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Efficient and eco-friendly synthesis of quinoxalines derivatives catalyzed by acidic ionic liquids at room temperature

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ABSTRACT

In this research, we have developed cheap recyclable and task-specific acidic ionic liquids (AILs) 1-hydrogen-3-methylimidazolium hydrogen sulfate [Hmim]HSO_4, 1-hydrogen-3-methylimidazolium chloride [Hmim]Cl, 2-pyrrolidonium hydrogensulfate [Hnhp]HSO_4 and applied them in acid catalyzed synthesis of quinoxaline derivatives from 1,2-phenylendiamines and 1,2-dicarbonyl compounds. The products could be separated from the catalyst simply by filtration and the catalyst could be recycled and reused several times without noticeable loss of efficiency.

***Keywords**: Acidic Ionic liquid, AILs, Quinoxaline, Green chemistry, Room temperature.*

1. Introduction

A number of compounds based on nitrogen-containing heterocycles constitute useful intermediates in organic synthesis [1,2]. The quinoxaline nucleus is present in many pharmaceutical agents, It can also exhibit a broad spectrum of biological activities, such as antitumor [3], antiviral [4], antiglucoma [5], antituberculosis [6], antiinflammatory [7,8] and anticancer activities [9]. Furthermore, they are potential building blocks for the synthesis of organic semiconductors [10], electroluminescent material [11], dehydroannulenes [12] and dyes [2]. A number of synthetic strategies have been developed for the preparation of substituted quinoxalines [13-15]. We focused on the modification of this reaction using 1,2-diketone alternatives and related catalysts [16-17]. Reactions using 1,2-diketone alternatives such as phenyl epoxides, phenacyl bromides, α-hydroxyketones, and *o*-phenylenediamines with the catalysts such as Bi(0) [18], $HClO_4/SiO_2$ [19], β-cyclodextrin (β-CD) [20], TMSCl [21], 1,4-diazabicyclo-[2,2,2]-octane (DABCO) [22], transition metals (Mn, Ru, Pd, and Cu) [23-27], ion-exchanged molybdophosphoric acid [28], SBSSA [29], IBX [30], oxalic acid [31], microwave/I_2 [32], $SnCl_2/SiO_2$, [33], I_2 [34], ultrasound irradiation [35], NH_4Cl [36], $(NH_4)_6Mo_7O_{24}.4H_2O$ [37], citric acid [38], ionic liquid [39], bentonite Clay K-10 [40], AcOH [41], BSA [42] and $Ga(ClO_4)_3$/EtOH [43] have been reported.

However, some of the mentioned methods have drawbacks such as requiring expensive reagents, long reaction times, low yields of the products and tedious work-up. Therefore, there is an urgent need for the introduction of new methods and inexpensive reagents for the synthesis of quinoxalines derivatives. Herein, we would like to report efficient, convenient mild and high yield in the presence of catalytic amount of acidic ionic liquids. The acidic ionic liquids such as 1-hydrogen-3-methylimidazolium hydrogen sulfate [44], 1-hydrogen-3-methylimidazolium chloride [45], 2-pyrrolidonium hydrogensulfate [46] (Scheme1), were prepared according to literature [44-46]. The reaction was easily carried out at room temperature under green conditions (Scheme 2).

2. Experimental

2.1. General

Products were characterized by comparison of their physical data, IR and ^{1}H NMR and ^{13}C NMR spectra with known samples. NMR spectra were recorded in DMSO on a Bruker Advance DPX 300 MHz instrument spectrometer using TMS as an internal standard.

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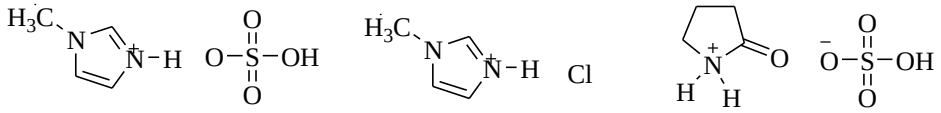


[Hmim]HSO_4 [Hmim]Cl [Hnhp]HSO_4

Scheme 1. The structure of acidic ionic liquids: [Hmim]HSO_4, [Hmim]Cl, [Hnhp]HSO_4.

The purity determination of the products and reaction monitoring were accomplished by TLC on silica gel polygram SILG/UV 254 plates. IR spectra of the compounds were obtained on a PerkinElmer spectrometer version 10.03.06 using a KBr disk.

2.2 General Procedure for the Preparation of Quinoxaline Derivatives (Scheme 2)

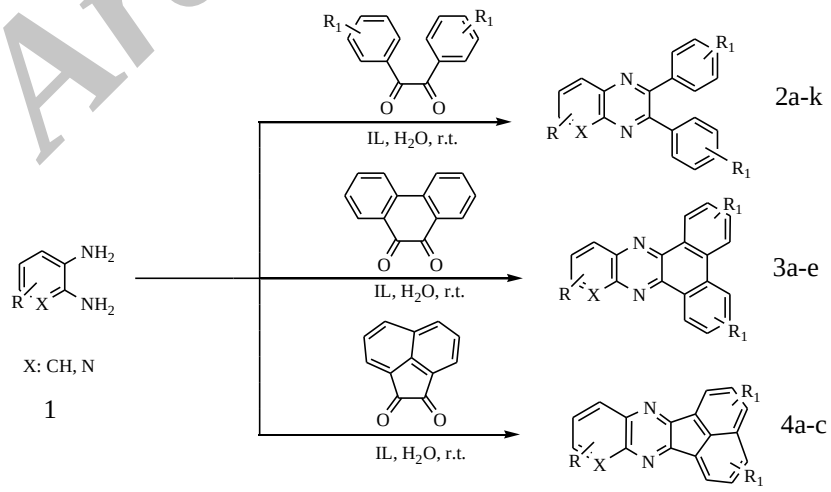
Phenylendiamine (1 mmol) and 1,2-diketone (1 mmol), acidic ionic liquid (10%) and H_2O (5mL) were stirred at room temperature in a 50 ml round-bottomed flask. The progress of the reaction was monitored by TLC (ethyl acetate: *n*-hexane, 1:20). Afterward, H_2O (20 ml) was added to the reaction mixture and was allowed to remain at room temperature for 30 mins. Crystals of the crude product were collected by filtration, and then were recrystallized from EtOH to give the pure product.

Selected specteral data

6-Bromo-2,3-diphenylpyrido[3,2-b]pyrazine **(2e)**: Cream solid m.p. 143-145 °C, $C_{19}H_{12}BrN_3$, Elem. Anal. Calc.: C, 63.00; H, 3.34; Br, 22.06; N, 11.60, Found: C, 62.54; H, 3.50; N, 11.50; FT-IR (KBr): 1610 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 7.35-7.46 (m, 6H, Ar-H), 7.58 (d, *J*=6.76, 2H, Ar-H), 7.66 (d, *J*=7.12, 2H, Ar-H), 8.71 (s, 1H, Ar-H), 9.19 (s, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 120.943, 128.265, 128.475, 129.625, 129.710, 129.841, 130.204, 136.405, 137.763, 138.065, 139.398, 148.224, 155.115, 155.486, 156.505; MS: m/z = 361 (M^+).

2,3-Bis(4-methoxyphenyl)-6-nitroquinoxaline **(2h)**: Ash-gray solid m.p. 188-189°C, $C_{22}H_{17}N_3O_4$, Elem. Anal. Calc.: C, 68.21; H, 4.42; N, 10.85; O, 16.52, Found: C, 68.28; H, 4.12; N, 11.05; FT-IR (KBr): 1613 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 3.90 (s, 3H, CH_3), 3.93 (s, 3H, CH_3), 7.01 (d, *J*=8.84, 4H, Ar-H), 7.60 (q, *J*=5.38, 1H, Ar-H), 7.99 (d, *J*=8.83, 4H, Ar-H), 8.27 (d, *J*=9.14, 1H, Ar-H), 9.06 (d, *J*=2.3, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 55.390, 55.656, 113.970, 114.295, 126.269, 130.413, 130.593, 131.345, 131.509, 132.386, 164.861, 193.526; MS: m/z = 387 (M^+).

(2,3-Bis (4-methoxyphenyl)pyrido[3,2-b]pyrazin-7-yl) (phenyl)methanone **(2j)**: Yellow solid m.p. 145-147°C, $C_{28}H_{21}N_3O_3$, Elem. Anal. Calc.: C, 75.15; H, 4.73; N, 9.39; O, 10.73, Found: C, 75.26; H, 4.83; N, 9.18; FT-IR (KBr): 1655 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 3.87 (s, 3H, OCH_3) 3.90 (s, 3H, OCH_3), 6.92 (q, *J*=4.18, 3H, Ar-H), 6.99 (d, 1H, Ar-H), 7.57 (q, *J*=6.9, 4H, Ar-H), 7.67 (t, *J*=7.2, 1H, Ar-H), 7.93-8.00 (dd, *J*=7.88, *J*=8.84, 2H, Ar-H), 8.27 (s, 1H, Ar-H), 8.53 (s, 1H, Ar-H);



Scheme 2. Synthesis of quinoxalines derivatives using acidic ionic liquids.

^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 55.351, 55.653, 113.882, 114.301, 126.255, 128.510, 129.486, 130.148, 131.059, 131.256, 131.457, 132.367, 132.768, 137.268, 137.856, 139.940, 142.782, 154.138, 154.637, 160.461, 160.618, 164.846, 193.505, 195.868; MS: m/z = 447 (M^+).

2,3-Bis(4-methoxyphenyl)-6-methylquinoxaline **(2k)**: Brown solid m.p. 122-123°C, $C_{23}H_{20}N_2O_2$, Elem. Anal. Calc.: C, 77.51; H, 5.66; N, 7.86; O, 8.98, Found: C, 77.49; H, 5.78; N, 7.76; FT-IR (KBr): 1608 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 2.65 (s, 3H, Ar-OCH_3), 3.88 (s, 6H, 2×OCH_3), 6.92 (d, *J*=8.55, 4H, Ar-H), 7.52-7.55 (dd, *J*=2.8, *J*=2.75, 4H, Ar-H), 7.62 (d, *J*=8.53, 1H, Ar-H), 8.02 (s, 1H, Ar-H), 8.12 (d, *J*=8.52, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 21.965, 55.337, 113.813, 114.296, 127.369, 128.205, 130.935, 131.085, 131.322, 131,407, 132.321, 139.173, 140.646, 152.043, 160.249, 160.349; MS: m/z = 356 (M^+).

11-Benzoil-dibenzo[a,c]phenazine **(3c)**: yellow solid m.p. 245-247°C, FT-IR (KBr): 1653, 1606 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 7.27 (s, 1H, Ar-H), 7.60 (s, 2H, Ar-H), 7.71 (s, 2H, Ar-H), 7.79 (s, 2H, Ar-H), 7.98 (s, 2H, Ar-H), 8.35 (s, 1H, Ar-H), 8.52 (s, 3H, Ar-H), 8.69 (s, 1H, Ar-H), 9.31 (s, 1H, Ar-H), 9.44 (s, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 123.048, 128.254, 128.601, 129.450, 130.220, 130.754, 131.056, 132, 916, 140.540, 140.980, 141.323, 141.876, 154.046; MS: *m/z* = 384 (M^+).

Pyrido[2,3-*b*]dibenzo[5,6-7,8]quinoxaline **(3e)**: Brown solid m.p. 221 -223°C, $C_{19}H_{11}N_3$, Elem. Anal. Calc.: C, 81.12; H, 3.94; N, 14.94, Found: C, 80.88; H, 3.99; N, 15.11; FT-IR (KBr): 1600 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 7.71 (bs, 5H, Ar-H), 8.39 (s, 2H, Ar-H), 8.60 (s, 1H, Ar-H), 9.10 (s, 1H, Ar-H), 9.29 (s, 1H, Ar-H), 9.39 (s, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): 122.773, 122.876, 124.759, 126.370, 127.335, 127.946, 128.105, 129.180, 129.385, 130.893, 131.282, 132.115, 132.407, 137.123, 138.966, 143.651, 145.015, 149.003, 153.807; MS: m/z = 281 (M^+).

Pyrido[2,3-b]acenaphto[1,2-b]pyrazine **(4c)**: Yellow solid m.p. 229-231°C, $C_{17}H_9N_3$, Elem. Anal. Calc.: C, 79.99; H, 3.55; N, 16.46, Found: C, 79.84; H, 3.60; N, 16.26; FT-IR (KBr): 1614 cm^{-1} (stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS, ppm) δ: 7.76 (q, *J*=3.97, 1H, Ar-H), 7.88 (q, *J*=7.7,2H, Ar-H), 8.16 (q, *J*=4.4, 2H, Ar-H), 8.42 (d, *J*=6.9, 1H, Ar-H), 8.55 (d, *J*=718, 1H, Ar-H), 8.59 (d, 7.18, 1H, Ar-H), 9.17 (d, *J*=2.09, 1H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) δ: 122.345, 123.374, 124.320, 128.703, 128.942, 129.923, 130.031, 130.251, 130.818, 131.121, 136.395, 137.181, 138.457, 150.523, 152.288, 154.955, 157.092; MS: m/z = 255 (M^+).

3. Results and Discussion

Our aim was also to study the effect of catalyst loading and temperature on the condensation reactions for the synthesis of 2,3-diphenylquinoxaline. The green reaction of benzene-1,2-diamine with benzyl in the presence of the acidic ionic liquids (AILs), 1-hydrogen-3-methylimidazolium hydrogen sulfate ([Hmim]HSO_4, 1-hydrogen-3-methylimidazolium chloride ([Hmim]Cl, 2-pyrrolidonium hydrogensulfate ([Hnhp]HSO_4) as catalyst was selected as a model (Table 1). The reaction was carried out with different amounts of acidic ionic liquids as our catalysts (3, 5, 10 mol %) and a variety of temperatures (25, 50, 70°C) (results are summarized in Table 1).

As it is evident from Table 1, 10 mol% of acidic ionic liquids (A, B, C) is best at room temperature and can properly afford to the corresponding product in 3, 7, 5 mins with 98%, 89%, 94% of yield of acidic ionic liquids A, B and C respectively.

Table 1. Optimization conditions for preparation 2,3-diphenylquinoxaline in the presence of different amounts of ionic liquids, A:[Hmim]HSO_4, B: [Hmim]Cl, C: [Hnhp]HSO_4, as catalyst under various temperature.

Entry	Temperature (°C)	Catalyst A			Catalyst B			Catalyst C		
		Cat. (%)	Time (min)	Yield[a] (%)	Cat. (%)	Time (min)	Yield[a] (%)	Cat. (%)	Time (min)	Yield[a] (%)
1	25	3	10	62	3	15	51	3	11	60
2	25	5	7	75	5	12	56	5	8	66
3	25	10	3	98	10	7	89	10	5	94
4	50	5	5	84	5	8	70	5	5	72
5	50	10	3	95	10	5	83	10	3	87
6	70	10	3	97	10	5	85	10	3	92

[a] Isolated yield.

Table 2. Synthesis of quinoxalines derivatives from the reaction of 1,2-diamines and α-diketones in the presence of A: $[Hmim]HSO_4$ (10 mol%), B: [Hmim]Cl (10 mol%), C: $[Hnhp]HSO_4$ (10 mol%), as catalysts.

Entry	R	R_1	X	Catalyst A		Catalyst B		Catalyst C		(m.p.°C) Found /Reported
				Time (min)	Yield[a] (%)	Time (min)	Yield[a] (%)	Time (min)	Yield[a] (%)	
2a	H	H	CH	3	98	7	89	5	94	126-128/128-129[47]
2b	4-Me	H	CH	3	96	7	85	5	91	115-117/116-117[48]
2c	4-PhCO	H	CH	15	92	25	83	17	88	140-142/139-140[36]
2d	4-NO2	H	CH	27	90	42	82	30	85	184-186/185-187[48]
2e	4-Br	H	N	30	88	45	80	30	83	143 -145/----------
2f	H	H	N	32	90	50	46	30	85	134-137/141-142[49]
2g	H	4-MeO	CH	5	95	10	88	8	92	145-146/148-150[30]
2h	4-NO2	4-MeO	CH	30	92	45	83	35	86	188-189/----------
2i	H	4-MeO	N	7	94	12	85	10	91	145-147/148-150[30]
2j	4-PhCO	4-MeO	N	20	91	28	83	25	87	145-147/----------
2k	4-Me	4-MeO	CH	5	97	10	90	8	92	122-123/----------
3a	H	H	CH	3	95	7	92	5	94	225-227/223-225[48]
3b	4-Me	H	CH	3	97	10	90	5	90	205-207/208-210[48]
3c	4-PhCO	H	CH	18	90	35	86	20	89	245-247/----------
3d	4-Br	H	N	7	92	15	83	10	86	230-232/235-238[50]
3e	H	H	N	30	85	55	78	40	81	221-223/----------
4a	H	H	CH	10	93	15	85	10	90	236-237/238-240[48]
4b	4-Me	H	CH	8	95	10	87	6	93	228-229/>300[48]
4c	H	H	N	35	87	55	77	35	83	229-231/----------
2a	H	H	CH	3	98	7	89	5	94	126-128/128-129[47]

[a] Isolated yield.

It can easily be seen that the condensation reaction proceeded smoothly in water as solvent and gave reasonable good to excellent yields. However, the electron-donating substituents connected with *o*-phenylenediamine (such as OMe) reacted in shorter reaction times in comparison with electron-withdrawing groups (such as NO_2, PhCO). Therefore, the order of the reactivity for condensation reaction was found to be: H, CH_3 > PhCO > NO_2 (Table 2).

We also applied $[Hmim]HSO_4$, [Hmim]Cl, $[Hnhp]HSO_4$ in condensation reaction of benzene-1,2-diamine with benzyl under green conditions for preparation of quinoxaline derivatives (Scheme 1).

In a plausible mechanism (Scheme 3), at first, 1,2-diketone compounds are activated by the acidic ionic liquids to produce I. Then, 1,2-phenylendiamines attack the carbonyl group of the activated 1,2-diketones, and afford intermediate II. Next, by removing H_2O from II, orthoquinone methide (*o*-QM, III) is prepared. Acidic ionic liquids again activate intermediate III. Afterward, by removing H_2O, 2,3-diphenylquinoxaline forms.

The advantages of the present procedure are simplicity of operation, very short reaction time and high yield. For recycling of catalysts, after washing solid products with water several times, the water containing acidic ionic liquids (AILs is soluble in water) was evaporated under reduced pressure and acidic ionic liquids were recovered and reused.

Catalyst reusability is of major importance in heterogeneous catalysis. The recovery and reusability of the catalyst was studied using 1,2-phenylendiamines with α-diketones as model reaction. Since the catalyst can be separated from the reaction mixture after removing starting materials and water. The catalyst was consecutively reused five times without any noticeable loss of its catalytic activity (Table 3).

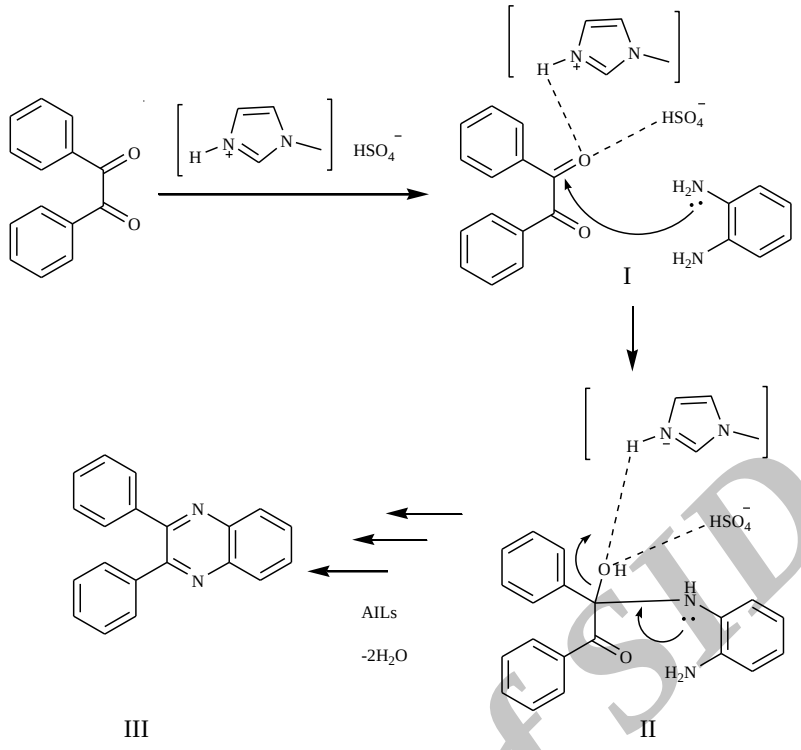
In order to evaluate the efficiency of our introduced method, more recently developed methods were compared with our present method on the basis of the yields and reaction times parameters, the results are given in Table 4.

4. Conclusion

In conclusion, we have developed a simple and green procedure for the synthesis of quinoxalines derivatives via the condensation of 1,2-diamines with α-diketones

Table 3. The catalyst reusability of $[Hmim]HSO_4$ in 5 cycles.

Run	1	2	3	4	5
Yield (%)	98	96	96	93	89



Scheme 3. The proposed mechanism for the synthesis of quinoxalines.

in water at ambient temperature. The merits of the present method are easy work-up, faster reactions, and a satisfactory yields without hazardous organic solvents and toxic catalysts. It can also be recovered and reused without noticeable loss of reactivity. In this reaction, the catalyst can be recyclable after removing starting materials and water.

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Table 4. Comparison of catalytic ability of catalysts.

Entry	Catalyst/solvent/temperature	Reaction time (min)	Yield %	Ref.
1	SBSSA[a]/ EtOH:H_2O/r. t.	5	96	29
2	$SnCl_2/SiO_2$/ CH_3OH/r.t.	4	100	33
3	Et_3N/toluene/90 °C	12 h	98	50
4	PTSA[b]/ NaPTS[c] /r.t.	7	96	51
5	[Hmim]HSO_4/H_2O/r. t.	3	98	This work
6	[Hmim]Cl/H_2O/r. t.	7	89	This work
7	[Hnhp]HSO_4/ H_2O/r. t.	5	94	This work

[a] silica bonded *S*-sulfonic acid.
[b] *p*-toluene sulphonic acid.
[c] sodium *p*-toluene sulphonate.

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