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Design, Characterization and Application of N^1,N^1,N^2,N^2-Tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium Trifluoroacetate as a Novel Ionic-Liquid Catalyst for the Production of Polyhydroquinolines

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Abstract

A novel Brønsted acidic ionic liquid namely N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium trifluoroacetate {[TMBSED][TFA]$_2$} was synthesized, and characterized by studying its ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and mass spectral data. Afterward, the ionic liquid was applied as catalyst for the production of polyhydroquinoline derivatives *via* the one-pot multi-component reaction of arylaldehydes with dimedone, β-ketoesters and ammonium acetate under solvent-free conditions. The protocol had several advantages, which include: (i) efficiency, (ii) generality, (iii) excellent yields of the products, (iv) short reaction times, (v) clean reaction profile, (vi) simplicity, (vii) usage of a few amount of the catalyst, (viii) easy preparation the catalyst from available and inexpensive starting materials, (ix) mild reaction conditions, (x) performing the reaction in solvent-free conditions, and (xi) easy work-up.

Keywords:*Brønsted acidic ionic liquid, N^1,N^1,N^2,N^2-Tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium trifluoroacetate {[TMBSED][TFA]$_2$}, Polyhydroquinoline, Multi-component reaction, Solvent-free.*

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Introduction

Ionic liquids (ILs) have diverse unique properties such as reasonable thermal and chemical stability, negligible vapor pressure, extensive liquid range, non-flammability, excellent ionic conductivity, wide electrochemical windows, green nature, changeable chemical and physical characteristics by varying cation or anion, and tunable hydrophobicity; consequently, they have been extensively used in industry and chemistry [1-10]; e.g. they have been applied in electrode position [1], electric double layer capacitors [2], lithium batteries [3], and solar cells [4]. Moreover, ILs has been utilized as solvent, reagent and catalyst for organic transformations [5-10]. Among the different classes of ILs, Brønsted acidic ones possess the helpful properties of solid acids and mineral liquid acids, and have been designed as catalysts to promote chemical reactions [7-10].

Multi-component reactions (MCRs) are defined as reactions in which three or more starting materials are reacted in a single vessel to produce aim product without separation of any intermediate; thus, they minimize synthetic time and effort. Moreover, MCRs are associated with high selectivity and atom economy, and are applicable for preparation of heterocyclic compounds [11-13].

The one-pot multi-component condensation of arylaldehydes with dimedone, β-ketoesters and ammonium acetate is of importance as this reaction provides a simple route toward synthesis of polyhydroquinolines (as a significant class of 1,4-dihydropyridine-containing heterocycles). The compounds bearing 1,4-dihydropyridine moiety have a variety of biological activities, consisting of anti-atherosclerotic, hepatoprotective, vasodilatory, antitumor, geroprotective, bronchodilatory, and anti-diabetic properties; furthermore, these compounds are applied as calcium channel modulators and curatives for cardiovascular diseases [14-18]. They are also utilized as chemosensitizers in tumor therapy, as neuroprotectants and platelet anti-aggregatory agents, and as cerebral anti-ischemic agents in the treatment of Alzheimer's disease [19,20]. Some catalysts have been applied to promote the synthesis of polyhydroquinolines, e.g.nanometasilica disulfuric acid [21], Co_3O_4-CNT [22], chitosan-$CuSO_4$ [23], $(NH_4)_2Ce(NO_3)_6$ [24], MoO_2Cl_2 [25], hafnium (IV) bis(perfluorooctanesulfonyl)imide complex [26],magnetic nanoparticles [27], $Yb(OTf)_3$ [28], *threo*-(1*S*,2*S*)-2-amino-1-(4′-nitrophenyl)-1,3-propanediol [29], and ZnO nanoparticles [30]. Nevertheless, most of the reported methods and catalysts have one or more of the following drawbacks: (i) the use of large amount of catalyst, (ii) application of toxic or expensive catalysts, (iii) long reaction times, (iv) moderate yields, (v) harsh reaction conditions, (vi) tedious work-up procedure, (vii) performing the reaction in volatile organic solvents, and (viii) poor agreement with the green chemistry protocols. So, search for finding more efficient, green and inexpensive catalysts for the preparation of polyhydroquinolines under milder reaction conditions is still of importance.

Having the above facts in mind, we report here preparation and characterization of a novel Brønsted acidic ionic liquid namely N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium trifluoroacetate {[TMBSED][TFA]$_2$} using ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and mass spectra, and then its application as a highly efficient and general catalyst for the one-pot multi-component condensation of arylaldehydes with dimedone (5,5-dimethylcyclohexane-1,3-dione), β-ketoesters and ammonium acetate under solvent-free conditions to furnish polyhydroquinolines as biologically important heterocycles.

Experimental

Materials and methods

All chemicals were purchased from Merck or Fluka Chemical Companies. All known compounds were identified by comparison of their melting points and spectral data with those reported in the literature. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. Progress of the reactions was monitored by TLC using silica gel SIL G/UV 254 plates. The ^{1}H NMR (300 or 500 MHz), ^{13}C NMR (75 or 125 MHz) and ^{19}F NMR (235 MHz) were run on a Bruker Avance DPX, FT-NMR spectrometers (δ in ppm). Mass spectra were recorded on spectrometer 5975C VL MSD model Tripe-Axis Detector.

Preparation of [TMBSED][TFA]$_2$

A solution of N^1,N^1,N^2,N^2-tetramethylethane-1,2-diamine (5 mmol, 0.581 g) in dry CH_2Cl_2 (30 mL) was added dropwise to a stirring solution of chlorosulfonic acid (10 mmol, 1.165 g) in dry CH_2Cl_2 (30 mL) over a period of 10 min, at 10 °C. After that, the reaction mixture was heated to room temperature (accompanied with stirring), and stirred for another 4 hours. The solvent was evaporated under reduced pressure, and the liquid residue was triturated with dry petroleum ether (3×2 mL), and dried under powerful vacuum at 90 °C to give [TMBSED][Cl]$_2$. Then, trifluoroacetic acid (10 mmol, 1.140 g) was added dropwise to [TMBSED][Cl]$_2$ (5 mmol, 1.746 g) over a period of 3 min at room temperature under pressure of nitrogen gas (to remove the HCl which produced during the reaction). The resulting mixture was stirred for 10 h at room temperature, and 2 hours at 60 °C under a continuous flow of nitrogen gas to give [TMBSED][TFA]$_2$as a viscous pale yellow liquid. ^{1}H NMR (500 MHz, DMSO-d$_6$, δ/ppm): 2.99 (s, 12H), 3.66 (s, 4H), 9.85 (br., 2H). ^{13}C NMR (125.7 MHz, DMSO-d$_6$, δ/ppm): 43.9, 51.7, 116.0 (q), 159.5 (q). ^{19}F NMR (235 MHz, DMSO-d$_6$, δ/ppm): -70.64. MS (*m/z*): 504 (M^+),505 (M^++1).

General procedure for the synthesis of polyhydroquinolines

To a mixture of arylaldehyde (1 mmol), dimedone (1 mmol, 0.140 g), β-ketoester (1 mmol) and ammonium acetate (1.4 mmol, 0.108 g), was added [TMBSED][TFA]$_2$(0.05 mmol, 0.025 g). The resulting mixture was firstly stirred magnetically at 60 °C, and after solidification of the reaction mixture, it was vigorously stirred with a small rod at the same temperature. After completion of the reaction, as monitored by TLC, the reaction mixture was cooled to room temperature, and the crude product was purified by recrystallization from ethanol (95%) or column chromatography eluted with *n*-hexane/ethyl acetate.

Selected spectroscopic data of polyhydroquinolines

*Ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1a**)*

^{1}H NMR (300 MHz, DMSO-d$_6$, δ/ppm): 0.85 (s, 3H), 1.00 (s, 3H), 1.13 (t, 3H, *J* = 7.0 Hz), 2.01-2.20 (m, 2H), 2.29 (s, 3H), 2.38-2.50 (m, 2H), 3.97 (q, 2H, *J* = 7.0 Hz), 4.82 (s, 1H), 7.05 (m, 1H), 7.18 (t, 2H, *J* = 6.7 Hz), 7.21 (t, 2H, *J* = 6.5 Hz), 9.12 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d$_6$, δ/ppm): 14.5, 18.8, 26.8, 29.5, 32.6, 36.5, 50.6, 59.6, 103.4, 109.9, 113.5, 126.9, 128.8, 130.5, 146.0, 150.3, 167.0, 194.7.

*Ethyl 2,7,7-trimethyl-4-(4-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1b**)*

^{1}H NMR (500 MHz, CDCl$_3$, δ/ppm): 0.92 (s, 3H), 1.10 (s, 3H), 1.19 (t, 3H, *J* = 7.1 Hz), 2.16 (d, 2H, *J* = 16.4 Hz), 2.24-2.29 (Distorted AB system, 2H), 2.41 (s, 3H), 4.07 (q, 2H, *J* = 7.1 Hz), 5.18 (s, 1H), 6.68 (s, 1H), 7.51 (d, 2H, *J* = 8.5 Hz), 8.09 (d, 2H, *J* = 8.5 Hz). ^{13}C NMR (125 MHz, CDCl$_3$, δ/ppm): 14.6, 19.8, 27.5, 29.8, 33.1, 37.7, 41.3, 51.0, 60.5, 105.3, 111.4, 123.7, 129.4, 145.0, 146.6, 149.6, 154.9, 167.3, 195.9.

*Ethyl 2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1c**)*

^{1}H NMR (500 MHz, DMSO-d$_6$, δ/ppm): 0.82 (s, 3H), 1.00 (s, 3H), 1.10 (t, 3H, *J* = 7.1 Hz), 1.98 (d, 1H, *J* = 16.1 Hz), 2.18 (d, 1H, *J* = 16.1 Hz), 2.31 (s, 3H), 2.43-2.99 (m, 2H), 3.96 (q, 2H, *J* = 7.1 Hz), 4.96 (s, 1H), 7.51 (t, 1H, *J* = 7.8 Hz), 7.61 (d, 1H, *J* = 7.7 Hz), 7.97 (d, 2H, *J* = 8.4 Hz), 9.25 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d$_6$, δ/ppm): 14.5, 18.8, 26.8, 29.5, 32.6, 36.9, 50.5, 59.7, 103.1, 109.7, 121.4, 122.5, 129.9, 134.8, 146.6, 147.8, 150.2, 150.6, 166.8, 194.8.

*Ethyl 2,7,7-trimethyl-4-(4-hydroxyphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1f**)*

^{1}H NMR (500 MHz, DMSO-d$_6$, δ/ppm): 0.86 (s, 3H), 0.99 (s, 3H), 1.13 (t, 3H, *J* = 7.1 Hz), 1.96 (d, 1H, *J* = 16.0 Hz), 2.14 (d, 1H, *J* = 16.0 Hz), 2.25 (s, 3H), 2.37-2.46 (m, 2H), 3.97 (q, 2H, *J* = 7.0

Hz), 4.73 (s, 1H), 6.55 (d, 2H, *J* = 8.4 Hz), 6.92 (d, 2H, *J* = 8.3 Hz), 8.97 (s, 1H), 9.04 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 14.6, 18.7, 26.9, 29.6, 32.2, 32.6, 35.2, 50.8, 59.4, 104.6, 110.7, 114.9, 128.9, 138.9, 144.8, 149.3, 155.7, 167.5, 194.7.

*Ethyl 4-(4-bromophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1h**)*

^{1}H NMR (300 MHz, DMSO-d_6, δ/ppm): 0.83 (s, 3H), 0.99 (s, 3H), 1.10 (t, 3H, *J* = 6.9 Hz), 1.96 (d, 1H, *J* = 16.0 Hz), 2.16 (d, 1H, *J* = 16.1 Hz), 2.29 (s, 3H), 2.38-2.49 (m, 2H), 3.97 (q, 2H, *J* = 7.0 Hz), 4.84 (s, 1H), 7.11 (d, 2H, *J* = 7.2 Hz), 7.37 (d, 2H, *J* = 7.2 Hz), 9.09 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d_6, δ/ppm): 14.6, 18.8, 26.9, 29.5, 32.6, 36.2, 50.6, 59.5, 103.5, 110.1, 119.1, 130.2, 131.0, 145.8, 147.4, 150.0, 167.1, 194.7.

*Methyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1l**)*

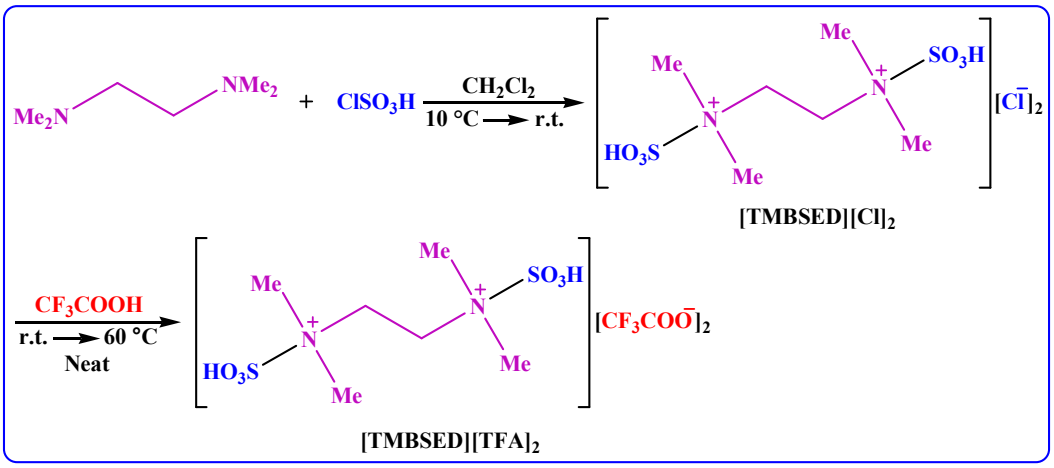
^{1}H NMR (300 MHz, DMSO-d_6, δ/ppm): 0.84 (s, 3H), 0.99 (s, 3H), 1.97 (d, 1H, *J* = 16.0 Hz), 2.15 (d, 1H, *J* = 16.1 Hz), 2.28 (s, 3H), 2.37-2.49 (m, 2H), 3.52 (s, 3H), 3.66 (s, 3H), 4.81 (s, 1H), 6.73 (d, 2H, *J* = 7.4 Hz), 7.05 (d, 2H, *J* = 7.4 Hz), 9.02 (s, 1H). ^{13}C NMR (75 MHz, DMSO-d_6, δ/ppm): 18.7, 26.9, 29.6, 32.6, 35.2, 50.7, 51.1, 55.3, 104.0, 110.7, 113.6, 128.7, 140.3, 145.4, 149.7, 157.7, 167.9, 194.7.

*Methyl 2,7,7-trimethyl-4-(4-tolyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (**1m**)*

^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 0.83 (s, 3H), 0.99 (s, 3H), 1.97 (d, 1H, *J* = 16.1 Hz), 2.15 (d, 1H, *J* = 16.1 Hz), 2.18 (s, 3H), 2.25-2.28 (Distorted AB system, 4H), 2.41 (d, 1H, *J* = 16.9 Hz), 3.51 (s, 3H), 4.83 (s, 1H), 6.97 (d, 2H, *J* = 7.9 Hz), 7.03 (d, 2H, *J* = 7.9 Hz), 9.05 (s, 1H). ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 18.7, 21.0, 26.9, 29.6, 32.6, 35.6, 39.9, 50.7, 51.1, 103.9, 110.6, 127.7, 128.8, 135.0, 145.1, 145.5, 149.8, 167.8, 194.7.

Results and discussion

Considering the unique properties of Brønsted acidic ionic-liquid catalysts, we decided to design a novel and attractive member of these catalysts. For this purpose, N^1,N^1,N^2,N^2-tetramethylethane-1,2-diamine(1 eq.) was reacted with chlorosulfonic acid (2 eq.), and then with CF_3CO_2H (2 eq.) to give N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminiumtrifluoroacetate [TMBSED][TFA]$_2$} as a Brønsted acidic IL (Scheme 1).



Scheme 1. The synthesis of [TMBSED][TFA]$_2$.

In this section, we characterize the ionic liquid by studying its ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and mass spectral data.In the ^{1}H NMR spectrum of [TMBSED][TFA]$_2$ (Figure 1), three peaks were observed; the corresponding data are summarized in Table 1.

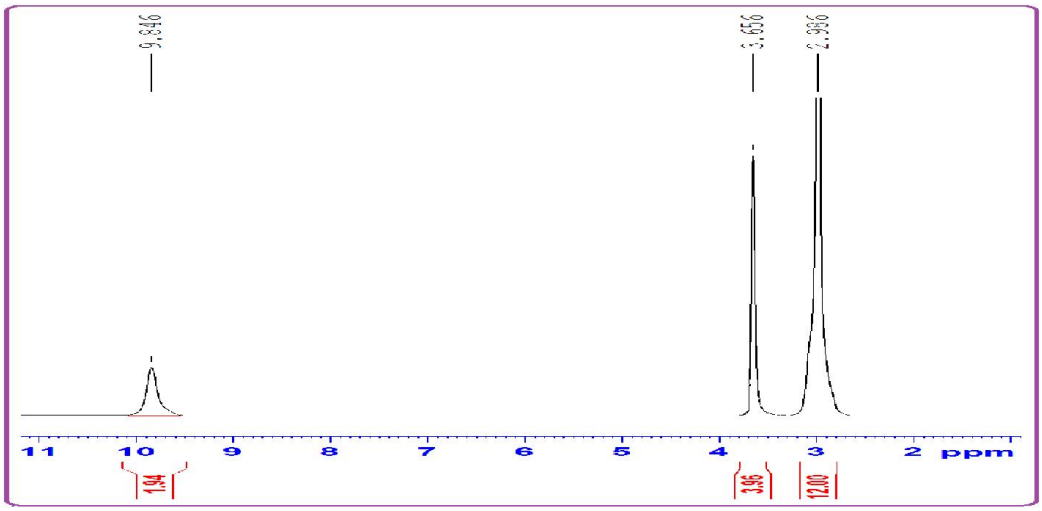


Figure 1. The ^{1}H NMR spectrum of the catalyst.

Table 1. The ^{1}H NMR data of [TMBSED][TFA]$_2$.

Chemical shift (ppm)	Splitting pattern	Integral	Corresponding hydrogens
2.99	Singlet	12	Four CH_3 groups
3.66	Singlet	4	Two CH_2 groups
9.85	Singlet	2	Two acidic hydrogens of the SO_3H groups

The ^{13}C NMR spectrum of [TMBSED][TFA]$_2$ is shown in Figure 2, and its data are displayed in Table 2.

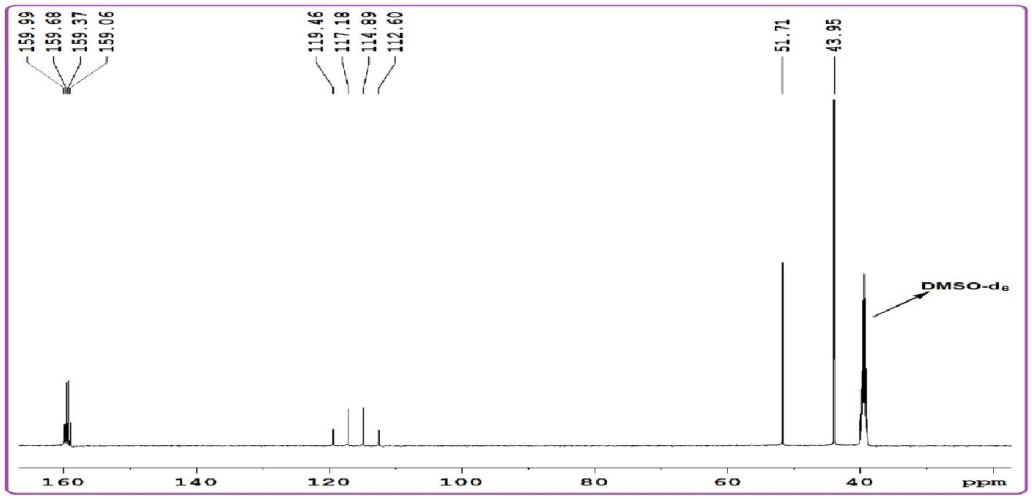


Figure 2. The ^{13}C NMR spectrum of [TMBSED][TFA]$_2$.

Table 2. The ^{13}C NMR data of the ionic liquid.

Chemical shift (ppm)	Splitting pattern	Corresponding carbons
43.9	Singlet	Four CH_3 groups
51.7	Singlet	Two CH_2 groups
116.0	Quartet	CF_3 groups of the two trifluoroacetate anions
159.5	Quartet	Carbonyl groups of the two trifluoroacetate anions

In the ^{19}F NMR spectrum (Figure 3), a singlet peak was observed at -70.64 ppm which corresponds to 6 fluorine atoms of the two trifluoroacetateanions.

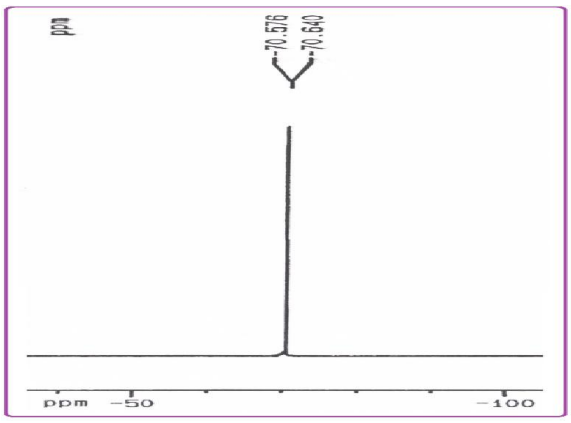


Figure 3. The ^{19}F NMR spectrum of the ionic liquid.

In the mass spectrum of [TMBSED][TFA]$_2$ (Figure 4), the peaks relevant to the molecular mass (M^+) and (M^++1) were observed at *m/z* 504 and 505, respectively.

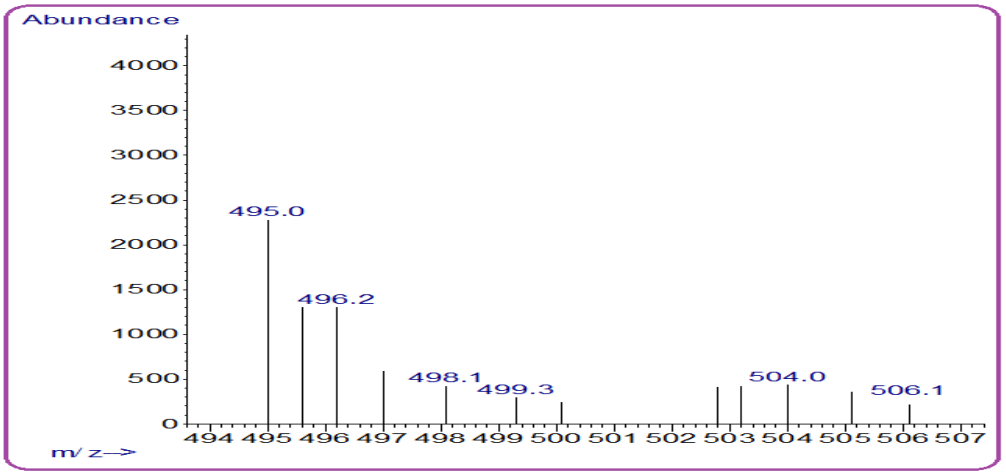
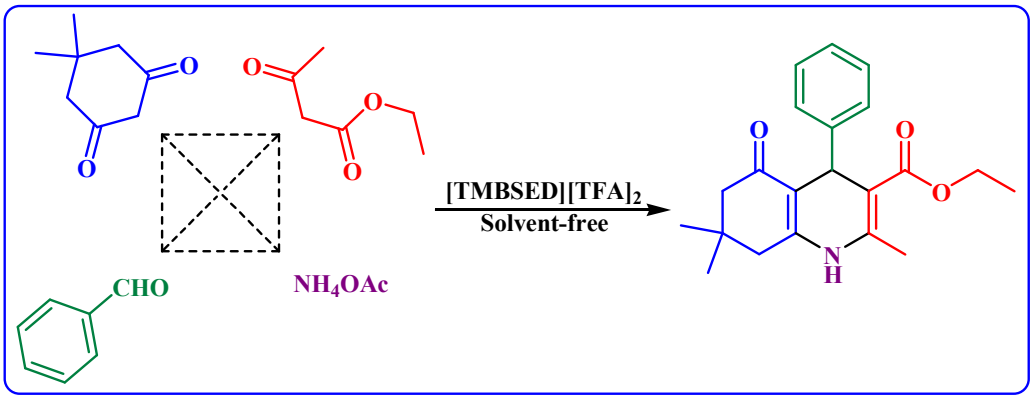


Figure 4. The mass spectrum of [TMBSED][TFA]$_2$.

After the full characterization of[TMBSED][TFA]$_2$, its catalytic applicability was examined to promote the synthesis of polyhydroquinolines. First of all, the condensation of dimedone (1 mmol) with benzaldehyde (1 mmol), ethyl acetoacetate (1 mmol) and ammonium acetate (1.4 mmol) was selected as a model reaction (Scheme 2), and studied in the presence of different mol percentages of the catalyst at 50-70 °C under solvent-free conditions. The results are displayed in Table 3. As it is clear from this Table, higher yield and shorter reaction time were obtained when 5 mol% of [TMBSED][TFA]$_2$ was applied at 60 °C (Table 3, entry 2). Increasing the reaction time or the catalyst amount decreased the yield.



Scheme 2. The model reaction.

Table 3. Effect of the catalyst amount and temperature on the reaction of dimedone with benzaldehyde, ethyl acetoacetate and ammonium acetate.

Entry	The catalyst amount (mol%)	Temp. (°C)	Time (min)	Yield[a] (%)
1	2.5	60	25	94
2	5	60	12	97
3	10	60	15	95
4	5	50	20	90
5	5	70	15	92

[a]*Isolated yield.*

To evaluate efficiency and generality of the catalyst, a variety of arylaldehydes (benzaldehyde as well as arylaldehydes possessing electron-withdrawing groups, electron-donating groups and halogens) were reacted with dimedone, β-ketoesters (ethyl and methyl acetoacetate) and ammonium acetate under the optimal reaction conditions. The respective results are summarized in Table 4. As Table 4 indicates, all reactions proceeded efficiently to furnish the desired polyhydroquinolines in excellent yields and in short reaction times. Thus, our acidic ionic liquid, was a highly efficient and general catalyst to promote a reaction that needs an acidic catalyst, i.e. the preparation of polyhydroquinolines.

Table 4. The solvent-free synthesis of polyhydroquinolines using $[TMBSED][TFA]_2$.

Product	X	R	Time (min)	Yield[a] (%)	m.p. (°C) [Lit.]
1a	H	CH_3CH_2	12	97	201-203 (203-205)[29]
1b	*p*-NO_2	CH_3CH_2	45	95	244-246 (245-247)[22]
1c	*m*-NO_2	CH_3CH_2	60	90	179-181 (177-179) [29]
1d	*p*-OCH_3	CH_3CH_2	10	96	256-258 (257-259)[28]
1e	*p*-CH_3	CH_3CH_2	20	97	261-263 (260-261)[28]
1f	*p*-OH	CH_3CH_2	10	94	231-233 (232-234)[28]
1g	*p*-$N(CH_3)_2$	CH_3CH_2	25	93	228-230 (229-231)[29]

1h	*p*-Br	CH_3CH_2	30	96	252-254 (255-257)[29]
1i	*m*-Br	CH_3CH_2	45	93	233-235 (235-237) [29]
1j	*m*-Cl	CH_3CH_2	40	96	230-232 (234-235) [24]
1k	H	CH_3	20	95	259-261 (260-262) [26]
1l	*p*-OCH_3	CH_3	35	93	256-258 (251-252)[30]
1m	*p*-CH_3	CH_3	12	96	281-283 (283-285) [26]

[a]*Isolated yield.*

Conclusion

In conclusion, we have introduced N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium trifluoroacetate as a novel Brønstedacidic ionic-liquid catalyst for an organic transformation which promote by an acidic catalyst. Application of this catalyst for the production of polyhydroquinolines is associated with many benefits; e.g. efficiency, generality, excellent yields of the products, short reaction times, cleaner reaction profile, simplicity, the use of a few amount of the catalyst, easy preparation the catalyst from available and inexpensive starting materials, mild reaction conditions, performing the reaction under solvent-free conditions, and easy work-up

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