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A novel imidazolium-based acidic ionic liquid as an efficient and reusable catalyst for the synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazoles

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Abstract A novel acidic ionic liquid based on imidazoliumcation is designed, synthesized, and successfully used as a catalyst for the one-pot synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives. The remarkable feature of this new catalyst is its ethyleneoxy bridge which participates in dissolving organic compounds in ionic liquids. The application of this acidic ionic liquid is studied in a new one-pot method for the synthesis of imidazole derivatives under solvent-free conditions. The advantages of this method are the reusability of the catalyst, high conversion, short reaction time, and simple experimental procedure.

Keywords Acidic ionic liquid · Imidazolium cation · Phenanthro[9,10-*d*]imidazole · Reusable catalyst

Introduction

Applications of solvent-free reactions in organic synthesis [1–3], particularly those based on imidazolium cations, have gained attention in recent years [4, 5]. Some of the unique physical and chemical properties of imidazolium ionic liquids are thermal stability, negligible vapor pressure, recyclability, ability to dissolve a wide range of organic and inorganic compounds, fast reaction rate, selectivity, and tendency to immobilize starting materials and catalyst [6]. For these reasons, they have been used in different areas of organic synthesis, as both catalyst and solvent [7, 8].

Imidazole and its derivatives are also receiving growing attention for their pharmacological properties, such as herbicidal, fungicidal, analgesic, anti-inflammatory, and antithromobotic [9]. Several methods have reported on the synthesis of

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2,4,5-triarylimidazoles in the presence of a catalyst such as copper(II) acetate [10], $ZrCl_4$ [11], AcOH [12, 13], $Yb(OTf)_3$ [14], $NbCl_3$, [14], $LaCl_3$ [14], $FeCl_3$ [14], $AlCl_3$ [14], $NiCl_2$ [15], and $[HBim]BF_4$ [16]. However, some of these methods suffer from one or more disadvantages such as prolonged reaction time, low yield of products, and volatile and hazardous organic solvents. Therefore, the search for a clean procedure using a green and eco-friendly catalyst with high catalytic activity and short reaction time for the production of 2,4,5-triarylimidazoles has gained considerable attention.

In recent years, dicationic ionic liquids (ILs) have become interesting research targets due to their high thermal stability, broad liquid range, and biological activities such as antiviral, antifungal, and anticancer. In particular, imidazolium-based dicationic ILs showing high thermal stability were used as effective reaction media for high temperature organic reactions [7, 17–19]. Some studies have reported imidazolium-based dicationic ILs that bridged via alkyl and aryl and alkoxy chains as a linker between imidazolium rings [20–23]. Here, we report for the first time a novel acidic ionic liquid (AIL) which contains an ethyleneoxy bridge and a sulfonic acid group, that participates in dissolving organic compounds in ILs with its ethyleneoxy bridge and SO_3H functionalizing as a Brønsted AIL catalyst.

The first ability of this novel catalyst is shown by the rapid synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives from 9,10 phenanthroquinone with various aromatic aldehydes and ammonium acetate in the presence of a catalytic amount of this AIL under solvent-free conditions. We believe that this novel AIL can be applied as a catalyst in many different organic transformations.

Experimental

Materials and instrumentation

All reagents were purchased from Merck and used without further purification. The melting points of the products were determined with an Electrothermal Type 9100 melting point apparatus. The FT-IR spectra were recorded on an Avatar 370 FT-IR Therma Nicolet spectrometer. The mass spectra were recorded on a 5973 Network Mass Selective Detector. The ^1H and ^{13}C NMR spectra were recorded on a Bruker AC 100 and Bruker DRX-400 Avance spectrometers at 400 and 100.65 MHz, respectively, using DMSO-d_6 or $CDCl_3$ as the deuterated solvents. Chemical shifts are reported in ppm downfield from TMS as internal standard; coupling constants *J* are given in Hz.

Synthesis of 1,2-bis(2-(1*H*-imidazol-1-yl)ethoxy)ethane (**1**):

A mixture of imidazole (32 mmol, 2.176 g) and potassium hydroxide (32 mmol, 1.792 g) in DMSO (25 ml) was stirred at 70 °C for 1 h. After this time, 1,2-bis(2-chloroethoxy)ethane (16 mmol, 2.48 ml) was added to the reaction mixture and stirred for 24 h. The resulting mixture was poured into 100 mL of water and extracted with methylene chloride (5 × 20 ml). The combined organic layer was

washed with water (3 × 20 ml), dried with anhydrous Na_2SO_4, and concentrated to give 1,2-bis(2-(1*H*-imidazol-1-yl)ethoxy)ethane (**1**) as a pale yellow viscous oil, 3.10 g, 77 % yield.^{1}H NMR ($CDCl_3$, 400 MHz) δ:3.45 (s, 4H), 3.59 (t, 4H, $J = 4.8$ Hz), 4.01 (t, 4H, $J = 4.8$ Hz), 6.90 (t, 2H, $J = 1.2$ Hz), 6.94 (t, 2H, $J = 1.2$ Hz), 7.45 (s, 2H). ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 46.98, 70.38, 70.49, 119.39, 128.93, 137.48. IR (KBr, cm^{-1}): 1,642, 1,525, 1,425. *m/z*, calcd. for $C_{12}H_{18}N_4O_2$ $[M]^+$: 250.30, found: 250.0.

Synthesis of 1,1′-((ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(3-sulfo-1*H*-imidazol-3-ium) chloride (**2**):

A 100-ml round-bottomed flask was charged with 1,2-bis(2-(1*H*-imidazol-1-yl)ethoxy)ethane (**1**) (12.4 mmol, 3.1 g) in dry CH_2Cl_2 (50 ml), and then chlorosulfonic acid (24.8 mmol, 1.64 ml) was added dropwise over a period of 15 min at room temperature. Afterward, the reaction mixture was stirred for 45 min, stand for 5 min, and the CH_2Cl_2 was decanted. The residue was washed with dry CH_2Cl_2 (3 × 20 mL) and dried under vacuum to give 1,1′-((ethane-1,2-diyl-bis(oxy))bis(ethane-2,1-diyl))bis(3-sulfo-1*H*-imidazol-3-ium) chloride (**2**) as a viscous colorless oil. ^{1}H NMR (DMSO-d_6, 400 MHz) δ:3.46 (s, 4H), 3.69 (t, 4H, $J = 4.8$ Hz), 4.33 (t, 4H, $J = 4.8$ Hz), 7.60 (t, 2H, $J = 1.6$ Hz), 7.66 (t, 2H, $J = 1.6$ Hz), 8.98 (t, 2H, $J = 1.6$ Hz), 14.32 (bs, 2H). ^{13}C NMR (DMSO-d_6, 400 MHz) δ: 48.80, 68.60, 69.75, 120.13, 122.71, 135.94. IR (KBr, cm^{-1}): 3425 (broad, SO_3H), 1,642, 1,580, 1,549. *m/z*, calcd. for $C_{12}H_{20}Cl_2N_4O_8S_2$ $[M]^+$: 482.01, found: 481.9.

General procedure for preparation of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives

A 10-ml round-bottom flask was charged with the mixture of phenanthraquinon (1 mmol), aromatic aldehyde (1 mmol), ammonium acetate (6 mmol), and IL (0.05 mmol). The mixture was stirred at 120 °C for 5–10 min. After completion of the reaction (monitored by thin-layer chromatography, TLC), the mixture was cooled to room temperature and extracted thoroughly with acetone (3 × 10 ml). The combined extracts were filtered and the solvent was removed under reduced pressure to afford the crude product, which was purified by recrystallization from ethyl acetate. The catalyst which does not dissolve in acetone, remained in the residue.

Selected spectroscopic data

*2-p-Tolyl-1H-phenanthro[9,10-d]imidazole (**5**)*

IR (KBr, cm^{-1}): 3,407, 1,616, 1,525, 1,425. ^{1}H NMR ($CDCl_3$, 100 MHz) δ: 2.40 (s, 3H), 7.40 (d, 2H, $J = 8$ Hz), 7.60–7.80 (m, 4H,), 8.30 (d, 2H, $J = 8$ Hz), 8.60 (d, 2H, $J = 7.6$ Hz), 8.75(d, 2H, $J = 8$ Hz).

*N,N-Diethyl-4-(1H-phenanthro[9,10-d]imidazol-2-yl)aniline (**6**)*

IR (KBr, cm^{-1}): 3,432, 3,068, 1,609, 1,525, 1,425. ^1H NMR (DMSO, 100 MHz) δ: 1.2 (t, 6H, $J = 5.4$ Hz), 3.45 (m, 4H), 6.81 (d, 2H, $J = 8$ Hz), 7.50-7.72 (m, 4H), 8.09 (d, 2H, $J = 8$ Hz), 8.53(d, 2H, $J = 7.4$), 8.83 (d, 2H, $J = 7.6$ Hz).

*2-(4-Isopropylphenyl)-1H-phenanthro[9,10-d]imidazole (**8**)*

mp 178 °C. IR (KBr, cm^{-1}): 3,434, 1,615, 1,540, 1,425. ^1H NMR ($CDCl_3$, 400 MHz) δ:1.35 (d, 6H, $J = 6.8$ Hz), 3.05 (m, 1H), 7.45 (d, 4H, $J = 8.4$ Hz), 7.75 (m, 8H), 8.34 (d, 4H, $J = 8.4$ Hz), 8.4 (d, 2H, $J = 7.6$ Hz), 8.68 (d, 2H, $J = 8$ Hz), 8.79 (t, 4H, $J = 8.4$ Hz). ^{13}C NMR ($CDCl_3$, 400 MHz) δ: 23.8, 34.2, 120.8, 121.1, 123.0, 123.4, 123.78, 125.2, 126.0, 126.3, 127.0, 127.2, 127.3, 127.4, 128.9, 129.2, 152.2. *m/z*, calcd. for $C_{24}H_{20}N_2$ $[M]^+$: 336.16, found: 336.0.

*2-(4-Hydroxyphenyl)-2H-phenanthro[9,10-d]imidazole (**11**)*

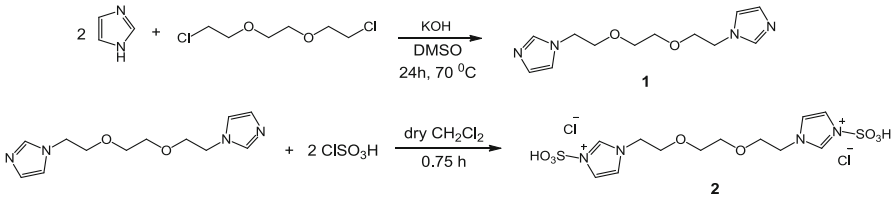
IR (KBr, cm^{-1}): 3,410, 3,357, 1,611, 1,552. ^1H NMR (DMSO, 100 MHz) δ: 6.95 (d, 2H, $J = 8$ Hz), 7.50–7.75 (m, 4H), 8.12 (d, 2H, $J = 8$ Hz), 8.52(d, 2H, $J = 7.4$ Hz), 8.82 (d, 2H, $J = 7.6$ Hz), 13.20 (s, OH).

Results and discussion

Preparation and characterization of dicationic acidic ionic liquid

We designed and synthesized a novel AIL based on imidazolium cations with a triethylenoxy spacer. In this protocol, we first synthesized 1,2-bis(2-(1*H*-imidazol-1-yl)ethoxy)ethane (**1**) through the reaction between imidazole and 1,2-bis(2-chloroethoxy)ethane in the presence of potassium hydroxide in DMSO. Then, for acidic functionalization, we used chlorosulfonic acid in dry dichloromethane and an AIL (**2**) was obtained in 95 % yield as a viscous oil (Scheme 1).

One of the key properties of Brønsted AILs is their Brønsted acidity which has a strong correlation to the catalytic activity of this compound in the many organic synthesis reactions [24]. SO_3H-functionalized ILs have been employed as Brønsted AIL catalysts for the synthesis of several acid-catalyzed organic reactions. In recent years, there have been reports about Brønsted AILs based on the sulfonic acid group which have good activity [25, 26]. The purpose of the preparation of this novel AIL is to increase the solubility of the organic compounds in the AIL through triethyleneoxy spacer that increase its flexibility and hydrophobisity. In addition, longer oligo (ethylene glycol) chains may improve the hydrophobisity of the overall AIL [29, 30]. In the synthesis of this novel AIL, the sulfonic acid group is covalently tethered to the AIL imidazolium cation and this part of the AIL can increase its hydrophilisity. So, features of both hydrophilisity and hydrophobisity are responsible for increasing the dissolving of organic compounds in this AIL. In addition, strong acidity by determination of pKa and high catalyst



Scheme 1 Synthesis of acidic ionic liquid

activity for the synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole were observed for this AIL. This catalyst can be described as a sulfuric acid derivative with the difference that the AIL participates in dissolving organic compounds by its ethyleneoxy bridge.

pH measurement of dicationic AIL

The pK_a value for the mentioned AIL was determined using a 0.1 mol L^{-1} solution of IL and was titrated with 0.1 mol L^{-1} of NaOH. The pH of the solution was measured using a calibrated glass electrode pH meter at 25 °C. As shown in Fig 1, the pK_a value of the AIL is 2.41. This novel AIL can be used as a catalyst in many different organic transformations.

The application of this AIL has been studied in a new one-pot method for the synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole from 9,10-phenanthraquinone with various aromatic aldehydes and ammonium acetate in the presence of a catalytic amount of the AIL under solvent-free conditions (Scheme 2). The corresponding desired products were isolated in excellent yields and the results are summarized in Table 1. Our results show that these reactions are rapid and completed in a few minutes. However, a logic substitution effect was not observed by changing the R group in the aldehyde.

Thermal stability of dicationic AIL

The thermal stability of the dicathionic AIL was measured by a thermogravimetric analyzer (TGA) and differential thermal analysis (DTA) under argon atmosphere with a heating rate of 10 °C/min. The TGA and DTA curves obtained for synthesized AIL are shown in Fig 2. Using TG–DTA, one can tell the melting or decomposition points of the material. Thermal decomposition temperature (T_d) values for the mentioned AIL were obtained at 348 °C. It was observed that the thermal stability of the synthesized dicationic AIL in this study was significantly higher than that of many traditional imidazolium-based monocationic analogs [18, 19]. In the DTA and TGA curves, a weight loss (8.84 %) was observed at 131 °C. With the loss of one equivalent HCl from AIL, a 7.5 % weight loss was calculated with the remaining 1.34 % pertaining to water removal. So both HCl and water were removed at 131 °C.

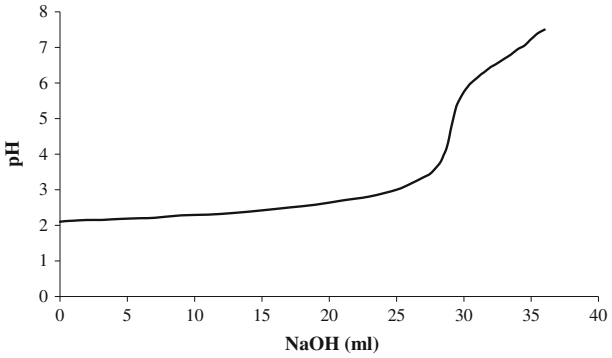
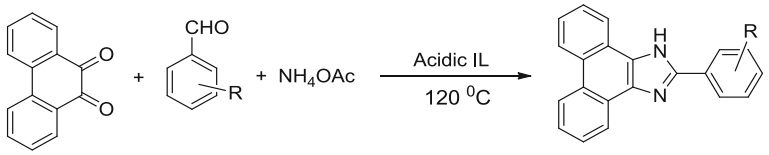


Fig. 1 pH meter titration curve of the novel dicationic AIL



Scheme 2 Synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives

Molecular geometry of AIL

The optimized geometry of the synthesized dicationic AIL is shown in Fig. 2 and the minimum energy geometries of the AIL were determined by performing ab initio geometry optimizations at the RHF/6-31G level [31]. All calculations were carried out using the Gaussian 09 series programs and the results are shown in Table 2. In the optimized molecular structure of the AIL demonstrated in Fig 3, there is a free HCl molecule which correlates with the acidity strength of sulfonic acid and hydrochloric acid. On the other hand, the distances of hydrogen from the nitrogen of the imidazolium ring, for two active sites, are H_{44}–N_{35} = 2.57712 and H_{46}–N_{36} = 3.17257, which shows that the distance for one side of the free HCl molecule is longer than the other side. This optimized structure reveals that the most stable form of AIL is anti-direction of the HCl molecule and chloride and also that the two imidazolium rings are opposite to each other.

Edgar et al. [32] isolated 9,l0-phenanthraquinone di-imine intermediates in the reactions of phenanthraquinone, aldehydes, and ammonium acetate in acetic acid solution and suggested a mechanism of reaction for imidazole formation under these conditions. In their mechanism, the initial reaction is the formation of the 9,l0-phenanthraquinone di-imine. The latter then undergoes an aminal condensation with the aldehyde to form trisubstitutedimidazoles. In a possible mechanism, in the initial step an aminal intermediate is formed which then reacts with phenantraquinone to give the desired product (Scheme 3) [33].

Table 1 Results of 2-phenyl-1*H*-phenanthro[9,10-*d*]imidazole derivatives synthesis using a novel acidic IL (**2**)

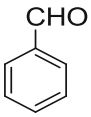
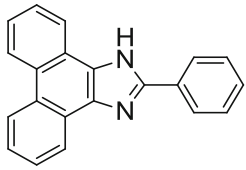
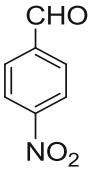
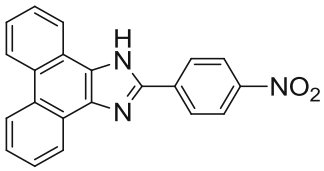
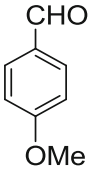
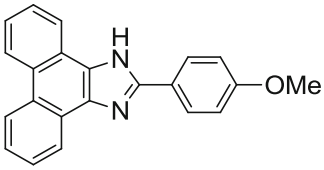
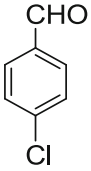
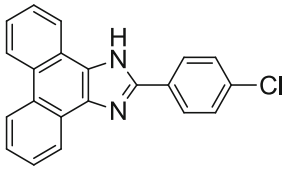
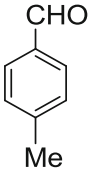
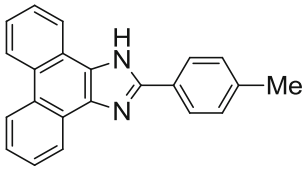
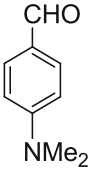
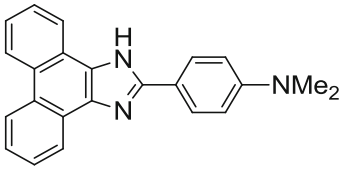
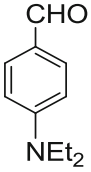
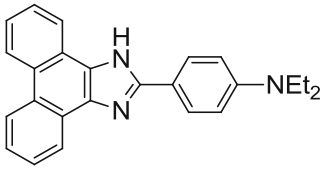
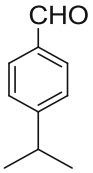
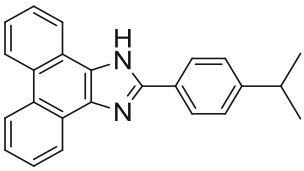
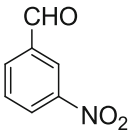
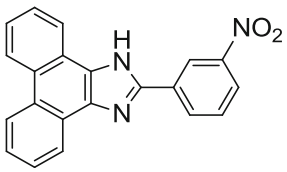
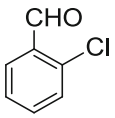
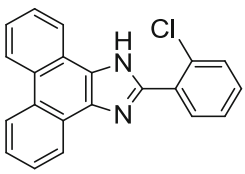
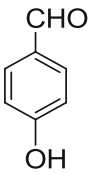
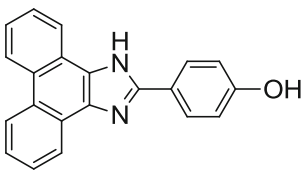
Entry	Aldehyde	Product	Time (min)	Yield (%)	Mp (°C)	Reported	Ref.
1			5	93	312	314	[27]
2			7	88	339	341	[27]
3			5	91	254	254–255	[27]
4			4	87	276	275–276	[28]
5			5	94	290	290–292	[12]
6			7	95	257	259–260	[27]
7			7	95	320	319–320	[13]

Table 1 continued

Entry	Aldehyde	Product	Time (min)	Yield (%)	Mp (°C)	Reported	Ref.
8			5	92	178	–	–
9			5	92	272	271–272	[27]
10			7	85	233	235	[27]
11			5	82	350	>360	[27]

[a] All compounds were characterized on the basis of ^{1}H NMR and IR spectral data, which were consistent with those reported in the literature

Because of high catalyst activity, the initial step, the formation of imine with aromatic aldehyde, is very farapidst and aromatic aldehydes with electron-donating and electron-withdrawing groups both gave the desired products in excellent yields within very short times, and the electronic effect and the steric effects of substitutes did not show significant effects on the yields and reaction times. For example, ortho-substituted aromatic aldehydes such as 2-chlorobenzaldehyde (Table 1, entry 10) gave 85 % yields in comparison to 87 % yields of para-substituted aromatic aldehydes (Table 1, entry 4).

Many of the reported synthetic methods for the synthesis of trisubstituted imidazoles suffer from one or more disadvantages such as harsh reaction conditions, poor yields, prolonged time periods, and use of hazardous organic solvents in the reaction (Table 3).

The synthesis of these heterocycles has usually been carried out in polar solvents such as ethanol, methanol, acetic acid, and dimethylsulfoxide (DMSO), which give complex isolations [16]. Therefore, the discovery of clean procedures and the use of a green and eco-friendly catalyst with high catalytic activity and short reaction time for the preparation of 2,4,5-triarylimidazoles has gained considerable attention.

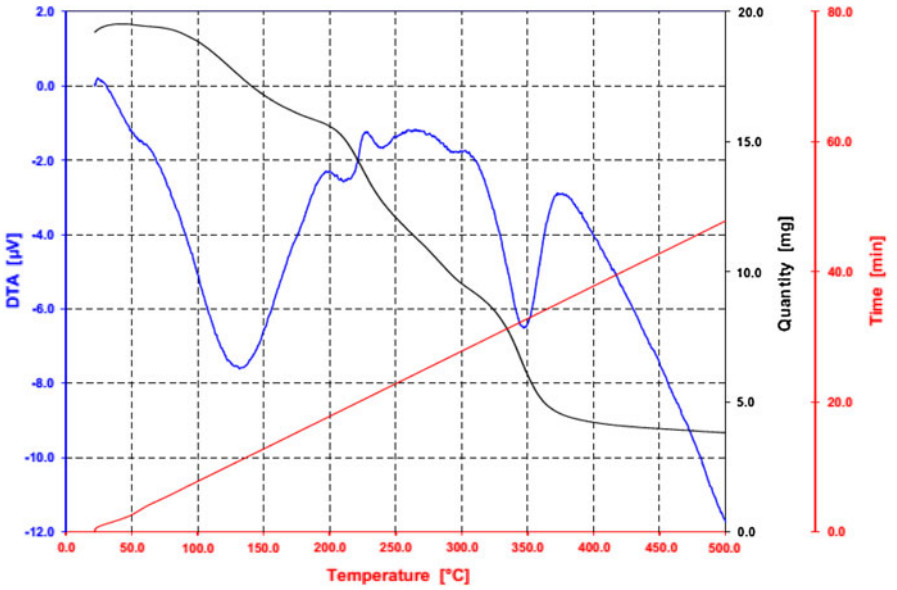


Fig. 2 Thermal analysis (TG–DTA) of synthesized dicationic RTILs

Table 2 The geometry parameters of AIL calculated at the RHF/6-31G basis set

Parameter		
Bond distance (Å)		
$(N)_2C$–H	C_3–H_7 = 1.06790	C_{30}–H_{33} = 1.06900
$(N)_2C$–H–Cl	H_7–Cl_{48} = 3.07264	H_{33}–Cl_{47} = 3.89662
SO_3–H	O_{43}–H_{44} = 1.02740	O_{45}–H_{46} = 1.93601
H–Cl	H_{44}–Cl_{48} = 1.84593	H_{46}–Cl_{47} = 1.28266
H–N	H_{44}–N_{35} = 2.57712	H_{46}–N_{36} = 3.17257
E (a.u.)	−2996.59632770	
Dipole moment (D)	12.5536	

Recycling of dicationic AIL

One of the advantages of this AIL is its ability to be recycled from the reaction medium. We were able to readily separate the catalyst from the residue by washing with ethyl acetate. The catalyst was recovered by evaporation of the water of the aqueous layer at 50–60 °C in a vacuum oven which gave 95 % yields and could be reused for the same experiment for four more times (Table 4). The ^{1}H NMR spectrum of the recycled AIL catalyst compared with the freshly prepared catalyst and at significant purity was observed.

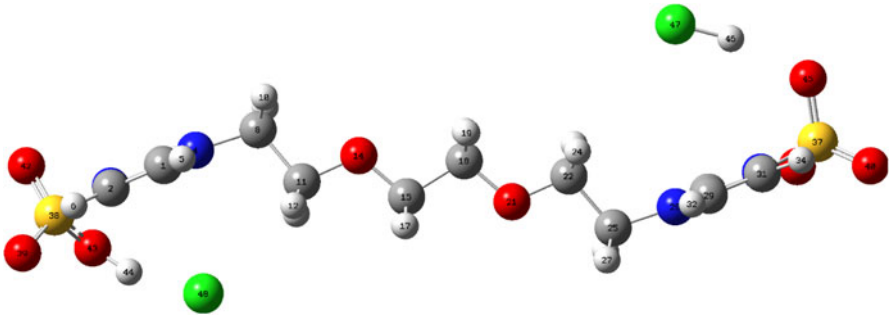
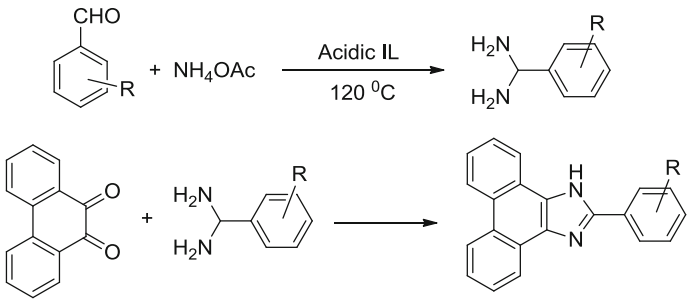


Fig. 3 Optimized molecular structure of **2** AIL using RHF/6-31G



Scheme 3 The suggested mechanism of synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives

Table 3 Reported synthetic methods for the synthesis of trisubstituted imidazole (**1**)

Entry	Catalyst	Solvent	Temperature(°C)	Time	Yield (%)	Ref.
1	NH_3SO_3	Ethanol	Reflux	5 h	93	[1]
2	CH_3COOH	Acetic acid	Reflux	1 h	41	[2]
3	$Cu(OAc)_2$/NaOH	Alcohol/NH_3(aq)	Heating	2 h	70	[27]
4	–	DMSO	95	0.5 h	85	[3]
5	AIL	–	120	5 min	93	This work

Table 4 Recycling of AIL in the reaction of phenanthraquinone with *N*,*N*-Diethylbenzaldehyde and ammonium acetate

Reusability[a]	AIL	1[b]	2[c]	3[d]	4[e]
Yield (%)[f]	95	92	90	91	89

[a] Reusability of the recovered catalyst.

[b–e] Reusability of the recovered catalyst in new runs from run 2(b) to run 5(e)

[f] Isolated yields

Conclusion

In conclusion, we have designed and synthesized a novel AIL based on imidazolium cations and have successfully used it as a catalyst for the one-pot synthesis of 2-aryl-1*H*-phenanthro[9,10-*d*]imidazole derivatives. The remarkable feature of this new catalyst is its ethyleneoxy bridge which participates in dissolving organic compounds. The application of this AIL is studied in a new one-pot method for the synthesis of imidazole derivatives under solvent-free conditions. The advantages offered by this protocol include reusability of the catalyst, high conversion, short reaction time, and simple experimental procedure.

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