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Facile Synthesis of Imidazo[1,2-*a*]pyridines *via* LED Light Induced Reaction between 2-Aminopyridines and Acetophenones

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Imidazo[1,2-*a*]pyridines are class of privileged motifs present in various important drugs. The majority of the processes involve metal catalysts, despite the fact that there are substantial benefits to develop new methodologies for the synthesis of imidazo[1,2-*a*]pyridines. In this work, an eco-friendly method is reported for the synthesis of imidazo[1,2-*a*]pyridines in the presence of LED light source under ambient conditions. The reaction proceeds smoothly with both electron donating and electron withdrawing groups to afford the products in excellent yields.

Keywords: LED light, Visible light, Imidazo[1,2-*a*]pyridines, Pyridine.

INTRODUCTION

Imidazo[1,2-*a*]pyridines are an significant class of nitrogen heterocycles and are found in various biologically active molecules [1]. Imidazo[1,2-*a*]pyridine exhibiting diverse biological activities such as antiviral [2,3], antibacterial [4], antifungal [5,6], K^+-stimulated ATPase inhibition [7], bradykinin B_2 receptor antagonists [8], antirhinoviral [9,10] and antiulcer [11]. They are also core skeletons in various commercially available drugs (Fig. 1) such as zolpidem, alpidem, zolimidine, necopidem and saripidem [12,13]. Due to the biological importance of imidazo[1,2-*a*]pyridine derivatives, several methods have been developed for the synthesis of these molecules. The most important precursors are (i) 2-aminopyridines, aldehydes and isonitriles [14,15], (ii) 2-aminopyridines, aldehydes and alkynes [16,17], (iii) Morita-Baylis-Hillman acetates of nitroalkenes [18], (iv) 2-aminopyridines and nitro-olefins [19], (v) 2-aminopyridine and alkynes [20-25]; (vi) 2-aminopyridine and acetophenone [23], *etc*.

However, some other approaches are also significant, leading to a variety of elegant strategies for synthesizing imidazo [1,2-*a*]pyridines using catalysts such as polymer supported bromine [24], copper(II) bromide [25], dioxane dibromide [26], I_2 [27], Ag_2CO_3 [28], copper(I) iodide/boron trifluoride etherate [29], iodine-ammonium acetate [30], iron(III) [31], CuI [32], *etc*. However, most of these methods are associated with various demerits such as expensive catalysts, metal catalysts, high temperature and low yields. Therefore, the development of new, efficient and eco-friendly protocol for the synthesis of imidazo-[1,2-*a*]pyridine derivatives has become a current research domain.

Recently, the use of LED bulb (visible light) instead of catalyst has become economic as well as environmental friendly [33,34]. It should be emphasized that light emitting diode (LED) lights never generate heat and has found to be a useful source for the synthesis of amino alcohols [35], 1,2,4-triazolines, 1,2,4-triazoles [36], 1,3,4-thiadiazole [37] and oxidation of alcohols [38]. To date, no reports has described the synthesis of imidazo-[1,2-*a*]pyridine derivatives under LED light irradiation. Thus, herein a catalyst free route to synthesize imidazo[1,2-*a*]-pyridine derivatives *via* LED light assisted one pot reaction of 2-aminopyridines and acetophenones at ambient conditions is described.

EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. ^1H and ^{13}C NMR (300 MHz and 75 MHz, respectively) in DMSO-d_6 solvent were analyzed on Bruker NMR spectrometer using TMS as an internal standard. All the compounds were recorded for HRMS on waters model: Synapt G2.

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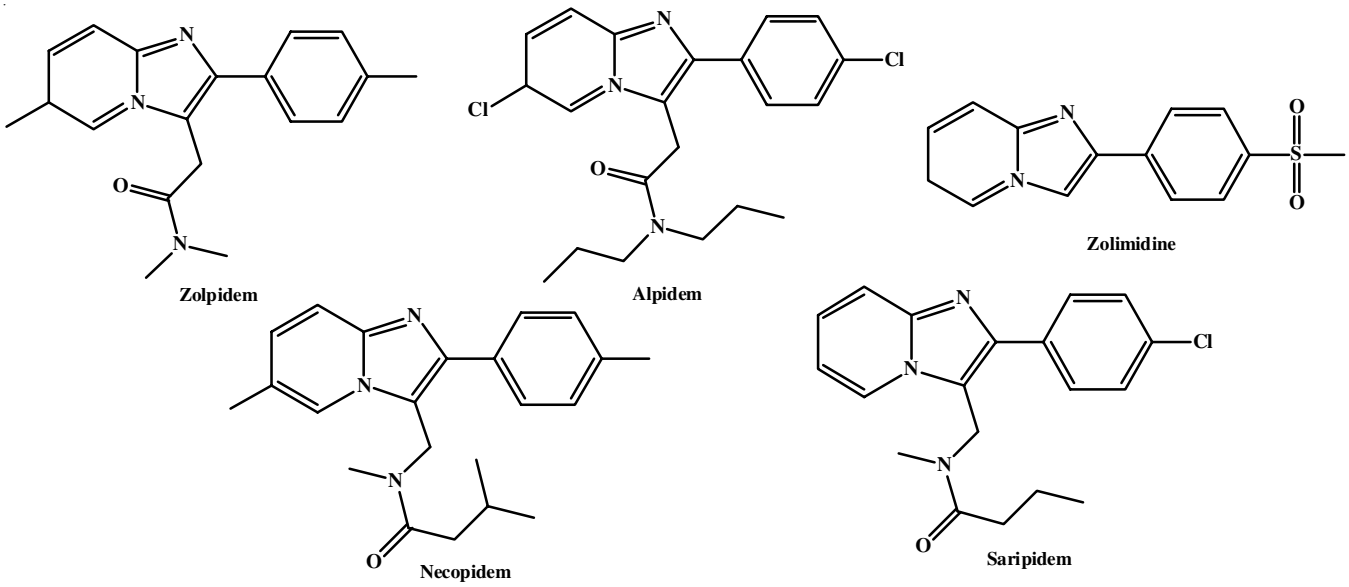
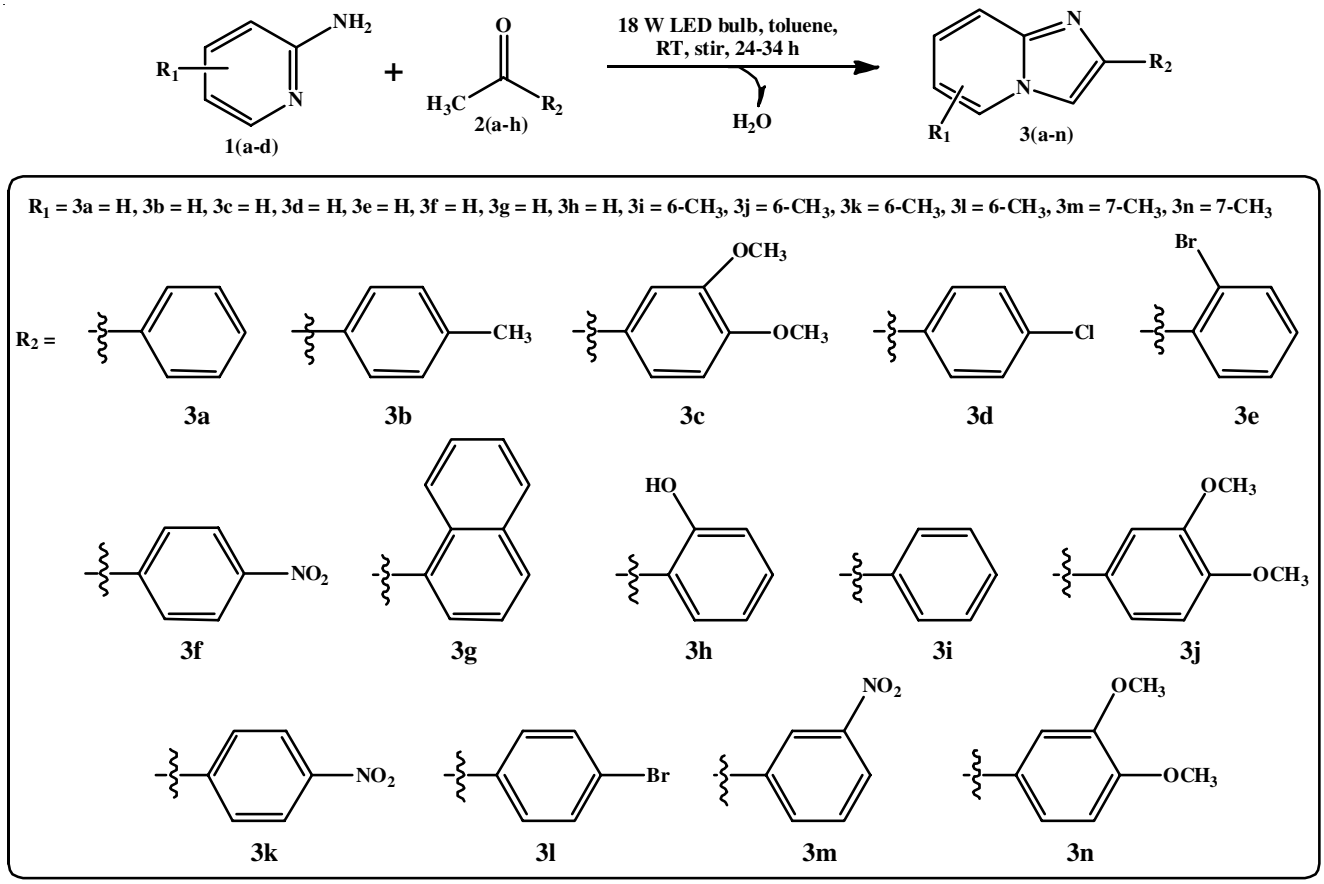


Fig. 1. Selected examples of bioactive imidazo[1,2-*a*]pyridines

General procedure for the LED light induced synthesis of imidazo[1,2-*a*]pyridines (3a-n): A mixture of 2-amino pyridines (**1a-d**), (1 mmol) and acetophenones (**2a-h**), (1 mmol) was stirred in toluene (10 mL) at room temperature under 18 W white LED light irradiation. The progress and completion of the reaction were monitored by TLC (eluent; EtOAc and *n*-hexane). Finally, the solvent was removed under vacuum to obtain the crude product which was purified by silica gel column chromatography to afford imidazo[1,2-*a*]pyridines (**3a-n**) (**Scheme-I**).

2-Phenylimidazo[1,2-*a*]pyridine (3a): White solid; 183.38 mg (yield: 94%); m.p.: 134-136 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.78 (t, J = 6.6 Hz, 1H), 7.17 (t, J = 7.9 Hz, 1H), 7.35-7.30 (m, 1H), 7.44 (t, J = 7.5 Hz, 2H), 7.65 (d, J = 9.1



Scheme-I: Synthetic route of imidazo[1,2-*a*]pyridines (**3a-n**) *via* LED light induced reaction

Hz, 1H), 7.96 (d, *J* = 7.6 Hz, 2H), 7.85 (s, 1H), 8.11 (d, *J* = 6.6 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 108.1, 112.5, 117.5, 124.7, 125.5, 126.1, 127.9, 128.6, 133.6, 137.9, 145.7. HRMS for $C_{13}H_{10}N_2$: *m/z* = 195.0955.

2-*p*-Tolylimidazo[1,2-*a*]pyridine (3b): Colourless solid; 190.30 mg (yield: 91%); m.p.: 144-146 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.37 (s, 3H), 6.69 (td, *J* = 6.8, 1.0 Hz, 1H), 7.14-7.06 (m, 1H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.59 (dd, *J* = 9.1, 0.6 Hz, 1H), 7.59 (dd, *J* = 9.1, 0.6 Hz, 1H), 7.75 (s, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 8.01 (dt, *J* = 6.8, 1.1 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 21.3, 107.7, 112.2, 117.3, 124.4, 125.5, 125.9, 129.4, 130.9, 137.7, 145.6, 145.8. ppm. HRMS for $C_{14}H_{12}N_2$: *m/z* = 209.1305.

2-(3,4-Dimethoxyphenyl)imidazo[1,2-*a*]pyridine (3c): Yellow solid; 207.09 mg (yield: 92%:); m.p.: 104-106 ºC; ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 3.91 (s, 3H), 3.99 (s, 3H), 6.72 (td, *J* = 6.8, 0.9 Hz, 1H), 6.93-6.88 (m, 1H), 7.17-7.09 (m, 1H), 7.43 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.57 (d, *J* = 1.9 Hz, 1H), 7.61 (d, *J* = 9.1 Hz, 1H), 7.76 (s, 1H), 8.06 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 55.9, 56.0, 107.4, 109.2, 111.3, 112.2, 117.2, 118.4, 124.4, 125.4, 126.9, 145.5, 145.7, 149.0, 149.2 ppm. HRMS for $C_{14}H_{12}N_2O$: *m/z* = 225.0999.

2-(4-Chlorophenyl)imidazo[1,2-*a*]pyridine (3d): Colourless solid; 205.24 mg (yield: 90%); m.p.: 201-203 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.79 (t, *J* = 6.8 Hz, 1H); 7.26-7.15 (m, 4H), 7.42-7.35 (m, 2H), 7.58 (d, *J* = 9.1 Hz, 1H), 8.09 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 108.4, 112.8, 116.8, 125.4, 125.7, 127.1, 128.8, 131.8, 133.7, 144.2, 145.5. HRMS for $C_{13}H_9ClN_2$: *m/z* = 228.0455.

2-(2-Bromophenyl)imidazo[1,2-*a*]pyridine (3e): Pale yellow solid; 247.52 mg (yield: 91%); m.p.: 80-82 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.78 (t, *J* = 6.7 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.68-7.62 (m, 2H), 8.17-8.13 (m, 2H), 8.28 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 111.9, 112.4, 117.6, 121.5, 124.7, 125.7, 127.5, 128.8, 131.6, 133.6, 134.4, 143.2, 144.5. HRMS for $C_{13}H_9BrN_2$: *m/z* = 272.0040.

2-(4-Nitrophenyl)imidazo[1,2-*a*]pyridine (3f): Yellow solid; 230.40 mg (yield: 96%); m.p.: 200-202 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.96 (td, *J* = 6.8, 0.9 Hz, 1H), 8.65 (s, 1H), 7.37-7.27 (m, 1H), 7.63 (d, *J* = 9.1 Hz, 1H), 8.23 (d, *J* = 9.0 Hz, 2H), 8.31 (d, *J* = 9.0 Hz, 2H), 8.58 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 112.1, 113.3, 117.4, 124.6, 126.4, 126.7, 127.7, 140.9, 142.4, 145.7, 146.9. HRMS for $C_{13}H_9N_3O_2$: *m/z* = 240.0090.

2-(Naphthalen-1-yl)imidazo[1,2-*a*]pyridine (3g): Yellow syrup; 215.83 mg (yield: 88%); m.p.: 186-188 ºC.^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.75 (td, *J* = 6.8, 1.1 Hz, 1H); 7.20-7.11 (m, 1H), 7.56-7.45 (m, 3H), 7.68 (dd, *J* = 9.1, 0.7 Hz, 1H), 7.78 (s, 1H), 7.92-7.79 (m, 3H), 8.65-8.57 (m, 1H), 8.09 (dt, *J* = 6.8, 1.1 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 111.2, 112.4, 117.7, 124.5, 125.4, 125.5, 125.8, 125.9, 126.4, 127.7, 128.3, 128.4, 131.5, 131.8, 133.9, 145.2,145.3. HRMS for $C_{17}H_{12}N_2$: *m/z* = 245.2650.

2-(Imidazo[1,2-*a*]pyridin-2-yl)phenol (3h): Colourless solid; 186.97 mg (yield: 89%); m.p.: 140-141 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 6.87 (t, *J* = 6.8 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 7.31-7.21 (m, 2H), 7.64-7.56 (m, 2H), 7.87 (s, 1H), 8.16 (d, *J* = 6.7 Hz, 1H), 12.76 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 106.7, 113.1, 116.2, 116.7, 117.7, 118.9, 125.1, 125.4, 125.7, 129.6, 143.4, 145.3, 157.3. HRMS for $C_{13}H_{10}N_2O$: *m/z* = 210.0880.

7-Methyl-2-phenylimidazo[1,2-*a*]pyridine (3i): White solid; 188.19 mg (yield: 90%); m.p.: 162-164 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.36 (s, 3H), 6.55 (dd, *J* = 6.9, 1.4 Hz, 1H), 7.33-7.23 (m, 1H), 7.43-7.37 (m, 3H), 7.71 (s, 1H), 8.00-7.84 (m, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 21.3, 107.5, 115.0, 115.8, 124.7, 125.9, 127.7, 128.6, 133.9, 135.5, 145.5, 146.1. HRMS for $C_{14}H_{12}N_2$: *m/z* = 209.1010.

2-(3,4-Dimethoxyphenyl)-7-methylimidazo[1,2-*a*]pyridine (3j): Yellow solid; 238.62 mg (yield: 89%); m.p. 135-137 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.28 (s, 3H), 3.91 (s, 3H), 3.99 (s, 3H), 6.90 (d, *J* = 8.4 Hz, 1H), 6.98 (dd, *J* = 9.2, 1.2 Hz, 1H), 7.41 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.50 (d, *J* = 9.2 Hz, 1H), 7.56 (d, *J* = 1.7 Hz, 1H), 7.68 (s, 1H), 7.84 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 18.0, 55.9, 56.0, 107.2, 109.1, 111.2, 116.5, 118.3, 121.8, 123.2, 127.0, 127.6, 144.6, 145.4, 148.8, 149.2. HRMS for $C_{16}H_{16}N_2O$: *m/z* = 268.1220.

7-Methyl-2-(4-nitrophenyl)imidazo[1,2-*a*]pyridine (3k): Yellow solid; 232.84 mg (yield: 92%); m.p.: 214-216 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.37 (s, 3H), 6.80 (dd, *J* = 6.9, 1.6 Hz, 1H), 7.39 (s, 1H), 8.20 (d, *J* = 9.1 Hz, 2H), 8.29 (d, *J* = 9.1 Hz, 2H), 8.45 (d, *J* = 7.0 Hz, 1H), 8.55 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 21.3, 111.6, 115.5, 115.8, 124.5, 126.6, 126.8, 136.9, 141.1, 142.2, 146.1, 146.7. HRMS for $C_{14}H_{11}N_3O_2$: *m/z* = 253.0880.

2-(4-Bromophenyl)-7-methylimidazo[1,2-*a*]pyridine (3l): Pale yellow solid; 263.21 mg (yield: 92%); m.p.: 210-212 ºC; ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.38 (s, 3H), 6.59 (dd, *J* = 6.9, 1.5 Hz, 1H), 7.35 (s, 1H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.71 (s, 1H), 7.78 (d, *J* = 8.5 Hz, 2H), 7.94 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 21.4, 107.6, 115.2, 115.8, 121.6, 124.7, 127.4, 131.7, 132.9, 135.8, 144.4, 146.2. HRMS for $C_{14}H_{11}BrN_2$: *m/z* = 286.0120.

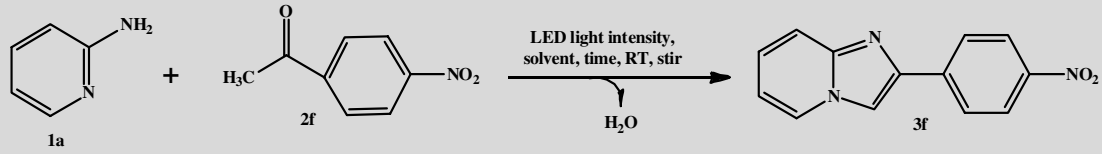
8-Methyl-2-(3-nitrophenyl)imidazo[1,2-*a*]pyridine (3m): Yellow solid; 227.77 mg (yield: 90%); m.p.: 167-169 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.56 (s, 3H); 6.84 (t, *J* = 6.8 Hz, 1H), 7.16-7.06 (m, 1H), 7.73 (t, *J* = 8.0 Hz, 1H), 8.19-8.12 (m, 1H), 8.42-8.35 (m, 2H), 8.59 (s, 1H), 8.79-8.74 (m, 1H). ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 17.1, 111.4, 113.1, 120.0, 122.4, 124.4, 125.2, 126.8, 130.7, 132.1, 136.3, 141.8, 145.9, 148.7. HRMS for $C_{14}H_{11}N_3O_2$: *m/z* = 253.0881.

2-(3,4-Dimethoxyphenyl)-8-methylimidazo[1,2-*a*]-pyridine (3n): White solid; 238.63 mg (yield: 89%); m.p.: 124-126 ºC. ^{1}H NMR (300 MHz, DMSO-d_6) δ ppm: 2.65 (s, 3H), 3.91 (s, 3H), 3.99 (s, 3H), 6.63 (t, *J* = 6.8 Hz, 1H), 6.94-6.88 (m, 2H), 7.45 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.58 (d, *J* = 1.9 Hz, 1H), 7.74 (s, 1H), 7.93 (d, *J* = 6.7 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ ppm: 17.1, 55.9, 55.9, 107.9, 109.4, 111.3, 112.2, 118.5, 123.1, 123.2, 127.2, 127.3, 145.1, 146.0, 148.8, 149.1. HRMS for $C_{16}H_{16}N_2O_2$: *m/z* = 268.1280.

RESULTS AND DISCUSSION

Inspired by reported research methods on the visible light (CFL bulb) irradiation [39-41], in the work, the reaction of 2-

TABLE-1
SYNTHESIS OF IMIDAZO[1,2-*a*]PYRIDINE (**3f**) IN INDICATED CONDITIONS



Entry	Solvent	Visible light intensity (W)	Time (h)	Yield (%)
1	Dioxane	14	35	75
2	THF	14	32	42
3	DMF	14	31	50
4	DMSO	14	32	45
5	Acetonitrile	14	29	55
6	Methanol	14	32	35
7	Water	14	33	Trace
8	Ethanol	14	32	49
9	Toulene	14	28	60
10	Toluene	9	36	50
11	Toluene	12	28	77
12	Toluene	18	24	96
13	Toluene	26	24	96

aminopyridine (**1a**) and 4-nitroactophenone (**2f**) in ethanol solvent (Table-1) were carried out under visible light irradiation with LED light source (14 W) at ambient conditions. Notably, the desired imidazo[1,2-*a*]pyridine (**3f**) was obtained in 49% yield (entry 8, Table-1). When the LED light source was not used, no reaction occurred and the starting materials were recovered.

The influences of solvent and light intensity were also investigated. Toluene proved to be the most suitable solvent in the reaction. In addition, 18W visible light intensity (entry 12, Table-1) obtained the maximum yield of 96%. It was also found that the reaction proceeds using LED light either in a daylight or night temperature, the output remains same.

The feasibility of the reaction based on the simple, catalyst free and ambient conditions for synthesizing imidazopyridines was also investigated. 2-Aminopyridines having electron donating methyl substituents delivered the desired products (**3a-n**) in 88-96% yields (Table-2). Acetophenones with electron withdrawing groups such as nitro reacted with 2-aminopyridines under optimized conditions to give excellent yields (Table-2, entries 6, 11, 13). Halogen atoms like chloro and bromo are also provide (Table-2, entries 4, 5, 12) good yields under these conditions. Electron-donating groups, including methoxy and hydroxy, are also generated in good yields (Table-2, entries 3, 8, 10, 14).

Mechanism: On the basis of above observations together with the literature reports [42-45], a radical pathway (**Scheme-II**) to account for the process is also proposed. Initially, imine (**A**) is formed *via* the condensation of 2-aminopyridine and acetophenone. Enamine (**B**) is produced by tautomerization of imine (**A**). Subsequently, intermediate **C** is formed *via* cyclization of enamine (**B**). The desired imidazo[1,2-*a*]pyridine is generated by the dehydrogenation of intermediate **C**.

Conclusion

In summary, a LED light induced reaction between 2-aminopyridines and acetophenones is successfully initiated,

TABLE-2
SCOPE OF 2-AMINOPYRIDINES AND ACETOPHENONES

Entry	Product	Time (h)	Yield (%)
1	**3a**	25	94
2	**3b**	29	91
3	**3c**	31	92
4	**3d**	33	90
5	**3e**	34	91
6	**3f**	24	96
7	**3g**	25	88
8	**3h**	30	89
9	**3i**	30	90
10	**3j**	34	89
11	**3k**	25	92
12	**3l**	32	92
13	**3m**	28	90
14	**3n**	28	89

leading to the facile synthesis of imidazo[1,2-*a*]pyridines in excellent yields. The room temperature and catalyst free methodology have successfully been applied to a wide range of 2-aminopyridines and acetophenones for the facile synthesis of imidazo[1,2-*a*]pyridines.

CONFLICT OF INTEREST

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

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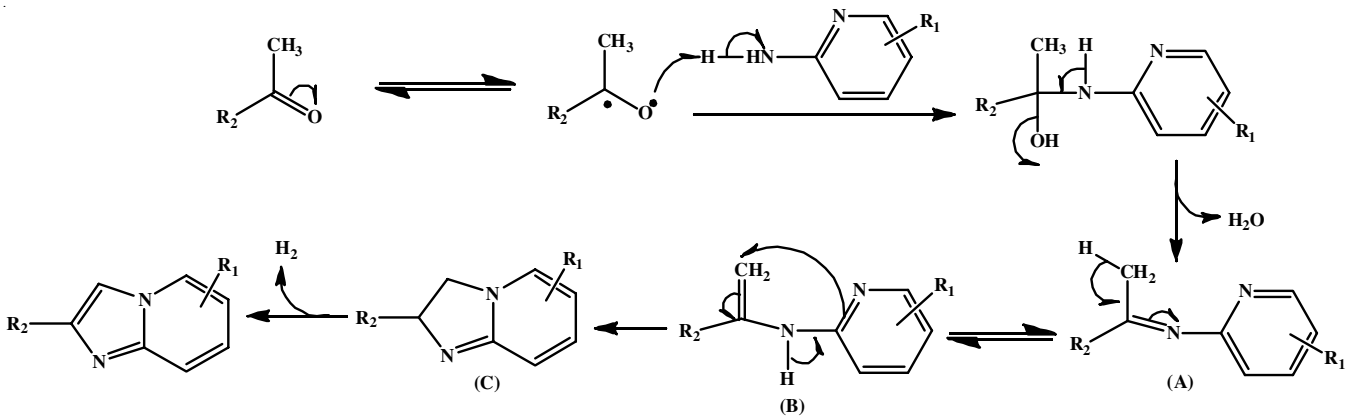

Scheme-II: Probable mechanism for LED light induced synthesis of imidazo[1,2-*a*]pyridines

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