STUDY OF USING [BMIM][PF₆] IONIC LIQUID AS A GREEN SOLVENT AND CATALYST FOR THE SCHIFF BASE SYNTHESIS

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ABSTRACT

The [BMIM][PF₆] ionic liquid that derived from dialkylimidazolium cations was synthesized using MW-assisted method under solvent-free condition. The synthesis of paraanisidineacetylacetone was then conducted in [BMIM][PF₆] ionic liquid as solvent with good conversion after 6 hours under ambient condition without added catalyst. Interestingly, the recycle of [BMIM][PF₆] ionic liquid was performed. The results showed that [BMIM][PF₆] ionic liquid can be recovered and successfully recycled into subsequent reactions without significant loss of activity.

Keywords: Ionic liquids, dialkylimidazolium, [BMIM][PF₆], *p*-anisidineacetylacetone, Schiff Base.

1. INTRODUCTION

Due to the environmental concerns, there has been an increasing interest in exploiting the application of ionic liquids as the green reaction medium alternatives to hazardous organic solvents. This claim rests on the fact that ionic liquids offer a great number of unique properties not achievable with any other organic solvent such as low volatility, negligible vapor pressure, high thermal stability, outstanding reusability and ease of separation of products from reactants. Room temperature ionic liquids, especially those containing the 1-alkyl-3-methylimidazolium cation, have been considered as a promising solvents due to their unique properties including non-volatility, non-flammability, high conductivity, high chemical and thermal stability, etc [1-3]. 1-alkyl-3-methylimidazoliumbased ionic liquids have been widely applied as greener solvents for a range of organic conversions, providing not only acceleration of the reaction rate and improvement of selectivity to desired products but also facilitation of product separation and solvent recycling [2, 4-5]. The promoting role of 1-alkyl-3-methylimidazolium-based ionic liquids in several organic transformations have been demonstrated by our group [6-8]. In our previous research, imidazolium-based ionic liquid was performed as an effective solvent for the Paalknorr reaction of 1,4-hexanedione and several aliphatic amines [9]. As a continuation of this potential orientation, herein, the using of ionic liquids as green solvents and catalyst for the Schiff base synthesis has been reported.

2. EXPERIMENT AND METHOD

2.1. Material and instrumentation

All chemicals were purchased from Sigma-Aldrich co and Merck, and used as received without further purification. ¹H and ¹³C NMR spectra were recorded using a Bruker av 500 spectrometer. Mass spectra were recorded using a thermos Finnigan tsq7000 triple quadrupole. Gas chromatographic (GC) analyzes were performed using a Shimadzu gc 17-a equipped with a flame ionization detector (FID) and an DB-5 column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μ m). The temperature program for GC analysis heated samples from 100 to 110 °C at 30 °C /min and held them at 110 °C for 1 min; then heated them from 110 to 200 °C at 45 °C/min; then heated them from 200 to 300 °C at 50 °C/min and held them at 300 °C for 3 min. Inlet and detector temperatures were set constant at 300 °C. Products were used as an internal standard to calculate reaction conversions. GC-MS analyzes were performed using a Hewlett Packard GC-MS 5972 with a RTX-5MS column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.5 μ m). The temperature program for GC-MS analysis heated samples from 60 to 280 °C at 10 °C/min and held them at 280 °C for 2 min. Inlet temperature was set constant at 280 °C. MS spectra were compared with the spectra gathered in the NIST library.

2.2. Synthesis of ionic liquid

The ionic liquid was synthesized according to previously reported procedures. In view of the green chemistry, it was decided to explore the synthesis of 1-butyl-3-methylimidazolium bromide ([BMIM][Br]) using microwave irradiation under solvent-free condition [10-12]. The anion metathesis reaction of 1-butyl-3-methylimidazolium bromide with hexafluorophosphoric acid was then carried out to prepare 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF₆]), according to a literature procedure (Scheme 1) [13]. 1-Hexyl-3-methylimidazolium hexafluorophosphate ([HMIM][PF₆]), and 1-octyl-3-methylimidazolium hexafluorophosphate ([OMIM][PF₆]) were also synthesized using similar procedure. The ionic liquids were characterized using ¹H and ¹³C-NMR, and MS, which were in good agreement with the literature.

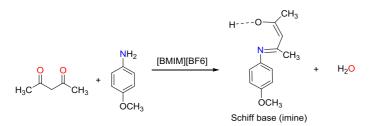
$$H_3C_N$$
 + RBr MW $\left[H_3C_N + N^R \right]^+ Br^-$

 $(R = C_4H_9, C_6H_{13}, C_8H_{17})$

Scheme 1. The synthesis of the 1-alkyl-3-methylimidazolium bromide ionic liquid [10-12]

2.3. General procedure for the Schiff base formation

Acetyl acetone (0.2 mL, 2 mmol) was placed into a round bottom flask of 25 mL, then 0.2 mL para-xylene was added as an internal standard in [BMIM][PF₆] solvent (0.5 mL), followed by adding recrystallized *p*-anisidine (0.35 g, 2.4 mmol) into the flask. Subsequently, the resulting mixture was stirred at room temperature. Reaction conversions were monitored by withdrawing aliquots (0.05 mL) from the reaction mixture at different time intervals. The organic components were extracted into diethyl ether (1 mL), dried over Na₂SO₄ and analyzed by GC with reference to para-xylene. Product identity was also further confirmed by GC-MS.



Scheme 2. The Schiff base formation of p-anisidine and acetylacetone

For investigation of $[BMIM][PF_6]$ recycling, after the reaction, the resulting mixture was cooled to room temperature and extracted with diethyl ether (5 x 10 mL) to remove the organic components. The ionic liquid layer was evaporated under vacuum (50 °C, 10 mmHg) for 1 hour to remove any excess solvent and then reused in further reaction under identical conditions to those of the first run.

3. RESULT AND DISCUSSION

The [BMIM]Br ionic liquid was synthesized *via* the microwave-assisted *N*-alkylation reaction of 1-methylimidazole with 1-bromobutane under solvent-free condition as previously reported (Scheme 1). 1-Hexyl-3-methylimidazolium ([HMIM]Br) and 1-octyl-3-methylimidazolium ([OMIM]Br) were also synthesized using similar procedure [6, 14]. The ionic liquids were characterized using spectroscopic techniques of ¹H and ¹³C-NMR, and MS, the data of which were consistent with the literature [6-8].

¹**H** NMR (500 MHz, DMSO): $\delta = 0.887$ (t, 3H; CH₃), 1.256 (m, 2H; CH₂CH₃), 1.770 (m, 2H; CH₂CH₂CH₃), 3.882 (s, 3H; N-CH₃), 4.204 (m, 2H; N-CH₂), 7.778 (t, 1H; N-CH=C), 7.856 (t, 1H; N-CH=C), 9.340 (s, 1H, N-CH=N). ¹³C NMR (125 MHz, DMSO): $\delta = 13.173$ (C-CH₃), 18.652 (CH₂), 31.279 (CH₂), 35.693 (N-CH₃), 48.357(N-CH₂), 122.172 (C=C-N), 123.461 (C=C-N), 136.435 (N-C=N). MS (ESI): m/z 139 [BMIM]⁺, 357 [(BMIM)₂Br]⁺.

¹**H** NMR (500 MHz, DMSO): $\delta = 0.848$ (t, 3H, CH₃); 1.256 (m, 6H, CH₂CH₂CH₂); 1.775 (m, 2H, CH₂); 3.863 (s, 3H, N–CH₃); 4.171 (t, 2H,N–CH₂); 7.732 (m, 1H, N–CH=C); 7.807 (m, 1H, N–CH=C); 9.240 (s,1H, N–CH=N). ¹³C NMR (125 MHz, DMSO): $\delta = 13.173$ (C–CH₃); 21.773 (CH₂); 25.049 (CH₂); 29.267 (CH₂); 30.455 (CH₂); 35.705 (N–CH₃); 48.687 (N–CH₂); 122.189(C=C–N); 123.504 (C=C–N); 136.442 (N–C=N). MS (ESI): m/z = 167 [HMIM]⁺; m/z = 413 [(HMIM)₂Br]⁺

¹**H NMR** (500 MHz, DMSO): $\delta = 0.840$ (t, 3H, CH₃); 1.245 (m, 10H, CH₂CH₂CH₂CH₂CH₂CH₂); 1.775 (m, 2H, CH₂); 3.870 (s, 3H, N–CH₃); 4.178 (t, 2H,N–CH₂); 7.756 (m, 1H, N–CH=C); 7.830 (m, 1H, N–CH=C); 9.295 (s,1H,N–CH=N). ¹³C NMR (125 MHz, DMSO): $\delta = 13.807$ (C–CH₃); 21.933 (CH₂); 25.389 (CH₂); 28.232 (CH₂); 28.357 (CH₂); 35.684 (N–CH₃); 48.648 (N–CH₂); 122.167(C=C–N); 123.464 (C=C–N); 136.437 (N–C=N). MS (ESI): m/z = 195 [OMIM]⁺; m/z = 469 [(OMIM)₂Br]⁺

The synthesis of three imidazolium based ILs, namely, 1-butyl-3-methyl-imidazolium hexafluorophosphate, [BMIM][PF₆], 1-hexyl-3-methyl-imidazolium hexafluorophosphate, [IMIM][PF₆], 1-octyl-3-methyl-imidazolium hexafluorophosphate, [OMIM][PF₆] was carried out through two consecutive steps. Especially, the process became easier and faster with the support of MW compared to that of conventional heating method. It was found that in the first stage, the increasing the length of alkyl chain led to a slight decrease in reaction yield, [BMIM][Br] achieved 96.07% compared to 92.13% [HMIM][Br] and 91.47% [OMIM][Br]. While in the second stage, the reaction yield reduced as the alkyl chain increased, [BMIM][PF₆] synthesized with the higher yield 90.91% compared to 85.20% in case of [HMIM][PF₆] and 78.36% [OMIM][PF₆].

¹H NMR (500 MHz, DMSO-d6): δ = 0.905 (t, 3H; CH₃), 1.262 (m, 2H; CH₂CH₃), 1.771 (m, 2H; CH₂CH₂CH₃), 3.846 (s, 3H; N-CH₃), 4.157 (m, 2H; N-CH₂), 7.668 (t, 1H; N-CH=C), 7.733 (t, 1H; N-CH=C), 9.071 (s, 1H, N-CH=N). ¹³C NMR (125 MHz, DMSO-d6): δ = 13.141 (C-CH₃), 18.711 (CH₂), 31.276 (CH₂), 35.651 (N-CH₃), 48.509 (N-CH₂), 122.193 (C=C-N), 123.542 (C=C-N), 136.444 (N-C=N). MS (ESI): *m*/*z* 139 [BMIM]⁺, 423 [(BMIM)₂PF₆]⁺.

¹H NMR (500 MHz, DMSO-d6) for [HMIM][PF₆]: $\delta = 0.873$ (t, 3H; *CH*₃), 1.272 (m, 6H; *CH*₂*CH*₂*CH*₂), 1.783 (m, 2H; *CH*₂), 3.846 (s, 3H; N-*CH*₃), 4.149 (m, 2H; N-*CH*₂), 7.7665 (t, 1H; N-*CH*=C), 7.734 (t, 1H; N-*CH*=C), 9.069 (s, 1H, N-*CH*=N). ¹³C NMR (125 MHz, DMSO-d6): $\delta = 13.708$ (C-*CH*₃), 21.799 (*CH*₂), 25.085 (*CH*₂), 29.266 (*CH*₂), 30.487 (*CH*₂), 35.651 (N-*CH*₃), 48.789 (N-*CH*₂), 122.191 (*C*=C-N), 123.540 (C=*C*-N), 136.436 (N-*C*=N). MS (ESI): *m/z* (%) 167 [HMIM]⁺, 479 [(HMIM)₂PF₆]⁺.

¹H NMR (500 MHz,DMSO-d6) for [OMIM][PF₆] : δ = 0.860 (t, 3H; CH₃), 1.265 (m, 10H; CH₂CH₂CH₂CH₂CH₂), 1.780 (m, 2H; CH₂), 3.845 (s, 3H; N-CH₃), 4.145 (m, 2H; N-CH₂), 7.674 (t, 1H; N-CH=C), 7.741 (t, 1H; N-CH=C), 9.076 (s, 1H, N-CH=N). ¹³C NMR (125 MHz, DMSO-d6): δ = 13.870 (C-CH₃), 22.390 (CH₂), 26.085 (CH₂), 28.772 (CH₂), 28.841 (CH₂), 30.137 (CH₂), 31.495 (CH₂), 36.642 (N-CH₃), 50.003 (N-CH₂), 121.860 (C=C-N), 123.641 (C=C-N), 137.076 (N-C=N). MS (ESI): *m*/*z* 195 [OMIM]⁺, 535 [(OMIM)₂PF₆]⁺

The imidazolium based ionic liquid was evaluated for its suitability as a solvent for the Schiff base formation of acetylacetone with *p*-anisidine to form *p*-anisidineacetylacetone as the major product (Scheme 2). The reactions were carried out at room temperature in 6 hours in order to examine its conversion. With the aim of obtaining a higher conversion, the effect of reaction conditions on the conversions should be investigated. The formation of Schiff base p-anisidine acetylacetone based on the reaction of p-anisidine with acetylacetone adopted as the model reaction and the molar ratio of two reactants acetylacetone: p-anisidine, the molar ratio of ionic liquid to acetylacetone and ionic liquids with various alkyl groups as well as different kinds of substituent on the reaction conversions were studied. As a result, the formation of p-anisidineacetlacetone was performed in [BMIM][PF₆] with the molar ratio of acetylacetone: p-anisidine and [BMIM][PF₆]: acetylacetone are 1:1.2 and 1.2:1 respectively, affording a reaction conversion of 99.21% obtained after 6 hours stirred in the mild condition. Remarkably, outstanding advantages of our approach include Schiff base effectively isolated in high conversion in ionic liquid, the recoverability and reusability of the ionic liquid without loss in the conversion and especially environmentally friendly characteristic

3.1. The effect of the reactants molar ratio of acetylacetone: *p*-anisidine

The reactions were carried out at room temperature in 6 hours with the molar ratio $[BMIM][PF_6]$: acetylacetone = 7.2:1. As shown in Figure 1, there was a remarkable increase in reaction conversion from 86.69% to 93.75% as the molar ratio of acetylacetone: *p*-anisidine increasing from 1:1 to 1:1.2.

Moreover, the reaction conversion was found not to change noticeably when increasing the reagent molar ratio. Therefore, using higher reagent molar ratio was unnecessary and it was decided to use the acetylacetone: *p*-anisidine molar ratio of 1.2:1 in further experiments.

Study of using [BMIM][PF₆] ionic liquid as a green solvent and catalyst for the Schiff base...

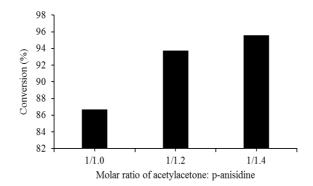


Figure 1. The effect of reactants molar ratio on the Schiff base synthesis conversions

3.2. The effect of the acetylacetone:ionic liquid molar ratio

The reactions were carried out at room temperature in 6 hours with the molar ratio acetylacetone: *p*-anisidine = 1:1.2. It was observed that when the [BMIM][PF₆]: acetylacetone molar ratio decreased from 7.2:1 to 4.8:1; 2.4:1 and 1.2:1 in that order, the product conversion increased steadily from 93.75% to 99.21% (Figure 2). Although the conversion increased, the lower molar ratio was not examined due to the fact that the suitable ratio was required to make the reactants dissolved. Another reason was that the reaction could not be conducted well with the smaller ratio in a flask. The lower product conversion corresponded with the higher [BMIM][PF₆]: acetylacetone molar ratio was explained that it was more difficult for the mixture of IL and reactants to be stirred well because of the IL's high viscosity.

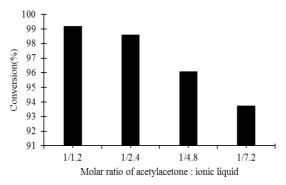


Figure 2. The effect of acetylacetone: [BMIM][PF₆] molar ratio on the Schiff base synthesis conversions

3.3. The effect of different ionic liquid on the Schiff base synthesis conversions

It was previously reported that Schiff bases were conventionally prepared by refluxing mixtures of the amine and the carbonyl compound in an organic solvent, such as, ethanol or methanol [15], CH_2Cl_2 [16]. However, it was observed that the product conversion was remarkably low in case of methanol compared to [BMIM][PF₆] as solvent. Moreover, the polar solvent such as ethyl acetate or non-polar solvent like toluene were clearly in-effective for this reaction. Consequently, it was very suitable to utilize IL for Schiff base synthesis in order to take advantage of their outstanding characteristics such as: low vapor pressure, ability to act as catalysts, chemical and thermal stability, non-flammability, high ionic conductivity [16]. Based on the fact that ionic liquid have proved its outstanding properties

as a very effective solvent with high conversion, the reaction was then conducted in the different ionic liquids. Different imidazolium hexafluorophosphate ionic liquids including [BMIM][PF₆], [HMIM][PF₆] and [OMIM][PF₆] were used as solvents in the synthesis of *p*-anisidineacetylacetone Schiff base. As shown in Figure 3 that increasing the alkyl chain resulted in a significant drop in reaction conversion, with 99.21% conversion being achieved for the case of [BMIM][PF₆] and 91.37% conversion for the case of [OMIM][PF₆]. This could be explained based on the fact that increasing the length of the alkyl group would decrease the polarity of the ionic liquid solvent, though the difference between butyl, hexyl and octyl groups were not significant. Additionally, the lowest viscosity of [BMIM][PF₆] among three ILs led to the best dispersion of reactant in the mixture and the conversion increased as a result. In summary, [BMIM][PF₆] could be considered as an efficient solvent for the Schiff base synthesis and would be employed for further studies.

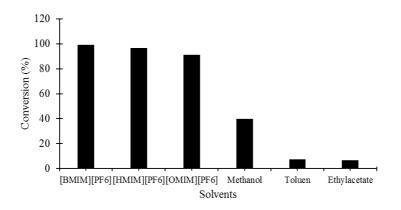


Figure 3. The effect of different ionic liquid on the Schiff base synthesis conversions

3.4. The effect of different substituents on the Schiff base synthesis conversion

It was thought that the condensation between a carbonyl compound and an amine leading to the formation of an imine would be a facile reaction due to the good electrophilic and nucleophilic properties of the carbonyl and amine groups, respectively. Therefore, the study was then extended to the Schiff base synthesis of both substituted-amine containing electron-donating and electron-withdrawing groups to investigate the effect of aminesubstituents on the reaction conversions.

In consideration of the electron's density in the Nitrogen atom that bonded directly to the phenyl group, it was noted that electron from Nitrogen atom moved to the ring by +C effect. Whereas in contrast, it was not the case for N connected to benzyl group. Although the presence of the methoxy in *p*-anisidine increased the nucleophilicity of the amine group through +C effect, the conversion reaction was achieved lower for the case of *p*-anisidine than for benzylamine, 2-chlorobenzylamine, 4-methylbenzylamine. Because the –Cl was the acceptor group, it was clearly found that reaction of 2-chlorobenzylamine did not occur as fast as benzylamine and 4-methylbenzylamine. In summary, the reaction rate was accelerated in the presence of electron – donating group to enhance the electron density in the N atom.

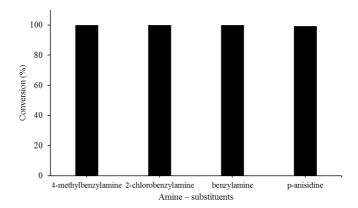


Figure 4. The effect of different amine-substituents on the Schiff base synthesis

3.5. The study on recyclability and reusability of ionic liquid as solvent in the Schiff base synthesis

The emerging use of ionic liquids was based on their important characteristics for the cleaner technologies such as: high polarity, immiscibility with a wide range of organic compounds, nonvolatile solvents and specially the recyclability and reusability for many times. Therefore, it was necessary to investigate the capability of ILs as recycling solvent in the synthesis of Schiff base *p*-anisidineacetylacetone. The reaction was carried out in the [BMIM][PF₆] ionic liquid under the optimal reaction condition as previously described. Since the products were weakly soluble in the ionic liquid, they were easily separated by simple extraction with ether. The ionic liquid phase was thoroughly washed with ether and recycled in subsequent reactions. After each run, the reaction product and unreacted starting materials were eliminated from the ionic liquid by extracting with diethyl ether, followed by the removing of the excess diethyl ether by a rotavapor at 50 °C.

The remaining ionic liquid [BMIM][PF₆] was recovered and recycled in subsequent reactions with only a gradual decrease in activity being observed (Figure 5). More specific, the recycling procedure was found to afford the product in 99.21% conversion (1st run) and changed very slightly in range of 90.12% - 98.71% in the next 9 runs. Therefore, the activity of the ionic liquid was proved to be consistent in runs and equally effective for this conversion. In conclusion, the use of an easily accessible and recyclable ionic liquid made this procedure convenient, economic and user friendly.

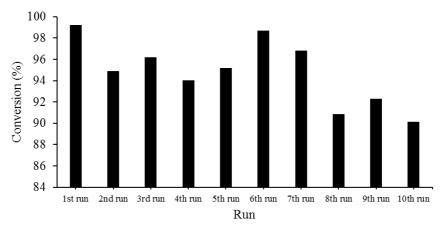


Figure 5. Effect of recycling ionic liquid

4. CONCLUSION

In summary, ionic liquid [BMIM][PF₆] had been demonstrated as an efficient and environmentally friendly reaction media as well as a promoter for the synthesis of Schiff base *p*-anisidineacetylacetone by investigating some main factors which influenced on the reaction conversions. In addition, the use of basic and polar organic solvents such as ethanol or methanol and high temperature reaction conditions could be avoided. As shown in the results above, the reaction between p-anisidine and acetylacetone should be carried out in $[BMIM][PF_6]$ ionic liquid as solvent without added catalyst, acetylacetone: p-anisidine molar ratio of 1.2:1, [BMIM][PF₆]: acetylacetone molar ratio of 1.2:1 to gain 99.21% conversion in 6 hours at ambient temperature. As results, the reaction rate was accelerated in the presence of electron – donating group to enhance the electron density in the N atom. The reaction of Benzylamine or 4-methylbenzylamine with acetylacetone achieved higher conversion compared to 2-chlorobenzylamine, followed by p-anisidine. The fact that the reaction could proceed readily in ionic liquids and that the ionic liquid solvent could be recycled and reused several times would be interested to the chemical industry. Finally, based on the good results above, it was noted that the formation of Schiff bases from the reaction of acetylacetone and primary amine p-anisidine carried out in IL had outstanding advantages compared to in organic solvents which are volatile, flammable and toxic.

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TÓM TẮT

NGHIÊN CỨU SỬ DỤNG CHẤT LỎNG ION [BMIM][PF₆] LÀM DUNG MÔI XANH VÀ XỨC TÁC TRONG TÔNG HỢP SCHIFF BASE

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Chất lỏng ion $[BMIM][PF_6]$ được tổng hợp trong điều kiện không dung môi với sự hỗ trợ vi sóng (MW). Chất lỏng ion $[BMIM][PF_6]$ sau tổng hợp được sử dụng làm dung môi cho quá trình tổng hợp para-Anisidineacetylacetone đạt độ chuyển hoá cao sau 6 giờ mà không cần thêm xúc tác khác. Ngoài ra, các chất lỏng ion 1-alkyl-3-methyl imidazolium được thu hồi và tái sử dụng trong các phản ứng nhiều lần mà hiệu quả giảm không đáng kể.

Từ khóa: Chất lỏng ion, dialkylimidazolium, [BMIM][PF₆], *p*-anisidine acetylacetone, Schiff Base