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Task-specific acidic ionic liquid [CmMIm]Cl-catalyzed one-pot multicomponent synthesis of β-aminocarbonyl compounds at room temperature

Dhaneshwar Prasad,[a] Kumari Anchal,[a] Saumy Sharma,[a] Pranali Hadole,[b] Sudip Monal*[b] and Subhash Banerjee *[a]

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Sustainable catalytic systems that promote multicomponent reactions under mild and environmentally benign conditions are highly desirable in modern organic synthesis. Herein, we report a task-specific ionic liquid, 3-(carboxymethyl)-1-methyl-3-1*H*-imidazole-3-ium chloride ([CmMIm]Cl), as a dual-functional catalyst and reaction medium for the one-pot synthesis of β-aminocarbonyl compounds *via* the Mannich reaction. The protocol proceeds efficiently at ambient temperature, affording excellent yields of products within short reaction times. Notably, the method avoids toxic metal catalysts, reduces solvent consumption, and enables straightforward product isolation. Mechanistic insights suggest that cooperative activation arising from the acidic (–COOH) and basic (imidazolium nitrogen) sites of the ionic liquid facilitates rapid imine formation and subsequent C–C bond formation with broad substrate scope. Furthermore, [CmMIm]Cl can be easily recovered and reused for successive cycles without significant loss of activity, highlighting its stability and practical applicability. Overall, this study demonstrates that [CmMIm]Cl represents an efficient and sustainable platform for C–C and C–N bond formation through Mannich-type multicomponent reactions under mild and green conditions.

1 Introduction

The Mannich reaction is an important organic transformation involving the simultaneous formation of carbon–carbon and carbon–nitrogen bonds[1] *via* three-component condensation reactions of aldehydes, amines, and carbonyl compounds, leading to the formation of β-aminocarbonyl compounds.[2] This reaction enables direct amino alkylation in the organic molecules.[3] These compounds are used as starting materials in the synthesis of amino alcohols, peptides, lactams, and optically active amino acids.[4] Due to its broad scope and reliability, the Mannich reaction and its modified forms are frequently used to synthesize structurally diverse molecules with applications extending from biologically active frameworks to material science.[5] In Fig. 1, a few examples of biologically active and therapeutic molecules formed by the Mannich reaction are shown.[6–9]

Mannich reactions were performed using homogeneous protic acid,[10] organo acid,[11–13] Lewis acids,[14–24] N-heterocyclic carbenes,[25] quinidine-thiourea derivatives,[26] molecular iodine,[27] *etc.*, as a catalyst. Despite efficient activity, these protocols also face several limitations. As Lewis acids involve toxic, moisture-sensitive metals, Brønsted acids suffer from a catalyst recovery issue, and organocatalytic or nitrogen-based systems often need high catalyst loadings with harsh and costly reaction conditions.

To overcome the limitations of homogeneous catalytic systems, several heterogeneous catalysts such as SiO_2–$OAlCl_2$,[28] $ZnCl_2/SiO_2$,[29] $FeCl_3$-supported rice husk ash,[30] montmorillonite K10 clay,[31] PS–SO_3H,[32] SBA-15-PrSO_3H,[33] $H_3PW_{12}O_{40}$,[34] GO/Fe_3O_4-POM,[35] ZnO-nanoparticles,[36] TiO_2 [37] have been introduced to perform the Mannich reaction. Some microwave-assisted methodologies[38] have also been introduced for this

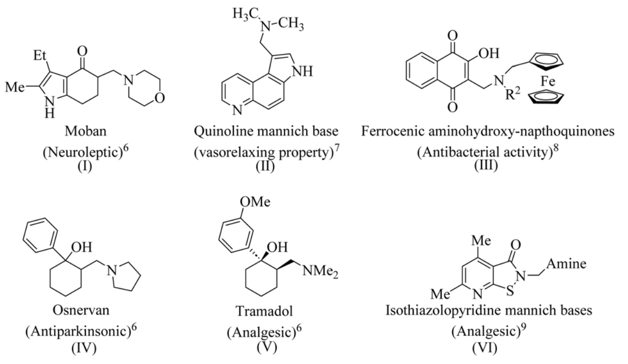

Fig. 1 Some biologically active molecules are synthesized *via* the Mannich reaction.

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[a] Department of Chemistry, Guru Ghasidas Vishwavidyalaya, Koni, Bilaspur-495009, Chhattisgarh, India. E-mail: ocsb2006@gmail.com

[b] Post Graduate Department of Chemistry, Seth Kesarimal Porwal College, RTM Nagpur University, Kamptee, Nagpur, 441001, India

purpose, which require a costly equipment setup. Most of these reported protocols involve the use of costly materials and hazardous volatile organic solvents. These drawbacks induce the need for greener, reusable catalysts operating under mild conditions.

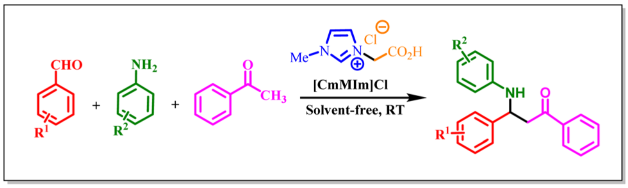
Room-temperature ionic liquids (RTILs) have gained considerable attention as environmentally benign substitutes for traditional organic solvents over the past several years.[39] Their growing use can be attributed to a unique combination of physicochemical features, such as low melting temperatures, negligible vapour pressure, reduced flammability, tunable polarity, and good compatibility with both organic and inorganic substrates[40] along with relatively low toxicity.[41] The emergence of task-specific RTILs has further expanded their usefulness by enabling them to serve the multifunctional role as catalysts, solvents as well as electrolytes in energy devices.[42] Ionic liquids have also been used as reaction media to perform Mannich reaction in combination with different metal salts like Fe_3O_4 nanoparticles,[43] and $RuCl_2(PPh_3)_3$.[44] As a part of our continued effort to establish ionic liquid as a true catalyst and reaction media,[42,45] we have successfully applied the task-specific ionic liquid [CmMIm]Cl in regioselective epoxide ring-opening reactions,[45l] α-halogenation of ketones[45p] as well as in the one-pot multicomponent synthesis of pyran–chromene derivatives.[45m]

Here, we report a task-specific 3-(carboxymethyl)-1-methyl-3-1*H*-imidazole-3-ium chloride [CmMIm]Cl ionic liquid as an efficient catalyst for one-pot three-component reactions of aldehyde, amine, and carbonyl compound, leading to the synthesis of β-aminocarbonyl compounds at room temperature under neat conditions (Scheme 1).

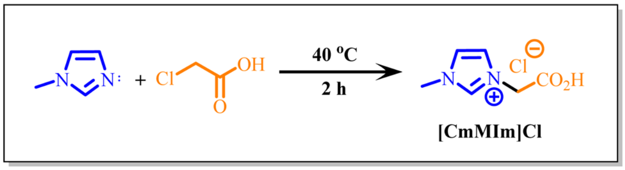
2 Results and discussion

2.1. Synthesis and characterization of [CmMIm]Cl

Initially, [CmMIm]Cl was prepared by the reaction of an equimolar amount of *N*-methyl imidazole (10 mmol) and 2-chloro acetic acid (10 mmol) at 40 °C for 2 h in acetonitrile (2 mL) following a previously reported procedure.[45l,m] The viscous yellow liquid was obtained, which was washed thoroughly with ether (3 × 2 mL) to remove any unreacted starting materials and further dried under reduced pressure (Scheme 2). [CmMIm]Cl was characterized by mass spectrometry and FT-IR spectroscopic study. A peak at *m*/*z* 141.0658 is observed corresponding to 3-(carboxymethyl)-1-methyl-3-1*H*-imidazole-3-ium cation (calculated *m*/*z* 141.06585). The FT-IR spectrum shows a strong band at 1732 cm^{-1}, which indicates the presence of a C=O group of carboxylic acid. The characteristic stretching vibrations of C=C and C=N in the imidazolium ring appear at around 1576 cm^{-1} and 1384 cm^{-1} respectively (see SI-1).



Scheme 1 Synthesis of β-aminocarbonyl compounds using [CmMIm]Cl.

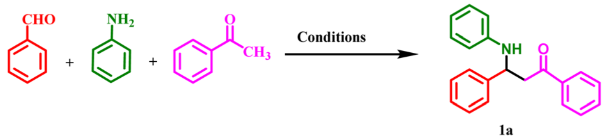


Scheme 2 Synthesis of [CmMIm]Cl.

2.2. [CmMIm]Cl-catalyzed synthesis of β-aminocarbonyl compounds *via* Mannich reaction using

The catalytic efficiency of [CmMIm]Cl was examined in the one-pot synthesis of β-aminocarbonyl derivatives. When a mixture of benzaldehyde (1 mmol), aniline (1 mmol), and acetophenone (1 mmol) was stirred in [CmMIm]Cl (100 mg, 57 mol%) for 1 h at room temperature, the reaction mixture solidified. The solid product was extracted using ethyl acetate (10 mL) twice and purified by recrystallization from hot ethanol. The pure product was identified as 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**) by ^{1}H NMR and ^{13}C NMR spectra (entry 1, Table 1). Encouraged by this result, the above reaction was selected as a model system to optimize the reaction conditions, and the corresponding results are summarized in Table 1. Initially, the amount of [CmMIm]Cl was varied. Notably, decreasing the catalyst loading to 50 mg (28 mol%) did not lead to any significant reduction in the yield of product **1a** (entry 2, Table 1). A slight decrease in yield to 80% was observed when the catalyst

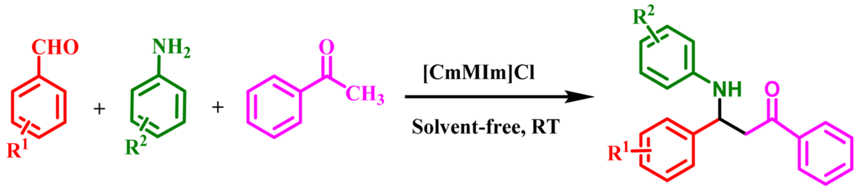
Table 1 Optimization of reaction conditions for the synthesis 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**) as a model reaction using [CmMIm]Cl[a]



Entry	Conditions	Time (hour)	Yield[b] (%)
1	[CmMIm]Cl (100 mg, 57 mol%)	1	97
2	**[CmMIm]Cl (50 mg, 28 mol%)**	**1**	**95**
3	[CmMIm]Cl (40 mg, 23 mol%)	1	80
4	[CmMIm]Cl (30 mg, 17 mol%)	1	52
5	[CmMIm]Cl (25 mg, 14 mol%)	1.5	45
6	No catalyst, neat	12	NR[c]
7	No catalyst, ethanol (1 mL)	12	NR[c]
8	*N*-Methyl imidazole	5	Trace
9	2-Chloroacetic acid	5	45
10	[pmIm]Br	5	NR[c]

[a] Benzaldehyde (1 mmol), aniline (1 mmol), and acetophenone (1 mmol) with continuous stirring in the given conditions at room temperature. [b] Yield of pure isolated products. [c] NR: no reaction.

Table 2 Substrate scope of [CmMIm]Cl-catalyzed synthesis of β-aminocarbonyl compounds *via* Mannich reaction[a]



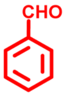
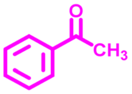
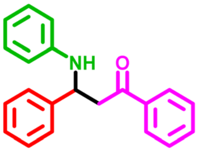
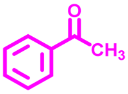
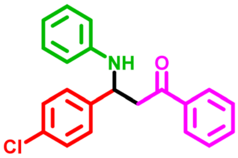
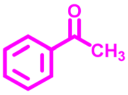
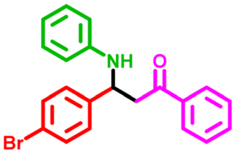
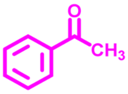
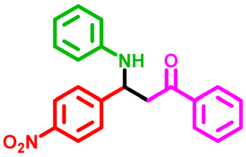
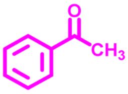
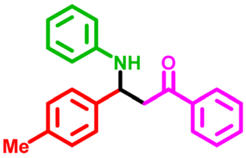
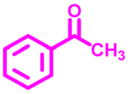
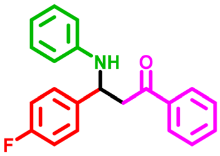
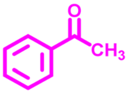
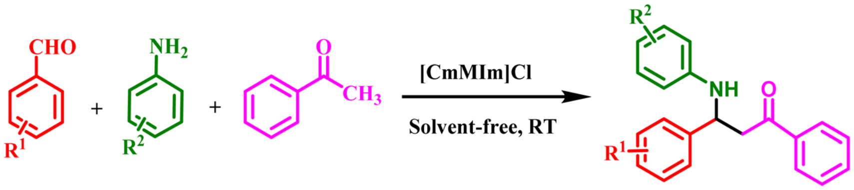
Entry	Aldehyde	Amine	Ketone	Substrate	Yield[b] (%)	Time	Melting point[c] (°C)	
							Observed	Reported
1				 **1a**	95	1 h	168–170	170–171 (ref. 14)
2				 **1b**	92	5 min	115–116	114–115 (ref. 17)
3				 **1c**	95	5 min	125–127	127–129 (ref. 30)
4				 **1d**	95	1 h	107–108	108–109 (ref. 14*a*)
5				 **1e**	90	1.5 h	130–131	131–132 (ref. 14*a*)
6				 **1f**	93	1 h	88–90	—[46]
7					90	1.5 h	140–142	—

Table 2 (*Contd.*)



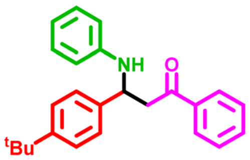
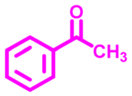
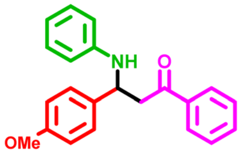
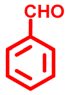
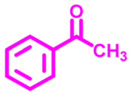
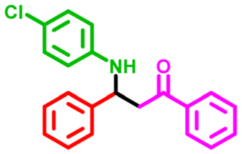
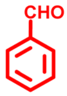
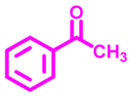
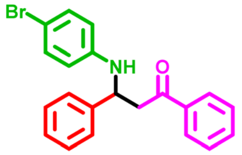
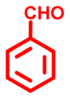
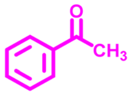
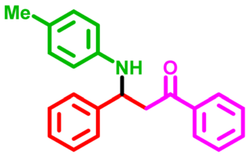
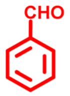
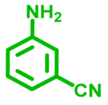
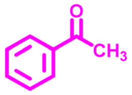
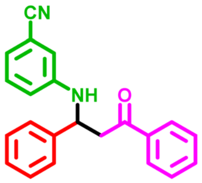
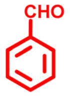
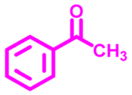
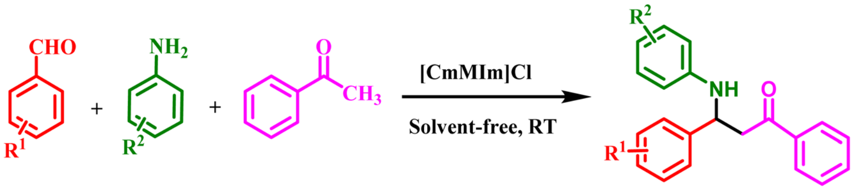
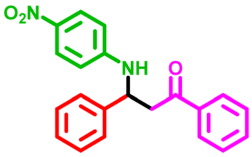
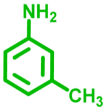
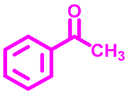
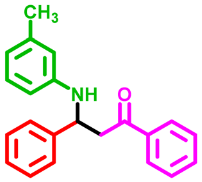
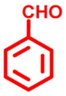
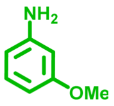
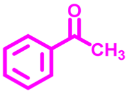
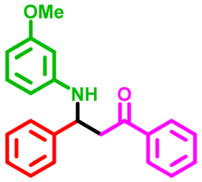
							Melting point^c (°C)	
Entry	Aldehyde	Amine	Ketone	Substrate	Yield^b (%)	Time	Observed	Reported
				 **1g**				
8				 **1h**	90	1.5 h	140–142	142–143 (ref. 17)
9				 **1i**	92	1.5 h	168–170	170–171 (ref. 14*a*)
10				 **1j**	91	1.5 h	180–181	180–182 (ref. 30)
11				 **1k**	93	1 h	167–168	168–169 (ref. 14*a*)
12				 **1l**	90	1.25 h	118–120	—
13					90	1.5 h	182–184	184–185 (ref. 14*a*)

Table 2 (*Contd.*)



Entry	Aldehyde	Amine	Ketone	Substrate	Yield^b (%)	Time	Melting point^c (°C)	
							Observed	Reported
				 **1m**				
14				 **1n**	95	1 h	130–131	131–132 (ref. 17)
15				 **1o**	92	1 h	129–130	—

[a] Reaction conditions: aryl aldehyde (1 mmol), amine (1 mmol), ketone (1 mmol), [CmMIm]Cl (28 mol%, 50 mg) with continuous stirring at room temperature. [b] Yield of pure isolated products. [c] The melting point was determined for the pure recrystallized products.

amount was further reduced to 40 mg (23 mol%) (entry 3, Table 1). Upon lowering the catalyst loading to 30 mg (17 mol%) and 25 mg (14 mol%), the yield of product **1a** declined noticeably to 52% and 45%, respectively (entries 4 and 5, Table 1). Control experiments were carried out in the absence of [CmMIm]Cl to confirm the role of IL. When the model reaction was carried out in ethanol in the absence of [CmMIm]Cl, starting materials were recovered (entries 6, Table 1) and when stirred only (entries 7, Table 1). The role of [CmMIm]Cl was further established by performing a model reaction using individual components of the ionic liquid, namely *N*-methyl imidazole and 2-chloroacetic acid, which afforded 20% and 35% yields, respectively (entries 8 and 9, Table 1). Again, the role of acid-functionality was established by performing a control experiment using a neutral ionic liquid [pmIm]Br, and no product formation was observed (entry 10, Table 1). These observations clearly demonstrate that the acidic carboxyl (–COOH) group

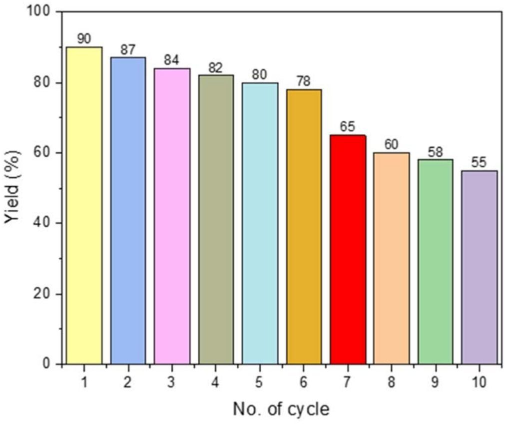
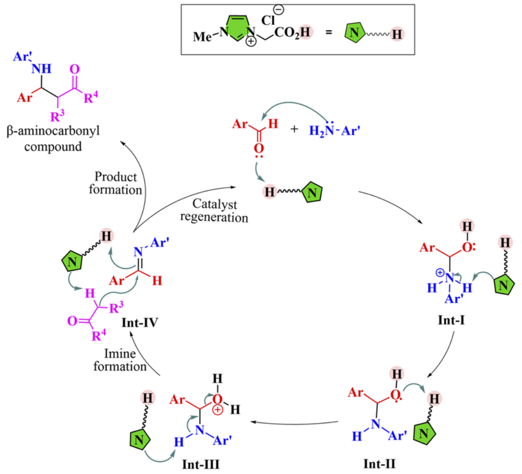


Fig. 2 Recyclability of [CmMIm]Cl for the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**) as a model reaction.



Scheme 3 Plausible mechanism for [CmMIm]Cl-catalysed synthesis of β-aminocarbonyl compound.

present in [CmMIm]Cl matrix plays a crucial role in promoting the reaction. Thus, stirring a mixture of benzaldehyde (1 mmol), aniline (1 mmol), and acetophenone (1 mmol) in [CmMIm]Cl (50 mg, 28 mol%) at room temperature was considered as the optimized reaction conditions.

Next, a series of β-aminocarbonyl compounds (**1a–1o**, Table 2) was synthesized *via* one-pot multicomponent reactions of various substituted aromatic aldehydes, aromatic amines, and acetophenone using optimized reaction conditions.

The results are summarized in Table 2, and these results demonstrate a broad substrate scope with excellent functional group tolerance. Both electron-donating (–Me, –OMe, –^{t}Bu) and electron-withdrawing substituents (–F, –Cl, –Br, –NO_2, –CN) on the aromatic aldehydes and amines afford the desired products with excellent yield percentage (90–95%). All the products were formed in a very short reaction time. Surprisingly, when the reaction was performed using *p*-bromo benzaldehyde and *p*-chlorobenzaldehyde, a significant increase in the rate of the reaction was observed, and the products were formed within 5 minutes, indicating their favourable influence on imine formation and subsequent C–C bond formation. The crude products were purified by recrystallization using hot ethanol and identified by comparing the observed melting points with previously reported values and NMR spectroscopic studies of the synthesized compounds (**1a–1o**), which showed good agreement with reported data.

To investigate the recyclability of [CmMIm]Cl in the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**), the experiment was carried out on a 2 mmol scale with 100 mg of [CmMIm]Cl. After completion of the reaction, the reaction mixture was extracted using ethyl acetate (10 mL) twice. The product **1a** was separated with ethyl acetate. After the extraction of the product, the remaining [CmMIm]Cl was then washed with ethyl acetate, dried using a rotary evaporator, and reused directly in subsequent cycles. The results are depicted in Fig. 2. The [CmMIm]Cl retained its catalytic activity for up to ten consecutive runs. After the sixth cycle, a slight decrease in the yield of product **1a** was noticed, which may be attributed to partial loss of the catalyst during the recovery steps.

A plausible mechanism for the synthesis of β-aminocarbonyl compound using [CmMIm]Cl is shown in Scheme 3. The reaction initiates with the protonation of the carbonyl oxygen atom by the acidic proton of [CmMIm]Cl,[45f] which activates the carbonyl carbon and promotes the addition of aromatic amine to give an unstable amino alcohol intermediate (Int-II). Dehydration of the Int-II produced the corresponding imine. The ketone is also converted to enol by [CmMIm]Cl, which attacks the electrophilic carbon of the imine, leading to the formation of the β-aminocarbonyl compound. The ionic liquid is regenerated for the next reaction cycle.

Table 3 Comparative study of present and previously reported methods for the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**)[a]

Entry	Catalyst/reaction condition	Solvent	Temperature (°C)	Time	Yield (%)
1	$Al(CH_3SO_3)_3\cdot 4H_2O$ (5 mol%)	Ethanol	RT	8 h	86 (ref. 19)
2	SiO_2–$OAlCl_2$	Ethanol	RT	5 h	93 (ref. 28)
3	Yb(PFO)$_3$ (1 mol%)	Ethanol	RT	8 h	94 (ref. 14*a*)
4	$NbCl_5$ (10 mol%)	Ethanol	RT	12 h	95 (ref. 16)
5	Yb(OPf)$_3$/perfluorodecalin	Toluene	60 °C	12 h	96 (ref. 14*b*)
6	DBSA	Water	23 °C	24 h	81 (ref. 23)
7	PS–SO_3H	Water	30 °C	24 h	75 (ref. 32)
8	$H_3PW_{12}O_{40}$ (0.12 mol%)	Water	RT	18 h	76 (ref. 34)
9	$BiCl_3$ (5 mol%)	Ethanol	RT	11 h	95 (ref. 17)
10	Hf(OTf)$_4$ (0.5 mol%)	Solvent-free	RT	4 h	93 (ref. 15)
11	Silzic ($ZnCl_2/SiO_2$) (0.03 g)	Solvent-free	RT	20 min	95 (ref. 29)
12	$FeCl_3$–RiH (0.04 g)	Ethanol	RT	8 h	95 (ref. 30)
13	TiO_2-NPs (20 mol%)	Solvent-free	RT	4 h	81 (ref. 37)
14	$ZrOCl_2\cdot 8H_2O$ (15 mol%)	Solvent-free	RT	4 h	65 (ref. 18)
15	SBA-15-Ph-PrSO_3H (5 mol%)	Water	RT	4 h	92 (ref. 33)
16	I_2 (5 mol%)	Solvent-free	RT	5 h	92 (ref. 27)
17	**[CmMIm]Cl (28 mol%)**	**Solvent-free**	**RT**	**1 h**	**95**[b]

[a] Comparative data is prepared for the Mannich reaction of benzaldehyde, aniline and acetophenone. [b] Present work.

Overall, [CmMIm]Cl mainly acts as a Brønsted acid and proton carrier, helping the reaction to proceed smoothly under mild reaction conditions.

A comparative study of the present and previously reported methods is provided in Table 3. Although several reported catalytic systems[14–17,28–30,33] afford comparable or slightly higher yields, these protocols are associated with various limitations, such as the use of hazardous volatile organic solvents, expensive metal salts and nanoparticles, and elevated temperatures. In this regard, the development of efficient and sustainable methodologies necessitates a more comprehensive approach to reaction design, taking into account principles such as atom economy and overall process efficiency.[47] In contrast, the [CmMIm]Cl offers a solvent- and metal-free approach for Mannich reactions at room temperature. Mild reaction conditions, operational simplicity, reusability and improved reaction efficiency render the present work a superior green alternative for Mannich reactions.

3 Conclusion

In conclusion, the present study clearly demonstrates that the task-specific carboxylic acid functionalized ionic liquid [CmMIm]Cl acts as a highly effective and reusable catalyst for the one-pot multicomponent synthesis of β-aminocarbonyl compounds *via* Mannich reaction. The transformation proceeds efficiently under ambient conditions, avoiding harsh reaction environments, corrosive Brønsted or Lewis acids, toxic nitrogen-containing bases, and excess volatile organic solvents. The strong ionic interactions of [CmMIm]Cl provide simultaneous acid–base functionality, resulting in enhanced reaction rates, excellent yields, and product purity. Notably, the catalyst exhibits remarkable operational simplicity and can be reused multiple times with minimal loss of activity, demonstrating its stability and economic advantage. Taken together, this protocol represents a greener, more sustainable, and synthetically powerful alternative to conventional Mannich methodologies. It highlights the strong potential of task-specific ionic liquids such as [CmMIm]Cl for multicomponent carbon–carbon and carbon–nitrogen bond-forming reactions.

Author contributions

Dhaneshwar Prasad: material preparation, synthesis of β-aminocarbonyl compounds, data analysis, primary draft preparation, *etc.* Kumari Anchal: characterization of the compounds. Saumy Sharma: primary draft preparation. Pranali Hadole: NMR study and data analysis. Sudip Mondal: data analysis, manuscript preparation, *etc.* Subhash Banerjee: conceptualization, supervision, review and editing, formal analysis, *etc.*

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: copies of 1H NMR and 13C NMR spectra of all the products listed in Table 2 are available in the SI. See DOI: https://doi.org/10.1039/d6ra01958g.

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