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ORIGINAL PAPER

Facile synthesis of imidazo[1,2-*a*]pyridines promoted by room-temperature ionic liquids under ultrasound irradiation

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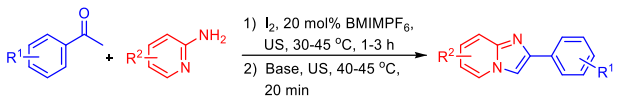
Abstract

A simple and efficient procedure for the synthesis of substituted imidazo[1,2-*a*]pyridines under ultrasound irradiation has been developed. The reactions were carried out using ionic liquids as catalyst. The reaction procedure demonstrated a broad substrate scope for both acetophenones and 2-aminopyridines, and provided convenient access to a wide variety of imidazo[1,2-*a*]pyridines. The present method offers several advantages compared to traditional heating methods such as higher yields, shorter reaction times, milder reaction conditions, and easier work-up procedure.

Graphical abstract



Keywords Cyclization · Ketones · One-pot synthesis · Heterocycles

Introduction

In recent years, the synthesis of imidazo[1,2-*a*]pyridines has drawn considerable attention due to their useful antiviral [1–3], antimicrobial [4], antiulcer [5], and anti-inflammatory therapeutic activities[6]. Additionally, their application has recently expanded to include biological imaging probes due to their remarkable fluorescence properties [7].

Various methods have been published for the synthesis of imidazo[1,2-*a*]pyridines. These methods have been continually modified and improved. These included oxidative coupling [8–12], oxidative amination [13], aminooxygenation [14], and hydroamination [15]. Of the many methods which have been reported, the condensation reactions of 2-aminopyridines with α-halogenoketones is the most attractive approach compared to other synthetic methods due to several advantages such as operational simplicity, and reduction in waste generation [16]. There are; however, some limitations of this method such as high toxicity and lachrymatory property of α-halogenoketones [17]. This limitation can be partly overcome by the use of α-ketomethylpyridinium iodide salts directly without isolation of the α-haloketone via Ortoleva–King reaction [18, 19]. This approach; however, has significant drawbacks such as long reaction times, high reaction temperatures, and low yields.

In recently years, ionic liquids (ILs) have attracted considerable attention for organic synthesis as alternative reaction media to volatile organic solvents owing to their distinctive physical properties such as low volatility, and non-flammability. Furthermore, the physical and chemical properties of the ionic liquids can be controlled by tailoring their structures. Although, ionic liquids were initially developed for use as solvents; recent studies have shown that ionic liquids can be used in catalysis as catalyst

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Scheme 1

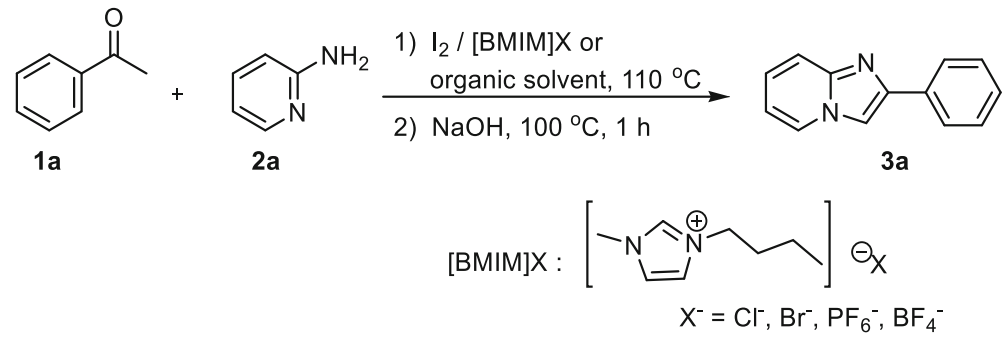


Table 1 Reaction conditions and yields for the synthesis of 2-phenylimidazo[1,2-*a*]pyridine (**3a**) using conventional method (CM) and ultrasound irradiation condition (US)

Entry	Condition		Time/h	Yield/%[a]
	Solvent	Method		
1	Benzene	CM[b]	24	56
2	Solvent-free	CM[b]	24	55
3	[BMIM]Br	CM[b]	24	50
4	[BMIM]Cl	CM[b]	24	52
5	[BMIM]BF_4	CM[b]	24	70
6	[BMIM]PF_6	CM[b]	24	73
7	Benzene + [BMIM]PF_6	CM[c]	24	69
8	Benzene	US[d]	2.5	55
9	Solvent-free	US[d]	2.5	58
10	[BMIM]Br	US[d]	2.5	59
11	[BMIM]Cl	US[d]	2.5	60
12	[BMIM]BF_4	US[d]	2.5	73
13	[BMIM]PF_6	US[d]	2.5	72[e]

Reaction conditions: **1a** (0.51 mmol), **2a** (1.17 mmol), iodine (0.61 mmol) under solvent-free or solvents 0.7 cm^3, carried out under specified conditions, followed by excess aqueous NaOH (45%), 100 °C, 1 h

[a]Isolated yield

[b]CM: conventional method performed by heating at 110 °C

[c]The reaction was carried out in benzene containing 4% (w/v) of [BMIM]PF_6

[d]US: ultrasound irradiation method performed at a frequency of 40 kHz at 30–35 °C

[e]The reaction under conventional heating for 2.5 h afforded **3a** in 12%

[20–22], catalyst activator [23], and co-catalyst for various reactions [24, 25].

The application of ultrasonic waves as alternative source of energy has received considerable attention over the past few decades, and it has made a major contribution in the field of organic synthesis. It is being used increasingly to speed up various organic reactions and provides new possibilities to stimulate new reactions that are difficult to synthesize by conventional methods. Recently, the sonochemical approach has been further extended to the syntheses of fused heterocyclics such as benzofuran [26], pyrimidopyrans [27], pyridopyrimidines [28], pyrazolopyridines [29], and dihydropyranopyrazoles [30].

In this paper, we wish to report an efficient method for one-pot synthesis of imidazo[1,2-*a*]pyridines using ionic liquids as catalyst with the assist of ultrasound irradiation. The method provides a significant achievement in comparison to conventional methods in terms of reducing reaction time, operational simplicity, and enhancing yield.

Results and discussion

To probe whether the reaction can be influenced by ionic liquids, we began our studies using acetophenone (**1a**) and 2-aminopyridine (**2a**) as a model reaction. The model

Table 2 Effect of $[BMIM]PF_6$ concentrations on the synthesis of **3a** under ultrasound irradiation

Entry	$[BMIM]PF_6$/mol%	Isolated yield/%
1	None	58[a]
2	700 (0.7 cm^3)[b]	72
3	100	74
4	30	72
5	20	80 (82)[c]
6	10	69

Reaction conditions: **1a** (0.51 mmol), **2a** (1.17 mmol), iodine (0.61 mmol) in the presence of various $[BMIM]PF_6$ concentrations, ultrasound irradiation at 30–35 °C, 2.5 h, followed by excess aqueous NaOH (45%), 100 °C, 1 h

[a]Reaction conditions: **1a** (0.51 mmol), **2a** (1.17 mmol), iodine (0.61 mmol) under solvent-free condition, ultrasound irradiation at 30–35 °C, 2.5 h, followed by excess aqueous NaOH (45%), 100 °C, 1 h

[b]$[BMIM]PF_6$ 0.7 cm^3 (700 mol%) was used as solvent

[c]Reaction conditions: **1a** (0.51 mmol), **2a** (1.17 mmol), iodine (0.61 mmol) and 20 mol% $[BMIM]PF_6$, ultrasound irradiation at 30–35 °C, 2.5 h, followed by excess aqueous K_2CO_3 (35%), ultrasound irradiation at 40–45 °C, 20 min

reaction was carried out in the presence of iodine at 110 °C for 24 h in three different systems, benzene (as reference reaction medium), solvent-free and room temperature ionic liquids, i.e., 1-butyl-3-methylimidazolium bromide [(BMIM)Br], 1-butyl-3-methylimidazolium chloride [(BMIM)Cl], 1-butyl-3-methylimidazolium tetrafluoroborate [$(BMIM)BF_4$], and 1-butyl-3-methylimidazolium hexafluorophosphate [$(BMIM)PF_6$], followed by adding excess amount of aqueous NaOH (45%) and heating at 100 °C for 1 h (Scheme 1). The results are summarized in Table 1.

As indicated in Table 1, the corresponding product **3a** was obtained in moderate yield after heating at 110 °C for 24 h under solvent-free condition which was comparable to the yield obtained in benzene, whereas slightly lower yields were obtained when [BMIM]Br and [BMIM]Cl were employed (Table 1, entries 1–4). However, when $[BMIM]BF_4$ and $[BMIM]PF_6$ were used, higher yields were obtained (Table 1, entries 5 and 6). In addition, a significant increase of yield was also observed, when the model reaction was carried out in benzene containing 4% (w/v) of $[BMIM]PF_6$ (Table 1, entry 7). These conditions have proved that $[BMIM]BF_4$ and $[BMIM]PF_6$ played a significant role in the model reaction. Although the reactions in $[BMIM]BF_4$ and $[BMIM]PF_6$ proceeded more smoothly and in the better yields, relatively heating at high temperature (110 °C) and long reaction times were required to reach completion (24 h). In an effort to increase the efficiency and decrease the temperature, we then explored the possibility of using the ultrasonication method to further improve the procedure. Hence, the model reaction was further investigated under the influence of ultrasound irradiation at 30–35 °C for 2.5 h. It was revealed that under ultrasound irradiation, in all cases, yields were comparable to or better than using conventional methods in shorter reaction time at lower temperature (Table 1, entries 8–13). Under ultrasound irradiation, higher yields, i.e., 58, 59, 60, and 73% (Table 1, entries 9, 10, 11, and 12) were obtained in comparison with the same reactions performed in the same reaction medium under conventional heating (Table 1, entries 2, 3, 4, and 5, respectively). The higher yield was obtained when $[BMIM]BF_4$ and $[BMIM]PF_6$ were used as compared to the reaction under solvent-free, benzene, and other ionic liquids. These results revealed that ionic liquids $[BMIM]BF_4$ and $[BMIM]PF_6$ could effectively accelerate the reaction under ultrasound irradiation.

Effect of ionic liquids concentrations

Prompted by these encouraging results, we next investigated the effect of ionic liquids concentrations on the reaction as catalyst. The system with $[BMIM]PF_6$ was chosen for this investigation due to its water immiscible property which provided an advantage over $[BMIM]BF_4$ in work-up process. The results are summarized in Table 2. It was observed that, decreasing the amount of $[BMIM]PF_6$ to 100 mol% led to an improvement of yield (74%) than that was obtained using $[BMIM]PF_6$ as solvent (Table 2, entries 2 and 3). However, decreasing the amount of $[BMIM]PF_6$ to 30 mol% only provided the desired product with comparable yield. The best result was obtained when 20 mol% of $[BMIM]PF_6$ was employed (Table 2, entry 5). An attempt to further decrease the amount of $[BMIM]PF_6$ (10 mol%) resulted in a significantly lower yield (69%; Table 2, entry 6). It was also observed that the second step of this reaction can be accomplished under milder conditions with slightly higher yield using aqueous K_2CO_3 (35%) under ultrasound irradiation at 40–45 °C for 20 min instead of heating with aqueous NaOH (45%) at 100 °C for 1 h. These results demonstrated that the use of ionic liquids in catalytic amount under ultrasound irradiation led to higher yields and the reactions were markedly accelerated using ultrasound irradiation.

Optimization the molar ratio of reagents under ultrasound irradiation

Next, we examined the influence of the molar ratio of reagents under ultrasound irradiation and the results of these findings are presented in Table 3.

The effect of molar ratio of the reagents was investigated under ultrasound irradiation at 30–35 °C in the

Table 3 Effect of substrate ratios

Entry	Ratio **2a**:**1a**	Iodine vs. **1a**/equiv	Isolated yield/%
1	1.2	1.2	41
2	1.5	1.2	52
3	2.0	1.2	69
4	2.3	1.2	82
5	2.5	1.2	83
6	2.7	1.2	83
7	3.0	1.2	80
8	2.3	0.5	29
9	2.3	1.0	67
10	2.3	1.5	68[a]
11	2.3	2.0	64[b]

Reaction conditions: 20 mol% [BMIM]PF_6, ultrasound irradiation at 30–35 °C, 2.5 h, followed by excess aqueous K_2CO_3 (35%), ultrasound irradiation at 40–45 °C, 20 min

[a] **4a** was isolated in 7%

[b] **4a** and **5a** were isolated in 19 and 10%, respectively

presence of 20 mol% [BMIM]PF_6. The molar ratio of 2-aminopyridine/acetophenone/iodine ranging from 1.2:1.0:1.2 to 3.0:1.0:1.2 was varied (Table 3). The results revealed that increasing the molar ratio of 2-aminopyridine from 1.2 to 2.3 resulted in higher yields (Table 3, entries 1–4). However, no significant change in product yield was observed, as the molar ratio of 2-aminopyridine was further increased to 3.0. Additionally, the reaction was also optimized by varying the amount of iodine. The yield of the product significantly decreased with increased the mole ratio of iodine from 1.2 to 2.0 (Table 3, entries 10 and 11). This was due to the formation of the side reaction products, 6-iodo-2-phenylimidazo[1,2-*a*]pyridine (**4a**) and 3-iodo-2-phenylimidazo[1,2-*a*]pyridine (**5a**). The observed minor side reaction product **4a** resulted from iodination of 2-aminopyridine which then further reacted with acetophenone (**1a**). On the other hand, **5a** was formed presumably by further iodination of the desired product **3a**. This was confirmed by reacting **3a** with 1 equivalent of iodine in [BMIM]PF_6 under the same condition for 2.5 h which provided **5a** in 48% (Scheme 2). The best condition for synthesizing imidazo[1,2-*a*]pyridine (**3a**) was obtained by carrying out the reaction under ultrasound irradiation with 2.3:1.0:1.2 molar ratios of 2-aminopyridine (**2a**), acetophenone (**1a**), and iodine in the presence of 20 mol% [BMIM]PF_6 at 30–35 °C for 2.5 h. When compared to the conventional method, this approach has greatly improved the total reaction time with milder conditions and easier operation.

Substrate scope of imidazo[1,2-*a*]pyridines synthesis

Having established the optimal reaction conditions, the substrate scope of [BMIM]PF_6-catalyzed system under sonication was then examined. A series of substituted acetophenones **1** containing electron-withdrawing and electron-donating groups were subjected to react with 2-aminopyridine or 2-amino-4-methylpyridine **2** using 20 mol% [BMIM]PF_6 as catalyst under ultrasound irradiation and compared with the conventional heating method and work-up procedure (Table 4). In general, shorter reaction time and highly significant differences in yields were obtained among entries when the reaction was carried out under ultrasound irradiation. It was also observed that, under sonication condition, the substituted acetophenones, such as *p*-bromo, *p*-methoxy, *m*-nitro, and *m*-methoxy groups on the aryl ring, reacted with **2a** efficiently and gave the desired products **3b**, **3c**, **3e**, and **3f** in high yields of 70–76% (Table 4, entries 2, 3, 5, and 6).

On the other hand, *p*-nitro and *o*-hydroxy acetophenones reacted with **2a**, to provide the expected product **3d** and **3g** in 53-58% (Table 4, entries 4 and 7). Whereas, the 2-amino-4-methylpyridine (**2b**) reacted efficiently with acetophenone and substituted acetophenone, such as *p*-bromo, *p*-methoxy, *p*-nitro, and *o*-hydroxy groups on the aryl ring, and gave the desired products **3h**–**3k** and **3m** in high-to-excellent yields of 72–86% (Table 4, entries 8–11 and 13). Similarly, the product **3l** was obtained in moderate

Scheme 2

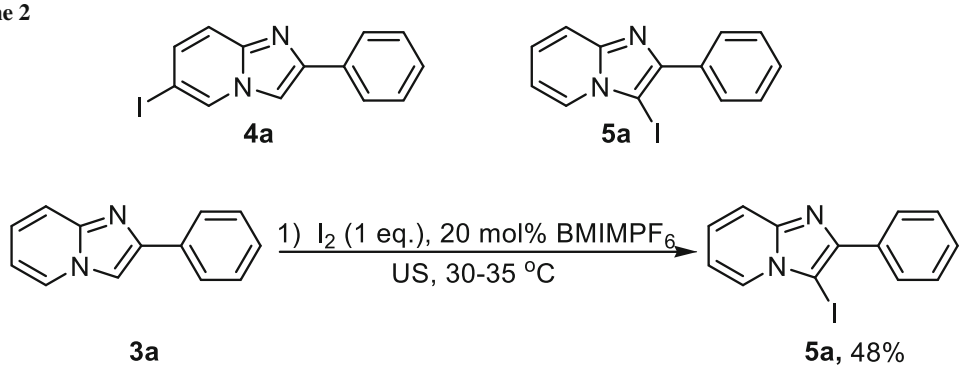
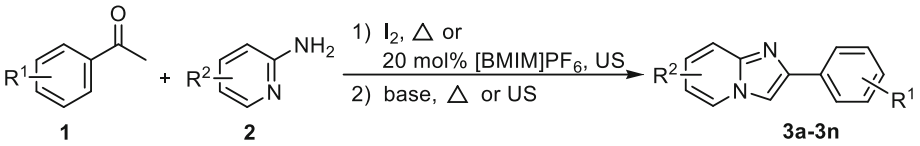


Table 4 One-pot synthesis of imidazo[1,2-*a*]pyridines **3** using conventional method (CM) and ultrasound irradiation condition (US)



Entry	R^1	R^2	Product	Isolated yield/% (Time/h)		M.p. (lit. m.p.)/°C
				CM[a]	US[b]	
1	H	H	**3a**	55 (24)	80 (2.5)	135–136 (136–137 [33])
2	4-Br	H	**3b**	30 (24)	70 (2)	213–215 (214–216 [34])
3	4-OMe	H	**3c**	42 (24)	70 (2)	133–134 (133–134 [35])
4	4-NO_2	H	**3d**	37 (24)	53 (1.5)[c]	265–267 (266–267 [36])
5	3-NO_2	H	**3e**	51 (24)	73 (3)[c]	201–202 (206–208 [37])
6	3-OMe	H	**3f**	62 (4)	76 (1)[c]	60–62 (61–62 [38])
7	2-OH	H	**3g**	51 (12)	58 (2.5)	143–145 (142–143 [39])
8	H	4-Me	**3h**	51 (24)	72 (1.5)	170–172 (174 [34])
9	4-Br	4-Me	**3i**	50 (6)	77 (2.5)	203–205 (202–204 [40])
10	4-OMe	4-Me	**3j**	42 (12)	74 (1.5)	160–162 (160–162 [41])
11	4-NO_2	4-Me	**3k**	36 (12)	86 (2.5)	215–217 (214–216 [41])
12	3-OMe	4-Me	**3l**	40 (6)	67 (1)	77–79 (76–77 [41])
13	2-OH	4-Me	**3m**	31 (12)	77 (2.5)	138–139 (136–137 [11])
14	4-SO_2Me	H	**3n**	53 (24)	72 (3)	244–245 (242 [42])

Reaction conditions: acetophenone **1** (0.51 mmol), 2-aminopyridine **2** (1.17 mmol), iodine (0.61 mmol); reactions were carried out under specified conditions

[a]CM: conventional method performed by heating at 110 °C under solvent-free condition, followed by excess aqueous NaOH (45%), 100 °C, 1 h

[b]US: ultrasound irradiation method performed at a frequency of 40 kHz at 30–35 °C in the presence of 20 mol% [BMIM]PF_6, followed by excess aqueous K_2CO_3 (35%), ultrasound irradiation at 40–45 °C, 20 min

[c]US: ultrasound irradiation method performed at a frequency of 40 kHz at 40–45 °C in the presence of 20 mol% [BMIM]PF_6, followed by excess aqueous K_2CO_3 (35%), ultrasound irradiation at 40–45 °C, 20 min

yield (67%) when *m*-methoxyacetophenone was used (Table 4, entry 12).

Moreover, by applying this method, we were able to synthesize the marketed drug zolimidine using metal-free one-pot synthesis in 72% (Table 4, entry 14) with milder condition and shorter reaction time (3 h) than the previously reported methods [4, 11, 31, 32].

Conclusion

In summary, a convenient, milder, and efficient one-pot procedure for the synthesis of imidazo[1,2-*a*]pyridines using catalytic amount of [BMIM]PF_6 assisted by sonication has been developed. This new method offers very attractive features such as reduced reaction times, higher yields and operational simplicity than the conventional method. The generality of the method was demonstrated by its application to various kinds of 2-aminopyridines and acetophenones. Additionally, this method is a fast and efficient method to synthesize the marketed drug zolimidine.

Experimental

All reagents were obtained from various commercial suppliers. They were further carefully purified before using. The melting points were determined using a MEL-TEMP from Laboratory device. The ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on Bruker AVANCE 400 spectrometer using the solvent peak as internal reference and $CDCl_3$ or DMSO-d_6 as solvents. Sonication was carried out using a HIQ-LAB ultrasonic cleaner Model ULA-4L (with a frequency of 40 kHz and a nominal power 150 W).

General procedure for the synthesis of imidazo[1,2-*a*]pyridines under conventional method

A mixture of acetophenone **1** (0.51 mmol), 2-aminopyridine **2** (1.17 mmol), and iodine (0.61 mmol) were added into a 5-cm^3 dried flat-bottom capped-vial equipped with

magnetic bar. The mixture was stirred at 110 °C and monitored by TLC. To complete the cyclization step, the excess aqueous NaOH (45%) was added and subsequently stirred at 100 °C for 1 h. Then, the reaction mixture was cooled to room temperature and diluted with $CHCl_3$. The mixture was neutralized with 3.5 M HCl. The organic phase was separated and aqueous layer was extracted with $CHCl_3$. The combined organic extract was dried over Na_2SO_4 and evaporated under reduced pressure to afford the crude product which was purified by column chromatography using ethyl acetate/hexane (1:9–2:8).

General procedure for the synthesis of imidazo[1,2-*a*]pyridines derivatives using ultrasonic irradiation

A mixture of acetophenone **1** (0.51 mmol), 2-aminopyridine **2** (1.17 mmol), iodine (0.61 mmol), and 20 mol% [BMIM]PF_6 (0.10 mmol) were added into a 5-cm^3 dried flat-bottom capped-vial equipped with magnetic bar and irradiated at 30–45 °C at a frequency of 40 kHz for 1–3 h. The reaction temperature of ultrasonic bath was controlled manually by addition or removal of small amounts water. Then, the excess aqueous K_2CO_3 (35%) was subsequently added and further irradiated at the same frequency at 40–45 °C for 20 min to accomplish the cyclization. After completion, the mixture was diluted with $CHCl_3$ and neutralized using 3.5 M HCl. The organic layer was separated and the aqueous layer was extracted with $CHCl_3$. The combined organic layer was dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was further purified by column chromatography using ethyl acetate/hexane (1:9–2:8) to give the desired product.

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