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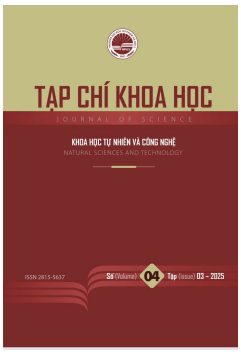

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The efficient and mild synthesis of phenacyl bromide *via* bromination using *N*-bromosuccinimide catalyzed by brønsted acidic ionic liquid under ultrasound irradiation

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Abstract

Phenacyl bromide is an important substrate for the synthesis of various biologically active heterocyclic compounds and industrial chemicals. Synthesis of phenacyl bromide via the bromination of the alpha carbon in acetophenone by using N-bromosuccinimide has been carried out in the presence of 1-(4-sulfobutyl)-3-methylimidazolium hydrogen sulfate (BAIL). Under the ultrasound irradiation, the yield of phenacyl bromide and 2,2-dibromo-2-phenylethanone has been achieved at 84% and 11%, respectively, in a very short time, ten minutes. Additionally, 1-(4-sulfobutyl)-3-methylimidazolium hydrogen sulfate was recovered and reused five times, without significantly reducing the yield of phenacyl bromide. This work has developed an approach that not only improves reaction time and product selectivity but also aligns with 'green chemistry' principles by employing a recoverable and reusable catalyst.

Keywords: phenacyl bromide, BAIL, ultrasound irradiation, bromination, N-bromosuccinimide

1. Introduction

Phenacyl bromide is a key reagent in organic synthesis, frequently employed as a precursor for various biologically active heterocyclic compounds [1], [2]. Due to its electrophilic nature, the bromoacetyl moiety readily undergoes nucleophilic substitution reactions [3]. In pharmaceutical chemistry, phenacyl bromide serves as a starting material for producing antibiotics, antifungal agents, and

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other bioactive compounds [1], [2]. It also plays a crucial role in the formation of ionic liquid precursors [4], [5] and polymeric materials [6], [7].

The synthesis of phenacyl bromide has been studied with several different starting substrates such as aryl ketones, secondary alcohols, haloalkenes, haloalkanes, haloalcohols, and 1-haloalkynes [8]. Among these, acetophenone is one of the most widely recognized substrates for the synthesis of phenacyl bromide through halogenation of the alpha carbon. A variety of methodologies have been employed, such as bromine (Br_2) in acidic condition [9], the reaction of NBS (*N*-Bromosuccinimide) in the presence of acid catalyst like *p*-TsOH [10], $NaHSO_4/SiO_2$ [11], urea-hydrogen peroxide–1-butyl-3-methylimidazolium tetrafluoroborate (UHP–BMIM[BF_4]) [12], $Cu(OTf)_2$ [13], 1-butyl-3-methylimidazolium hexafluorophosphate (BMIM[PF_6]) [14], 1-methyl-3-(4-sulfobutyl)imidazolium triflate (BMIM(SO_3H)[OTf]) [15], or nano TiO_2 [16], and the reaction of HBr with H_2O_2 [17], [$EtNH_3^+$][NO_3^-] [18], DMSO [19], $NaNO_2$-KI system [20], and $Cu(NO_3)_2$ [21]. Another synthesis of α-halo carbonyls using metal halides $CuBr_2$ [22], [hydroxy(tosyloxy)iodo]benzene with $MgBr_2$ [23], $(COBr)_2$ with DMSO [24] bis(1,3-dimethyl-2-imidazolidinone)hydrotribromide (DITB),[25] have also been investigated.

In continuation of our work on homogeneous acidic catalyst combined with ultrasonic irradiation,[26] an efficient and mild method using 1-(4-sulfobutyl)-3-methylimidazolium hydrogen sulfate as a catalyst was employed for the bromination of acetophenone using NBS under ultrasound irradiation with respect that reducing the reaction time, green activation, and good catalyst recycles (Figure 1).

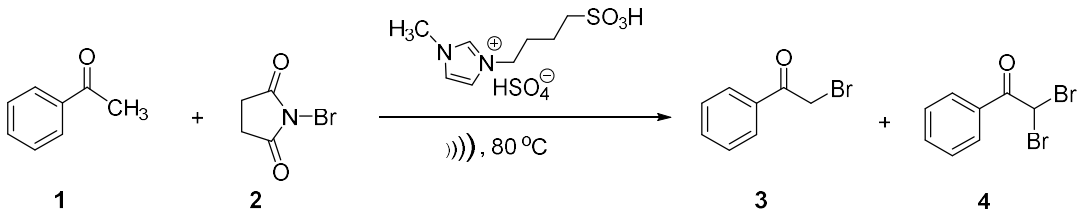

Figure 1. The bromination of acetophenone with N-Bromosuccinimide promoted by a Brønsted acidic ionic liquid.

2. Experimental

2.1. Materials and Methods

Chemicals, including acetophenone (purity ≥ 98%), 1-methylimidazole (purity ≥ 99%), 1,4-butane sultone (purity ≥ 99%), *N*-bromosuccinimide (purity ≥ 99%), Amberlyst 15, and Montmorillonite K10, were purchased from Thermo Fisher Scientific and Sigma-Aldrich. The reaction was conducted in an Elma S30H ultrasonic bath (frequency 30 kHz). High-performance liquid chromatography with UV/Vis detection (HPLC-UV/Vis) was performed using a Phenomenex Ultracarb 5 µm ODS (30) column (150 x 4.6 mm). Nuclear magnetic resonance (NMR) spectra were obtained on a Bruker Avance II at 500 MHz for ^{1}H-NMR and 125 MHz for ^{13}C-NMR. The melting point was determined using a Büchi melting point analyzer.

2.2. Preparation of 1-(4-sulfobutyl)-3-methylimidazolium hydrogen sulfate

1-Methylimidazole (1.5 mmol, 0.1230 g) and 1,4-butane sultone (1.5 mmol, 0.2040 g) were added to a 5 mL flask and subjected to ultrasound irradiation in an 80 °C bath for 5 minutes. A white solid (zwitterion) formed, which was washed with diethyl ether (6×5 mL). The residual solvent was removed by rotary evaporation at 80 °C for 30 minutes. Then, 98% H_2SO_4 (1.5 mmol, 0.1470 g) was added to the

flask, and the reaction continued under ultrasound irradiation at 60 ºC for 60 minutes. After completion, the reaction mixture was washed with diethyl ether (6×5 mL), and the solvent was evaporated by rotary evaporation, yielding a clear, light-yellow liquid. The structure of the BAIL catalyst was confirmed by using ^{1}H NMR and found to be compatible with previous literature [27]. 1-(4-Sulfobutyl)-3-methylimidazolium hydrogen sulfate. ^{1}H-NMR (500 MHz, D_2O) δ (ppm) = 8.51 (s, 1H), 7.28 (t, J = 2.5 Hz, 1H), 7.22 (t, J = 2.5 Hz, 1H), 4.03 (t, J = 9.0 Hz, 2H), 3.68 (s, 3H), 2.73 (t, J = 9.5 Hz, 2H), 1.81 (p, J = 9.0 Hz, 2H), 1.55–1.50 (m, 2H). ^{13}C-NMR (125 MHz, D_2O) δ (ppm) = 135.8, 123.5, 122.0, 49.9, 48.8, 35.5, 27.9, 20.8.

2.3. Procedure for the bromination of acetophenone

BAIL (0.25 mmol, 0.0788 g), acetophenone (1.5 mmol, 0.1802 g), and *N*-bromosuccinimide (1.65 mmol, 0.2669 g) were sequentially added to a 5 mL flask. The mixture was then subjected to an 80 °C ultrasound bath for the appropriate reaction time. After completion, 5 mL of the ethyl acetate-water mixture (50% v/v) was added to the reaction flask, and then the two layers were separated by a separating funnel. The aqueous layer was continuously extracted with ethyl acetate (3×20 mL). Subsequently, all combined extracts were rinsed with 10% Na_2CO_3 solution (2×5 mL) and water (3×30 mL) until pH reached 7, then dried over anhydrous Na_2SO_4. The solvent was removed by rotary evaporation to yield the crude product, which was subsequently purified by column chromatography using *n*-hexane as eluent to obtain pure products. The structure of pure products was identified by using ^{1}H NMR and found to be compatible with the previous reports as follows:

Phenacyl bromide, or 2-bromo-1-phenylethanone (3), is a white solid with a melting point at 49.8–50.2ºC (lit. 46.8–48.2°C).[14] ^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm) = 7.99 (dd, J = 10.5 Hz, J = 2.0 Hz, 2H), 7.62 (tt, J = 9.5 Hz, J = 2.0 Hz, 1H), 7.50 (tt, J = 9.5 Hz, J = 2.0 Hz, 2H), 4.46 (s, 2H) [15]. ^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm) = 191.4, 134.1 (2C), 129.1 (2C), 129.0 (2C), 31.0. HR–ESI–MS: *m/z* $[M + Na]^+$ calcd for $[C_8H_7BrNaO]^+$: 220.9572 and 222.9552; found: 220.9573 and 222.9544.

2,2-Dibromo-2-phenylethanone (4) is a transparent light-yellow liquid. ^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm) = 8.09 (dd, J = 10.0 Hz, J = 2.0 Hz, 2H), 7.64 (tt, J = 9.5 Hz, J = 2.0 Hz, 1H), 7.51 (tt, J = 10.0 Hz, J = 2.0 Hz, 2H), 6.71 (s, 1H).[15] ^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm) = 186.1, 134.6, 131.0, 129.8 (2C), 129.1 (2C), 39.8.

3. Results and Discussion

At the beginning of this work, several acidic catalysts were chosen randomly to be used for the bromination of acetophenone under ultrasound irradiation at room temperature (Table 1). As a result, solid acid catalysts, *e.g,* Amberlyst 15, zeolite, and Mont K-10, did not promote but even prohibited the reaction from producing phenacyl bromide, in comparison with that under catalyst-free conditions. This matter could be explained by obstacles in the interaction of reagents, which were caused by zeolite, Mont K-10, under solvent-free conditions. Among the homogeneous catalysts applied, BAIL, a Brønsted acidic catalyst, was evaluated as a better catalyst to improve the yield of phenacyl bromide than choline chloride•$ZnCl_2$, a Lewis acidic catalyst.

Table 1. The nature of the catalysts influenced the alpha carbon bromination of acetophenone.[a]

Entry	Catalyst	Solvent	Yield[b] (LC[c]) %	
			3	4
1	None	-	40 (48)	3 (5)
2	Amberlyst 15 (0.2 g)	CH_3CN (5 mL)	39 (57)	3 (6)
3	Amberlyst 15 (0.2 g)	EtOH-H_2O (3:1, 5 mL)	37 (69)	12 (31)
4	Amberlyst 15 (0.2 g)	-	42 (72)	4 (9)
5	Zeolite (0.2 g)	-	5 (9)	2 (4)
6	Montmorillonite K10 (0.2 g)	-	1 (2)	0 (0)
7	Choline Chloride•$ZnCl_2$ (1:2, 0.5 mmol, 0.206 g)	-	25 (35)	13 (26)
8	BAIL (0.5 mmol, 0.158 g)	-	76 (78)	9 (12)

[a] The reaction of acetophenone (1,5 mmol) and NBS (1,8 mmol) was carried out in the presence of various acidic catalysts under ultrasound irradiation at room temperature for 3 hours.
[b] Yields were calculated based on HPLC-UV/Vis analyses.
[c] LC: The composition was measured by the HPLC-UV/Vis method.

After choosing BAIL as the best catalyst for the α–bromination of acetophenone, the reaction temperature was the next factor investigated and was varied at room temperature, 60 °C, and 80 °C. The results in Table 2 showed that increasing the temperature led to an increase in the yield of phenacyl bromide from 76 to 84% (Entries 1–3, Table 2). Since the reaction reached 100% conversion at 180 minutes, the reaction time should be reduced. Shortening the reaction time from 180 minutes to 5 minutes did not cause any significant changes in product ratios and yields; therefore, a reaction time of 10 minutes was chosen as the ideal time to achieve a complete conversion (100%) and used for further investigation.

Table 2. The influence of temperature and reaction time on the bromination of acetophenone.[a]

Entry	Temperature (°C)	Time (minutes)	Yield[b] (LC[c]) %	
			3	4
1	Room temperature	180	76 (78)	9 (12)
2	60	180	77 (75)	16 (22)
3	80	180	84 (82)	14 (18)
4	80	120	80 (80)	15 (20)
5	80	60	78 (83)	12 (17)
6	80	30	77 (77)	16 (23)
7	80	10	79 (77)	17 (23)
8	80	5	79 (77)	15 (21)

[a] The reactions of acetophenone (1.5 mmol) with NBS (1.8 mmol) were performed in the presence of BAIL (0.5 mmol) under ultrasound irradiation.
[b] Yields were calculated based on HPLC-UV/Vis analyses.
[c] LC: The composition was measured by the HPLC-UV/Vis method.

A subsequent series of experiments was conducted to investigate the influence of BAIL catalyst amount on the reaction, with the results summarized in Figure 2. Notably, increasing the catalyst amount over 0.25 mmol resulted in a decrease in the yields of phenacyl bromide. This phenomenon could be explained by the larger amount of BAIL, which caused a further bromination of the phenacyl bromide and formed 2,2-dibromo-1-phenylethanone under ultrasonic irradiation. The value of 0.25 mmol was chosen as a suitable quantity for further experiments, owing to its good effectiveness on the yield and selectivity of phenacyl bromide.

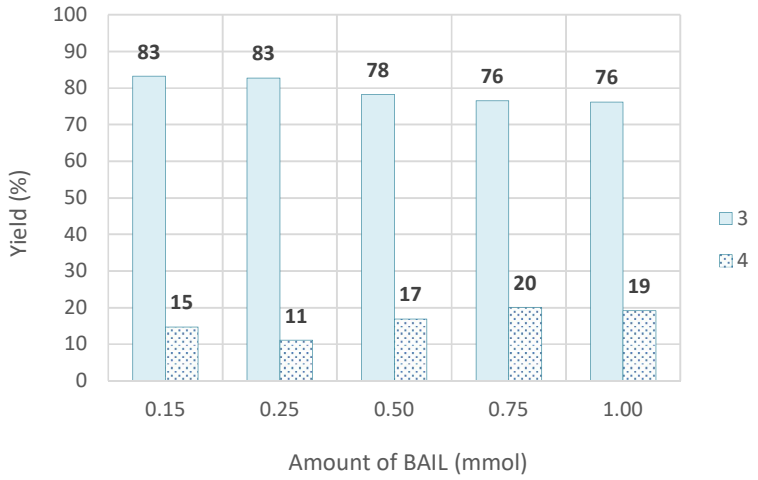

Figure 2. Influences of the amount of BAIL catalyst on the α–bromination of acetophenone (1.50 mmol) and NBS (1.80 mmol) under ultrasound irradiation at 80 oC in 10 minutes.

Next, the molar ratios of acetophenone and NBS were also paid attention to and studied by changing values as described in Figure 3. Consequently, both molar ratios of acetophenone and NBS, being 1.50 mmol:1.80 mmol and 1.50 mmol:1.65 mmol, resulted in complete reaction conversion (LC = 100%) and a good yield of phenacyl bromide at 84%. Therefore, the optimal ratio for acetophenone and NBS was determined to be 1.50 mmol:1.65 mmol.

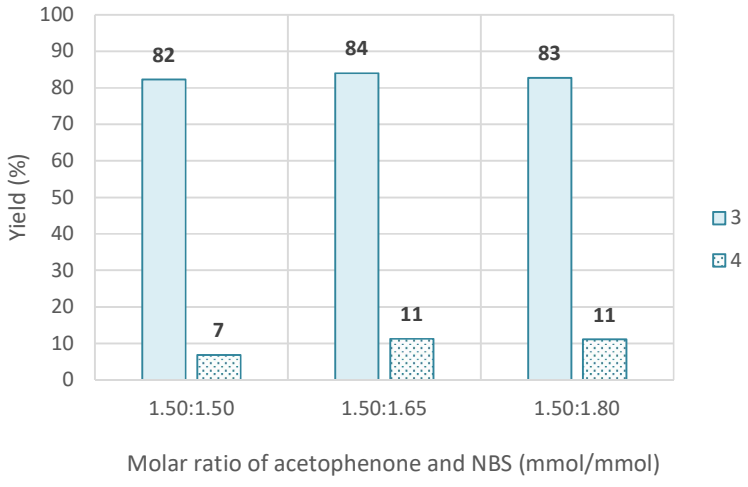

Figure 3. Influences of the molar ratio of acetophenone and NBS in the synthesis of phenacyl bromide catalyzed by BAIL (0.25 mmol) under ultrasound irradiation at 80 ºC in 10 minutes.

To enhance its efficiency in the reaction, the recovery and reusability of BAIL were examined as part of the reaction's sustainability. After extracting with ethyl acetate, the aqueous phase was evaporated using a rotary evaporator at 80°C for 30 minutes, yielding a viscous liquid with an average recovered yield of approximately 83%. This recovered ionic liquid could be used immediately or stored in a desiccator for subsequent experiments. The recycled capability was evaluated in a solvent-free reaction between acetophenone and NBS under ultrasound irradiation at 80°C for 10 minutes, following the previously optimized conditions. The results, presented in Figure 4, demonstrate the effectiveness of BAIL after five recycles without dropping the yield of phenacyl bromide considerably; however, BAIL's appearance had changed from a transparent, light-yellow liquid to a dark-brownish liquid at the fifth recycle.

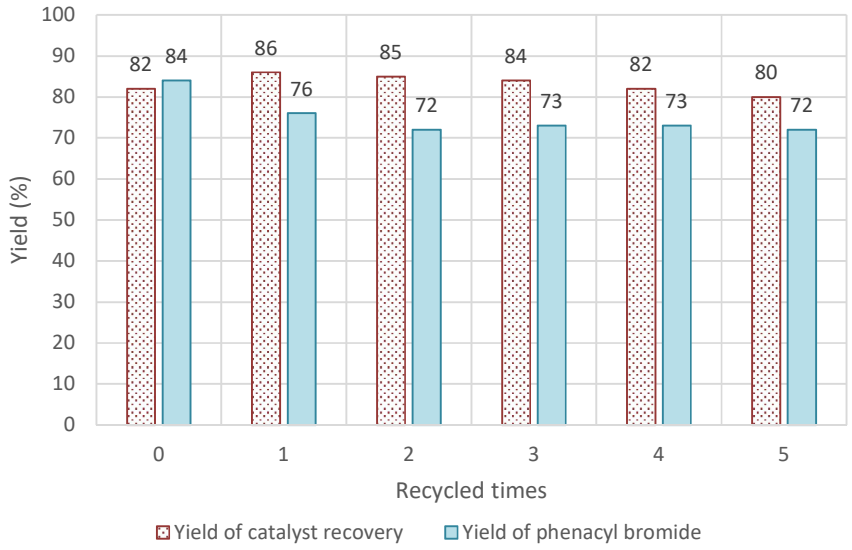

Figure 4. The reusability and recovery of the catalyst BAIL in the alpha carbon bromination of acetophenone.

Based on the previous literature on the synthesis of phenacyl bromide, several reported protocols required non-recyclable catalysts and hazardous organic solvents as reaction media [9]–[13], [16]–[25] Moreover, harmful or synthetically demanding brominating agents were utilized, such as Br_2 [9], HBr [17] –[21] $(COBr)_2$ [24], and DITB [25]. Several of these methods also involved complicated procedures, requiring specialized equipment or harsh conditions [9], [13], [16]–[18], [20], [21], [23], [24]. Overall, most of the reported reactions were time-consuming [9]–[15], [17]–[22], [24], [25] and energy-intensive [16, 23]. In summary, our reported method demonstrated a highly efficient and sustainable approach: the Brønsted acidic ionic liquid (BAIL) served as a reusable catalyst, enabling the reaction to proceed rapidly with high yields in the presence of ultrasound irradiation.

4. Conclusions

The synthesis of phenacyl bromide via the bromination of acetophenone using 1-(4-sulfobutyl)-3-methylimidazolium hydrogen sulfate as a catalyst under ultrasound irradiation demonstrated remarkable advantages over traditional methods. This procedure was not only performed in a significantly shorter time, with low energy requirements and simple operation, but also achieved high product selectivity and yield. Optimal reaction conditions consisting of acetophenone (1.50 mmol), NBS (1.65 mmol), and BAIL (0.25 mmol) have been found under ultrasonic irradiation at 80 °C for 10 minutes, yielding 84% of

phenacyl bromide. Notably, the recycling ability of the BAIL has been illustrated through five-time recycling without a significant reduction of the phenacyl bromide.

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