DE GRUYTER Green Process Synth 2018; 7: 191–197

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Choline taurinate: a new biocompatible amino-functionalized ionic liquid as basic catalyst and extraction solvent

DOI 10.1515/gps-2017-0009
Received January 30, 2017; accepted April 25, 2017; previously published online June 29, 2017

Abstract: A new biocompatible amino-functionalized ionic liquid composed solely of biomaterials was synthesized from taurine and choline hydroxide, and its physical properties (density, viscosity, miscibility and thermal stability) were also determined. The classic base-promoted three-component cyclic condensation of aromatic aldehydes, malononitrile and dimedone was chosen as the test reaction to demonstrate the catalytic activity of choline taurinate ([Ch][Tau]). The catalyst could be recovered and reused several times with no significant loss of activity. As a basic ionic liquid, [Ch][Tau] can extract acidic compounds from immiscible solvents through acid-base interaction. The extraction process of fluorescent dye Rhodamine B was monitored using ultraviolet-visible (UV-Vis) spectroscopy, by which the percentage extraction rate was determined as 95%.

Keywords: catalysis; choline taurine; extraction; ionic liquids; recyclability.

1 Introduction

Room temperature ionic liquids (ILs) exhibit many unique and astonishing properties such as negligible vapor pressure, excellent thermal stability, low flammability, wide liquidus temperature range as well as tunable solvation properties. As a result, their application as potential alternative solvents has been intensively investigated over a long period [1, 2]. Because their physical and chemical properties can be finely tuned through slight alterations to the cations and/or anions, they can address very specific requirements as “designer solvents”. More recently, research attention has focused on the preparation and application of functionalized ILs with special tasks (“task-specific” ILs) as catalysts, soluble supports, adsorbents and so on [3–5]. Most studies on ILs are about 1,3-dialkylimidazolium and pyridinium salts. However, the scope of possible applications of traditional imidazolium and pyridinium ILs is restricted due to their high toxicity to certain organisms and their environmental persistence due to poor biodegradability [6–8].

An alternative approach to overcoming these drawbacks is the development of ILs from non-toxic and biorenewable components (so-called “drinkable” solvents). Choline is a natural component of plants and meat [9]. Choline chloride (also known as vitamin B_4) is a non-toxic and cheap organic salt that is a globally accepted additive to chicken feed. Greener amino-functional ILs could be obtained by the combination of the cholinium cation, $[Me_3N(CH_2CH_2OH)]^+$, with amino acid-based anions. Hu et al. reported their pioneer work in this area [10]. L-proline based cholinium IL ([Ch][Pro]) was synthesized by ion exchange and neutralization (Scheme 1, **1**), and then applied as a catalyst in direct aldol reactions. In another example, amino ILs were prepared starting from glycine, alanine, L-phenylalanine, threonine, and histidine (Scheme 1, **2–6**) [11]. These ILs were successfully used to catalyze a Knoevenagel reaction between benzaldehyde and different active methylene compounds. In addition, an amino acid-based cholinium IL could also have been applied for the pre-treatment of biomass [12].

One of the important features of ILs is their chemical stability. Generally speaking, the stability of a sulfonate is superior to the corresponding carboxylate (with the same counterion). In other words, a sulfonate IL is theoretically more stable than a carboxylate one. Taurine (ethylamine sulfonic acid) is a non-toxic compound widely distributed in animal tissue. It is essential for cardiovascular function and the development and function of skeletal muscle, the retina and the central nervous system [13]. Therefore, taurine is also a possible candidate as the anion component in an eco-friendly amino-functionalized IL. Mu and coworkers [14] reported their results about the synthesis of an amino-functionalized IL with imidazolium cation and taurinate anion, and its application in the absorption of

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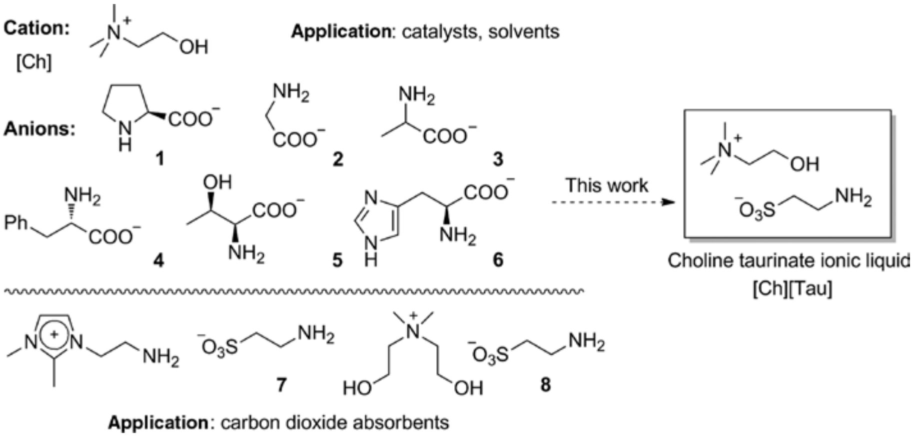


Scheme 1: Previously reported amino acid cholinium ionic liquids and taurinate–based ionic liquids.

carbon dioxide (Scheme 1, **7**). However, the imidazolium cation made this kind of IL undesirable from the viewpoint of biocompatibility and biodegradability. In 2013, the Maier group reported another taurinate IL for carbon dioxide capture, in which the imidazolium unit was replaced by dihydroxyethyldimethylammonium [15]. This cation had an improved biodegradability but was still not a non-toxic component (Scheme 1, **8**).

We have previously reported the use of an imidazolium-type amino-functional IL as a heterogeneous catalyst and scavenger reagent for combinatorial synthesis [16–18]. In continuation of this work, we report herein the first preparation and characterization of choline taurinate [Ch][Tau] as a biocompatible and biodegradable IL. [Ch][Tau] can be treated as a task-specific IL with low toxicity, as this IL is composed of animal feed additives and of additives for human nutrition; although the environmental ecotoxicity of [Ch][Tau] has not been evaluated by biological assay in the present study. Furthermore, the application of this amino-functionalized IL as an eco-friendly basic catalyst and extraction solvent was demonstrated.

2 Materials and methods

2.1 General

Choline base (choline hydroxide) was obtained from Taminco Co. Ltd. (Shanghai, China) as a 45% w/w aqueous solution. Other chemicals were purchased from commercial suppliers (Aldrich or Shanghai Aladdin Reagent Co. Ltd., Shanghai, China) and used without further purification. Fourier transform infrared (FTIR) spectra were recorded with a Thermo Nicolet (USA) 6700 IR spectrophotometer in KBr pellets. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker (Switzerland) AC 400 instrument in DMSO-d_6 [tetremethylsilane (TMS)]. The viscosity of [Ch][Tau] was measured with a Thermo HAAKE (Germany) RheoStress 600 viscometer at 25°C. Thermogravimetric analysis (TGA) was performed under nitrogen atmosphere using a Netzsch (Germany) STA 409 PC thermogravimetric analyzer.

2.2 Preparation of [Ch][Tau] IL

Taurine (6.25 g, 50 mmol) was introduced into a 100 ml flask containing 20 ml of water. To the stirred mixture was slowly added a 45% aqueous solution of choline hydroxide (13.44 g, 50 mmol) under cooling (iced water). After 3 h of stirring, the water in the solution was removed by evaporation under reduced pressure for 2 h at 50°C. Absolute ethanol (20 ml) was then employed to remove the residual water by azeotropic distillation. Afterwards, the residue was further dried *in vacuo* for 24 h at 50°C. The IL was obtained in 93% yield (10.61 g) as a viscous and hydrophilic liquid which was identified by ^{1}H NMR, ^{13}C NMR, IR and HRMS (ESI).

2.3 Typical procedure for the synthesis of 4*H*-chromene-3-carbonitrile derivatives

A solution of aromatic aldehyde (2 mmol), malononitrile (0.13 g, 2 mmol), dimedone (0.28 g, 2 mmol) and [Ch][Tau] (0.02 g, 0.1 mmol, 5 mol%) in 4 cm^3 of 50% aqueous ethanol was stirred under reflux. On completion, as monitored by thin layer chromatography (TLC), the ethanol in the mixed solvent was evaporated using a rotary evaporator to ensure the full precipitation of the product. The crude product was collected by filtration. Finally, the crude product was recrystallized from ethanol to afford pure 4*H*-chromene-3-carbonitrile derivatives. All products were known and characterized by comparison of their physical and spectra data with those already reported (see online supplemental material). IL [Ch][Tau] could be recovered by removal of water from the filtrate.

2.4 Extraction of fluorescent dye rhodamine B from acetone by [Ch][Tau]

The extraction of rhodamine B was conducted in a batch system. Typically, 4 ml of rhodamine solution (5 mM in acetone) was added into a glass vial charged with 4 ml of IL [Ch][Tau]. The mixture was agitated at room temperature with a magnetic stirring bar. The samples were drawn from the vial at predetermined times until adsorption equilibrium was achieved. The concentrations of rhodamine B in the organic phase were determined by measuring the solution's UV absorbance at 542 nm.

3 Results and discussion

3.1 Synthesis and characterization of [Ch][Tau]

Amino-functionalized IL [Ch][Tau] was synthesized by neutralization of choline hydroxide with taurine as illustrated in Scheme 1. Starting with the commercially available choline hydroxide (45 wt% aqueous solution, checked *via* titration before use), neutralization with equimolar of crystalline taurine at room temperature afforded the desired task-specific IL (97% yield) as a colorless liquid which was characterized by NMR, FTIR and high-resolution mass spectroscopy (HRMS). The IL formed is viscous and hygroscopic but moisture-stable which allows easy use and storage without any precautions. In contrast to its salts, choline base is unstable, especially under elevated temperature. It should be mentioned that the aqueous solution of choline hydroxide turned brown during long-term storage. As a result, [Ch][Tau] was often obtained as a slightly brown liquid when using a precursor after prolonged storage. The color could not be diminished by solvent extraction. However, the existence of colored impurities has no influence on the application of the IL (Caution*:* the choline hydroxide solution must be stored in a refrigerator and used as soon as possible due to its unstable nature). The water content in the IL was determined as 3.6 wt% by Karl Fischer titration. The trace water is difficult to remove thoroughly due to the hygroscopic nature of this IL.

Some physical data for [Ch][Tau] is listed in Table 1. The density of [Ch][Tau] is determined as 1.19 g/cm^3 using a glass densitometer; similar to that of proline-based cholinium IL (1.13 g/cm^3) [10]. The viscosity of [Ch][Tau] was measured in duplicate with a Thermo HAAKE RS600 viscometer at 25°C (the temperature of the sample was maintained by external temperature controller). Its viscosity (210 cP) is somewhat higher than the viscosity of our previously reported imidazolium-type amino-functional IL [aemim][PF_6] (195 cP) [18]. This is no surprise as the terminal hydroxy functionality of the cholinium cation might give intermolecular hydrogen bonding. The miscibility of [Ch][Tau] with several organic solvents was also identified in this study. This IL is fully miscible with polar organic solvents such as water, methanol, ethanol, dimethylformamide and dimethyl sulfoxide, while it is partially miscible with acetonitrile and *t*-butyl alcohol. As expected, it is immiscible with less polar organic solvents such as tetrahydrofuran, diethyl ether, ethyl acetate, dichloromethane, acetone and toluene.

Table 1: Some physical data for [Ch][Tau] at 25°C.

Density (g/cm^3)	1.19
Viscosity (cP)	210
Miscibility	
H_2O	Miscible
MeOH	Miscible
EtOH	Miscible
t-BuOH	Partially miscible
DMF	Miscible
DMSO	Miscible
MeCN	Partially miscible
THF	Immiscible
Et_2O	Immiscible
EtOAc	Immiscible
CH_2Cl_2	Immiscible
Acetone	Immiscible
PhMe	Immiscible

The TGA curve showed the mass loss of organic materials as they decomposed upon heating. Furthermore, the thermal stability of [Ch][Tau] was characterized by TGA over the temperature range 100–500°C under nitrogen, heating at 10°C/min (Figure 1). It can be observed that [Ch][Tau] is stable to at least 192°C, which is lower than for choline chloride (T_{dec} = 300°C). There are two steps in the TGA curve. The weight loss in the range of 200–250°C might be contributed to the decomposition of

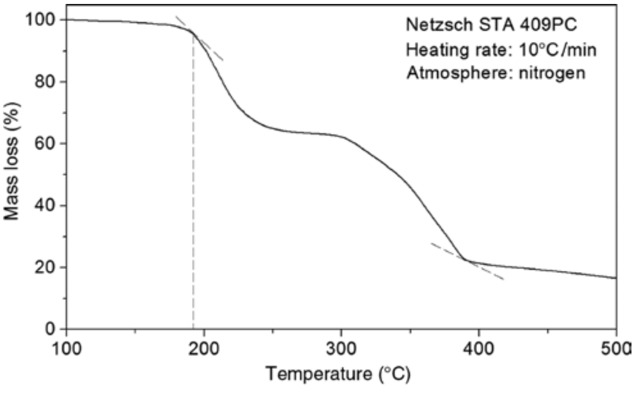


Figure 1: TGA of [Ch][Tau] IL. The TGA curve showed the mass loss of organic materials as they decomposed upon heating. The inflection points at 192°C and 390°C could be defined as the start and finish of rapid pyrogenic decomposition.

the cholinium cation, while the weight loss from 300 to 390°C might be due to the decomposition of the taurinate anion. According to these results, the thermal stability of [Ch][Tau] is higher than that of corresponding amino acid-based cholinium ILs [12].

3.2 Activity as basic catalyst

After the characterization, we turned our attention to demonstrating the practicability of this IL in catalysis and isolation processes. The three-component cyclic condensation of aromatic aldehyde, malononitrile and dimedone was firstly chosen as the test reaction. This three-component reaction is a classic base-promoted cyclic condensation which has previously been catalyzed by N-methylimidazole [19], amino-β-cyclodextrin [20], supported poly(4-vinylpyridine) [21], DABCO [22] and poly (ethylene glycol) bridged amine functionalized dicationic IL [23].

To develop the optimal conditions, benzaldehyde, malononitrile and dimedone were initially selected as the model reactants. The results in condition screening are summarized in Table 2. Ethanol was chosen as the solvent because it is considered to be an inexpensive, non-toxic and biorenewable conventional solvent with significant solvency to both catalyst and substrates. The control reaction run without catalyst in refluxing ethanol gave only a 26% yield of desired product after 2 h. In comparison, the addition of [Ch][Tau] (3 mol%) led to a much better result (82% yield, 2 h) under similar conditions; indicating the important role of [Ch][Tau] as a catalyst. The influence of catalyst loading on the reaction was subsequently examined. As can be seen in Table 2, the yield was promoted with increasing the catalyst loading from 3% to 5% and 10 mol%, while the resulting product was obtained with 82%, 91% and 94% yield after 2 h, 2 h and 1.5 h, respectively. In terms of yield and time, 5 mol% of catalyst was considered as the optimum amount. Interestingly, the mixed solvent EtOH:H_2O (1:1) also gave an excellent result. Our investigation proved pure water to be an inappropriate solvent in terms of the poor yield due to incomplete transformation (a large amount of benzylidenemalononitrile, the intermediate from the Knoevenagel reaction, remained unchanged). Furthermore, a less polar solvent such as acetonitrile gave a satisfactory yield of product after a relatively prolonged reaction time (88% yield, 3 h). This fact might be explained by a general knowledge about the solvent effects on the transition state; namely, the transition state is better solvated by polar solvents, so the reaction rate and yield were thus enhanced. It is worth noting that the exploration of this reaction in neat IL led to much better results. The target product could be obtained in high yield even at room temperature by using [Ch][Tau] as a catalytically active solvent rather than as a catalyst. As a comprehensive consideration from the aspects of catalyst loading and reaction efficiency, finally, the optimum reaction condition was chosen as follows: 5 mol% of catalyst in 50% aqueous EtOH solvent at refluxing temperature.

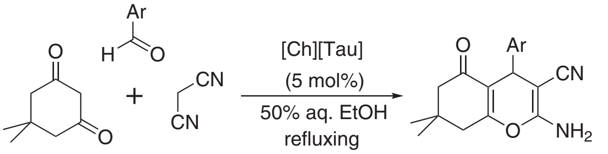
To establish the substrate generality of the present method, we conducted the reaction using various aromatic aldehydes bearing different substituent groups under the conditions optimized in model reactions. The results are summarized in Table 3. All aromatic aldehydes having

Table 2: Screening of reaction conditions in model reaction.

No.	Catalyst loading (mol%)	Solvent	Time (h)	Yield (%)[c]
1[a]	0	EtOH	2	26
2[a]	3	EtOH	2	82
3[a]	5	EtOH	2	91
4[a]	10	EtOH	1.5	94
5[a]	5	50% EtOH	2	92
6[a]	5	H_2O	3	33
7[a]	5	MeCN	3	88
8[b]	Neat [Ch][Tau] (2 ml)		2 (r.t.)	90

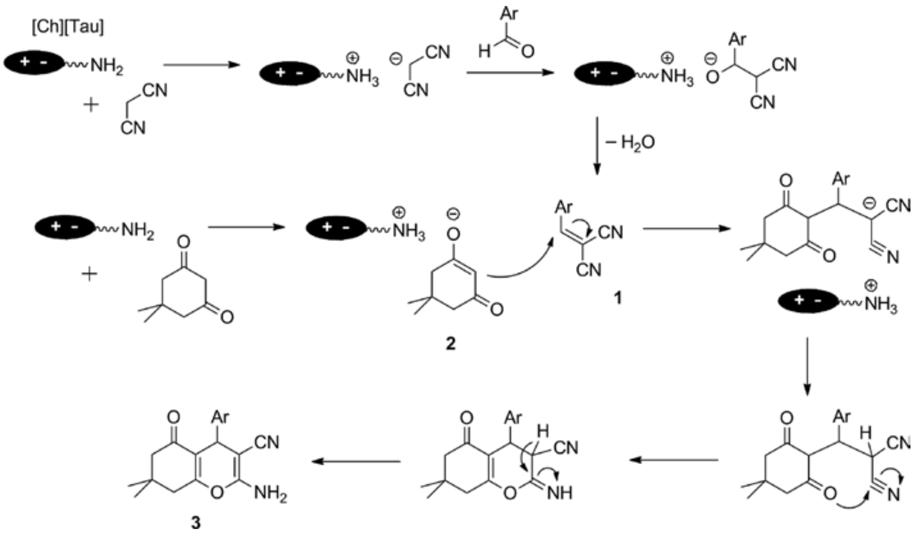
[a]Reaction conditions: benzaldehyde (2 mmol), malononitrile (2 mmol), dimedone (2 mmol), required amount of the catalysts, 4 cm^3 of solvent, refluxing. [b]Reaction conditions: benzaldehyde (2 mmol), malononitrile (2 mmol), dimedone (2 mmol), 2 cm^3 of [Ch][Tau], room temperature. [c]Isolated yield.

Table 3: Synthesis of 4*H*-benzo[b]pyrans catalyzed by [Ch][Tau].



Entry	Ar	Time (h)[a]	Yield (%)[b]
1	C_6H_5	2 (Run1)	91
		2 (Run2)	87
		2 (Run3)	90
		2 (Run4)	85
		2 (Run5)	89
2	$4\text{-CH}_3\text{O-C}_6\text{H}_4$	2.5	90
3	$4\text{-F-C}_6\text{H}_4$	2	85
4	$4\text{-Cl-C}_6\text{H}_4$	2	94
5	$4\text{-CH}_3\text{-C}_6\text{H}_4$	2.5	92
6	$3\text{-Br-C}_6\text{H}_4$	3	88
7	$3,4\text{-OCH}_2\text{O-C}_6\text{H}_3$	2	78

[a]Reaction conditions: aldehyde (2 mmol), malononitrile (2 mmol), dimedone (2 mmol), catalyst (5 mol%), 4 cm^3 of 50% EtOH, refluxing. [b]Isolated yield.



Scheme 2: Proposed mechanism for [Ch][Tau] catalyzed synthesis of 4*H*-benzo[b]pyrans.

electron withdrawing groups (Cl^-, Br^-, and F^-) and electron donating groups (MeO^-, 3,4-OCH_2O^-, and Me^-) reacted smoothly to afford the desired product in 2–3 h with good to excellent isolated yields (ranging from 78% to 92%). It has been reported that aromatic aldehydes bearing electron-withdrawing groups generally exhibit higher reactivity in this reaction compared to those bearing electron-releasing groups [23]. In our experiments, however, no specific substituent effects could be observed.

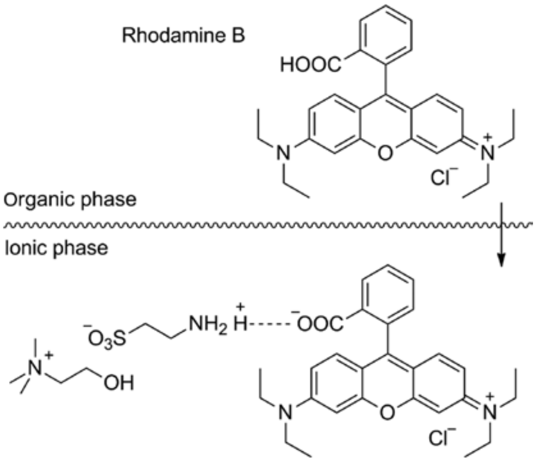
In order to evaluate the ability of [Ch][Tau] to be recycled, the cyclic condensation was carried out using the aforementioned model reaction with benzaldehyde (Table 3, entry 1). After the completion of the reaction, 2 ml of water was added to the reaction vessel and the mixture was left to stand for several hours. The precipitated crude product was collected by filtration while the filtrate was distilled to remove ethanol and water. The recovered IL was further washed with an additional 5 ml of ethyl acetate and dried *in vacuo* for 1 h at 80°C before reuse. As can be seen in Table 3, the reactivity of the recovered same batch of [Ch][Tau] was retained in the subsequent four test runs under identical reaction conditions. The yields remain around 85%–91% clearly illustrating the reusability of the functional IL.

A possible mechanism for this reaction has been proposed as shown in Scheme 2. In the first step, malononitrile reacts with amino IL to afford the carboanion of malononitrile in a reversible route. Ylidemalononitrile **1** is formed by Knoevenagel condensation with aryl aldehyde. Amino IL can also catalyze the generation of an enolate ion **2**, which reacts with the alkene **1**. Michael addition between **1** and **2** takes place followed by cyclization and imine tautomerization to give the desired 4*H*-benzo[b] pyrans **3**.

3.3 Application in extraction

On completion of the catalysis demonstration, the performance of [Ch][Tau] in extraction was subsequently investigated. As a basic IL, [Ch][Tau] might have the ability to extract acidic compounds from immiscible solvents through acid-base interaction. In this work, fluorescent dye rhodamine B was selected as a representative acidic compound (Scheme 3).

Rhodamine B can be extracted from an organic solvent immiscible with the IL phase. The rose red solution of



Scheme 3: Reactive extraction rhodamine B *via* ion pair formation.

rhodamine B in acetone (5 mM, 5 ml) was introduced into a vessel containing IL [Ch][Tau] (5 ml), forming a biphasic system because acetone is immiscible with the IL. The progress of the extraction could be observed intuitively by the color changes of the two phases. Chromogenic Rhodamine B moved across the phase boundary to the ionic phase (lower layer) as could be seen under visible and UV light (365 nm). After the mixture was stirred for 3 h at room temperature, the ionic phase showed a brilliant yellow fluorescence of rhodamine B while no distinguishable fluorescence emission could be identified in the organic layer; indicating a large majority of Rhodamine B was extracted into the IL (Figure 2A, 2 and 4). The significant color change could also be observed by the naked eye (Figure 2A, 1 and 3). To quantitatively evaluate the extraction efficiency further, the residual dye in the organic phase was determined using UV-Vis spectroscopy by measuring the absorbance at a wavelength of maximum absorbance. As can be seen from the absorption spectra, the intensity of peaks at 542 nm associated with rhodamine B decreased remarkably within 5 h, demonstrating an excellent ability of [Ch][Tau] in the extraction process. About 95% of rhodamine B was extracted as calculated through these curves (Figure 2B). The IL-acetone partition coefficient of rhodamine B was estimated as 19.

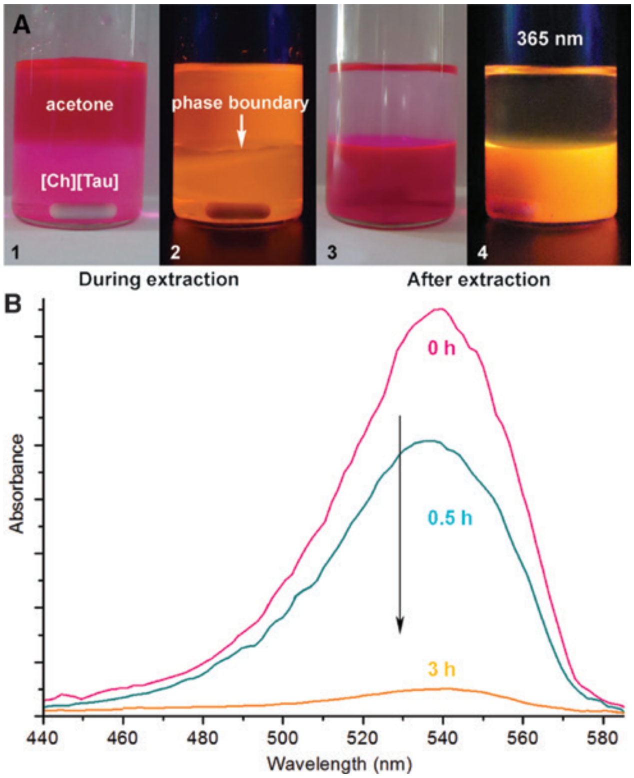


Figure 2: Photographs of the vessel during and after extraction under visible light and UV (A); UV-Vis spectroscopic tracing of the extraction of Rhodamine B (5 mM) with [Ch][Tau] (B). (A) Digital photographs of dye solutions during (left) and after (right) extraction. A significant color change in organic layer from bright rose red to nearly colorless could be observed by the naked eye. Under UV light the initial RB solution showed a brilliant yellow fluorescence, which changed to very weak fluorescence emission after extraction. (B) Absorption curves, respectively correspond to the UV absorption spectra after 0 h (pink), 0.5 h (green), and 3 h (orange). The direction of arrow indicates the downward trend of UV absorption.

4 Conclusions

In conclusion, we present a new amino-functionalized IL which was prepared by the neutralization of taurine and choline hydroxide, and its physical properties such as density, viscosity, miscibility and thermal stability were determined. This IL is eco-friendly due to the biocompatible and biodegradable nature of both choline and taurinate ions. It can be employed as an effective catalyst for the three-component cyclic condensation of aromatic aldehydes, malononitrile and dimedone, affording the corresponding 4*H*-chromene-3-carbonitrile derivatives in good to excellent yields. This catalyst can be recovered and recycled for several runs without a significant loss in activity. Besides its application in catalysis, amino-functionalized IL [Ch][Tau] can also be used as a reactive extractant as demonstrated by the extraction of rhodamine B fluorescent dye from immiscible organic solvent. The extraction process was monitored using UV-Vis spectroscopy, the extraction rate of which was determined as 95%.

Acknowledgments: The authors gratefully acknowledge the financial support from the Shanghai Commission of Science and Technology (Project no. 08431901800).

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Supplemental Material: The online version of this article (DOI: 10.1515/gps-2017-0009) offers supplementary material.

Bionotes

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