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Synthesis of a novel poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid and its application in one-pot synthesis of 2-amino-2-chromene derivatives in water

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ABSTRACT

A novel poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid has been prepared and characterized. It was successfully used as an efficient catalyst for the three-component synthesis of 2-amino-2-chromene derivatives in water. The simple workup, mild reaction conditions, wide substrate flexibility, excellent yields, and environmental friendliness are notable advantages of this protocol. In addition, the catalyst could be reused several times without appreciable reduction of catalytic activity.

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1. Introduction

2-Amino-2-chromene derivatives represent an important class of chemicals being the main components of many natural products, and are widely employed as pigments, cosmetics, photoactive materials, and potential biodegradable agrochemicals [1–3]. These compounds are generally prepared through the multicomponent reactions of aromatic aldehyde, malononitrile and activated phenol in the presence of a basic catalyst in organic solvent. Recently, various modified procedures have been reported to construct this heterocyclic system, such as tetrabutylammonium bromide (TBAB) [4], γ -alumina [5], hexadecyltrimethylammonium bromide (HTMAB) [6], nanosized magnesium oxide [7], diazabicyclo[2.2.2]octane (DABCO) [8], 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) [9], nanostructured diphosphate $\text{Na}_2\text{CaP}_2\text{O}_7$ [10], disodium hydrogen

phosphate (Na_2HPO_4) [11], benzyltrimethylammonium hydroxide (triton B) [12], thiourea dioxide [13], nanozeolite clinoptilolite [14], hydroxyapatite and sodium-modified hydroxyapatite [15], amberlyst A21 [16], $[\text{Cu}(2,2'\text{-bipyridine } 1,1'\text{-dioxide})_2 \cdot 2\text{H}_2\text{O}]^{2+}$ -supported SBA-15 [17], guanidine supported on magnetic nanoparticle Fe_3O_4 [18], amino-functionalized MCM-41 [19], potassium phthalimide [20], 4-dimethylaminopyridine functionalized polyacrylonitrile fiber ($\text{PAN}_{\text{DMAPF}}$) [21], potassium sodium tartrate [22], and silica-supported piperazine [23]. At the same time, some methods including the use of microwave irradiation [24–25], ultrasonic irradiation [26] and grinding [27] have also been developed to promote this reaction. However, some of the reported methods require prolonged reaction time, volatile organic solvents, special apparatus and laborious workup procedure.

Poly(ethylene glycols) (PEGs) [28–30] and ionic liquids (ILs) [31–33] have attracted considerable attention in the last decade, due to their intrinsic chemical and physical properties, such as good thermal and chemical stability, low or null vapor

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pressure, the possibility of recycling, and the ability to act as efficient solvents or supports for reagent and catalyst immobilization. The functionalized ionic liquids which incorporated poly(ethylene glycol) moieties into cationic units make the link between the two distinct but very similar fluids, generating an attractive group of compounds that find applications across a range of disciplines, including extractions, biphasic systems, gas separations, carbon capture, carbohydrate dissolution, polymer chemistry, organic synthesis and catalysis [34–39]. Recent studies have demonstrated that ether-functionalized ionic liquids tend to reduce toxicity than their aliphatic-substituted counterparts [40]. Some functionalized ionic liquids, such as 1-butyl-3-methyl imidazolium hydroxide ([Bmim][OH]) [41], *N,N*-dimethylaminoethylbenzyl dimethyl-ammonium chloride ([PhCH₂Me₂NCH₂CH₂NMe₂][Cl]) [42], 1-(2-aminoethyl)-3-methylimidazolium imidazolidate ([2-aemim][im]) [43], 2-hydroxyethanaminium formate, 3-hydroxypropanaminium formate, 2-hydroxyethanaminium acetate and 3-hydroxypropanaminium acetate [44], have been successfully used as catalysts for the synthesis of 2-amino-2-chromenes.

Due to the wide-ranging applications of these heterocyclic compounds and the advantages of poly(ethylene glycol)-based ionic liquids, the further development of catalysts that are recyclable easily and efficiently is a desirable goal. In continuation of our work about green synthesis in the presence of the poly(ethylene glycol)-grafted functionalized dicationic ionic liquid [45–53], herein we designed and prepared a novel poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid ([TEA-PEG₈₀₀-DIL][Cl]) and applied it as an efficient and recyclable catalyst for the synthesis of 2-amino-2-chromenes in aqueous medium.

2. Results and discussion

The synthetic route of the poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid is illustrated in Scheme 1. Two methods for preparing [TEA-PEG₈₀₀-DIL][Cl] were investigated. The first method was the classical approach with the quaternization of PEG-800 bridged di-

imidazolium compound (**2**) and 2-chloro-*N,N*-diethylethan-1-amine hydrochloride. However, this method suffered from tedious workup and it was impossible to purify the ionic liquid. Alternatively, the other method was found to be much better. 2-Chloro-*N,N*-diethylethan-1-amine hydrochloride reacted with imidazole and NaOH to afford *N,N*-diethyl-2-(1*H*-imidazol-1-yl)ethanamine (**3**) firstly, and then quaternized with PEG-800 dichloride (**1**); the target ionic liquid (**4**) could be obtained in high purity. According to the thermal gravimetric analysis (TGA), [TEA-PEG₈₀₀-DIL][Cl] is quite stable since it does not decompose below 250 °C (Fig. 1). In addition, the solubility of [TEA-PEG₈₀₀-DIL][Cl] was determined at room temperature. In general, it is soluble in dichloromethane, ethyl acetate, acetonitrile, ethanol, *N,N*-dimethylformamide (DMF) and water, while insoluble in hexane, cyclohexane, petroleum ether and diethyl ether.

In order to evaluate the catalytic activity of [TEA-PEG₈₀₀-DIL][Cl], the reaction of 4-methylbenzaldehyde, malononitrile and 1-naphthol was selected as a model reaction, and selected results from our screening experiments are listed in Table 1. When [TEA-PEG₈₀₀-DIL][Cl] was used as both catalyst and solvent, only 64% yield was obtained, which might result from the inefficient mass

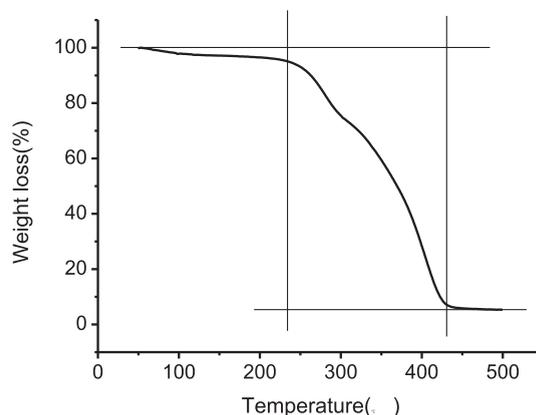
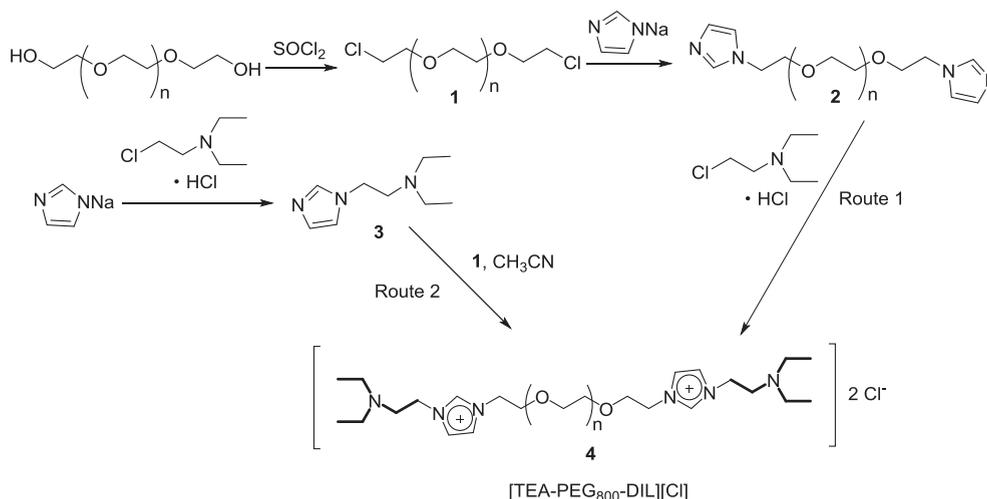


Fig. 1. The TGA spectrum of [TEA-PEG₈₀₀-DIL][Cl].

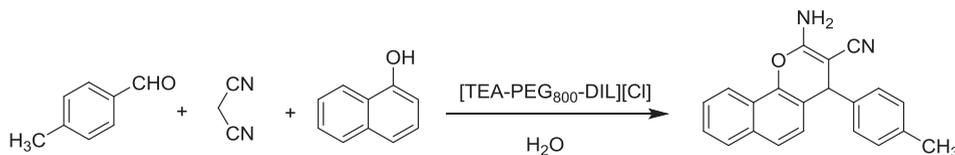


Scheme 1. The synthesis of [TEA-PEG₈₀₀-DIL][Cl].

transfer due to the high viscosity of the ionic liquid (Table 1, entry 1). However, none of the desired product was detected when the reaction was carried out in water under reflux for 25 min without any catalyst (Table 1, entry 2). Further optimization suggested that the amount of catalyst

had a strong effect on this process. The yield was increased with the addition of [TEA-PEG₈₀₀-DIL][Cl] and the optimal amount of catalyst was 5 mol% (Table 1, entries 3–7). Furthermore, low temperatures decelerated the reaction and led to lower yields (Table 1, entries 8–9).

Table 1
Optimization of the reaction conditions.^a

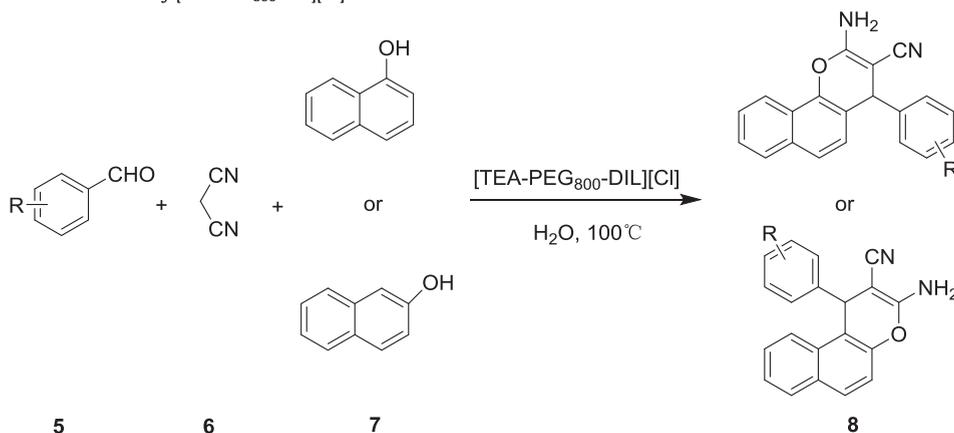


Entry	Amount of catalyst	Temperature/°C	Solvent	Time/min	Yield/% ^b
1	10%	100	—	25	70
2	—	100	H ₂ O	25	—
3	1%	100	H ₂ O	25	38
4	3%	100	H ₂ O	25	74
5	5%	100	H ₂ O	25	92
6	7%	100	H ₂ O	25	92
7	10%	100	H ₂ O	25	93
8	5%	80	H ₂ O	35	78
9	5%	90	H ₂ O	25	86

^a Reaction conditions: 4-methylbenzaldehyde (2 mmol), malononitrile (2 mmol), 1-naphthol (2 mmol), water (2 mL).

^b Isolated yield.

Table 2
Synthesis of 2-amino-2-chromenes by [TEA-PEG₈₀₀-DIL][Cl] in water.^a



Entry	R	Naphthol	Time/min	Product	Yield/% ^b	Mp/°C	Lit Mp/°C
1	4-CH ₃	1-Naphthol	25	8a	92	205–206	206–207 [41]
2	4-OH	1-Naphthol	25	8b	91	250–251	249–251 [4]
3	4-CH ₃ O	1-Naphthol	30	8c	91	191–192	190–192 [42]
4	3-CH ₃ O-4-OH	1-Naphthol	35	8d	89	136–137	136–138 [42]
5	H	1-Naphthol	20	8e	93	216–217	215–217 [8]
6	4-Cl	1-Naphthol	15	8f	95	246–247	245–248 [8]
7	4-Br	1-Naphthol	15	8g	94	240–242	241–243 [8]
8	4-F	1-Naphthol	12	8h	96	232–234	232–233 [41]
9	2-NO ₂	1-Naphthol	15	8i	95	241–242	241–242 [8]
10	3-NO ₂	1-Naphthol	10	8j	95	218–220	217–219 [8]
11	4-NO ₂	1-Naphthol	10	8k	96	233–235	233–235 [14]
12	2-Furyl	1-Naphthol	25	8l	92	168–170	169–171 [42]
13	H	2-Naphthol	25	8m	90	286–288	287–288 [41]
14	4-CH ₃	2-Naphthol	30	8n	87	271–273	270–272 [27]
15	4-Cl	2-Naphthol	15	8o	92	223–225	222–224 [27]

^a Reaction conditions: aromatic aldehyde (2 mmol), malononitrile (2 mmol), naphthol (2 mmol), [TEA-PEG₈₀₀-DIL][Cl] (0.1 mmol), water (2 mL), 100 °C.

^b Isolated yield.

To explore the application of this method, the scope of the substrates was evaluated with a variety of aromatic aldehydes under the optimal condition. The results are summarized in Table 2. It is found that the substituents of the aromatic aldehyde and the position of the hydroxyl on the naphthol dramatically influence the reaction. Aromatic aldehydes bearing electron-donating groups (such as methyl, hydroxy, methoxy) (Table 2, entries 1–4) required longer reaction times and provided lower yields than those bearing electron-withdrawing groups (such as halide and nitro) (Table 2, entries 6–11). It is worth noting that *ortho* and *meta* substituents did not significantly hamper the reaction (Table 2, entries 9–11). The heterocyclic aldehyde (such as furyl aldehyde) was also demonstrated to be an efficient reagent for this reaction (Table 2, entry 12). Furthermore, the activity of 2-naphthol was lower than that of 1-naphthol because of the electronic effect (Table 2, entries 13–15).

In view of green chemistry, the catalyst was further explored for the reusability by the model reaction of 4-methylbenzaldehyde, malononitrile and 1-naphthol. Upon the completion of the reaction, the product was isolated by filtration while the filtrate containing the catalyst was reused in subsequent reactions without further treatment. As shown in Fig. 2, the reaction medium could be recycled at least five times without considerable decrease of activity.

The efficiency of our newly synthesized catalyst for the synthesis of 2-amino-2-chromenes was further evaluated by comparing its performance with previously reported methods in the literatures. These comparative experiments were based on the synthesis of compounds **8a** from 4-methylbenzaldehyde, malononitrile and 1-naphthol, and the results are listed in Table 3. The results show that this method is superior to some of the earlier methods in terms of yield and reaction time.

A probable mechanism for the synthesis of 2-amino-2-chromene derivatives is also outlined in Scheme 2. Only one side is displayed because the [TEA-PEG₈₀₀-DIL][Cl] is symmetrical. We assume that [TEA-PEG₈₀₀-DIL][Cl] acts as the Brønsted proton scavenger in the reaction. The first step is the formation of the olefin catalyzed by [TEA-PEG₈₀₀-DIL][Cl], which is formed in situ by Knoevenagel condensation of aromatic aldehyde and malononitrile. [TEA-PEG₈₀₀-DIL][Cl] also catalyzes the generation of the naphtholate anion, which reacts with the dicyanoolefin, followed by

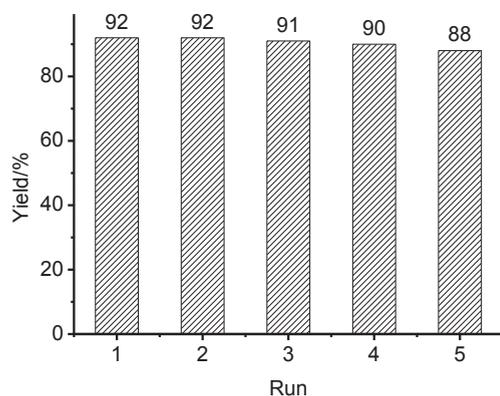


Fig. 2. The recycling of the [TEA-PEG₈₀₀-DIL][Cl]/H₂O system.

Table 3

Comparison of different catalysts for the synthesis of **8a**.

Entry	Catalyst	Conditions	Time (min)	Yield (%)
1	[TEA-PEG ₈₀₀ -DIL][Cl]	100 °C, H ₂ O	25	92 (present work)
2	[Bmim][OH]	100 °C, H ₂ O	30	91 [41]
3	Silica-supported piperazine	Reflux, CHCl ₃	1860	63 [23]
4	Nanozeolite clinoptilolite	Reflux, H ₂ O	30	88 [14]
5	Sodium-modified hydroxyapatite	Reflux, H ₂ O	180	79 [15]
6	Nanostructured diphosphate Na ₂ CaP ₂ O ₇	Reflux, H ₂ O	300	72 [10]

cyclization to give the iminium ion. After hydrolysis, the final product could be obtained. Thus, the existence of [TEA-PEG₈₀₀-DIL][Cl] is essential for both steps.

3. Conclusions

In conclusion, we have synthesized a novel poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid from the commercially available starting materials, and successfully used as a catalyst for the synthesis of substituted 2-amino-2-chromenes through one-pot three-component condensations of aromatic aldehydes, malononitrile, and naphthol in aqueous media. The attractive features of this method are environmentally friendly, mild reaction conditions, compatible with various functional groups, easy workup and recyclability of reaction medium.

4. Experimental

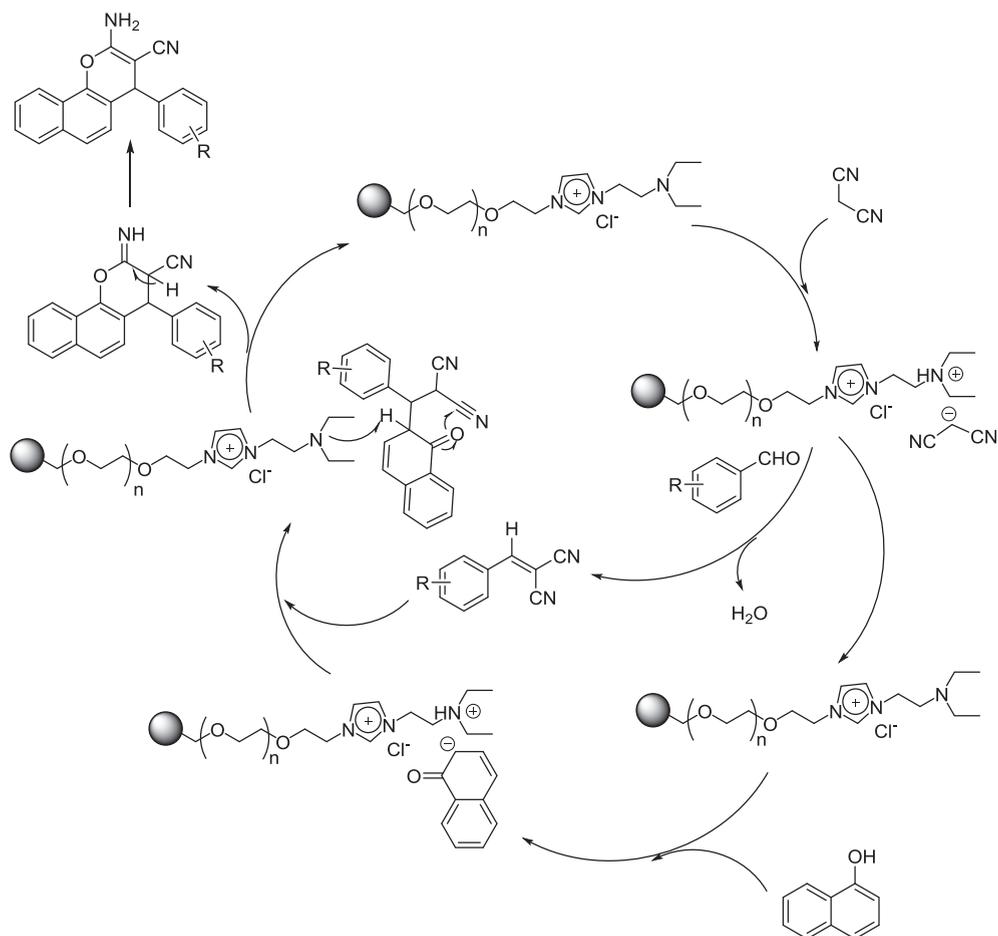
4.1. General remarks

All of the reagents and solvents were purchased from commercial suppliers and used without further purification. ¹H NMR and ¹³C NMR were recorded on a Bruker (Rheinstetten, Germany) Avance III 500, and tetramethylsilane (TMS) was used as the internal standard. Mass spectra were taken on an Agilent (Santa Clara, CA, USA) liquid chromatography–mass spectrometry (LC–MS) 1100 series instrument in the electrospray ionization (positive electrospray ionization (ESI)) mode. IR spectra were recorded from KBr disks with a Shimadzu (Kyoto, Japan) IRPrestige-21 FT-IR spectrometer. The thermo gravimetric analysis (TGA) was performed on a TGA/SDTA851e thermal analyzer (Switzerland-Mettler Toledo). Samples were loaded into an aluminum oxide crucible and heated at a rate of 20 °C · min⁻¹ from 50 °C to 600 °C under N₂. All melting points were determined on a digital melting-point apparatus.

4.2. Synthesis of the poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid

4.2.1. Synthesis of PEG-800 dichloride (**1**)

A stirred solution of PEG-800 (20.0 g, 25 mmol) and pyridine (4.0 mL, 50 mmol) in CH₂Cl₂ (120 mL) was cooled in an ice bath to 0 °C, and SOCl₂ (4.0 mL, 55 mmol) was



Scheme 2. The proposed mechanism for the synthesis of 2-amino-2-chromene derivatives by [TEA-PEG₈₀₀-DIL][Cl].

added dropwise within 30 min, and then the ice bath was removed and stirring was continued at room temperature for 12 h. After completion, the solvent was removed under vacuum, the residue was then dissolved in ethyl acetate to allow the pyridine hydrochloride to be precipitated and filtered off. Evaporation of the solvent under reduced pressure yielded PEG-800 dichloride (18.6 g, 89% yield). ¹H NMR (500 MHz, CDCl₃) δ: 3.75–3.73 (t, *J* = 5.0 Hz, 4H), 3.67–3.60 (m, 65H)

4.2.2. Synthesis of the PEG-800 bridged di-imidazolium compound (2)

Imidazole (3.7 g, 2.4 equiv) was melted at around 100 °C and NaOH (1.8 g, 2.0 equiv) was added while stirring. After the solid NaOH disappeared, toluene (50 mL) was added for azeotropic removal of water for 3 h. Then, the toluene was evaporated under reduced pressure. Subsequently, PEG-800 dichloride (18.6 g) and CH₃CN (80 mL) were added. The mixture was stirred under reflux for another 12 h. After completion, the solvent was removed by distillation. The residue was dissolved in CH₂Cl₂ and washed with water several times to remove unreacted materials. After removal of solvent, the intermediate PEG-800 bridged di-imidazolium compound was obtained. (17.2 g, 86% yield).

¹H NMR (500 MHz, CDCl₃, ppm) δ: 7.49 (s, 2H), 6.97 (d, *J* = 13.4 Hz, 4H), 4.06 (t, *J* = 5.2 Hz, 4H), 3.70 (t, *J* = 5.2 Hz, 4H), 3.66–3.48 (m, 71H).

4.2.3. Synthesis of *N,N*-diethyl-2-(1*H*-imidazol-1-yl)ethanamine (3)

Imidazole (8.9 g, 130 mmol) was melted at around 100 °C and NaOH (4.8 g, 120 mmol) was added while stirring. After the solid NaOH disappeared, toluene (50 mL) was added for azeotropic removal of water for 3 h. Then, the toluene was evaporated under reduced pressure. Subsequently, 2-chloro-*N,N*-diethylethan-1-amine hydrochloride (11.1 g, 60 mmol) and CH₃CN (80 mL) were added. The mixture was stirred under reflux for another 12 h. After completion, the solvent was removed by distillation. The residue was dissolved in CH₂Cl₂ and washed with water several times to remove inorganic salts and excess imidazole. After removal of solvent, the desired product *N,N*-diethyl-2-(1*H*-imidazol-1-yl)ethanamine was obtained (8.2 g, 81% yield). ¹H NMR (500 MHz, CDCl₃, ppm) δ: 7.49 (s, 1H), 7.01 (s, 1H), 6.94 (d, *J* = 1.1 Hz, 1H), 3.95 (t, *J* = 6.6 Hz, 2H), 2.71 (t, *J* = 6.6 Hz, 2H), 2.51 (q, *J* = 7.1 Hz, 4H), 0.96 (t, *J* = 7.1 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃, ppm) δ: 137.40, 129.07, 119.19, 54.05, 47.40, 46.00, 11.95.

4.2.4. Synthesis of the poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid

A mixture of PEG-800 dichloride (**1**) (18.6 g) and *N,N*-diethyl-2-(1*H*-imidazol-1-yl)ethanamine (**3**) (8.2 g) in CH₃CN (80 mL) was stirred under reflux for 7 days. After completion, the solvent was evaporated in a vacuum. The residue was dissolved in water and washed with CH₂Cl₂ several times to remove excess unreacted materials. After removal of water, the poly(ethylene glycol) grafted triethylamine functionalized dicationic ionic liquid [TEA-PEG₈₀₀-DIL][Cl] was finally obtained. (21.9 g, 84% yield). ¹H NMR (500 MHz, CDCl₃, ppm) δ: 9.94 (s, 2H), 7.60 (s, 2H), 7.52 (s, 2H), 4.53–4.43 (m, 4H), 4.27 (s, 4H), 3.80–3.72 (m, 4H), 3.51 (s, 69H), 2.74 (t, *J* = 5.2 Hz, 4H), 2.44 (q, *J* = 7.0 Hz, 8H), 0.82 (t, *J* = 7.1 Hz, 12H); ¹³C NMR (126 MHz, CDCl₃, ppm) δ: 137.28, 122.74, 122.34, 70.38, 70.28, 70.20, 70.15, 69.12, 52.62, 49.51, 48.03, 46.79, 11.56; IR (cm⁻¹): 3382.00, 2866.97, 1664.08, 1563.61, 1453.93, 1348.39, 1294.61, 1249.31, 1092.35, 945.85, 844.01, 730.66; ESI-MS: 489.36 (M²⁺/2, *n* = 16), 511.28 (M²⁺/2, *n* = 17), 533.40 (M²⁺/2, *n* = 18), 555.36 (M²⁺/2, *n* = 19).

4.3. General procedure for the synthesis of 2-amino-2-chromene derivatives and recycling of catalyst

A mixture of aromatic aldehyde (**5**) (2 mmol), malononitrile (**6**) (2 mmol), naphthol (**7**) (2 mmol), H₂O (2 mL), and [TEA-PEG₈₀₀-DIL][Cl] (0.1 mmol) was taken in a round bottom flask. This reaction mixture was stirred at 100 °C for a certain time. The progress and completion of reaction were monitored by TLC. After reaction, the reaction mixture was cooled to room temperature, and the precipitated solid was broken up, filtered off and washed with water. The crude product was purified by recrystallization from methanol to give the pure product (**8**). All of the products are known and the data are found to be identical to those reported in the literature.

The catalyst that remained in water was reused for the next catalytic cycle without any treatment.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.crci.2016.03.002>.

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