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ORIGINAL ARTICLE

A simple and efficient synthesis of imidazolo[1,2-a]pyridines using MgO in aqueous medium

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Abstract Various imidazolo[1,2-a]pyridines were synthesized from amino pyridines and aromatic phenacyl bromides by one step process in the presence of MgO in aqueous medium at room temperature. The salient feature of this method includes mild conditions, short reaction time, high yields, easy purification and simple procedure.

1. Introduction

It has long been known that imidazolo[1,2-a]pyridine derivatives exhibit diverse biological activities like antiviral (Gudmundsson et al., 1997), antiulcer (Kaminsky et al., 1989), antiprotozoal (Ismail et al., 2008) and anti-inflammatory (Rupert et al., 2003). Drug formulation containing imidazolo[1,2-a]pyridines available in the market include alpidem (anxiolytic), zolpidem (hypnotic), and zolimidine (antiulcer). The majority of reported imidazolo[1,2-a]pyridine syntheses proceed via the condensation reaction of α-bromo carbonyl compounds with 2-amino pyridine derivatives under neutral (Sharma et al., 2007) and basic conditions (Gudmundsson and Johns, 2003). A proposed mechanism (Cai et al., 2004) for the reaction involves the nucleophilic substitution of bromide by pyridine-nitrogen of 2-amino pyridine derivatives. Imidazolo[1,2-a]pyridine derivatives were also synthesized on solid support (Kazzouli et al., 2003) and using catalysts such as Al_2O_3 (Ponnala et al., 2005) and $TiCl_4$ (Cai et al., 2006). Other methodologies include reaction of 2-amino pyridines with α-tosyl ketones (Xie et al., 2002), polymer supported [hydroxyl (sulphonyloxy) iodo] benzene with ketones or alcohol (Ueno and Togo, 2004), alkynyl (phenyl) iodinium salts (Liu et al., 2004), alpha diazoketones (Yadav et al., 2007) and using iodine catalyst (Chang et al., 2010), $ZnCl_2$ (Amanda et al., 2007), scandium triflate (Ireland et al., 2003; Mandair et al., 2002), ammonium chloride (Parchinsky et al., 2006), perchloric acid (Bienayme and Bouzid, 1998) and using montmorillonite clay k-10 (Varma and Kumar, 1999).

However the above mentioned synthetic routes have several drawbacks such as low yields, use of expensive reagents, long reaction time, tedious workup procedure and harsh reaction conditions.

Catalyst free and solvent free synthesis by grinding was also reported (Dong et al., 2009) but this method is limited to a small scale and commercially not useful. Therefore, the search continues for the synthesis of imidazolo[1,2-a]pyridine in terms

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of operational simplicity and economic viability. The use of environmentally benign solvents like water (Li and Chan, 1997; Lindstrom, 2002) represents very powerful green chemical technology procedures from both the economic and synthetic points of view. They not only reduce the burden of organic solvent disposal, but also enhance the rate of many organic reactions. Water is safe, nontoxic, environmentally friendly, and cheap and this inspired us to focus on heterocyclic synthesis in water. Therefore, efforts have been made to perform the synthesis of imidazolo[1,2-a]pyridine derivatives in aqueous medium. In pursuit of a simple and environmentally benign process we herein report a new method for the synthesis of imidazolo[1,2-a]pyridine derivatives using MgO, a cheap catalyst in aqueous medium. The method is simple and convenient for commercial synthesis.

2. Materials and methods

2.1. Experimental

Chemicals were purchased from Aldrich Chemical Co. Reactions were monitored and purity of the products was checked by thin layer chromatography (TLC). TLC was performed on Merck 60 F-254 silica gel plates with visualization by UV-light. The melting points were determined on a Buchi Melting Point B-545 apparatus. The IR spectra of the compounds were recorded on a Nicolet 6700 FT-IR spectrometry using KBr pellets. NMR spectra were recorded on Brucker either at 400 or 300 MHz (^{1}H NMR) and 75 MHz (^{13}C NMR) spectrophotometer instruments in $CDCl_3$ and DMSO-d_6. Chemical shifts were recorded in parts per million downfield from tetramethylsilane. Mass spectra were recorded on a MS-3200Q trap. Elemental analysis was performed on a HOSLI CH-analyzer. Column chromatography was performed on silica gel (230–400 mesh) supplied by Acme Chemical Co. The chemicals and solvents used were of LR grade and were purified as per literature methods.

2.2. General method for the preparation of imidazolo[1,2-a]pyridines (**2a**)

2-Amino pyridine (0.94 gm, 10 mmole), 4-chloro phenacyl bromide (1.89 gm, 10 mmole) and MgO powder (0.4 gm, 10 mmole) were taken in water (20 ml) in a round bottom flask and stirred at room temperature (2–3 h). The progress of the reaction was monitored on TLC (ethyl acetate: *n*-hexane, 3:7). After completion of the reaction, the reaction mixture was poured in ice water and extracted with ethyl acetate. The organic solvent was distilled and the product obtained was purified by column chromatography. Similar procedure was followed for synthesis of **2b–j**.

2.3. Spectral analysis

2.3.1. 2-(4-Chlorophenyl)H-imidazolo[1,2-a]pyridine (**2a**)

Yield: 92%; mp 206–208 °C (Shailesh and Devi, 2008); ^{1}H NMR (400 MHz, DMSO-d_6): 7.45(t, J = 6.5 Hz, 1H); 7.65(d, J = 8.5 Hz, 2H); 7.88(t, J = 6.7 Hz, 2H); 7.98(d, J = 8.4 Hz, 2H); 8.8(s, 1H); 8.86(d, J = 6.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 108.15, 112.60, 117.41, 124.97, 125.58, 127.18, 128.84, 132.14, 133.64, 144.46, 145.59; ms *m*/*z* (relative intensity, %): 228(M^+ 100), 230(M + 2, 32), 229(M + 1, 15); Anal. Calcd for $C_{13}H_9ClN_2$: C, 68.28; H, 3.97; N, 12.25. Found: C, 68.11; H, 3.81; N, 12.03; IR (KBr, cm^{-1}): 1614(C═N), 1550(C═C), 3320, 3021(Ar-H), 1100, 910, 1045, 750, 650.

2.3.2. 2-(4-Bromophenyl)H-imidazolo[1,2-a]pyridine (**2b**)

Yield: 89%; mp 214–216 °C (Shailesh and Devi, 2008); ^{1}H NMR (400 MHz, DMSO-d_6): 7.90(d, J = 8.8 Hz, 2H), 7.61(d, J = 8.8 Hz, 2H), 7.23(m, 1H), 6.88(m, 1H), 7.55(d, J = 9.2 Hz, 1H), 8.41(s, 1H), 8.5(d, J = 6.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 108.44, 111.50, 116.48, 125.11, 125.10, 126.76, 129.00, 134.67, 135.14, 145.45, 147.61; ms *m*/*z* (relative intensity, %): 271(M^+, 100), 273(M + 2, 98), 272(15); Anal. Calcd for $C_{13}H_9BrN_2$: C, 57.17; H, 3.32; N, 10.26. Found: C, 58.05; H, 3.81; N, 11.03; IR(KBr cm^{-1}): 1620 (C═N), 1555 (C═C), 3323, 3021(Ar-H), 1100, 910, 1660, 1065, 760, 550.

2.3.3. 2-(4-Nitrophenyl)H-imidazolo[1,2-a]pyridine (**2c**)

Yield: 90%; mp 203–207 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 7.98(d, J = 8.3 Hz, 2H), 7.72(d, J = 8.3 Hz, 2H), 7.3(m,1H), 6.9(m, 1H), 7.6(d, J = 8.6 Hz, 1H), 8.44(s, 1H), 8.52(d, J = 7.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 111.50, 112.55, 117.78, 127.41, 128.60, 125.56, 129.20, 136.57, 135.54, 146.55, 148.21; ms *m*/*z* (relative intensity, %): 239 (M^+, 100), 240(M + 1, 14); Anal. calcd for: $C_{13}H_9N_3O_2$: C, 65.27; H, 3.79; N, 17.56. Found: C, 66.01; H, 4.01; N, 17.81; IR(KBr, cm^{-1}): 1640(C═N), 1550(C═C), 3320, 3025 (Ar-H), 1530, 1340, 1100, 910, 1650, 1035, 650.

2.3.4. 2-(4-Methoxyphenyl)H-imidazolo[1,2-a]pyridine (**2d**)

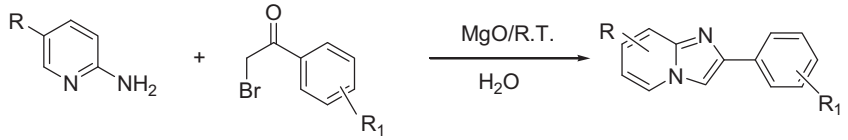
Yield: 89%; mp 132–134 °C (Shailesh and Devi, 2008); ^{1}H NMR (400 MHz, DMSO-d_6): 7.7(d, J = 8.5 Hz, 2H), 7.6(d, J = 8.5 Hz, 2H), 7.23(m, 1H), 7.5(d, J = 8.0 Hz, 1H), 8.6(d, J = 7.9 Hz, 1H), 8.7(s, 1H), 7.3(m, 1H), 7.71(m, 1H), 4.3(s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): 106.45, 111.16, 116.51, 123.07, 124.43, 126.54, 127.81, 130.22, 131.51, 142.04, 143.92, 55.39; ms *m*/*z* (relative intensity, %): 224(M^+, 100), 225(M + 1, 15), 272(M + 2, 15); Anal. Calcd for $C_{14}H_{12}N_2O$: C, 74.98; H, 5.39; N, 12.49. Found: C, 74.91; H, 4.01; N, 12.82; IR(KBr, cm^{-1}): 1665(C═N), 1550(C═C), 3320, 3020(Ar-H), 1100, 910, 1660, 1065, 760, 550.

2.3.5. 2-(3-Nitrophenyl)H-imidazolo[1,2-a]pyridine (**2e**)

Yield: 87%; mp 207–209 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 8.54(d, J = 6.8 Hz, 1H), 7.61(d, J = 9.2 Hz, 1H), 7.28(m, 1H), 6.9(m, 1H), 8.62(s, 1H), 8.76(m, 1H), 8.15(m, 1H), 7.29(m, 1H), 8.38(d, J = 7.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 109.04, 113.087, 117.68, 120.73, 122.49, 125.58, 125.82, 129.69, 131.80, 135.49, 143.23, 143.78, 148.68.

2.3.6. 6-Chloro-2-(4-chlorophenyl)H-imidazolo[1,2-a]pyridine (**2f**)

Yield: 88%; mp 205–207 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 8.18(s, 1H); 7.4(d, J = 8.4 Hz, 2H), 7.89(d, 8.4 Hz, 2H), 7.85(s, 1H), 7.6(d, J = 9 Hz, 1H), 7.18(d, J = 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 108.52, 117.67, 121.06, 123.43, 126.71, 127.32, 129.032, 131.34, 134.24, 143.81, 145.28; ms *m*/*z* (relative intensity, %): 262(M^+, 100), 264(M + 2, 33.9),



2a-j

R= H, Cl . R_1= 4-Cl, 4-Br, 4-NO_2, 3-NO_2, 4-OMe.

Scheme 1 Synthetic route of **2a–j**.

263(17); Anal. Calcd for $C_{13}H_8Cl_2N_2$: C, 59.34; H, 3.06; N, 10.65. Found: C, 60.21; H, 3.66; N, 11.05; IR(KBr, cm^{-1}): 1670(C=N), 1560(C=C), 3325, 3022(Ar-H), 970, 1120, 850, 725.

*2.3.7. 2-(4-Bromophenyl)-6-chloro H-imidazolo[1,2-a]pyridine (**2g**)*

Yield: 85%; mp 200–202 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 7.39(d, J = 8.3 Hz, 2H), 7.84(d, J = 8.3 Hz, 2H), 8.17(s, 1H), 7.82(s, 1H), 7.6(d, J = 8.5 Hz, 1H), 7.2 (d, J = 8.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 108.65, 116.67, 120.80, 122.23, 124.91, 126.72, 128.24, 130.34, 134.74, 143.11, 145.18; ms m/z (relative intensity, %): 305.95(M^+,100), 307.95(M + 2, 98); Anal. Calcd for $C_{13}H_8ClBrN_2$: C, 50.76; H, 2.62; N, 9.11. Found: C, 50.18; H, 2.56; N, 9.76.

*2.3.8. 6-Chloro-2-(4-nitrophenyl) H-imidazolo[1,2-a]pyridine (**2h**)*

Yield: 89%; mp 255–258 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 8.1(d, J = 8.1 Hz, 2H), 7.5(d, J = 8.1 Hz, 2H), 8.00(s, 1H), 7.91(s, 1H), 7.63(d, J = 8.4 Hz, 1H), 7.2 (d, J = 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 107.02, 114.47, 123.70, 125.55, 126.65, 128.21, 130.44, 132.76, 135.87, 145.79, 146.43; ms m/z (relative intensity, %): 273(M^+, 100%), 275(M + 2, 32), 274(18); Anal. Calcd for $C_{13}H_8ClN_3O_2$: C, 57.05; H, 2.95; N, 15.35. Found: C, 58.01; H, 3.01; N, 15.83; IR(KBr, cm^{-1}): 1665(C=N), 1550(C=C), 3321, 3021(Ar-H), 970, 1110, 850, 720, 1535, 1330.

*2.3.9. 6-Chloro-2-(4-methoxyphenyl) H-imidazolo[1,2-a]pyridine (**2i**)*

Yield: 84%; mp 202–204 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 7.8(d, J = 8.3 Hz, 2H), 7.3(d, J = 8.3 Hz, 2H), 8.00(s, 1H); 7.91(s, 1H), 7.5(d, J = 8.4 Hz, 1H), 7.1(d, J = 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 106.54, 118.57, 119.43, 122.56, 124.17, 126.23, 127.21, 128.87, 133.43, 142.16, 144.30, 56.03; ms m/z (relative intensity, %): 258(M^+, 100), 260(M + 2, 33), 259(M + 1, 18); Anal. Calcd for $C_{14}H_{11}ClN_2O$: C, 65; H, 4.29; N, 10.83. Found: C, 65.41; H, 4.21; N, 10.20; IR(KBr, cm^{-1}): 1660(C=N), 1550(C=C), 3323, 3021(Ar-H), 960, 1120, 850, 720.

*2.3.10. 6-Chloro-2-(3-nitrophenyl) H-imidazolo[1,2-a]pyridine (**2j**)*

Yield: 88%; mp 194–198 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 7.34(m, 1H), 7.67(d, J = 9.6 Hz, 1H), 8.83(d, J = 0.8 Hz, 1H), 8.59(s, 1H), 8.75–8.76(m, 1H), 8.37–8.76(m, 1H), 7.71–7.75 (m, 1H), 8.15–8.18(m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 109.32, 117.94, 120.730, 121.280, 122.76, 123.57, 127.39, 129.76, 131.76, 134.94, 144.10, 148.64; ms m/z (relative intensity, %): 273(M^+, 100), 275(M + 2, 33), 274(M + 1, 18).

Table 1 Effect of various concentrations of MgO catalyst.

Entry	MgO concn (equiv)	Time (h)	Yield (%)
1	0.0	12	No reaction
2	0.2	10	50
3	0.4	8	60
4	0.6	6	63
5	0.8	5	65
6	1.0	2.1	92
7	1.2	2.5–4	92

Table 2 Effect of catalysts (1 equiv) on the reaction of 2-aminopyridine with *p*-chlorophenacyl bromide in water at room temperature.

Entry	Catalyst	Time (h)	Yield (%)
1	MgO	2.1	92
2	ZnO	3.4	60
3	CaO	4.1	58
4	K_2CO_3	4.3	56
5	Triethyl amine	5.1	55

Table 3 Effect of solvent for synthesis of 2-(4-chlorophenyl)*H*-imidazolo[1,2-a]pyridine.

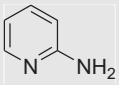
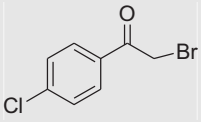
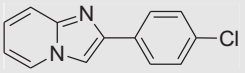
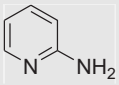
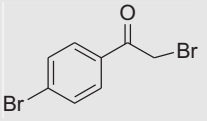
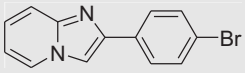
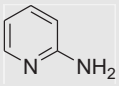
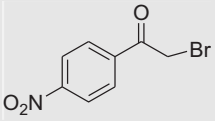
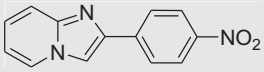
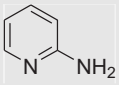
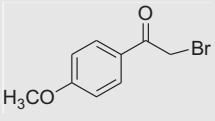
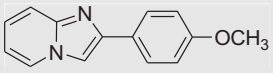
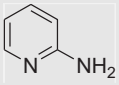
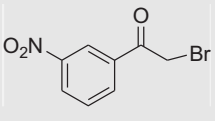
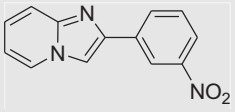
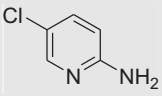
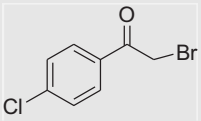
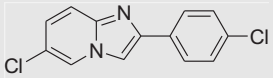
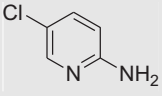
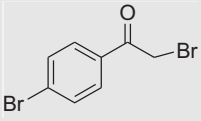
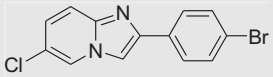
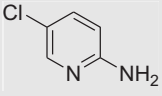
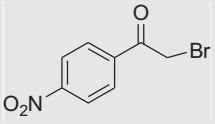
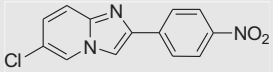
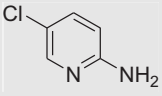
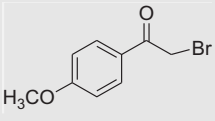
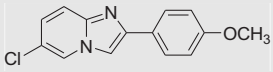
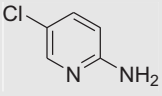
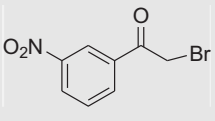
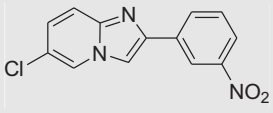
Entry	Solvent	Time (h)	Yield (%)
1	H_2O	2.1	92
2	DMF	4.5	60
3	EtOH	6	50
4	CH_3CN	9	40
5	$CHCl_3$	12	35
6	CH_2Cl_2	12	25
7	Hexane	24	20

3. Results and discussion

Homogeneous catalytic reactions suffer the disadvantage of separation and regeneration of catalyst. The use of heterogeneous catalyst is therefore desirable as the catalyst can be separated at the end of the reaction and can be reused. This makes the use of heterogeneous catalyst environmental friendly (Hasan and Ayoob, 2009).

Xu and co-workers investigated the reaction of high surface area form of MgO as a catalyst for a number of reactions (Bartley et al., 2005). MgO is environmentally safe, non

Table 4 Synthesis of imidazolo[1,2-a]pyridine derivatives.

Entry	Amino pyridine	α-Haloketones	Product	Time (h)	Yield (%)
2a				2.1	92
2b				2.3	89
2c				2.2	90
2d				2.4	89
2e				2.3	87
2f				2.4	88
2g				2.5	85
2h				2.3	89
2i				2.4	84
2j				2.2	88

volatile, non hygroscopic, odorless with outstanding physical properties and stability. It is commercially available and is a very cheap chemical. Recently it was shown that MgO has the prospect to be used as a substitute for conventional basic catalytic material. It has been used as an efficient heterogeneous catalyst in many important organic reactions (Kabaudin and Karimi, 2006).

We sought to develop a route that is simple, efficient and commercially useful. We focused on aqueous medium where the product would precipitate out from reaction mixture after completion of reaction.

During our present work on reaction in aqueous medium, we investigated the synthesis of imidazopyridine by reaction of phenacyl bromide, amino pyridine and MgO in water as a

solvent at room temperature. To select the favorable reaction condition, we first examined the model reaction of 4-chloro phenacyl bromide, 2-amino pyridine and MgO in water at room temperature (Scheme 1). It was observed that the reaction without MgO failed (Table 1, entry 1) whereas using MgO as a catalyst, the reaction proceeded with excellent yield in less time. To optimize the amount of MgO required for the reaction, various amounts of MgO were tried. The yield of product (**2a**) obtained using 1 equiv MgO was 92% which indicate that 1 equiv MgO is suitable for the reaction (Table 1). The reaction progress was monitored by thin layer chromatography (*n*-hexane: ethyl acetate 7:3). To examine the catalytic activity of MgO, we carried out the reaction using different catalysts like MgO, ZnO, CaO, K_2CO_3, triethyl amine etc. (Table 2). It was found that MgO was the most suitable catalyst for synthesis of imidazolo[1,2-a]pyridine derivatives.

To compare the results obtained using water and organic solvents, the reaction was carried out using different organic solvents (Table 3). Compared to the reaction using the organic solvents, the reaction using water as a solvent was much faster, requiring only 2–3 h for completion. Both yield and purity of the product was greater in the reaction using water than the organic solvents. Encouraged by these results, we screened a few reactions of commercially available α-haloketones with 2-amino pyridine as well as 5-chloro 2-amino pyridine. The results are summarized in Table 4. All products were confirmed by melting point assay, NMR spectroscopy, mass spectrometry and elemental analysis.

4. Conclusion

In summary the present procedure, using MgO as catalyst in water provides a convenient and synthetically useful method for the preparation of various imidazolo[1,2-a]pyridines. The major advantage of this method over existing methods includes good yield and cheap catalyst with water as solvent which is ecofriendly. This makes the process attractive and commercially viable.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.arabjc.2012.04.017.

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