

Facile synthesis of *N, N'*-alkylidene bisamides catalyzed by trimethyl-tris(4-pyridinium)benzene trifluoroacetate: An efficient and rapid approach

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Abstract:

In the current study, a new Brønsted-acidic ionic liquid, namely trimethyl-tris(4-pyridinium)benzene trifluoroacetate ([TMPB][TFA]), was synthesized as a high-performance, reproducible acidic-basic catalyst. The structure of the ionic liquid was characterized by various physicochemical techniques. In continuation, the catalytic effect of [TMPB][TFA] in the production of *N, N'*-alkylidene bisamides was monitored via a one-pot pseudo three-component condensation of a wide range of aldehydes with benzamide/acetamide. The enhancement of this synthetic pathway was carried out using 10 mol% ionic liquid in ethanol solvent at 60 °C. The important features of this process include: the dual function of IL as an acid-base dual in the activation of reactants, comprehensiveness of the protocol in the rapid production of a variety of derivatives in high yields (**1a-17a**, 85 to 98%, 9 to 30 min), the ability to recover and reuse the IL for up to three cycles while maintaining catalytic efficiency, moderate reaction conditions, easy work-up, producing and characterizing new compounds (**8a** and **9a**), and green reaction environment without using expensive metal precursors, harsh temperature conditions as well as non-benign solvents.

Keywords: Ionic liquid; Aldehydes; Green solvent; *N, N'*-alkylidene bisamides

Introduction

In the quest for innovative and efficient strategies to facilitate reactions under environmentally friendly, mild, and rapid conditions, ionic liquids have emerged as compelling candidates that have swiftly integrated into contemporary synthetic methodologies [1–3]. Ionic liquids are characterized by several advantageous properties that make them highly attractive for chemical processes [4]. Firstly, their non-flammability significantly enhances safety during application. Additionally, their thermal stability allows them to withstand a wide range of temperatures without decomposing, making them suitable for reactions that require elevated temperatures [5–9]. Furthermore, ionic liquids exhibit low vapor pressure, which minimizes the release of volatile organic compounds (VOCs) into the atmosphere, thereby contributing to a reduction in environmental pollution. A notable category within the realm of ionic liquids is Brønsted acidic ionic liquids (BAILs) [10, 11]. These

compounds have garnered attention as excellent alternatives to traditional mineral acids, which are often corrosive and environmentally detrimental [12, 13]. Brønsted acidic ionic liquids can function effectively as solvents, catalysts, or reagents in various chemical reactions, providing a more sustainable and less hazardous option for acid-catalyzed processes [14, 15]. Their ability to dissolve a wide range of substrates and promote reactions under mild conditions makes them particularly valuable in organic synthesis [16]. The motivation of the present study is to introduce a new class of BAIL with three functional acidic and basic centers and investigate its catalytic performance in facilitating the leading synthetic route.

In green synthesis, using ionic liquids in multicomponent reactions (MCRs) is a promising approach with high atom economy [17, 18]. In traditional linear synthesis, multiple steps are often required to construct complex molecules, leading to the generation of waste and the need for exten-

sive purification processes [19]. In contrast, MCRs allow for the simultaneous combination of three or more reactants in a single reaction vessel, resulting in the formation of a diverse array of products in fewer synthetic steps [20–22]. This not only minimizes the amount of raw materials consumed but also reduces the overall environmental impact of the synthesis process [23].

Amides are indispensable in the chemical industry and organic synthesis due to their diverse applications and functional properties [24]. Their role as intermediates in producing pharmaceuticals, polymers, and other chemical products, combined with their utility in synthetic methodologies, underscores their versatility and importance in modern chemistry [25]. As research continues to explore new applications and reactions involving amides, their significance is likely to grow even further in the future. Specifically, bisamides are of considerable interest in synthesizing peptidomimetic compounds [26]. The growing interest in bisamides within the realm of peptidomimetic compound synthesis reflects their promise as versatile building blocks in medicinal chemistry, offering exciting opportunities for innovation in drug design and development. As research continues to evolve, bisamides are likely to play an increasingly important role in addressing medical needs and advancing the frontiers of science [27, 28]. The synthesis of *N, N'*-methylene bisamides usually involves the direct reaction of aldehydes with amides, which is facilitated by suitable catalytic conditions. In recent years, the literature has documented a limited number of catalysts for synthesizing *N, N'*-methylene bisamides [29]. Notable examples include NiFe₂O₄@SiO₂-PPA [30], polymer-supported Brønsted acid ionic liquids [31], Fe₃O₄@nano-cellulose/B(III) [32], H₃BO₃ [33], hydroxyapatite [34], POM-MIMBS ionic liquid nano-hybrids [35], Nano-SnCl₄.SiO₂ [36], and ZnO/KIT-6@NiFe₂O₄ [37]. However, many of these methods present certain disadvantages, including toxicity, instability, high costs, the necessity for microwave usage, lack of homogeneity or reusability, challenging work-up processes, stringent reaction conditions, and complex synthetic procedures.

In this research work, by integrating the unique properties of Brønsted acidic ionic liquids, multicomponent reactions, acidic-basic catalysts, and green chemistry approaches to overcome some of the mentioned limitations, we present the application of [TMPB][TFA] as a new catalyst for the an ef-

ficient and rapid synthesis of *N, N'*-alkylidene bisamides in ethanol solvent at 60 °C. The synthesis of novel derivatives **8a** and **9a**, which have not been synthesized and characterized in the previous literature, is another noteworthy aspect of this protocol.

Experimental

General approach to producing [TMPB][TFA]

A mixture of trimethyl-tris(4-pyridyl)benzene (2.811 g, 8 mmol) and CH₂Cl₂ (25 mL) was added to trifluoroacetic acid (2.736 g, 24 mmol) dissolved in CH₂Cl₂ (20 mL) over a period of 20 min in an ice bath. The resulting mixture was stirred at room temperature for 60 min. Next, dichloromethane was evaporated, and the residue washed several times with petroleum ether (4 × 5 mL) and dried under vacuum (65 °C) to synthesize [TMPB][TFA] as white powder with a melting point of 96 °C, and a yield of 96% equivalent to 5.327 g (Fig. 1).

General method for the preparation of *N, N'*-alkylidene bisamide derivatives

A mixture of [TMPB][TFA] (0.069 g, 0.1 mmol), aldehyde (1 mmol), and amide (2 mmol) in 1 mL EtOH was stirred in an oil bath at 60 °C. The progress of the reaction was monitored by TLC. After complete consumption of the starting material, the ionic liquid was separated, and the solid residue was recrystallized from ethanol (95%) to provide the pure product (Fig. 2).

Results and discussion

Catalyst characterization

The FT-IR pattern of the ionic liquid [TMPB][TFA] is presented in Fig. 3. The literature-based data of this spectrum support the generation of our catalyst (Table 1), as it includes peaks corresponding to all bonds and functional groups of the IL.

The structure of the ionic liquid was evaluated using NMR spectroscopy. The results of this study confirmed its high purity as follows: ¹H NMR (500 MHz, DMSO-*d*₆) δ (ppm): 2.35 (s, 9H, 3CH₃), 8.21 (d, *J* = 8.3 Hz, 6H, H_{Ar}), 8.91 (d, *J* = 8.3 Hz, 6H, H_{Ar}), 16.97 (br., 3H, 3NH), (Fig. 4); ¹⁹F NMR (235 MHz, DMSO-*d*₆) δ (ppm): -70.89 ppm (Fig. 5); ¹³C NMR (125 MHz, DMSO-*d*₆) δ (ppm): 12.21,

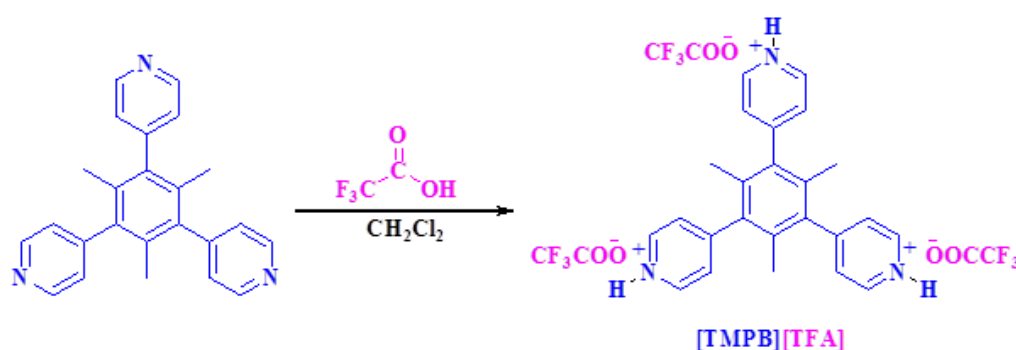


Figure 1. The synthesis of [TMPB][TFA].

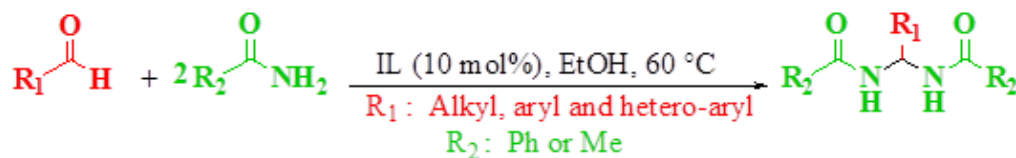


Figure 2. The preparation of *N,N'*-alkylidene bisamides.

112.57 – 119.46 (q, $^1J_{C-F} = 287.0$ Hz), 123.03, 134.66, 137.28, 142.94, 153.63, 159.04-160.04 (q, $^2J_{C-F} = 42.0$ Hz) (Fig. 6).

The thermal resistance of the ionic liquid [TMPB][TFA] was investigated using the TGA technique, which is shown in Fig. 7. The results revealed that this IL exhibits similar behavior to its counterparts in the literature [42, 43], meaning that it decomposes in two stages in the temperature range of 50 to 600 °C: 50 – 195 °C (–12%), and 195 – 500 °C (–88%). The reason for the weight loss of the catalyst in the first stage is the removal of moisture absorbed in it, and in the other stage, the decomposition of its organic components. Therefore, this ionic liquid has adequate thermal

stability for catalyzing an organic reaction at 60 °C.

Our final studies aimed to characterize the structure of the [TMPB][TFA] catalyst using CHN elemental analysis and mass spectrometric techniques. The agreement between experimental and calculated CHN analysis data Experimental (calculated) C, 51.85 (51.96); H, 3.39 (3.49); N, 5.98 (6.06), along with the appearance of the desired peak in the mass spectrum, confirmed the successful production of [TMPB][TFA] while confirming the results of previous analyses. The most important mass data (M/z) were as follows: 693 (M^+), 694 ($M^+ + 1$), 693 ($M^+ + 5$), 579 ($M^+ - CF_3COOH$), 465 ($M^+ - 2 CF_3COOH$), and 349 ($M^+ - 3 CF_3COOH$, and -H).

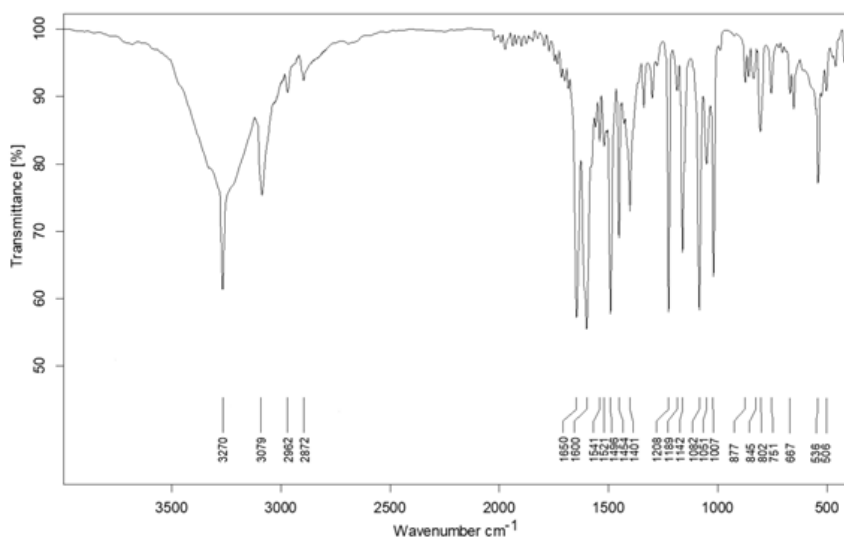


Figure 3. The FT-IR of [TMPB][TFA].

Table 1. FT-IR data of [TMPB][TFA].

Peak (cm ⁻¹)	Assignment
3270	N-H stretching [38, 39]
2600 – 3400	N-H stretching with hydrogen bonding [38, 40]
3075	C-H stretching [40]
2872 and 2962	CH ₃ stretching [39]
1650	C=O stretching [39, 40]
1541	N-H bending [39]
1496	C=N stretching [41]
1600 and 1521	C=C stretching [40, 41]
1189	C-O stretching [39]
1208 and 1142	C-F stretching [39]
751 and 506	C-F bending [39, 41]
802	N-H out-of-plane [39]
536	N-H wagging [39]
845	C-H in-plane and out-of-plane [40]

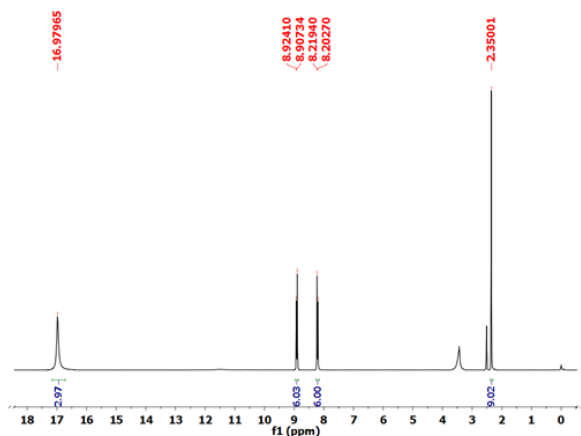


Figure 4. The ^1H NMR of [TMPB][TFA].

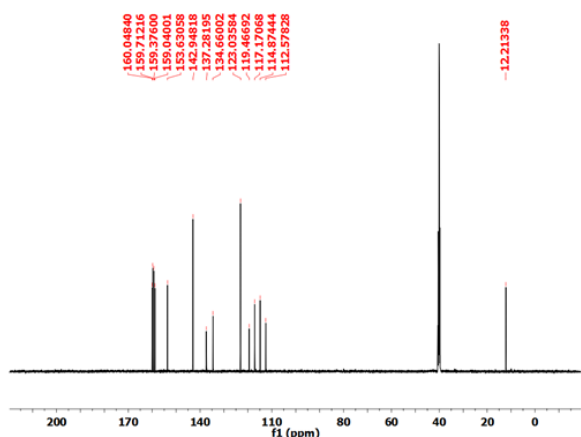


Figure 5. The ^{13}C NMR of [TMPB][TFA].

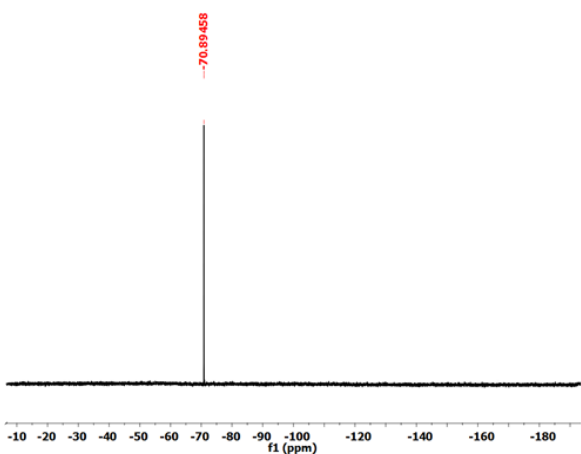


Figure 6. The ^{19}F NMR of [TMPB][TFA].

In addition, in the positive (Fig. 8 (a)), and negative (Fig. 8 (b)) mode mass spectra of this ionic liquid, the peak at m/z 354, correspond to the IL cation, and the peak at m/z 113 associated to the IL anion, respectively.

Measuring the acidity level of the catalyst

A practical approach to assessing the acidity level of an acid in organic solvents involves employing the Hammett method in conjunction with UV-visible spectroscopy [44].

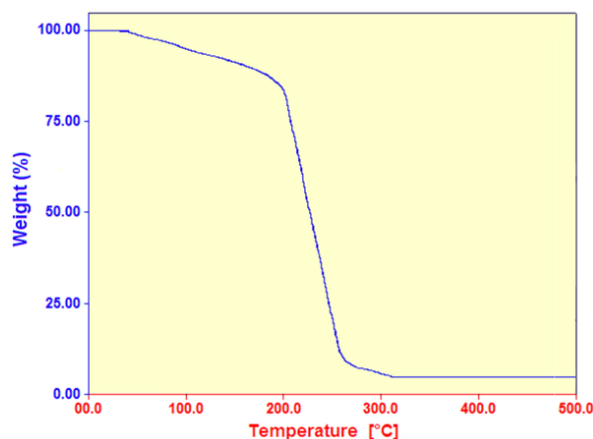


Figure 7. The TGA pattern of [TMPB][TFA].

The Hammett function, denoted by H_0 , is calculated from equation (1).

$$H_0 = \log\left(\frac{[\text{I}]_s}{[\text{IH}^+]_s}\right) + \text{pK}(\text{I})_{\text{aq}} \quad (1)$$

In this model, the molar concentrations of the unprotonated and protonated forms of the indicator in the solvent are denoted as $[\text{I}]_s$ and $[\text{IH}^+]_s$, respectively, while $\text{pK}(\text{I})_{\text{aq}}$ represents the pK_s value of the indicator in its aqueous solution. The concentration of $[\text{I}]_s/[\text{IH}^+]_s$ can be determined using the UV-visible spectrum data in accordance with Lambert-Beer's Law [44]. To do this, carbon tetrachloride (CCl_4) and 4-nitroaniline (with $\text{pK}(\text{I})_{\text{aq}}$ equal to 0.99) were used as solvent and the basic indicator, respectively. The highest absorbance of the un-protonated form of the indicator was observed at 338 nm in carbon tetrachloride (CCl_4) within the UV-visible spectrum, as illustrated in Fig. 9. After adding [TMPB][TFA] to the indicator solution, there was a noticeable decrease in the absorbance of the un-protonated form of the indicator, indicating that a partial conversion to the $[\text{IH}^+]$ form had indeed occurred. The results presented

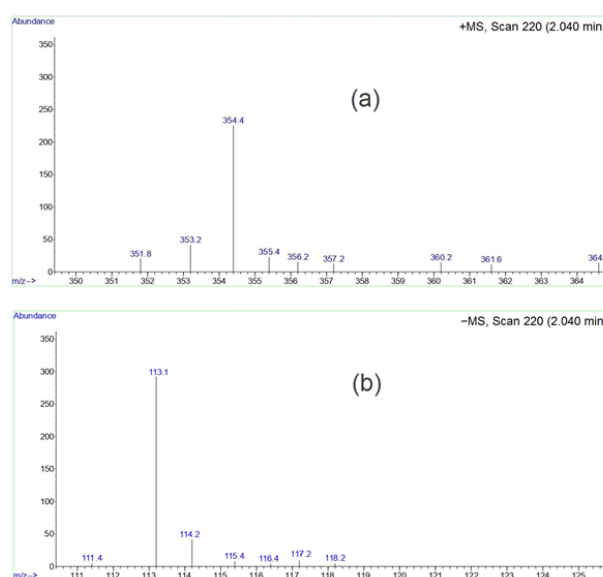


Figure 8. MS^+ (a), and MS^- (b) for the cation and anion of [TMPB][TFA].

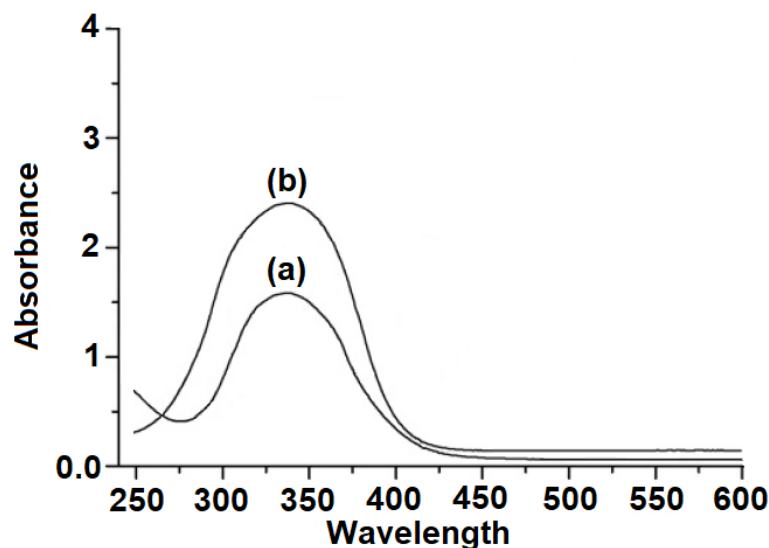


Figure 9. Absorption spectra of [TMPB][TFA] (a), and 4-nitroaniline (indicator) (b) in CCl_4 .

in Table 2 illustrate the acidity strength of [TMPB][TFA].

Catalytic performance

Checking catalytic activity of [TMPB][TFA] for the synthesis of *N, N'*-alkylidene bisamides

After synthesizing and characterizing the [TMPB][TFA] catalyst, we proceeded to assess its effectiveness in the synthesis of *N, N'*-alkylidene bisamides. In order to identify the best conditions of the catalyst and solvent system, we selected a model reaction using benzamide (2 mmol), and 4-nitrobenzaldehyde (1 mmol), as shown in (Fig. 10). The data presented in Table 2 clearly show that the use of [TMPB][TFA] (10 mol%) in ethanol solvent (1 mL) leads to the highest yield at an ideal time (Table 3, entry 7). It is worth noting that in methanol, the model reaction is completed in a slightly shorter time (Table 3, entry 13). However, the insignificant difference in the reaction period compared to the ethanol medium caused entry seven to remain in the

optimal state, considering the principles of green chemistry. The faster progress of the reaction in methanol solvent is probably related to the greater solubility of benzamide in it [45].

In subsequent experiments, various derivatives of *N, N'*-alkylidene bisamides were synthesized through the reaction of alkyl/aryl/hetero-aryl aldehydes with benzamide (or acetamide) under optimized conditions, as outlined in Table 4. The results of the table indicate that *N, N'*-alkylidene bisamides were successfully produced with high yields and in a short duration, utilizing aldehydes that contained electron-withdrawing, electron-donating, and halogen substituents. These findings confirm the significant efficacy and broad applicability of [TMPB][TFA] as a catalyst for this reaction.

Based on the previous performance of ionic liquids as a dual acid-base [46], an interesting mechanism for the synthesis of *N, N'*-alkylidene bisamides by the acidic (hydrogen bonded to a positively charged nitrogen) and basic (nega-

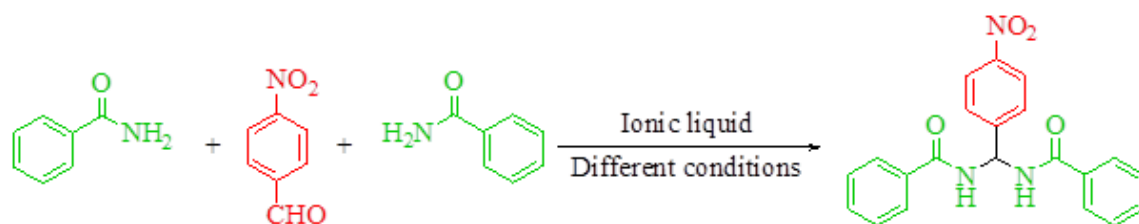


Figure 10. Model reaction.

Table 2. computation of Hammett acidity equation (H_0) for [TMPB][TFA]^a.

Entry	Catalyst	A_{\max}	$[I]_s\%$	$[IH^+]_s\%$	H_0
1	-	2.2263	100	0	0
2	[TMPB][TFA]	1.5845	67.70	26.90	1.39

^a Conditions for calculating visible-ultraviolet spectrum: Carbon tetrachloride: solvent, 4-nitroaniline: indicator with $0.144 \times 10^{-3} \text{ mol L}^{-1}$ (10 mL), $\text{pK}(I)_{\text{aq}} = 0.99$; [TMPB][TFA]:10 mg, ambient temperature.

Table 3. Optimization of various parameters in the synthesis of **2a**.

Entry	Catalyst	Catalyst quantity (mol%)	Temp. (°C)	Solvent	Solvent amount (mL)	Time (min)	Yield ^a (%)
1	-	-	120	-	-	45	trace
2	[TMPB][TFA]	10	120	-	-	30	71
3	[TMPB][TFA]	10	25	EtOH	2	25	54
4	[TMPB][TFA]	10	40	EtOH	2	20	60
5	[TMPB][TFA]	10	50	EtOH	2	15	78
6	[TMPB][TFA]	10	60	EtOH	2	11	97
7	[TMPB][TFA]	10	60	EtOH	1	9	98
8	[TMPB][TFA]	10	60	EtOH	3	15	98
9	[TMPB][TFA]	10	Reflux	EtOH	1	9	98
10	[TMPB][TFA]	8	60	EtOH	1	9	80
11	[TMPB][TFA]	12	60	EtOH	1	9	98
12	[TMPB][TFA]	14	60	EtOH	1	9	93
13	[TMPB][TFA]	10	60	MeOH	1	8	98
14	[TMPB][TFA]	10	60	H ₂ O	1	15	72
15	[TMPB][TFA]	10	60	PEG 400	1	13	83
16	[TMPB][TFA]	10	60	H ₂ O/EtOH (3:1)	1	12	84
17	[TMPB][TFA]	10	60	Isopropanol	1	10	87
18	Trimethyl-tris (4-pyridyl)benzene	10	60	EtOH	1	20	23
19	TFA	10	60	EtOH	1	20	45

^a Isolated yield.

tively charged oxygen of the trifluoroacetate group) sites of our ionic liquid is designed in Fig. 11 [32–37]. Initially, the reaction between the **1** and the **2** activated through the acidic and basic sites of [TMPB][TFA], correspondingly, generates intermediate **11** (I). Then the active sites of the catalyst help remove a water molecule from **11** (I) to produce **11** (II). Subsequently, the interaction of the carbonyl group of **11** (II) with the acidic site of the catalyst renders the

β -carbon of **11** (II) susceptible to nucleophilic attack by the second molecule **2** to produce **11** (III). Finally, intermediate **11** (III) undergoes an ionic liquid-catalyzed tautomerization process to give the target molecule.

Catalyst recycling

The ability to reuse catalysts not only contributes to more sustainable and efficient synthetic processes but also offers

Table 4. Production of *N, N'*-alkylidene bisamide derivatives by [TMPB][TFA].

Entry	R ¹	R ²	Time (min)	Yield ^a (%)	TON	TOF (min ⁻¹)	M.p. °C (Lit.)
1a	2-O ₂ NC ₆ H ₄	C ₆ H ₅	9	95	9.5	1.05	257 – 259 (257 – 259) [30]
2a	4-O ₂ NC ₆ H ₄	C ₆ H ₅	9	98	9.8	1.08	258 – 260 (257 – 259) [30]
3a	C ₆ H ₅	C ₆ H ₅	10	97	9.7	0.97	213 – 215 (213 – 215) [30]
4a	4-MeC ₆ H ₄	C ₆ H ₅	12	94	9.4	0.78	242 – 244 (242 – 244) [30]
5a	4-BrC ₆ H ₄	C ₆ H ₅	14	95	9.5	0.67	254 – 256 (254 – 256) [30]
6a	4-ClC ₆ H ₄	C ₆ H ₅	10	97	9.7	0.97	249 – 251 (250 – 252) [30]
7a	2,4-Cl ₂ C ₆ H ₃	C ₆ H ₅	15	93	9.3	0.62	197 – 199 (198 – 200) [30]
8a	4-(4-MePh)C ₆ H ₄	C ₆ H ₅	15	95	9.5	0.63	263 – 265
9a	4-(4-MePh)C ₆ H ₄	CH ₃	18	93	9.3	0.51	288 – 290
10a	4-O ₂ NC ₆ H ₄	CH ₃	12	96	9.6	0.80	272 – 274 (271 – 273) [31]
11a	3-O ₂ NC ₆ H ₄	CH ₃	12	95	9.5	0.79	230 – 232 (230 – 232) [30]
12a	C ₆ H ₅	CH ₃	14	96	9.6	0.68	234 – 236 (233 – 236) [31]
13a	4-MeC ₆ H ₄	CH ₃	17	92	9.2	0.54	270 – 272 (270 – 273) [31]
14a	4-ClC ₆ H ₄	CH ₃	14	95	9.5	0.67	255 – 257 (254 – 257) [31]
15a	4-MeOC ₆ H ₄	CH ₃	17	90	9.0	0.52	218 – 220 (216 – 219) [31]
16a	(CH ₃) ₂ CH	C ₆ H ₅	30	85	8.5	0.28	124 – 126 (125 – 127) [32]
17a	4-Pyridyl	C ₆ H ₅	14	94	9.4	0.67	210 – 212 (208 – 212) [33]

^a Isolated yield.

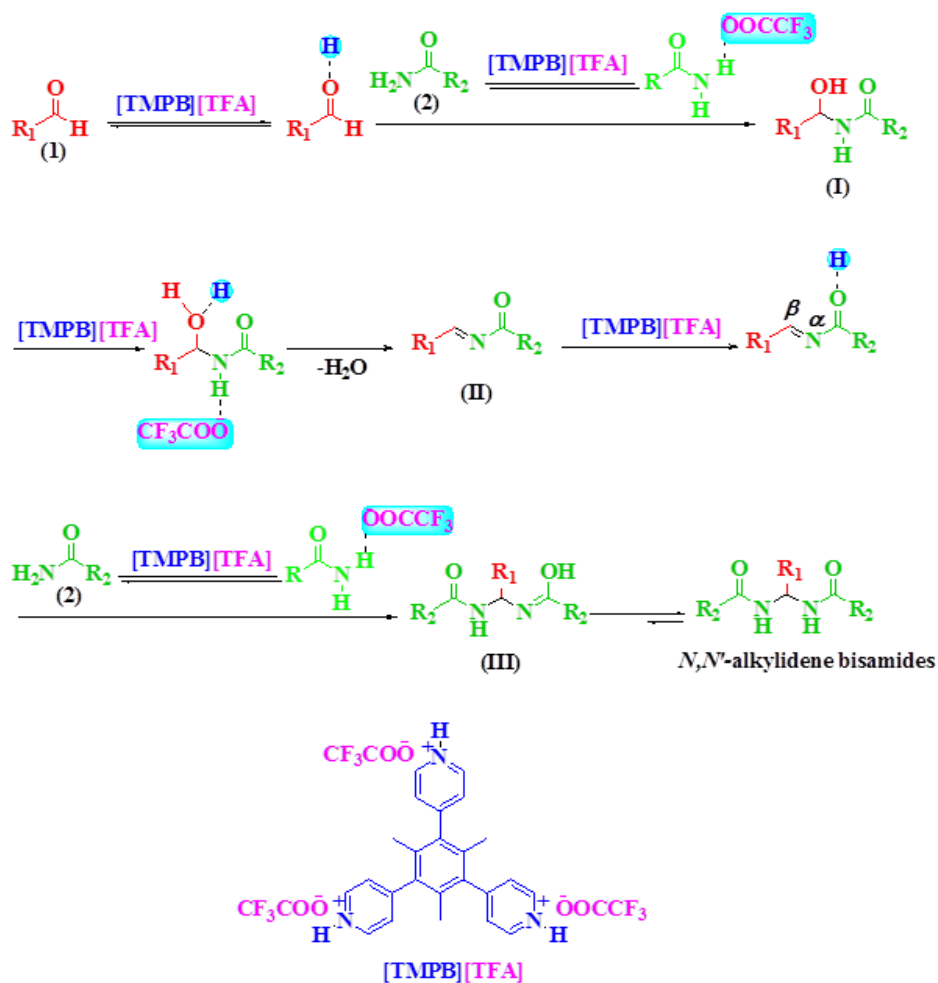


Figure 11. Mechanism of the synthetic route facilitated by [TMPB][TFA].

considerable economic benefits, making it a valuable aspect of modern chemical manufacturing. In consideration of this, an examination was conducted on the feasibility of reusing [TMPB][TFA] in the production of product **2a**. To extract the ionic liquid, after the reaction was complete, 25 mL of hot ethanol/water (4/1) solution was added to the residue, and the soluble bisamide was extracted from the insoluble [TMPB][TFA]. The separated ionic liquid was prepared for further use after trituration (5×3 mL with petroleum ether) and drying at 70 °C. The study demonstrates that this ionic liquid can be utilized in the synthesis for up to three cycles, showing only a negligible decline in performance. This minor reduction in efficacy is linked to a 4% loss of the ionic liquid that takes place during the recycling process (Fig. 12).

In another study, the structure of the recovered ionic liquid was investigated by ^1H NMR spectroscopy (Fig. 13). The results of this study showed that the spectrum of the recovered sample was in high agreement with the fresh sample, meaning that the catalysis had maintained its stability well during the recovery steps.

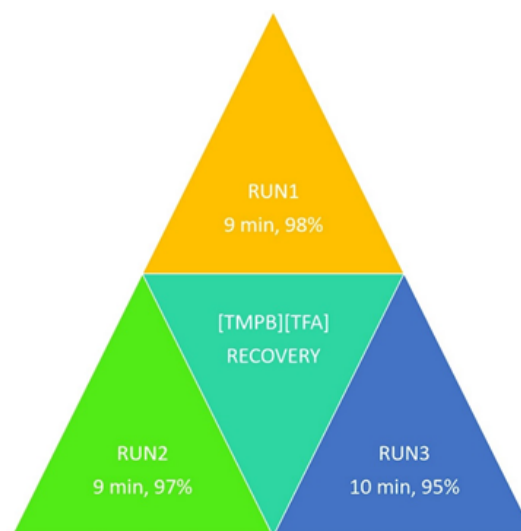


Figure 12. Results of recovery of [TMPB][TFA] in the synthesis of product **2a**.

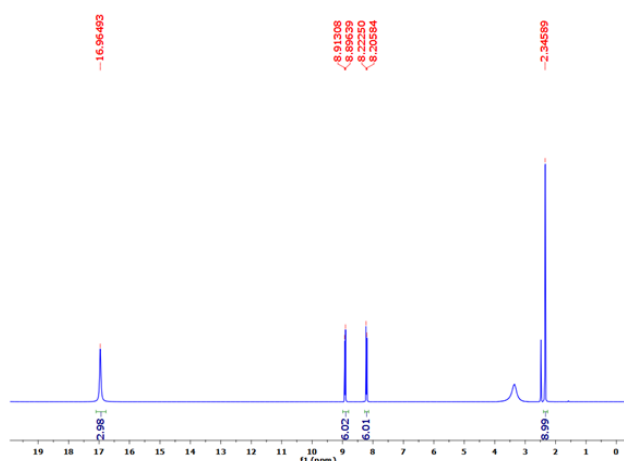


Figure 13. The ^1H NMR of recovered [TMPB][TFA].

Comparison

The subsequent investigation focused on the synthesis of *N, N'*-alkylidene bisamide and assessed the effectiveness of [TMPB][TFA] compared to previous studies in this domain. Various parameters, including catalyst amount, nature of solvent, temperature, time, and percent yield, were considered during the synthesis of product **2a**. The findings, detailed in Table 5, showed that our catalyst produced the corresponding product in the shortest time, with the highest yield under the mildest temperature conditions listed in the table, compared to all competitors. The absence of the use of expensive metal precursors in the catalyst preparation, the green nature of the solvent used, no need for unusual energy sources such as microwaves (Table 5, Entry 1 vs. 6), and the dual function of [TMPB][TFA] as an acid-base dual in the activation of the reactants are other advantages of our protocol that highlight the distinctive performance of our ionic liquid.

Table 5. Comparison of the performance of [TMPB][TFA] with competitors in similar literature.

Entry	Catalyst	Conditions and results	Ref.
		Catalyst amount, Solvent, Temp, Time, Yield (%) ^a	
1	[TMPB][TFA]	10 mol%, EtOH, 60 °C, 9 min, 98%	-
2	NiFe ₂ O ₄ @SiO ₂ -PPA	0.1 g, CH ₃ OH, reflux, 80 min, 86%	[30]
3	Polymer-supported Bronsted acid IL	0.07 mmol, Toluene, Reflux, 30 min, 85%	[31]
4	Fe ₃ O ₄ @nano-cellulose/B(III)	0.06 g, Solvent-free, 70 °C, 10 min, 97%	[32]
5	H ₃ BO ₃	0.3 mmol, Toluene, 120 °C, 960 min, 92%	[33]
6	H ₃ BO ₃	0.3 mmol, CH ₂ Cl ₂ , Microwave, 20 min, 85%	[33]
7	Hydroxyapatite	0.05 g, CH ₃ CN, Reflux, 180 min, 92%	[34]
8	POM-MIMBS IL nano-hybrid	0.02 g, Solvent-free, 80 °C, 15 min, 89%	[35]
9	Nano-SnCl ₄ .SiO ₂	0.1 g, n-hexane, Reflux, 126 min, 90%	[36]
10	ZnO/KIT-6@NiFe ₂ O ₄	0.05 g, Solvent-free, 125 °C, 10 min, 57	[37]

^a Isolated yield.

Conclusion

As a result, we have created a protic ionic liquid with H⁺ on cation, namely [TMPB][TFA], which has three acidic (-NH⁺) and basic (CF₃COO⁻) groups; it can catalyze synthetic routes that demand acidic-basic catalyst. In this protocol, we effectively used this ionic liquid as a catalyst for the synthesizing *N, N'*-alkylidene bisamide from alkyl/aryl/hetero-aryl aldehydes (1 eq.) and benza-mide/acetamide (2 eq.) (**1a-17a**, 85 to 98%, 9 to 30 min); the highlights of this procedure includes the non-metallic nature of the catalyst, wide domain, short-time and excellent yield of derivatives, capability of the protocol to create the bisamides from hetero-aromatic, aromatic and aliphatic amides, utilization of a green solvent at mild temperatures, easy work-up, and the repeatability of the IL up to three cycles without significant decline in its activity. Given the high performance, the potential application of this catalyst in other organic reactions, especially on an industrial scale, is a prospect for this protocol.

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Authors contributions

Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Conflict of interests

The author declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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