NMR spectrum (DMSO- d_6): 2.42 s (3H), 7.25 m (3H), 7.47 d (2H), 7.67 d (3H), 7.85 d (1H).

8-(4-Bromophenyl)-7*H***-acenaphtho[1,2-***d***]imidazole (4d). Yield 92%, brown solid, mp 262–264°C [28]. IR spectrum (KBr), v, cm⁻¹: 3436 (N–H), 3094 (C–H_{arom}), 1656 (C=N), 1607 (\deltaN–H), 1484, 1417 (C=C), 1384 (C–N), 762 (\deltaC–H, out-of-plane), 683 (C–Br). ¹H NMR spectrum (DMSO-***d***₆), \delta, ppm: 8.88 d (1H,** *J* **= 7.6 Hz), 8.77 d (1H,** *J* **= 8 Hz), 8.43 d (1H,** *J* **= 6.8 Hz), 8.35 s (1H), 8.31 d (1H,** *J* **= 7 Hz), 8.15 d (1H,** *J* **= 7 Hz), 8.08–8.10 m (2H), 7.90–7.83 m (4H), 7.79– 7.81 m (1H), 7.64–7.70 m (2H).**

8-(4-Fluorophenyl)-7*H***-acenaphtho[1,2-***d***]imidazole (4e). Yield 94%, light brown solid, mp 301– 303°C [28]. IR spectrum (KBr), v, cm⁻¹: 3480 (N–H), 3130 (C–H_{arom}), 1674 (C=N), 1607 (\deltaN–H), 1511, 1417 (C=C), 1384 (C–N), 1236 (C–F), 762 (\deltaC–H, out-of-plane). ¹H NMR spectrum (DMSO-***d***₆), \delta, ppm: 8.88 d.d (1H,** *J* **= 7.2, 1.2 Hz), 8.77 d.d (1H,** *J* **= 7.2, 1.2 Hz), 8.43 d (1H,** *J* **= 6.4 Hz), 8.33 d.d (1H,** *J* **= 8, 1.2 Hz), 8.08–8.10 m (2H), 7.81–7.89 m (2H), 7.79 s (1H), 7.63–7.70 m (2H).**

8-Phenyl-7*H*-acenaphtho[1,2-*d*]imidazole (4f). Yield 91%, yellow solid, mp 270–272°C [30]. IR spectrum (KBr), cm⁻¹: 3434 (N–H), 1664 (C=N), 1603 (δ N–H), 1590 (C=C), 1227 (C–N), 776 (δ C–H, out-ofplane). ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 12.91 s (1H), 7.26 m (2H), 7.46 m (1H), 7.53 m (2H), 7.62 m (2H), 8.18 m (2H). **2-(7***H***-Acenaphtho[1,2-***d***]imidazol-8-yl)phenol (4g). Yield 94%, brown solid, mp 294–297°C [28]. IR spectrum (KBr), cm⁻¹: 3435 (N–H, O–H), 3055 (C–H_{arom}), 1697 (C=N), 1657 (C=C), 1591 (\deltaN–H), 1384 (C–N), 1287 (C–O), 760 (\deltaC–H, out-of-plane). ¹H NMR spectrum (DMSO-***d***₆), \delta, ppm: 6.86–6.93 d (2H,** *J* **= 7.5 Hz), 6.97–7.01 d (2H,** *J* **= 7.8 Hz), 7.2– 7.52 m (10H), 12.49 br.s (NH).**

2-(7*H***-Acenaphtho[1,2-***d***]imidazol-8-yl)-6-methoxyphenol (4h). Yield 96%, brown solid, mp 102– 105°C. IR spectrum (KBr), v, cm⁻¹: 3436 (N–H, OH), 3098 (C–H_{arom}), 1653 (C=N), 1583 (N–H), 1619 (C=C), 1584 (C–N), 1253 (C–O), 719 (\deltaC–H, out-ofplane). ¹H NMR spectrum (DMSO-***d***₆), \delta, ppm: 6.99– 7.04 d (2H,** *J* **= 8 Hz), 7.51–7.86 m (10H), 7.87–7.90 d (2H,** *J* **= 8.5 Hz), 12.61 br.s (NH). Mass spectrum:** *m/z* **314.10 (***I***_{rel} 21%) [***M* **+ 1]⁺. Found, %: C 76.40; H 4.46; N 8.88. C₂₀H₁₄N₂O₂. Calculated, %: C 76.42; H 4.49; N 8.91.**

8-(2-Methoxyphenyl)-7*H*-acenaphtho[1,2-*d*]imidazole (4i). Yield 94%, orange solid, mp 220– 225°C. IR spectrum (KBr), v, cm⁻¹: 3435 (N–H), 1603 (C=N), 1583 (δN–H), 1478 (C=C), 1384 (C–N) 1243 (C–O), 767 (δC–H, out-of-plane). ¹H NMR spectrum (DMSO-*d*₆), δ, ppm: 3.07 s (3H, OMe), 7.80–7.82 m (5H, H_{arom}), 8.05 d (4H, H_{arom}, *J* = 7.0 Hz), 8.26 d (4H, H_{arom}, *J* = 8.3 Hz), 8.40 d (1H, H_{arom}, *J* = 8.0 Hz). Mass spectrum: *m*/*z* 298.11 (*I*_{rel} 20%) [*M* + 1]⁺. Found, %: C 80.50; H 4.71; N 9.36. C₂₀H₁₄N₂O. Calculated, %: C 80.52; H 4.73; N 9.39.

8-(2,6-Dichlorophenyl)-7*H***-acenaphtho[1,2-***d***]imidazole (4j). Yield 95%, yellow solid, mp 250– 258°C. IR spectrum (KBr), cm⁻¹: 3413 (N–H), 3055 (C–H_{arom}), 1678 (C=N), 1591 (\deltaN–H), 1480 (C=C), 1227 (C–N) 819 (\deltaC–H, out-of-plane), 770 (C–Cl). ¹H NMR spectrum (DMSO-***d***₆): 7.82–7.90 m (4H, H_{arom}), 8.12 d (3H, H_{arom},** *J* **= 7.0 Hz), 7.29–8.36 m (4H, H_{arom}), 8.36 d (1H, H_{arom},** *J* **= 7.3 Hz). Mass spectrum:** *m***/***z* **336.02 (***I***_{rel} 20%) [***M* **+ 1]⁺. Found, %: C 67.60; H 2.95; Cl 21.00; N 8.29. C₁₉H₁₀C₁₂N₂. Calculated, %: C 67.68; H 2.99; Cl 21.03; N 8.31.**

CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

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