Brønsted acidic ionic liquid-promoted direct C3-acylation of N-unsubstituted indoles with acid anhydrides under microwave irradiation†

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A green and efficient pathway for the synthesis of 3-acylindoles using a Brønsted acidic ionic liquid as a catalyst has been developed for the first time. The C3-acylation of N-unsubstituted indoles with acid anhydrides affords the desired products in good to excellent yields with high regioselectivity under microwave irradiation. Moreover, the Brønsted acidic ionic liquid can be recycled up to four times without significant loss of catalytic activity.

Results and discussion

Preparation of [[4-SO₃H]BMIM]HSO₄ ionic liquid

[[4-SO₃H]BMIM]HSO₄ ionic liquid has been prepared by two steps: formation of the zwitterionic intermediate and subsequent protonation by concentrated sulphuric acid. However, these methods suffered from some drawbacks such as low yields and long reaction times.† Thus, the ultrasound irradiation was chosen to improve the reaction efficiency for the synthesis of [[4-SO₃H]BMIM]HSO₄. It should be noted that the two-step procedure provided [[4-SO₃H]BMIM]HSO₄ in excellent yield within very short reaction time under solvent-free sonication (please see ESI, Tables S1 and S2). The structure of ionic liquid was characterized by NMR, FT-IR, and HR-MS (ESI). The ionic liquid is hygroscopic but moisture-stable and should be constantly stored in a desiccator.

Effect of reaction time and temperature for the C3-propionylation of indole

The catalytic activity of the prepared [[4-SO₃H]BMIM]HSO₄ was evaluated for the Friedel–Crafts acylation of indoles with acid anhydrides under solvent-free microwave irradiation. To optimize the reaction conditions, the reaction of indole and propionic anhydride under microwave irradiation was conducted as a model reaction. Initially, the Friedel–Crafts propionylation of indole in the presence of [[4-SO₃H]BMIM]HSO₄ (25 mol%) under microwave irradiation at 60 °C for 5 min was employed to prepare the desired product. To our surprise, the model reaction afforded the propionylindole in 46% yield with 100% regioselectivity in C3-position, and no impurity was found in TLC and GC (Table 1, entry 1). After screening the reaction

Introduction

Acylindoles are commonly found in many biologically active natural products and pharmaceutical compounds.1–3 Among these, 3-acylindole derivatives potentially show biological activities including anti-diabetic,7–9 anti-cancer,4,6 and anti-HIV.7–10 However, their synthesis remains challenging and includes N-protection,9 low yields,1,2,13 poor regioselectivity, excess reactants,14 high temperature, and long reaction time.11,15,16 Traditional methods involved the use of stoichiometric amounts of Lewis or Brønsted acids for the Friedel–Crafts acylation of indole.11,17,18 Recently, the preparation of 3-acylindoles has received much attention and metal-based catalytic pathways have been studied extensively.13,18 However, some of the existing methods are often harmful to the environment. Thus, developing new catalysts for the synthesis of 3-acylindoles with excellent yields and high selectivity under mild and environmentally friendly conditions is highly desirable.19

Ionic liquids have received much attention in many fields including catalysis,19–25 electrolytes,26–29 extraction desulfurization,30–32 biomass processing,33–35 and biodiesel.28–29 Friedel–Crafts C3-acylation of indoles using ionic liquids has been reported in previous literature.36,41 Recently, Brønsted acidic ionic liquids (ionic liquids with acidic properties) have been widely used for various organic transformations due to their unique properties.19,42–56 Nevertheless, to the best of our knowledge, they have never been used for the direct C3-acylation of N-unsubstituted indoles. Thus, we now report the development of a high-yielding and regioselectivity method for the C3-acylation N-unsubstituted indoles using Brønsted acidic ionic liquid as an efficiently homogeneous catalyst under microwave irradiation.

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† Electronic supplementary information (ESI) available. See DOI: 10.1039/c7ra11362e
In the presence of 25 mol% of [(4-SO$_3$H)BMIM]HSO$_4$, a 92% yield was observed for the desired product when a detectable catalyst absence was used (Table 2, entry 1). As only a small amount of catalyst was presented in Table 2, the desired product was not detected in the study. To examine the scope of reaction, the reactions of various indoles and a wide range of acid anhydrides were investigated.

### Effect of reaction time and amount of catalyst for the C3-propionylation of indole

Next, the amount of catalyst was varied from 5 to 100 mol% to ascertain the optimal conditions at 100 °C for 5 min (Table 1, entry 3). The control experiments were also carried out under conventional heating and ultrasound irradiation, but only a trace amount of 3-propionylindole was detected.

### Table 1: Optimization of the reaction conditions

| Entry | Time (min) | Temperature (°C) | Yield (%) | Selectivity (1:2:3) |
|-------|------------|------------------|-----------|---------------------|
| 1     | 5          | 60               | 46        | 0:0:100             |
| 2     | 5          | 80               | 79        | 1:0:99              |
| 3     | 5          | 100              | 92        | 0:0:100             |
| 4     | 5          | 120              | 93        | 2:0:98              |
| 5     | 5          | 140              | 90        | 2:0:98              |
| 6     | 1          | 100              | 62        | 0:0:100             |
| 7     | 3          | 100              | 78        | 1:0:99              |
| 9     | 7          | 100              | 90        | 2:1:97              |
| 10    | 10         | 100              | 91        | 3:1:96              |

| Reaction condition: indole (1.0 mmol), propionic anhydride (1.0 mmol), and [(4-SO$_3$H)BMIM]HSO$_4$ (25 mol%) under solvent-free microwave irradiation.

### Effect of acylating reagents for the C3-propionylation of indole

With optimal reaction conditions in hand, we extended the current method to other acid anhydrides such as acetic, butyric, isobutyric, pivalic, and benzoic anhydride. The results were presented in Table 2. The desired products were obtained in good to excellent yields (65–92%) with over 95% selectivity in C3-position. The low yields of pivalic and benzoic anhydride were obtained under current method presumably due to steric effect (Table 3, entries 5 and 6). Interestingly, no 1,3-diacylated and polymerized products were obtained under current method.

### Table 2: Effect of the catalytic amount

| Entry | [(4-SO$_3$H)BMIM]HSO$_4$ (mol%) | Yield (%) | Selectivity (1:2:3) |
|-------|---------------------------------|-----------|---------------------|
| 1     | 0                               | 0         | 0                   |
| 2     | 5                               | 35        | 0:0:100             |
| 3     | 15                              | 60        | 0:0:100             |
| 4     | 25                              | 92        | 0:0:100             |
| 5     | 50                              | 92        | 1:0:99              |
| 6     | 100                             | 93        | 2:1:97              |

| Reaction condition: indole (1.0 mmol), propionic anhydride (1.0 mmol) under solvent-free microwave irradiation at 100 °C for 5 min.

### Table 3: Effect of acylating reagents

| Entry | R–                  | Yield (%) | Selectivity (1:2:3) |
|-------|---------------------|-----------|---------------------|
| 1     | CH$_3$–              | 88        | 0:0:100             |
| 2     | C$_5$H$_5$–           | 92        | 1:0:99              |
| 3     | CH$_3$2CH$_2$–       | 91        | 1:0:99              |
| 4     | (CH$_3$)$_2$CH–      | 85        | 2:1:97              |
| 5     | (CH$_3$)$_3$C–       | 70        | 3:2:95              |
| 6     | C$_3$H$_7$–           | 65        | 1:0:99              |

| Reaction condition: indole (1.0 mmol), acid anhydrides (1.0 mmol), and [(4-SO$_3$H)BMIM]HSO$_4$ (25 mol%) under solvent-free microwave irradiation.

a Determined by GC-MS.

### Effect of substrate scope for the C3-acylation of indole derivative

To examine the scope of reaction, the reactions of various indoles and a wide range of acid anhydrides were investigated under current method. The results were presented in Table 4. A wide range of indoles bearing the substituent on the position C5 could proceed well and afford the corresponding products in good yields (Table 2, entries 6–20). Non-substituted indole and indoles bearing electron-donating groups such as 5-methylindole and 5-methoxyindole gave the desired products in high yields.
Table 4  [(4-SO$_3$H)BMIM]HSO$_4$-catalyzed acylation of free (NH) indoles with acid anhydrides under solvent-free microwave irradiation

| Entry | Indoles | Acid anhydrides | Condition | Products | Yield$^b$ (%) | Selectivity$^c$ (1 : 2 : 3) |
|-------|---------|-----------------|-----------|----------|----------------|-----------------------------|
| 1     | (CH$_3$CO)$_2$O | 100 °C, 8 min | | | 90 | 0 : 0 : 100 |
| 2     | (C$_2$H$_5$CO)$_2$O | 100 °C, 5 min | | | 92 | 1 : 0 : 99 |
| 3     | (n-C$_3$H$_7$CO)$_2$O | 100 °C, 10 min | | | 92 | 1 : 0 : 99 |
| 4     | (t-C$_4$H$_9$CO)$_2$O | 100 °C, 10 min | | | 82 | 1 : 0 : 99 |
| 5     | (C$_6$H$_5$CO)$_2$O | 100 °C, 10 min | | | 78 | 1 : 0 : 99 |
| 6     | (CH$_3$CO)$_2$O | 100 °C, 5 min | | | 92 | 4 : 0 : 96 |
| 7     | (t-C$_4$H$_9$CO)$_2$O | 100 °C, 5 min | | | 90 | 5 : 0 : 95 |
| 8     | (i-C$_3$H$_7$CO)$_2$O | 100 °C, 5 min | | | 89 | 5 : 0 : 95 |
| Entry | Indoles | Acid anhydrides | Condition | Products | Yield$^b$ (%) | Selectivity$^c$ (1 : 2 : 3) |
|-------|---------|----------------|-----------|----------|--------------|----------------------------|
| 9     | MeO ![Indole](image) | (CH$_3$CO)$_2$O | 100 °C, 5 min | ![Product](image) | 92 | 5 : 0 : 95 |
| 10    | ![Indole](image) | (i-C$_3$H$_7$CO)$_2$O | 100 °C, 5 min | ![Product](image) | 90 | 6 : 0 : 94 |
| 11    | ![Indole](image) | (CH$_3$CO)$_2$O | 100 °C, 10 min | ![Product](image) | 88 | 1 : 0 : 99 |
| 12    | ![Indole](image) | (C$_2$H$_5$CO)$_2$O | 100 °C, 10 min | ![Product](image) | 84 | 1 : 1 : 98 |
| 13    | ![Indole](image) | (CH$_3$CO)$_2$O | 100 °C, 10 min | ![Product](image) | 84 | 1 : 1 : 98 |
| 14    | ![Indole](image) | (C$_2$H$_5$CO)$_2$O | 100 °C, 10 min | ![Product](image) | 80 | 2 : 1 : 97 |
| 15    | ![Indole](image) | (n-C$_3$H$_7$CO)$_2$O | 100 °C, 10 min | ![Product](image) | 76 | 3 : 1 : 96 |

Table 4 (Contd.)
Table 4  (Contd.)

| Entry | Indoles          | Acid anhydrides | Condition       | Products | Yield\(^b\) (%) | Selectivity\(^c\) (1 : 2 : 3) |
|--------|-----------------|-----------------|-----------------|----------|----------------|------------------------------|
| 16     | \((t-C\textsubscript{3}H\textsubscript{7}CO)\textsubscript{2}O\) | 100 °C, 10 min  | 82              | 2 : 1 : 97 |
| 17     | \((t-C\textsubscript{4}H\textsubscript{9}CO)\textsubscript{2}O\) | 100 °C, 10 min  | 72              | 1 : 1 : 98 |
| 18     | \((C\textsubscript{6}H\textsubscript{5}CO)\textsubscript{2}O\) | 100 °C, 10 min  | 70              | 2 : 1 : 97 |
| 19     | \((C\textsubscript{2}H\textsubscript{5}CO)\textsubscript{2}O\) | 120 °C, 10 min  | 80              | 3 : 1 : 98 |
| 20     | \((t-C\textsubscript{4}H\textsubscript{9}CO)\textsubscript{2}O\) | 120 °C, 10 min  | 65              | 4 : 1 : 95 |
| 21     | \((C\textsubscript{2}H\textsubscript{5}CO)\textsubscript{2}O\) | 120 °C, 10 min  | 56              | 25 : 0 : 75 |
| 22     | \((t-C\textsubscript{4}H\textsubscript{9}CO)\textsubscript{2}O\) | 120 °C, 10 min  | 42              | 49 : 0 : 51 |

\(^a\) Reaction condition: indoles (1.0 mmol), acid anhydrides (1.0 mmol), and [(4-SO\textsubscript{3}H)BMIM]HSO\textsubscript{4} (25 mol%) under solvent-free microwave irradiation. \(^b\) Isolated yield. \(^c\) Determined by GC-MS.
excellent yields (Table 4, entries 1–10). Indoles bearing electron-poor group such as 5-fluoroindole, 5-chloroindole, 5-bromoindole were also reactive under high temperature and/or extended reaction times (Table 4, entries 11–19). The reaction was highly governed by steric effect as the reaction of 4-bromoindole with propionic and pivalic anhydride afforded the corresponding products in low yields and poor regioselectivity to C3-acylation (Table 4, entries 21–22).

Investigation on recycling of [(4-SO3H)BMIM]HSO4 ionic liquid for the C3-propionylation of indole

The reusability of [(4-SO3H)BMIM]HSO4 ionic liquid was carried out in the model reaction at 100 °C for 5 min under microwave irradiation. The results were reported in Table 5. Four consecutive runs were tested with less reduction in the catalytic activity. FT-IR spectra of fresh and recovered [(4-SO3H)BMIM]HSO4 confirmed that the structure of ionic liquid kept unchanged.

Mechanism of C3-acylation of indoles by [(4-SO3H)BMIM]HSO4 ionic liquid

Based on the previous literatures, we propose a plausible mechanism depicted in Scheme 1. First, the nucleophilic addition of indole at the C-3 position to acid anhydride in the presence of [(4-SO3H)BMIM]HSO4 provided the intermediate A. Next, the removal of carboxylic acid resulted in the formation of the intermediate B which can be subsequently undergo the elimination of proton to afford the desired product and regenerate [(4-SO3H)BMIM]HSO4 to finish the catalytic cycle.

| Table 5 Reusability of [(4-SO3H)BMIM]HSO4 ionic liquid for the synthesis of 3-propionylindole |
|-------|-------------------------------|-----------------|
| Run   | Isolated yield (%) | Selectivity (1 : 2 : 3) |
|-------|----------------|-------------------|
| 0     | 90             | 0 : 0 : 100       |
| 1     | 90             | 1 : 0 : 99        |
| 2     | 89             | 1 : 0 : 99        |
| 3     | 88             | 1 : 1 : 98        |
| 4     | 89             | 1 : 0 : 99        |

| Table 6 The comparative study of the current method with previous literaturesa |
|-------|-------------------------------|-----------------|----------------|-----------------|
| Entry | Catalyst                       | Acetylating reagent | Condition | Yield (%) | Selectivity (1 : 2 : 3) |
|-------|--------------------------------|---------------------|-----------|-----------|------------------------|
| 1     | ZrCl4 (1.5 equiv.) DCE, N2    |                     | 30 °C, 4 h | 67 (ref. 61) | nd                     |
| 2     | TfOH (0.15 equiv.)            |                     | 80 °C, 24 h | 64 (ref. 17) | nd                     |
| 3     | Y(OTf)3 (0.01 equiv.)/[BMI]BF4 (1.0 equiv.) |                     | 80 °C, 24 h | 88 (ref. 62) | 0 : 0 : 100             |
| 4     | Pd(OAc)2 (0.05 equiv.); 2,2′-bipyridine (6 mol%), CSA (0.6 mmol) |                     | 120 °C, 36 h | 84 (ref. 63) | nd                     |
| 5     | Hβ zeolite (0.05 g)           |                     | 120 °C, 8 min | 70 (ref. 64) | 9 : 0 : 77a            |
| 6     | Current work                  |                     | 100 °C, 8 min | 90         |                        |

a 1,3-Diacetylindole was obtained in 14% yield.
Comparison of [(4-SO3H)BMIM]HSO4 ionic liquid with other previous catalysts

A comparison of current work with previous literatures is presented in Table 6. The acetylation of indole with acetic anhydride in the presence of [(4-SO3H)BMIM]HSO4 provided the 3-acetylimidazole in higher yield within shorter reaction time. Moreover, the [(4-SO3H)BMIM]HSO4 ionic liquid was easily recovered and reused several times without decrease in catalytic activity.

Experimental

Chemicals, supplies, and instruments

All starting materials were purchased from Sigma-Aldrich and immediately used without further purification. Silica gel 230–400 mesh (for flash chromatography) and TLC plates were obtained from Merck. Microwave irradiation was used on a CEM Discover BenchMate. GC-MS spectra were performed on an Agilent GC System 7890 equipped with a mass selective detector Agilent 5973N. FT-IR spectra were analyzed by a Bruker Vertex 70. 1H and 13C NMR spectra were recorded on a Bruker Advance 500. HRMS (ESI) data were performed on Bruker microTOF-QII MS at 80 eV (please see in ESI† Section S1).

Preparation of [(4-SO3H)BMIM]HSO4 catalyst under solvent-free sonication

The two-step procedure for the synthesis of ionic liquids from 1-methylimidazole, 1,4-butanetone, and sulfuric acid under ultrasound irradiation were reported in the Section S2 (please see in ESI†).

General procedure for Friedel–Crafts acylation of indole

Indole (1.0 mmol, 0.117 g), propionic anhydride (1.0 mmol, 0.130 g) and [(4-SO3H)BMIM]HSO4 (25 mol%) was heated under microwave irradiation at 100 °C for 5 min in a CEM Discover apparatus. The completion of the reaction was checked by TLC and GC. The mixture was then extracted with diethyl ether (5 × 5 mL). The organic layer was decanted, washed with aqueous NaHCO3 (2 × 15 mL), water (15 mL) and brine (15 mL), and dried with Na2SO4. The solvent was removed under vacuum. The crude product was purified by silica gel chromatography using ethyl acetate–hexane (1 : 9). The purified product was then characterized by 1H and 13C NMR, GC-MS or HR-MS (ESI).

Recycling of [(4-SO3H)BMIM]HSO4

The recycling of [(4-SO3H)BMIM]HSO4 was also carried out under microwave irradiation in the model reaction between indole and propionic anhydride. After completion of the reaction, diethyl ether was used to extract the reaction mixture until both starting materials and products were entirely separated from the ionic liquid. The recovered [(4-SO3H)BMIM]HSO4 was immiscible in diethyl ether was dried under vacuum at 80 °C for 60 min. The recycled [(4-SO3H)BMIM]HSO4 was used for four consecutive runs. Remarkably, the yield of the product decreased slightly after each run. The simple process of recycling [(4-SO3H)BMIM]HSO4 could be potentially developed on a large scale.

Conclusions

In summary, we have developed a green and efficient method using [(4-SO3H)BMIM]HSO4 as an effective catalyst for the highly regioselective C3-acylation of N-unsubstituted indoles with acid anhydrides. Remarkably, the accelerated microwave-assisted Friedel–Crafts acylation catalyzed by [(4-SO3H)BMIM]HSO4 was reported for the first time. Furthermore, the [(4-SO3H)BMIM]HSO4 possesses several advantages including mild preparation from commercially available materials, easy handling, and recyclability without loss of reactivity. Finally, it provides a simple, facile, and efficient alternative to the existing synthetic methods of 3-acylimidazoles.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

The Vietnam National Foundation for Science and Technology Development (NAFOSTED) is acknowledged for financial support through project code 104.01-2016.59. We thank Duc-Khiem Nguyen Chau, Hoang-Tan Le Doan (VNU-HCM), and Ngoc-Mai Hoang Do (IPH-HCM) for their valuable discussions.

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