Synthesis and Characterization of Hydroxy Substituted Pyridinium Type of Ionic Liquids via Conventional/ Silica Supported Approaches and Their Applications

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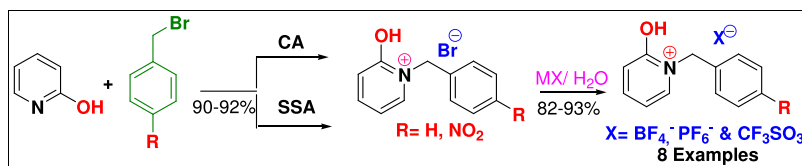PG and Research Department of Chemistry, Presidency College (Autonomous), Chennai 600 005, India
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Synthesis of 2-hydroxy pyridinium type of ionic liquids is obtained via conventional as well as silica-supported approaches. We found that solid-supported reaction under muffle furnace condition has reduced the reaction nearly 20 times shorter than the conventional method. We have studied the catalytic activities of our synthesized pyridinium salts for one-pot preparation of substituted quinolone derivatives and radical bromination of toluidine with optimized reaction conditions.

INTRODUCTION

Ionic liquids play several important roles in the areas of enzyme-catalyzed reaction [1], dye-sensitized solar cell [2], replacement of toxic organic solvent for organic synthesis [2,3] used as stationary phase in chromatography [4], and lubricants [5,6]. The behavior of the ionic liquids is varied from the nature of cations and anions such as water-soluble and insoluble character [7]. Some of the ionic liquids are acted as dual role for unusual oxidative esterification of different aldehydes [8]. Over the few decades, green chemistry has become the fastest growing and more popular research field [9]. Synthesis regioselective and sterioselective Furocou marines using multicomponent reaction in the presence of ionic liquids [10]. Heterocyclic compounds are more useful, and interesting observations are made in the area of organometallic chemistry [11,12]. Zhao *et al* reported that some of the pyridinium-type ionic liquids showed that better catalytic behavior for Marital–Bailys–Hillman reaction [13]. The catalytic activity of chiral pyridinium ionic liquids is very effective in Michael reaction [14]. Chlorination of non-allyl hindered aromatic substrate from oxidative methodology in the presence of ionic liquids [15]. Quartinization of heterocyclic compounds like imidazole/pyridine moieties in the presence of halo alkane generally need prolonged heating [16] whereas in the assistance of ionic liquids will be easier [17]. Substituted imidazolium type of ionic liquids acted as solvent and promoting the sonochemical approach for Heck and Suzuki reactions at room temperature [18,19]. Recently, we have reported that pyridinium-type ionic liquids have played better catalytic activity for Mannich reaction [20,21].

Herein, we have discussed the synthesis of hydroxy substituted pyridinium-type ionic liquids via conventional and silica-supported methodologies and studied its catalytic activities for the one-pot preparation of substituted quinolone and α aralkyl bromination reaction under free radical methodologies.

RESULTS AND DISCUSSION

n-Alkylation of 2-hydroxypyridine (1.051×10^{-2} mmol; 1.0 equiv.) is treated with benzyl bromide/4-nitrobenzyl bromide (1.156×10^{-2} mmol; 1.05 equiv.) in the presence of dry MeCN under refluxing condition for about 5–8 h afforded the *n*-alkylated products of compound (**1a–b**) in quantitative yields after the purification. The same reaction we have tried under silica-supported methodology; 2-hydroxy pyridine and benzyl/4-nitrobenzylbromide with silica gel (60–120 mesh) followed by fine grinding and to be kept in a muffle furnace at 100°C between 20–35 min. 2-Hydroxy pyridine with 4-nitro benzyl bromide under silica-supported reaction is completed within 20 min, whereas the same reaction under conventional approaches needs 8 h to complete. We have tried similarly benzyl bromide with 2-hydroxy pyridine under silica-supported n-alkylation reaction under muffle furnace at 100°C; the reaction is completed within 35 min, whereas the conventional reflux condition with dry acetonitrile will take 8 h before completion. So silica-supported n-alkylation reaction is the most efficient methodology compared with the conventional refluxing approach. 4-Nitrobenzylbromide is reacted with 2-hydroxypyridine much faster than benzyl bromide because of weaker C-Br bond. After the removal of excess benzyl bromide, anion

exchange reaction is carried out with different anion containing inorganic salt such as $NaBF_4$, K_4PF_6, and $LiCF_3SO_3$ in the presence of minimum amount of deionized water at room temperature for about 2 h to give anion exchanged products of ionic liquids (**2a–f**) in quantitative yield (Scheme 1).

CATALYTIC ACTIVITY

Catalytic Activity for One-Pot Preparation of Substituted Quinolone. One-pot operation of multicomponent reaction gives an opportunity for the joining of three or more simple and flexible building units to form a giant or complex structure by the simultaneous formation of two or more new bonds [22]. The number of quinoline and its derivatives are showed a broad range of applications like antimalarial [23], antidiabetic [24], antibacterial [25], and antiasthmatic [26] activities. One-pot model reaction between 4-nitroaniline, 2-nitrobenzaldehyde, and butanal are performed in the presence of 10 mL of dry MeCN along with our synthesized ionic liquids. We have found that our ionic liquids are showed from good to excellent catalytic behaviors to synthesis of substituted quinoline derivative, and another interesting observation is the percentage of yield for the corresponding quinoline high with optimized catalyst concentration.

Catalytic study with 4-nitroaniline (1.810×10^{-3} mmol; 1.0 equi.) 2-nitrobenzaldehyde (1.846×10^{-3} mmol; 1.02 equi.) are treated with butanal (1.846×10^{-3} mmol; 1.02 equi.) with optimized concentration of our synthesized pyridinium type of ionic liquid in the presence of 10 mL of dry MeCN between of 70 to 110 min afforded the 3-methyl-6-nitro-2-(2-nitrophenyl) quinoline, which was confirmed by spectral and analytic data.
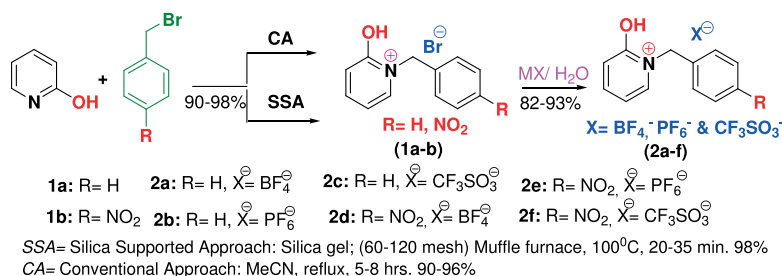
We have tried catalytic activities of our synthesized pyridinium type of ionic liquids for the quinolone reaction using our synthesized ionic liquid at ambient reaction condition. We have used different aryl amine like aniline, p-nitroaniline, p-toluine, and o/p-hydroxyaniline for quinoline derivative formation. The reaction facilitates with p-nitroaniline o/p-hydroxy aniline than other anilines our target to prepare 7-nitro quinoline under one-pot approach with various our synthesized ionic liquids. We have observed that ionic liquid with bromide counter ion showed better catalytic behavior than the others. Herein, we have employed two types of bromide counter ions containing pyridinium salts; among these, pyridinium bromide **1a** showed good catalytic activity than the 4-nitro substituted pyridinium bromide **1b.**

One-pot synthesis of various substituted quinoline derivatives (Scheme 2) from equal molar mixture of 2-nitrobenzaldehyde, butanal, and different aryl amine with 1.44×10^{-4} mmol concentration of our synthesized ionic liquid in the presence of 10 mL of dry MeCN between 70 and 110 min afforded the desired product in quantitative yield (Table 1).

Substituted hydroxy pyridinium bromide assisted one-pot synthesis of quinoline under ambient reaction condition. We have compared Tables 1 and 2 from the results we have observed that while an increase from 4.82×10^{-5} mmol to 9.64×10^{-5} mmol of our synthesized ionic liquids same reaction condition the reaction mixture with 9.64×10^{-5} mmol showed lesser reaction time than the 9.64×10^{-5} mmol concentration therefore while increase the concentration of ionic liquid catalyst role is enhanced (Table 2).

We have monitored the formation of substituted quinoline with different concentrations like 4.82×10^{-5} mmol, 9.64×10^{-5} mmol, and 1.44×10^{-4} mmol of synthesized ionic liquids interesting observation when moving from

Scheme 1. Synthesis of 2-hydroxy pyridinium type of ionic liquids *via* conventional and silica-supported approaches. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

1a: R= H   2a: R= H, X= $BF_4^{\ominus}$ 2c: R= H, X= $CF_3SO_3^{\ominus}$ 2e: R= NO_2, X= $PF_6^{\ominus}$
1b: R= NO_2 2b: R= H, X= $PF_6^{\ominus}$ 2d: R= NO_2, X= $BF_4^{\ominus}$ 2f: R= NO_2, X= $CF_3SO_3^{\ominus}$

SSA= Silica Supported Approach: Silica gel; (60-120 mesh) Muffle furnace, 100⁰C, 20-35 min. 98%
CA= Conventional Approach: MeCN, reflux, 5-8 hrs. 90-96%

Scheme 2. One-pot preparation of quinoline under different concentration of our synthesized ionic liquids. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

R= H, NO_2, Me, o-OH, p-OH
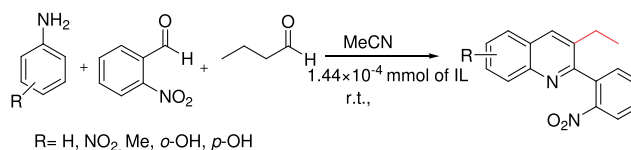
Table 1

Multi components reaction with 4.82 x 10^{-5} mmol concentration of our pyridinium salts.

| Catalyst | *p*-NO$_2$ | | *p*-Me | | −H | | *o*-OH | | *p*-OH | |
	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %
1a	70	85	110	84	95	90	80	92	85	95
2a	75	82	115	82	100	88	85	89	90	92
2b	80	78	120	81	115	85	90	87	100	89
2c	80	75	120	77	115	79	95	85	105	85
1b	90	82	130	81	105	87	100	89	100	93
2d	105	80	150	79	104	84	108	85	107	90
2e	105	78	150	78	119	81	110	82	112	85
2f	110	75	160	75	124	75	115	78	120	81

Table 2

Multi components reaction with 9.64 x 10^{-5} mmol concentration of our pyridinium salts.

| Catalyst | *p*-NO2 | | *p*-Me | | −H | | *o*-OH | | *p*-OH | |
	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %
1a	55	89	90	87	60	93	58	94	57	96
2a	58	87	105	85	63	90	60	92	59	94
2b	60	82	110	83	65	87	62	89	60	91
2c	60	80	110	81	65	82	62	85	60	89
1b	60	85	100	85	67	90	65	92	60	92
2d	70	82	109	83	75	87	72	89	65	89
2e	70	79	112	80	87	85	78	85	75	85
2f	80	76	119	78	102	79	82	82	85	81

Table 3

Multi components reaction with 1.44 x 10^{-4} mmol concentration of our pyridinium salts.

| Catalyst | *p*-NO2 | | *p*-Me | | −H | | *o*-OH | | *p*-OH | |
	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %
1a	25	93	55	93	30	95	30	95	35	93
2a	28	90	58	89	33	93	33	93	38	91
2b	30	89	60	85	33	90	33	90	40	86
2c	30	85	60	81	35	87	35	88	40	82
1b	30	95	60	91	35	93	40	93	43	90
2d	33	92	65	87	49	89	42	90	49	85
2e	38	90	69	83	49	83	47	87	55	81
2f	43	88	74	79	55	80	59	83	65	79

4.82×10^{-5} mmol to 9.64×10^{-5} mmol of ionic liquids. The ionic liquids are reducing the reaction time and increased the percentage of yield. We have extended the reaction with 1.92×10^{-4} mmol, ionic liquids there is no interesting observation for reaction time and percentage of yield. So one-pot preparation of substituted quinoline with 1.44×10^{-4} mmol of ionic liquid is sufficient to complete the reaction with higher yield (Tables 3 and 4).

Therefore optimized concentration of ionic liquid for quinoline preparation is 1.44×10^{-4} mmol.

Table IV: One-Pot Synthesis of Quinolone by Using 5th-Cycle recycled Iionic Liquids. Our synthesized ionic liquids are potential candidates to accelerate the quinoline reaction with better yield. Our synthesized ionic liquids are recycled up to four cycles and used for quinoline reaction with the same reaction condition. Even after

Table 4

Catalytic efficiency of our recycled pyridinium salts upto 5th cycles.

S. No	Iionic liquid	Yield (%)
1	**1a**	87–89
2	**2a**	85–88
3	**2b**	86–88
4	**2c**	87–88
5	**1b**	84–86
6	**2d**	82–86
7	**2e**	82–85
8	**2f**	84–85

the fourth recycle, the product obtained was the same as we observed in the fresh use shown in the Table 5.

Catalytic Activity for Free Radical Bromination Reaction.
Bromination of aralkyl substrate using a different brominating agent like molecular bromine, HBr which cause serious environmental problem. NBS is the most effective and inexpensive brominating agent for benzylic, allylic, and aralkyl substrate under a milder condition. NBS is a more suitable reagent because the after completion of the reaction will give succinimide as the by product and work up are also very easier (readily soluble in water) and can be recycled with suitable reaction condition. Most of our synthesized pyridinium type of ionic liquids acted as both a solvent as well as a catalytic promoter for aralkyl bromination under free radical reaction condition. The advantages of our substituted pyridinium type of ionic liquid are hydrophilic and hydrophobic residues are present, which will show good catalytic properties.

Free radical initiator benzoyl peroxide (BPO) catalytic study with p-toluidine (1.866×10^{-3} mmol; 1.0 equi.) is treated with NBS (1.959×10^{-3} mmol; 1.05 equi.) with 1.69×10^{-4} mmol optimized concentration of our synthesized pyridinium type of ionic liquid in the presence of 10 mL of dry CCl_4 of free radical initiator BPO for 3 to 30 min afforded the p-amino benzyl bromide, which was confirmed by spectral and analytic data.

We have tried catalytic activities of our synthesized pyridinium type of ionic liquids for the bromination reaction using N-bromosuccinimide under free radical reaction mechanism.

The rates of the reactions in the presence of ionic liquids are much faster, because our ionic liquid activates the initiation of free radical formation because of increase charge separation of N-Br bond in NBS. We have tried free radical bromination reaction with different concentrations of our synthesized pyridinium type of ionic liquids such as 5.63×10^5, 1.12×10^{-4}, and 1.69×10^{-4} mmol among these concentration; we found that 1.69×10^{-4} mmol concentration of ionic liquid is optimized catalytic concentration; while increasing the concentration of ionic liquids, there is no appreciable response in reaction time and percentage of yield. So free radical bromination reaction with optimized concentration catalyst is 1.69×10^{-4} mmol.

Rajagopal and coworkers reported that conversion of toluene into benzyl bromide with NBS type of with ionic liquid afforded only 35% yield. We have tried with 4-toluidine absence of catalyst and there is no appreciable observation. Whereas conversion of 4-amino toluene into the respective benzyl bromide in the presence of varies synthesized pyridinium salts along with NBS/BPO at room temperature. We have observed that nitro benzyl pyridinium salts showed better catalytic activity than the benzyl substituted pyridinium salts (Scheme 3). We have used four different nitro benzyl pyridinium salts among these **1b** bromide counter ions containing pyridinium salt that showed effective catalytic behavior than the others. Due to size of bromide ion bulkier than the other counter ions. So our synthesized pyridinium type of ionic liquid showed better catalytic activities than the earlier reports (Table 5).

Scheme 3. Free radical bromination reaction with optimized concentration of ionic liquid.
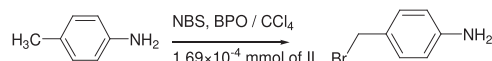
Table 5

	Different concentration of ionic liquids					
	5.63×10^{-5} mmol		1.12×10^{-4} mmol		1.69×10^{-4} mmol	
Ionic liquid	Time (minutes)	Yield %	Time (minutes)	Yield %	Time (minutes)	Yield %
Absence of ionic liquid	No appreciable changes even after 3 h					
1a	30	88	14	86	09	87
1b	22	82	10	91	03	90
2a	32	80	19	84	09	86
2b	28	82	20	88	09	85
2c	28	80	21	88	09	83
2d	19	89	13	88	04	88
2e	19	86	11	90	04	81
2f	20	85	10	72	04	88

CONCLUSION

We have prepared hydroxy substituted pyridinium salts using easily available starting material under conventional and silica-supported approaches. We have examined the catalytic activities of our synthesized ionic liquid for aralkyl substrate under free radical bromination using NBS. We have conducted different concentration of ionic liquids for these reactions, and the optimized catalyst concentration is 1.69×10^{-4} mmol. We found that pyridinium-type ionic liquids acted as a potential candidate to reduce the reaction time with higher yield. We have examined the catalytic activity for one-pot preparation of quinolone by using our synthesized pyridinium type of ionic liquids during one-pot preparation; we have screened the catalyst concentration from 4.82×10^{-5} mmol; 9.64×10^{-5} mmol among the concentration we have found that 1.44×10^{-4} mmol is optimized concentration. One-pot quinoline derivative preparation bromide counter ion containing ionic liquids showed better catalytic activity than the other BF_4^-, PF_6^-, and $CF_3SO_3^-$ counter ions. Among the two bromide counter ions, pyridinium bromide showed better activity than the 4-nitro pyridinium salts.

EXPERIMENTAL

General Procedure For n-Alkylation.   2-hydroxy pyridine $(1.051 \times 10^{-2}$ mmol; 1.0 equi.) is treated with benzyl bromide/4-nitrobenzyl bromide $(1.156 \times 10^2$ mmol; 1.05 equi.) in the presence of 20 mL of dry MeCN under refluxing condition between 5 to 8 h will give n-alkylated products of compound **1a** and **1b** in quantitative yield.

[BHPy]Br⁻ 1a.   Yield: 2.53 g, 90 %; Liquid; ^{1}H NMR (D$_2$O) δ: 4.69 (s, 2H); 6.75–8.06 (m, 9H); ^{13}C NMR δ: 54.2, 112.3, 115.3, 117.9, 127.3, 127.7, 128.4, 129.0, 134.5, 139.4; MS: m/e: 266; Anal. Calcd. for C$_{12}$H$_{12}$BrNO: C, 54.13; H, 4.50; N, 5.26; Found: C, 54.02; H, 4.42; N , 5.18.

[NBHPy]Br⁻ 1b.   Yield: 3.17 g, 96 %; Mp.: 148–150°C; ^{1}H NMR (D$_2$O) δ: 5.30 (s, 2H); 7.16–7.69 (m, 9H); ^{13}C NMR δ: 72.1, 119.4, 127.6, 128.4, 129.1, 135.0, 138.2, 141.7, 142.7, 162.0; MS: m/e: 311 Anal. Calcd. for C$_{12}$H$_{11}$BrN$_2$O$_3$: C, 46.30; H, 3.53; N, 9.00; Found: C, 46.18; H, 3.38; N, 8.92.

General Procedure for Anion Exchange Reaction. Compound **1a/b** with bromide salt with different inorganic salts like NaBF$_4$, K$_4$PF$_6$, and LiCF$_3$SO$_3$ for anion exchange reaction. Substituted pyridinium bromide **1a/b** $(1.878 \times 10^{-3}$ mmol; 1.0 equi) mixed with required anion containing inorganic salt $(1.916 \times 10^{-3}$ mmol; 1.02 equi) in the presence of 10 mL of deionized water at room temperature for stirring for 2 h afforded the anion exchanged products of

compounds (**2a–f**) along with metal bromide. After completing the anion reaction with the assistance of Sox let extraction by using dry THF for extraction, we have separated metallic bromide free ionic liquids in quantitative yield.

[BHPy]PF₆ 2a.   Yield: 0.51 g, 82%; Mp.: 132–134°C; ^{1}H NMR (D$_2$O) δ: 4.56 (s, 2H); 6.80–8.09 (m, 9H); ^{13}C NMR δ: 54.4, 112.5, 115.6, 118.0, 127.7, 127.9, 128.6, 129.2, 134.7, 139.7; MS: m/e: 331; Anal. Calcd. for C$_{12}$H$_{12}$F$_6$NOP: C, 43.50; H, 3.62; N, 4.22; Found: C, 43.40; H, 3.52; N, 4.04.

[BHPy]BF₄ 2b.   Yield: 0.43 g, 84%; Liquid; ^{1}H NMR (D$_2$O) δ: 4.62 (s, 2H); 6.82–8.12 (m, 9H); ^{13}C NMR δ: 54.5, 112.6, 115.8, 118.2, 127.4, 127.9, 128.6, 130.2, 134.8, 140.7; MS: m/e: 273; Anal. Calcd. for C$_{12}$H$_{12}$BF$_4$NO: C, 52.74; H, 4.39; N, 5.12; Found: C, 52.68; H, 4.28; N, 5.02.

[BHPy]CF₃SO₃ 2c.   Yield: 0.52 g, 82%; Liquid; ^{1}H NMR (D$_2$O) δ: 4.60 (s, 2H); 6.94–8.09 (m, 9H); ^{13}C NMR δ: 54.2, 112.5, 115.4, 117.9, 127.8, 127.7, 128.7, 129.0, 134.6, 139.4; MS: m/e: 335; Anal. Calcd. for C$_{13}$H$_{12}$F$_3$NO$_4$S: C, 46.56; H, 3.58; N, 4.17; Found: C, 46.42; H, 3.42; N, 4.14.

[NBHPy]PF₆ 2d.   Yield: 0.56 g, 93 %; Mp.: 149–151°C; ^{1}H NMR (D$_2$O) δ: 5.28 (s, 2H); 7.20–7.70 (m, 9H); ^{13}C NMR δ: 72.4, 119.6, 127.8, 128.7, 129.5, 135.4, 138.6, 141.8, 142.9, 162.6; MS: m/e: 376; Anal. Calcd. for C$_{12}$H$_{11}$F$_6$ N$_2$O$_3$P: C, 38.29; H, 2.92; N, 7.44; Found: C, 38.08; H, 2.80; N, 7.32.

[NBHPy]BF₄ 2e.   Yield: 0.50 g, 98 %; Liquid; ^{1}H NMR (D$_2$O) δ: 5.34 (s, 2H); 7.19–7.76 (m, 9H); ^{13}C NMR δ: 72.1, 119.6, 127.6, 128.9, 129.5, 135.4, 138.2, 141.7, 142.7, 162.4; MS: m/e: 318.03 Anal. Calcd. for C$_{12}$H$_{11}$BF$_4$N$_2$O$_3$: C, 45.28; H, 3.45; N, 8.80; Found: C, 45.16; H, 3.36; N, 8.72.

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