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Oxidative Esterification of Aldehydes and Alcohols Catalyzed by Camphor-Based Imidazolium Salts

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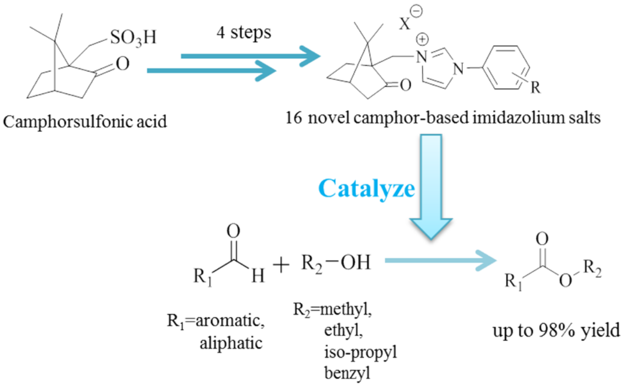
Abstract

Sixteen new camphor-based imidazolium salts have been synthesized with renewable camphorsulfonic acid as the starting material. The chemical shifts of the characteristic proton of C_2 on the imidazolium ring (N−C=N) were discussed thoroughly and all of these imidazolium salts exhibit good thermal stability. Furthermore, the excellent catalytic performance of the synthesized imidazolium salts were observed in the oxidative esterification between aromatic or aliphatic aldehydes containing electron-withdrawing or electron-donating groups on aromatic ring and primary or secondary alcohol by air as the sole oxidant.

Graphic Abstract



Keywords Camphorsulfonic acid · Imidazolium salts · Catalysis · Oxidative esterification

1 Introduction

Imidazolium salts, derived from imidazoles, consist of a pair of cation and anion, and have been found widespread utility in various fields. They are widely applied in organic synthesis [1–8], antitumor and antimicrobial activities [9–17], antioxidative activities [18, 19] and probes [20]. Therefore, more and more attention are being paid to synthesizing novel kinds of imidazolium salts. Moreover, imidazolium salts can be tuned and modified by changing the substituent of the cation or anion specifically, which has made imidazolium salts much more tunable or designable. The direct attachment of functional groups to the nitrogen of the imidazolium

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cation makes it a easy way to prepare task specific imidazolium salts efficiently.

Camphor, as a versatile building block, has shown great value in medicinal chemistry [21–23], material science [24–26] and sensors [27]. Camphor and their derivatives are bulky and possess multiple stereocenters, and they are widely used as source of chiral auxiliaries, chiral reagents and asymmetric catalyst [28–31]. Camphor-based imidazolium salts have been emerging in recent years and can be applied in enantioselective organic reactions and have particular thermotropic behavior [32]. However, there were no camphor-based imidazolium salts with phenyl ring connecting to the imidazolium core, which could increase the rigidity and stability of the salts.

The traditional ester formation was most accessed via the carboxylic acid and alcohol in the presence of acidic catalysts usually sulfuric acid, known as Fischer esterification [33]. While with the demand for a higher yield and a broader substrate scope, a stoichiometric activation of the carboxylic acid to the corresponding acyl halide was necessary. Other activators like DCC (dicyclohexylcarbodiimide) and EDC (1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide) can also achieve this aim smoothly [34]. However, it was obvious that these activation way was not atom-economic for the stoichiometric consumption of the activators. Recently, imidazolium salts have also been reported to catalyze the oxidative esterification of aromatic aldehydes as pre-catalysts. Inspired by the seminal work reported by Feroci et al. [35–37], herein. We reported the synthesis of sixteen novel camphor-based imidazolium salts. Their catalytic activities on oxidative esterification of aldehydes and alcohols were investigated with air as a mild oxidant and short reaction time and high yield could be obtained in this way.

2 Experimental

2.1 General Procedure for Synthesis of 10-Iodocamphor (1) and Phenylimidazoles (2a–2d)

10-iodocamphor **(1)** and phenylimidazoles **(2a–2d)** were prepared following a modified literature procedure [38–40] (see Supplementary Data for details).

2.2 General Procedure for the Synthesis of Camphor-Based Imidazolium Salts (3a–6a)

To a solution of 10-iodocamphor (5 mmol, 1.39 g) in DMF (3 ml), substituted phenylimidazoles (5 mmol) were added in a pressure tube and N_2 was used to exclude oxygen. The resulting reaction mixture was heated at 130 °C for 5 h. After the reaction completed, EtOAc (ca. 6 ml) was added in the mixture. Precipitation was formed and the filtrate was removed. The crude product was recrystallized in methanol and dried under vacuum.

2.3 General Procedure for the Synthesis of Camphor-Based Imidazolium Salts (3b–3d; 4b–4d; 5b–5d; 6b–6d)

A reported procedure [41] with some modifications was as followed: the synthesized iodide salt (2 mmol) was dissolved in 10 ml of DCM and 3 equivalent of the metal salts has been added. The reaction mixture was then stirred for 10 h at room temperature. The solid residue was removed by filtration and the filtrate was washed with water, and then concentrated and dried under vacuum to afford good yields of the targeted camphor-based imidazolium salts **(3b–3d; 4b–4d; 5b–5d; 6b–6d)**.

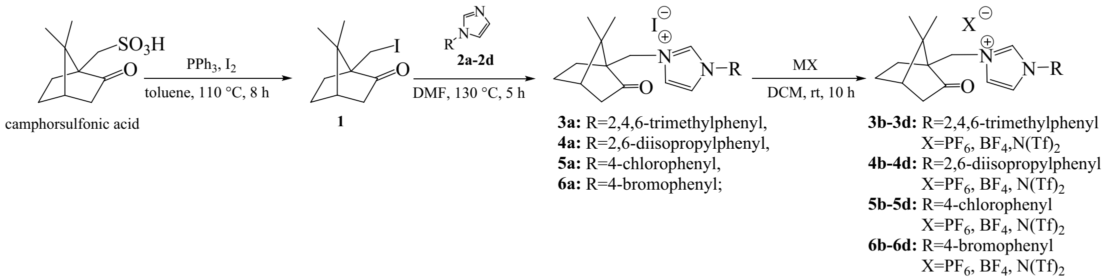
2.4 General Procedure for Oxidative Esterification of Aldehydes and Alcohols

A three-neck flask was charged with aldehyde **7a–7p** (1.0 mmol), camphor-based imidazolium salts (5 mol%), Cs_2CO_3 (163 mg, 0.5 mmol), alcohol (5 mmol) and 10 ml of toluene in an air atmosphere, then the mixture was stirred for 3 h at 60 °C. The reaction mixture was extracted with diethyl ether (3×30 ml), concentrated under reduced pressure and the residue was purified by flash column chromatography on silica gel (PE (petroleum ether):EA (ethyl acetate) = 20:1) to give the desired product. All products were identified by ^1H and ^{13}C NMR and in accord with literatures [42–52] (for spectra see Supplementary Data).

3 Results and Discussion

3.1 Synthesis and Characterization

In this report, we synthesized sixteen new camphor-based imidazolium salts with commercially available camphorsulfonic acid as starting material, as shown in Scheme 1. Firstly, 10-iodocamphor **(1)** and substituded phenylimidazoles **(2a–2d)** were prepared in an efficient way in accord with the literature [31, 38–40]. The S_N2 reaction between **(1)** and **(2a–2d)** occurred in the presence of a small amount of DMF to give high yield of corresponding iodide salts **(3a**, **4a**, **5a**, **6a)**. After that, anion exchange reactions were



Scheme 1 Synthesis of camphor-based imidazolium salts

Table 1 Chemical shift of C_2 proton on the imidazolium ring

Salts	δ (ppm)	Salts	δ (ppm)
3a	9.97	**5a**	11.03
3b	10.04	**5b**	10.82
3c	10.00	**5c**	11.04
3d	9.39	**5d**	9.70
4a	9.98	**6a**	11.02
4b	10.09	**6b**	10.75
4c	9.87	**6c**	10.92
4d	9.39	**6d**	9.87

Table 2 Physical properties of imidazolium salts **(3a–6d)**

Salts	Abbreviation	Physical state	T_m (°C)	$T_{5\%}$ (°C)
3a	[MesCamIm]I	Off-white solid	218–220	319
3b	[MesCamIm]PF_6	White solid	199–201	320
3c	[MesCamIm]BF_4	White solid	212–214	315
3d	[MesCamIm]$N(Tf)_2$	Yellowish solid	90–92	328
4a	[IprCamIm]I	Light-brown solid	238–239	312
4b	[IprCamIm]PF_6	Yellowish solid	222–224	309
4c	[IprCamIm]BF_4	Light-brown solid	239–240	307
4d	[IprCamIm]$N(Tf)_2$	Yellowish solid	76–78	312
5a	[4-Br-PhCamIm]I	Light-brown solid	144–145	310
5b	[4-Br-PhCamIm] PF_6	Brown solid	140–141	313
5c	[4-Br-PhCamIm] BF_4	Brown solid	158–160	315
5d	[4-Br-PhCamIm] $N(Tf)_2$	Brown viscous liquid	− 15.4[a]	358
6a	[4-Cl-PhCamIm]I	Light-brown solid	194–195	313
6b	[4-Cl-PhCamIm] PF_6	Off-white solid	186–187	311
6c	[4-Cl-PhCamIm] BF_4	Yellowish solid	197–198	309
6d	[4-Cl-PhCamIm] $N(Tf)_2$	Yellow viscous liquid	− 11.1[a]	328

[a] Glass transition temperature (T_g)

performed in DCM at room temperature for 10 h, furnishing camphor-based imidazolium salts **(3b–3d**; **4b–4d**; **5b–5d**; **6b–6d)** with 78–86% yields.

^1H NMR spectra used here was not only a tool to illuminate the structure and purity of target compounds but also providing information on the electron density distribution between cations and anions [53]. In this context, the most significant chemical shifts were observed in the case of the C_2 proton (proton on the carbon of N−C=N) in imidazolium ring. As shown in the ^1H NMR spectrum of these imidazolium salts (for spectra see supplementary data), the signal of C_2 proton is registered in the lowest field, which means the C_2 proton has the lowest electron density surrounding itself. The chemical shifts of C_2 proton from these camphor-based imidazolium salts were all listed in Table 1 and investigated thoroughly. It is obvious to see that the substituents on the aryl part of these imidazolium salts have a tremendous effect on C_2 proton chemical shifts. The imidazolium salts with electron-withdrawing groups on the phenyl ring (4-chlorophenyl, 4-bromophenyl) have a larger chemical shift than that with electron-donating groups (2,4,6-trimethylphenyl, 2,6-diisopropylphenyl). This can be understood that the electron-withdrawing groups make the imidazolium ring much more electron deficient and the proton prone to be more acidic [54]. With the cation fixed, the substitution of the I^- anion with PF_6^- and BF_4^- resulted in limited changes in the electron density near the quaternary nitrogen atom. While a significant upfield variation was observed when the I^- was exchanged by the $N(Tf)_2^-$, resulting from the increasing shielding capacities of $N(Tf)_2^-$.

3.2 Thermal Properties

As observed in Table 2, not all of these imidazolium salts have low melting points. Similar condition could be observed in the case of phenyl/alkyl-substituted pyrazolium, as well as other kinds of imidazolium-based salts [55–59]. Unlike *N*,*N*-dialkyl imidazolium salts, these imidazolium salts have much higher melting points, which can be attributed to the introducing of aryl moiety and hence the enhancement of intermolecular π–π stacking occurred [57]. Four imidazolium salts (**3d**, **4d**, **5d**, **6d**) among these 16 imidazolium salts could be considered as ionic liquids for their melting points were below 100 °C.

According to the results shown in Table 2, both the electron-withdrawing substituents (4-chlorophenyl, 4-bromophenyl) and the electron-donating substituents (2,4,6-trimethylphenyl, 2,6-diisopropylphenyl) on the phenyl ring and the types of anions affect the melting points of these imidazolium salts. The melting points of imidazolium salts with electron-donating groups on the phenyl ring are higher than those of the salts containing electron-withdrawing groups, when the anions are fixed.

Considering the effect of the counterion on the melting points, we found that the anion size was an important factor that influence the phase transition temperatures of these salts. Imidazolium salts containing I^- and BF_4^- displayed highest melting temperatures. While in the case of PF_6^-, the melting points are a little lower for a given cation than the imidazolium salts with I^- and BF_4^-, which is corresponding to the trend observed in literature [58]. Moreover, this effect was more apparent when the weakest coordinating anion $N(Tf)_2^-$ was applied, and as a result, the imidazolium salts with $N(Tf)_2^-$ as anion have the lowest melting points below 100 °C. Thus, it can be predicted that the melting point of studied ILs decreases in the following order: $N(Tf)_2^- < PF_6^- < BF_4^- \approx I^-$.

Thermal stability is an important criterion and basis for evaluating the properties and applications of imidazolium salts. In this report, thermal stability of these imidazolium salts was investigated with thermogravimetric analysis and $T_{5\%}$ (temperature at which 5% weight loss of sample was

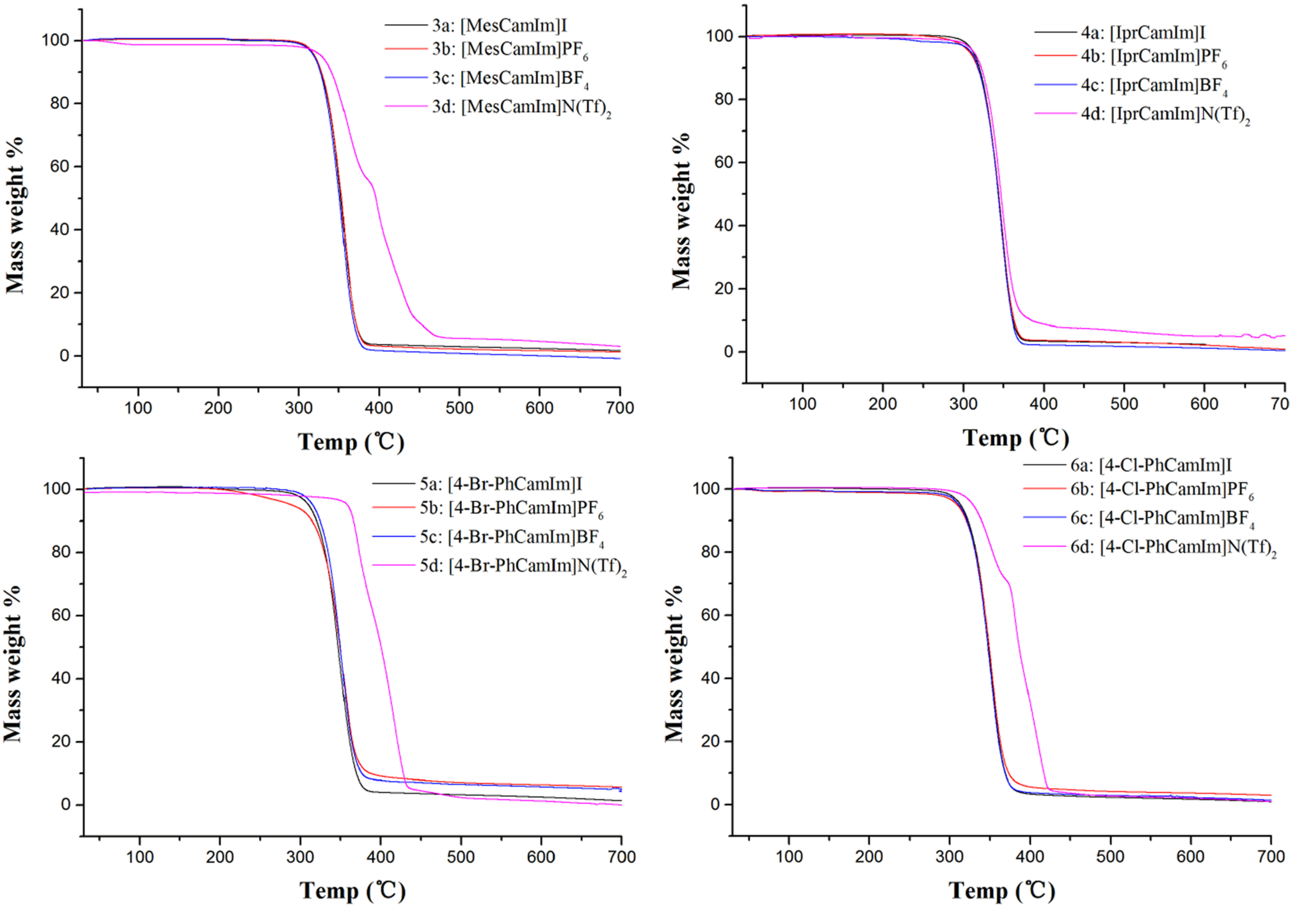
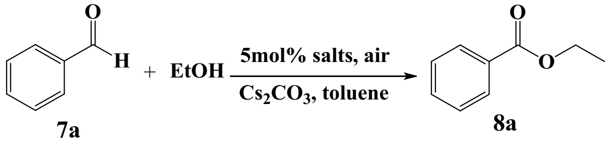


Fig. 1 TGA curves for camphor-based imidazolium salts **3a**–**6d** with mass weight% against temperature (°C)

Table 3 Catalytic performance of these imidazolium salts with benzaldehyde and ethanol as model reaction



Entry	Salts	Base	Temp (°C)	Time (h)	Yield (%)[a]
1	None	Cs_2CO_3	80	3	0
2	3a	Cs_2CO_3	80	3	98
3	3b	Cs_2CO_3	80	3	95
4	3c	Cs_2CO_3	80	3	Trace
5	3d	Cs_2CO_3	80	3	91
6	4a	Cs_2CO_3	80	3	80
7	4b	Cs_2CO_3	80	3	95
8	4c	Cs_2CO_3	80	3	93
9	4d	Cs_2CO_3	80	3	76
10	5a	Cs_2CO_3	80	3	85
11	5b	Cs_2CO_3	80	3	Trace
12	5c	Cs_2CO_3	80	3	68
13	5d	Cs_2CO_3	80	3	10
14	6a	Cs_2CO_3	80	3	13
15	6b	Cs_2CO_3	80	3	Trace
16	6c	Cs_2CO_3	80	3	Trace
17	6d	Cs_2CO_3	80	3	Trace

[a] Yields determined by GC analysis in the presence of internal standard

observed) was chosen as the onset decomposition temperature, which was especially important for TAAILs (in this text **3d**, **4d**, **5d**, **6d**). As shown in Table 2 and Fig. 1, thermal decomposition temperatures of all these imidazolium salts were found higher than 300 °C ($T_{5\%} > 300$ °C). With regard to the anion effect, the thermal stability of I^- containing imidazolium salts were almost the same with PF_6^- and BF_4^- containing imidazolium salts. While the most thermal stable imidazolium salts were obtained when the anions was exchanged to $N(Tf)_2^-$ (up to 358 °C). Surprisingly, there were no much difference in thermal stability when electron-donating groups on the phenyl ring were switched by electron-withdrawing groups (excepted **5d**, 358 °C).

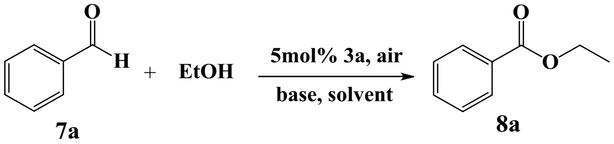
3.3 Catalytic Performance in Aldehyde Oxidative Esterification

With these camphor-based imidazolium salts in hand, we examined the catalytic potential in oxidative esterification of aldehyde with alcohol, and the test results were listed in Table 3. We first conducted reactions in the following condition: 1 equivalent of benzaldehyde, 5 mol% imidazolium salts, 0.5 equivalent of Cs_2CO_3 and 10 equivalent of ethanol were added to the flask containing toluene equipped with an oxygen balloon and the mixture was stirred at 80 °C for 3 h. As comparison, the reaction, in the absence of catalyst, can hardly go on. In general, several camphor-based imidazolium salts exhibited moderate to good catalytic performance (**3a, 3b, 3d, 4a, 4b, 4c, 4d, 5a, 5c**) with yields ranging from 68 to 98%.

The reaction was then optimized by using benzaldehyde and ethanol as model substrates, and **3a** as catalyst. Firstly, when the temperature was lowered to 60 °C or the amount of ethanol was decreased to 5 equivalents, the reaction could still proceed smoothly without any yield loss (Table 4, entries 2 and 3). Decreasing the catalyst loading to 2 mol% slightly decreased the product yield (Table 4, entry 2). However, yield decreasing was observed when temperature was lowered below 60 °C (Table 4, entries 4 and 5) and time was reduced less than 3 h (Table 4, entries 6 and 7). The reaction can be carried out in MeCN and THF with yields as 56% and 59% respectively (Table 4, entries 8 and 10), while in TBME, trace of product was obtained (Table 4, entry 8). The bases as another important factor were also investigated including organic and inorganic bases, including KOH, K_2CO_3, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), DIPEA (N,N-diisopropylethylamine) and DABCO (1,4-diazabicyclo[2.2.2]octane). It was found that the organic base DBU and the inorganic base K_2CO_3 could promote the reaction in a moderate yield (Table 4, entries 12 and 13), while KOH, DIPEA and DABCO only obtained low yield of products (Table 4, entries 11, 14 and 15).

The substrate scope of aldehydes and alcohols were then investigated and the results were listed in Table 5. As shown in Table 5, all the primary, including benzyl alcohol, and secondary alcohols were suitable for this catalytic system and achieved good yields (Table 5, entries 1, 2, 3 and 4). However, when a more steric hindered *t*-BuOH (Table 5, entry 5) was applied, only trace of product could be obsreved, indicating that the steric *t*-butyl group significantly affected the activity of the reaction. The aromatic aldehydes with electron-withdrawing substituents, including nitro (NO_2), chloro (Cl), bromo (Br), fluoro (F), trifluoromethyl (CF_3) and cyano (CN) groups had much higher yields than the aromatic aldehydes with electron-donating substituents. Increasing the equivalent of alcohol or prolonging the reaction time to 5 h could still not promote the yield of the corresponding ester significantly (Table 5**,** entries 15, 16 and 17). This makes sense because electron-withdrawing groups make the carbonyl much more electrophilic and thus more

Table 4 Condition optimization with benzaldehyde and ethanol as model reaction



Entry	EtOH (mmol)	Base	Solvent	Temp (°C)	Time (h)	Yield (%)[a]
1	10	Cs_2CO_3	Toluene	80	3	98
2	10	Cs_2CO_3	Toluene	60	3	98 (86[b])
3	5	Cs_2CO_3	Toluene	60	3	98
4	5	Cs_2CO_3	Toluene	40	3	77
5	5	Cs_2CO_3	Toluene	rt	Over night	80
6	5	Cs_2CO_3	Toluene	60	1	45
7	5	Cs_2CO_3	Toluene	60	2	75
8	5	Cs_2CO_3	THF	60	3	56
9	5	Cs_2CO_3	TBME	60	3	Trace
10	5	Cs_2CO_3	MeCN	60	3	59
11	5	KOH	Toluene	60	3	Trace
12	5	K_2CO_3	Toluene	60	3	51
13	5	DBU	Toluene	60	3	78
14	5	DIPEA	Toluene	60	3	Trace
15	5	DABCO	Toluene	60	3	37

Unless otherwise noted, reactions were performed with following optimized conditions: benzaldehyde (1 mmol), imidazolium salts **3a** (5 mol%), base (0.5 mmol), solvent (10 ml)

[a] Yields determined by GC analysis in the presence of internal standard

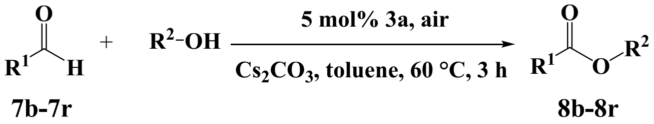
[b] 2mol% catalyst loading

easily attacked by alcohols in the basic condition. Excellent yields were observed for various substitutions at the *ortho*-, *meta*- and *para* positions of the phenyl ring, when the functional group on the phenyl ring was fluoride (Table 5, entries 10, 11 and 12). As a contrast, a poor yield was observed with the nitro group located at the *ortho*-position (Table 5, entry 7). We deemed that the nitro group, as a larger functional group than fluoro group, had a larger steric hindrance and thus reduced the reaction activity. Other kinds of aromatic aldehyde, including 1-pyrenecarboxaldehyde and 2-thenaldehyde, could also proceed smoothly with yield over 79% (Table 5, entries 18 and 19). Compared to aromatic aldehyde, aliphatic aldehydes could also achieve good yields in such condition (Table 5, entries 20 and 21).

3.4 Recycling of Catalyst 3a and Characterization

The hypothesis of the reaction mechanism has been described in Supplementary data (Scheme S1). To test the reusability of our synthesized catalyst **3a**, we first conducted the parallel reactions with/without substrate (with 3-nitrobenzaldehyde and ethanol as model reaction) and the corresponding ^1H NMR of **3a** were almost identical with the original spectrum as shown in Fig. 2, which demonstrated the stability of the catalyst **3a** under the reaction condition. Then we put our efforts on the recycling of the catalyst **3a**. The solvent was first evaporated under vacuum and diethyl ether (ca. 20 ml) was poured into the flask to form a suspension. Catalyst **3a** along with the base could be obtained by

Table 5 Catalytic performance of **3a** with variation of substrates and alcohols



Entry	R^1	R^2	Product	Yield[a]
1	3-NO_2 Phenyl	Et	**8b**	95
2	3-NO_2 Phenyl	Me	**8c**	95
3	3-NO_2 Phenyl	i-Pr	**8d**	95
4	3-NO_2 Phenyl	Benzyl	**8e**	92
5	3-NO_2 Phenyl	t-Bu	–	Trace
6	4-NO_2 Phenyl	Et	**8f**	94
7	2-NO_2 Phenyl	Et	–	Trace
8	4-Cl Phenyl	Et	**8g**	95
9	4-Br Phenyl	Et	**8h**	90
10	2-F Phenyl	Et	**8i**	94
11	3-F Phenyl	Et	**8j**	94
12	4-F Phenyl	Et	**8k**	92
13	3-CF_3 Phenyl	Et	**8l**	92
14	3-CN Phenyl	Et	**8m**	95
15	4-OMe Phenyl	Et	**8n**	73
16	4-OMe Phenyl	Et	**8n**	75[b]
17	4-OMe Phenyl	Et	**8n**	75[c]
18	2-Thienyl	Et	**8o**	88
19	1-Pyrenyl	Me	**8p**	79
20	CH_3CH_2	Et	**8q**	84
21	$CH_3(CH_2)_8$	Et	**8r**	80

[a] Yields of isolated product, relative to the starting aldehyde

[b] 10 eq of alcohol was used

[c] Reaction time was prolonged to 5 h

filtration and the resulting solid was washed completely with diethyl ether. Regenerated base and **3a** could be reutilized in four runs (20% of original amount of **3a** was added additionally but no additional base was needed each run) with no significant decline in yields. The results were listed in Fig. 3

4 Conclusion

To summarize, a series of camphor-based imidazolium salts have been synthesized by an efficient method, with anions as I^-, PF_6^-, BF_4^- and $N(Tf)_2^-$. Four of these salts can be rendered as TAAILs (**3d, 4d, 5d, 6d**). All these salts have been fully characterized using ^1H, ^{13}C, ^{19}F NMR and HR-MS techniques. These imidazolium salts exhibited good thermal stability, all of which have $T_{5\%}$ larger than 300 °C. Several of these new synthesized camphor-based imidazolium salts (**3a, 3b, 3d, 4a, 4b, 4c, 4d, 5a, 5c**) showed moderate to good catalytic performance in aldehyde oxidative esterification. Furthermore, **3a** was successfully employed in such reaction and high yields were obtained with a broad scope of aldehyde.

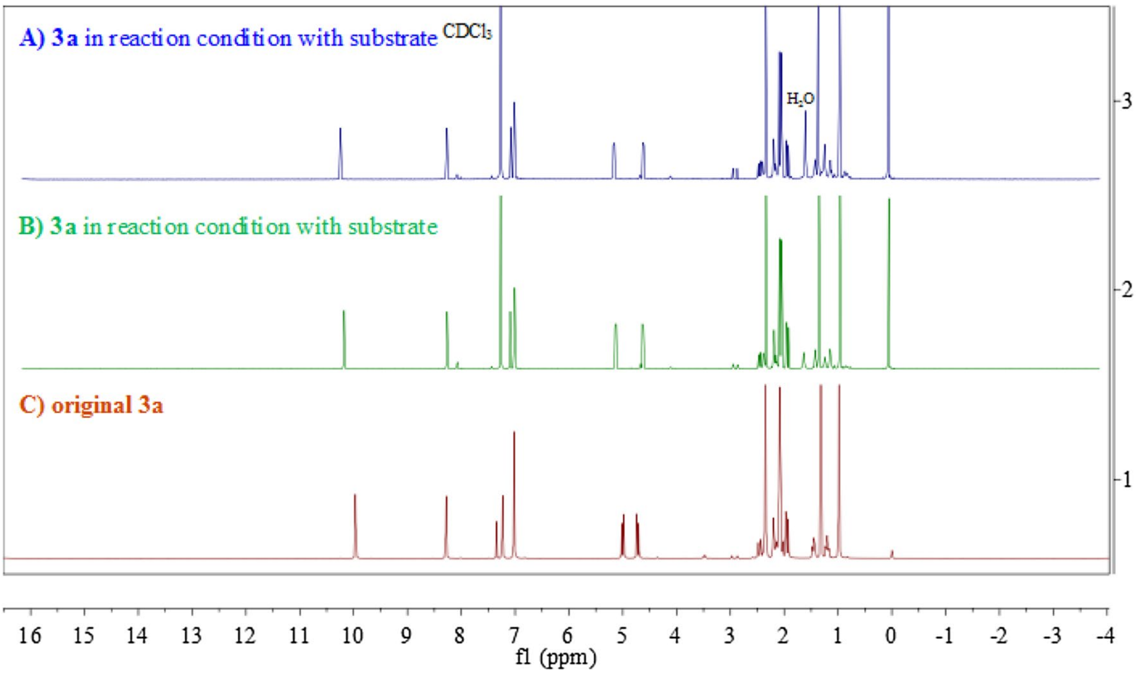


Fig. 2 ^{1}H NMR of catalyst **3a**. **a** Regenerated with substrate under reaction condition; **b** regenerated without substrate under reaction condition; **c** original spectrum

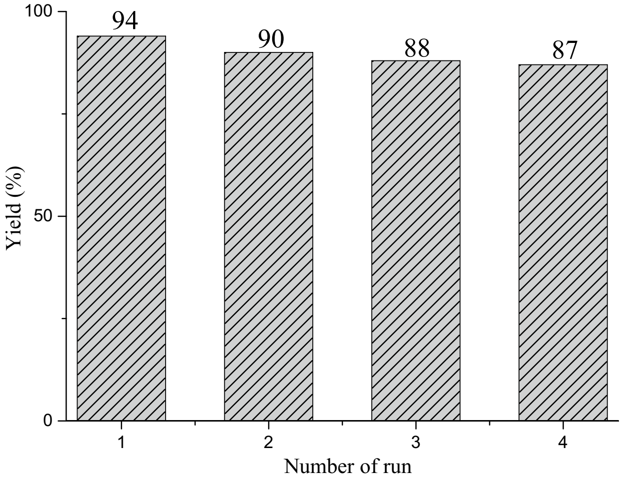


Fig. 3 Reusability of catalyst **3a** for model reaction

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