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ORIGINAL PAPER

An efficient one-pot three-component synthesis of imidazol-2-ones using [Bmim]BF_4 ionic liquid

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Abstract A mild and efficient method was developed for preparation of imidazol-2-ones via one-pot three-component condensation of 1,3-cyclic diketones, aryl glyoxals and urea using [Bmim]BF_4 ionic liquid. Excellent yields, short reaction time, simple work-up, and reusability of IL are advantages of this procedure.

Keywords One-pot · [Bmim]BF_4 · 1,3-Cyclic diketone · Aryl glyoxals · Imidazole-2-one

Introduction

The imidazole nucleus is present in large number of natural products. Compounds with an imidazole ring system have many pharmacological properties and play important roles in biochemical processes [1–6]. Many of the substituted imidazoles are known as of fungicides, herbicides, plant growth regulators and therapeutic agents [7, 8]. Imidazole-2-one derivatives exhibit antioxidant and cardiotonic properties [9–20]. Recent advances in green chemistry and organometallic chemistry have extended the boundary of imidazoles to the synthesis and application of a large class of imidazoles as ionic liquids as well as imidazole-related *N*-heterocyclic carbenes [21, 22]. Therefore, the interest is continuously growing on simplifying the synthesis of imidazole derivatives.

Literature survey reveals that the number of methods [23] was reported on Biginelli reaction of 1,3-cyclic diketones with aromatic aldehydes and urea or thiourea, but analogous three-component condensations with participation of aryl glyoxals were not found in the literature.

One-pot multi-component condensation represents a possible instrument for ideal synthesis because by this way complex molecules could be built-up with maximum simplicity [24, 25]. They offer significant advantages over conventional linear step syntheses by the way of reducing time, saving money, energy and raw-materials, thus resulting in both economical and environmental benefits [26].

Recently, ionic liquids have emerged as very potential green alternatives to the volatile and hazardous organic solvents and have been used as efficient reaction media for organic synthesis due to their special properties such as good solvating capability, wide liquid range, negligible vapour pressure, easy recycling, and high thermal stability and rate enhancing ability [27–38]. In continuation of our research work on the synthesis of heterocyclic compounds using ionic liquids [39, 40], herein, we have reported an efficient one-pot three-component synthesis of novel imidazole-2-ones using 1,3-cyclic diketones, aryl glyoxals and urea in the presence of ionic liquid [Bmim]BF_4 at RT in excellent yields with short reaction times (Scheme 1).

Results and discussion

In order to find the optimum reaction conditions, a model reaction of dimedone (1 mmol), 4-bromoglyoxal (1 mmol), and urea (1 mmol) was performed in various solvents and

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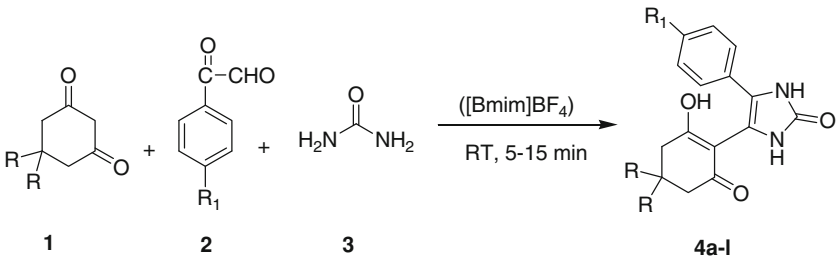
Scheme 1 Synthesis of imidazole-2-ones using [Bmim]BF4 ionic liquid



ionic liquids at different temperatures. The efficiency and the yield of the reaction in $[Bmim]BF_4$ at RT were higher than the reaction conducted in other solvents and ionic liquids. The results are summarized in Table 1.

Based on the optimum reaction conditions, one-pot three-component condensation of various glyoxals, 1,3-cyclic diketones and urea was then explored. As can be seen from Table 2, the reactions were carried out efficiently and the desired products were obtained in excellent yield (92–96 %) and very short reaction time (5–15 min).

Table 1 Solvent optimization for the synthesis of **4c**

Entry	Solvent	Temperature (°C)	Time (min)	Yield (%)[a]
1	CH_3CN	Reflux	480	Trace
2	EtOH	Reflux	420	32
3	*n*-Propanol	Reflux	450	25
4	EtOH + AcOH	Reflux	360	62
5	[Bmim]Br	80	120	72
6	[Bmim]Cl	90	100	65
7	$[Bmim]PF_6$	100	150	60
8	$[Bmim]BF_4$	RT	5	95, 93, 91, 90, 89[b]

[a] Yield of isolated product

[b] $[Bmim]BF_4$ was reused five times

Table 2 Synthesis of imidazol-2-one derivatives **4a–l**

Entry	Compound	R	R_1	Time (min)	Yield (%)[a]
1	**4a**	Me	H	5	96
2	**4b**	Me	Cl	6	93
3	**4c**	Me	Br	5	95
4	**4d**	Me	Me	8	94
5	**4e**	Me	OMe	10	92
6	**4f**	Me	NO_2	15	93
7	**4g**	H	H	7	95
8	**4h**	H	Cl	9	93
9	**4i**	H	Br	12	94
10	**4j**	H	Me	14	92
11	**4k**	H	OMe	10	93
12	**4l**	H	NO_2	15	92

[a] Isolated yield

The IL could be reused up to five runs without evident loss in activity (Table 1, entry 8). The results are summarized in Table 2. All the synthesized compounds were characterized by elemental analysis, IR, ^{1}H NMR, mass and ^{13}C NMR techniques.

IR spectra of compound **4c** exhibited absorptions at 3,423 cm^{-1} for OH, 3,250 cm^{-1} for NH, 3,059 cm^{-1} for aromatic C–H stretching, 1,695 cm^{-1} for C=O, and 1,665 cm^{-1} for C=O, amide. The ^{1}H NMR of compound **4c** showed singlet at δ 11.05, 10.30 and 9.79 ppm for OH, and two NH protons, respectively. Four aromatic protons were observed as doublets at δ 7.42 and 7.19 ppm, it also showed multiplet at δ 2.21–2.43 ppm for 2 × CH_2 and singlet at δ 1.05 ppm for 2 × CH_3 of dimedone. The ^{13}C NMR spectrum is in good agreement with the structure assigned. The carbonyl carbon was observed at δ 193.4 ppm and the signal at δ 185.3 ppm is assigned to carbon attached with OH group. The signal at δ 154.1 ppm is due to C=O of amide. All aromatic carbons were observed at around δ 124.2–131.0 ppm. The structure was further confirmed by its mass spectral studies. It gave molecular ion peak at *m/z* 378 (M + 1) corresponding to molecular formula $C_{17}H_{17}BrN_2O_3$. The elemental analysis values are in good agreement with theoretical data. Similarly, all these compounds were characterized on the basis of spectral studies.

Conclusion

In conclusion, we have demonstrated an efficient method for the synthesis of substituted imidazol-2-ones by one-pot three-component condensation of 1,3-cyclic diketones, aryl glyoxals and urea using ($[Bmim]BF_4$) ionic liquid. The reactions are very fast (5–15 min) and high yielding; IL was reused for five times.

Experimental

Melting points were recorded in open capillary and were uncorrected. IR spectra (ν_{max}) were recorded on a Bruker WM-4(X) spectrometer (577 model) using KBr disks. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on Bruker AC-300 spectrometer in DMSO-d_6

with TMS as an internal standard. Mass spectra (EI-MS) were determined on Perkin Elmer (SCIEX API-2000, ESI) at 12.5 eV. CHN analysis was done by Carlo Erba EA 1108 automatic elemental analyzer. All the solvents and chemicals used were pure, purchased from commercial sources and were used without further purification unless otherwise stated.

Typical procedure

A mixture of cyclic 1,3-diketone (1 mmol), aryl glyoxal (1 mmol) and urea (1 mmol) in $[Bmim]BF_4$ ionic liquid (5 mL) was stirred at RT for 5–15 min. The solid separated was filtered and washed with water to leave the crude product which was recrystallized from ethanol to afford colorless solid in excellent yield (92–96 %). The IL was recovered by evaporating the solvent under vacuum and it was reused for five runs without evident loss of activity. This procedure was followed for the synthesis of all the compounds **4a**–**l**.

5-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-4-phenyl-1*H*-imidazol-2)-one (**4a**)

M.p.: 235–237 °C; IR (KBr, cm^{-1}) $v = 3{,}391$, 3,260, 3,058, 1,705, 1,666; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.05 (s, 6H, 2 × CH_3), 2.26–2.42 (m, 4H, 2 × CH_2), 7.12–7.33 (m, 5H, ArH), 9.79 (s, 1H, NH), 10.27 (s, 1H, NH), 11.01 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.2, 185.3, 154.1, 136.4, 130.2, 126.6, 122.1, 117.0, 111.0, 105.4, 51.6, 47.2, 31.1, 29.1, 27.9 ppm; MS: $m/z = 299$ ($M^+ + 1$); Anal. Calcd for $C_{17}H_{18}N_2O_3$: C, 68.44; H, 6.08; N, 9.39. Found: C, 68.50; H, 6.01; N, 9.45.

4-(4-Chlorophenyl)-5-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-1*H*-imidazol-2-one (**4b**)

M.p.: 267–269 °C; IR (KBr, cm^{-1}) $v = 3{,}420$, 3,155, 3,057, 1,694, 1,662; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.06 (s, 6H, 2 × CH_3), 2.20–2.42 (m, 4H, 2 × CH_2), 7.16 (d, $J = 8.4$ Hz, 2H, ArH), 7.40 (d, $J = 8.4$ Hz, 2H, ArH), 9.79 (s, 1H, NH), 10.28 (s, 1H, NH), 11.02 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.2, 185.3, 154.0, 131.0, 130.1, 126.0, 124.2, 117.2, 111.3, 105.3, 51.6, 47.1, 31.1, 29.3, 27.7 ppm; MS: $m/z = 333$ ($M^+ + 1$); Anal. Calcd for $C_{17}H_{17}ClN_2O_3$: C, 61.36; H, 5.15; N, 8.42. Found: C, 61.42; H, 5.11; N, 8.46.

4-(4-Bromophenyl)-5-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-1*H*-imidazol-2-one (**4c**)

M.p.: 276–278 °C; IR (KBr, cm^{-1}) $v = 3{,}423$, 3,250, 3,059, 1,695, 1,665; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.05 (s, 6H, 2 × CH_3), 2.21–2.43 (m, 4H, 2 × CH_2), 7.19 (d, $J = 8.4$ Hz, 2H, ArH), 7.42 (d, $J = 8.4$ Hz, 2H, ArH), 9.79 (s, 1H, NH), 10.30 (s, 1H, NH), 11.05 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.4, 185.3, 154.1, 131.0, 130.3, 126.1, 124.2, 117.7, 111.7, 105.5, 51.8, 47.2, 31.4, 29.6, 27.9 ppm; MS: $m/z = 378$ ($M^+ + 1$); Anal. Calcd for $C_{17}H_{17}BrN_2O_3$: C, 54.13; H, 4.54; N, 7.43. Found: C, 54.07; H, 4.57; N, 7.47 %.

5-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-4-(4-methylphenyl)-1*H*-imidazol-2-one (**4d**)

M.p.: 223–225 °C; IR (KBr, cm^{-1}) $v = 3{,}398$, 3,253, 3,054, 1,702, 1,664; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.02 (s, 6H, 2 × CH_3), 2.20 (s, 3H, CH_3), 2.28–2.40 (m, 4H, 2 × CH_2), 7.12 (d, $J = 8.0$ Hz, 2H, ArH), 7.37 (d, $J = 8.0$ Hz, 2H, ArH), 9.69 (s, 1H, NH), 10.25 (s, 1H, NH), 10.93 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 192.9, 185.0, 154.0, 136.7, 133.2, 127.3, 122.2, 117.3, 111.1, 105.4, 51.4, 47.0, 30.9, 29.2, 24.6, 27.5 ppm; MS: $m/z = 313$ ($M^+ + 1$); Anal. Calcd for $C_{18}H_{20}N_2O_3$: C, 69.21; H, 6.45; N, 8.97. Found: C, 69.27; H, 6.36; N, 8.92.

5-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-4-(4-methoxyphenyl)-1*H*-imidazol-2-one (**4e**)

M.p.: 252–254 °C; IR (KBr, cm^{-1}) $v = 3{,}411$, 3,254, 3,056, 1,710, 1,666; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.06 (s, 6H, 2 × CH_3), 2.26–2.43 (m, 4H, 2 × CH_2), 3.82 (s, 3H, OCH_3), 7.08 (d, $J = 8.2$ Hz, 2H, ArH), 7.32 (d, $J = 8.2$ Hz, 2H, ArH), 9.77 (s, 1H, NH), 10.28 (s, 1H, NH), 10.95 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.0, 185.2, 154.2, 142.9, 134.4, 122.7, 120.2, 117.6, 111.5, 105.3, 56.3, 51.5, 47.1, 31.2, 29.5, 27.7 ppm; MS: $m/z = 329$ ($M^+ + 1$); Anal. Calcd for $C_{18}H_{20}N_2O_4$: C, 65.84; H, 6.14; N, 8.53. Found: C, 65.76; H, 6.21; N, 8.49.

5-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-4-(4-nitrophenyl)-1*H*-imidazol-2-one (**4f**)

M.p.: 292–294 °C; IR (KBr, cm^{-1}) $v = 3{,}407$, 3,257, 3,056, 1,698, 1,662; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.04 (s, 6H, 2 × CH_3), 2.29–2.43 (m, 4H, 2 × CH_2), 7.52 (d, $J = 8.7$ Hz, 2H, ArH), 8.35 (d, $J = 8.7$ Hz, 2H, ArH), 9.75 (s, 1H, NH), 10.24 (s, 1H, NH), 10.97 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.1, 185.2, 154.0, 143.2, 138.1, 126.5, 124.2, 117.4, 111.5, 105.2, 51.5, 47.1, 31.2, 29.6, 27.6 ppm; MS: $m/z = 344$ ($M^+ + 1$); Anal. Calcd for $C_{17}H_{17}N_3O_5$: C, 59.47; H, 4.99; N, 12.24. Found: C, 59.54; H, 5.04; N, 12.14.

5-(2-Hydroxy-6-oxocyclohex-1-enyl)-4-phenyl-1*H*-imidazol-2-one (**4g**)

M.p.: 235–237 °C; IR (KBr, cm^{-1}) $v = 3{,}361$, 3,245, 3,068, 1,701, 1,666; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.91–1.95 (m, 2H, CH_2), 2.26–2.31 (m, 2H, CH_2), 2.61–2.64 (m, 2H, CH_2), 7.20–7.53 (m, 5H, ArH), 9.75 (s, 1H, NH), 10.26 (s, 1H, NH), 10.97 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.0, 185.6, 154.5, 136.7, 130.2, 126.8, 123.1, 117.9, 111.0, 105.7, 36.6, 27.3, 20.4 ppm; MS: $m/z = 271$ ($M^+ + 1$); Anal. Calcd for $C_{15}H_{14}N_2O_3$: C, 66.66; H, 5.22; N, 10.36. Found: C, 67.60; H, 5.19; N, 10.29.

4-(4-Chlorophenyl)-5-(2-hydroxy-6-oxocyclohex-1-enyl)-1*H*-imidazol-2-one (**4h**)

M.p.: 267–269 °C; IR (KBr, cm^{-1}) $v = 3{,}378$, 3,245, 3,057, 1,699, 1,662; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.90–1.93 (m, 2H, CH_2), 2.22–2.30 (m, 2H, CH_2), 2.60–2.63 (m, 2H, CH_2), 7.16 (d, $J = 8.2$ Hz, 2H, ArH), 7.70 (d, $J = 8.2$ Hz, 2H, ArH), 9.80 (s, 1H, NH), 10.38 (s, 1H, NH), 10.95 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.5, 185.4, 154.6, 131.3, 130.7, 126.2, 124.5, 118.1, 109.5, 106.2, 36.1, 27.1, 20.2 ppm; MS: $m/z = 305$ ($M^+ + 1$); Anal. Calcd for $C_{15}H_{13}ClN_2O_3$: C, 59.12; H, 4.30; N, 9.19. Found: C, 59.21; H, 4.37; N, 9.06.

4-(4-Bromophenyl)-5-(2-hydroxy-6-oxocyclohex-1-enyl)-1H-imidazol-2-one (**4i**)

M.p.: 276–278 °C; IR (KBr, cm^{-1}) $v = 3{,}384$, 3,265, 3,056, 1,712, 1,674; ^{1}H NMR (δ, DMSO-d_6): δ 1.92–1.96 (m, 2H, CH_2), 2.25–2.32 (m, 2H, CH_2), 2.62–2.66 (m, 2H, CH_2), 7.14 (d, $J = 8.2$ Hz, 2H, ArH), 7.72 (d, $J = 8.2$ Hz, 2H, ArH), 9.83 (s, 1H, NH), 10.44 (s, 1H, NH), 11.12 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.8, 185.5, 154.4, 131.7, 130.5, 126.3, 124.7, 118.5, 109.9, 105.5, 36.7, 27.4, 20.5 ppm; MS: $m/z = 350$ ($M^+ + 1$); Anal. Calcd for $C_{15}H_{13}BrN_2O_3$: C, 51.60; H, 3.75; N, 8.02. Found: C, 51.72; H, 3.83; N, 7.95.

5-(2-Hydroxy-6-oxocyclohex-1-enyl)-4-(4-methylphenyl)-1*H*-imidazol-2-one (**4j**)

M.p.: 223–225 °C; IR (KBr, cm^{-1}) $v = 3{,}371$, 3,243, 3,061, 1,698, 1,664; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.87–1.91 (m, 2H, CH_2), 2.18–2.23 (m, 2H, CH_2), 2.59–2.62 (m, 2H, CH_2), 7.11 (d, $J = 7.9$ Hz, 2H, ArH), 7.67 (d, $J = 7.9$ Hz, 2H, ArH), 9.66 (s, 1H, NH), 10.22 (s, 1H, NH), 10.91 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.2, 185.1, 154.5, 136.9, 133.6, 127.8, 122.7, 117.6, 109.0, 106.3, 36.1, 27.4, 26.5, 19.7 ppm; MS: $m/z = 285$ ($M^+ + 1$); Anal. Calcd for $C_{16}H_{16}N_2O_3$: C, 67.59; H, 5.67; N, 9.85. Found: C, 67.53; H, 5.50; N, 9.93.

5-(2-Hydroxy-6-oxocyclohex-1-enyl)-4-(4-methoxyphenyl)-1*H*-imidazol-2-one (**4k**)

M.p.: 252–254 °C; IR (KBr, cm^{-1}) $v = 3{,}374$, 3,276, 3,063, 1,714, 1,666; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.94–1.96 (m, 2H, CH_2), 2.24–2.31 (m, 2H, CH_2), 2.63–2.67 (m, 2H, CH_2), 7.18 (d, $J = 8.1$ Hz, 2H, ArH), 7.81 (d, $J = 8.1$ Hz, 2H, ArH), 9.77 (s, 1H, NH), 10.31 (s, 1H, NH), 10.99 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.3, 185.5, 154.3, 143.4, 134.7, 122.5, 120.6, 118.4, 111.2, 105.6, 56.3, 37.1, 27.6, 20.8 ppm; MS: $m/z = 301$ ($M^+ + 1$); Anal. Calcd for $C_{16}H_{16}N_2O_4$: C, 63.99; H, 5.37; N, 9.33. Found: C, 63.86; H, 5.21; N, 9.44.

5-(2-Hydroxy-6-oxocyclohex-1-enyl)-4-(4-nitrophenyl)-1*H*-imidazol-2-one (**4l**)

M.p.: 292–294 °C; IR (KBr, cm^{-1}) $v = 3{,}380$, 3,267, 3,056, 1,697, 1,662; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.90–1.93 (m, 2H, CH_2), 2.21–2.33 (m, 2H, CH_2), 2.63–2.66 (m, 2H, CH_2), 7.17 (d, $J = 8.6$ Hz, 2H, ArH), 7.77 (d, $J = 8.6$ Hz, 2H, ArH), 9.77 (s, 1H, NH), 10.28 (s, 1H, NH), 10.96 (s, 1H, OH) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz): δ 193.4, 185.4, 154.7, 143.6, 138.8, 127.2, 124.5, 118.2, 111.0, 106.1, 37.2, 27.4, 20.6 ppm; MS: $m/z = 316$ ($M^+ + 1$); Anal. Calcd for $C_{15}H_{13}N_3O_5$: C, 57.14; H, 4.16; N, 13.33. Found: C, 57.20; H, 4.21; N, 13.28.

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