

Preparation and Application of Silica Films Supported Imidazolium-Based Ionic Liquid as Efficient and Recyclable Catalysts for Benzoin Condensations

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Abstract

Two silica films -immobilized imidazolium-based ionic liquids (TMICl @silica films) were prepared, characterized and utilized as efficient catalysts for the benzoin reaction. Combined characterization results from FT-IR, elemental analysis, N_2 adsorption–desorption isotherms and SEM, suggested that the imidazolium-based ionic liquids were successfully immobilized on the silica films. Moreover, the catalytic performance tests demonstrated that silica films immobilized imidazolium-based ionic liquids (ILs) exhibited excellent activity for the benzoin reactions of aromatic aldehydes. The influence of catalyst concentration, temperature and reaction duration on the catalytic activity were investigated by employing 0.7TMICl @ silica film(catalyst C) as the catalyst. The results also showed that the benzoin condensations of aromatic aldehydes could give desired products with satisfactory yields under the optimized conditions. Additionally, the catalyst can be effortlessly separated by filtration and reused more than five times without significant loss of activity.

Graphic Abstract



 Wide substrate variety
 Easy separation and recycle
 Good potential application

 0.7TMICl@ silica films (catalyst C) exhibited robust catalytic performance in the benzoin reaction
 Content of the benzoin reaction

Keywords Silica films · Immobilized · Imidazolium-based ionic liquids · Catalysis · Benzoin reaction

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1 Introduction

 α -Hydroxyketones (acyloins) are valuable intermediates for the synthesis of a wide range of natural and unnatural compounds [1]. Thus the efficient synthesis of α -hydroxyketones has been pursued for decades [2–8]. In its traditional form, only aromatic aldehydes or glyoxals could be coupled using cyanide anion as a catalyst [9, 10]. However, the toxicity of cyanide anion and finite suitable substrates limit the large-scale industrial application of the reaction. In 1943, Ugai et al. [11] discovered that VB1 could promote benzoin condensation reaction with the aid of alkali, which avoided the involvement of highly tox cyanide as catalyst. Further studies showed that thiazolium salt in VB1 was converted into nucleophilic *N*-heterocyclic carbene (NHCs) under the action of alkali, which was the actually active catalyst.

Accordingly, considerable attention has been paid to the use of NHCs as organocatalysts [12, 13]. Many NHCs derived from triazolium or thiazolium salts have been identified as efficient catalysis for benzoin condensation reaction [14, 15]. Although NHCs catalysts have made great achievements in benzoin condensation as organic catalysts, these homogeneous catalysts tend to cause difficulties in catalyst recovery and product purification. Moreover, because the reactants and ionic liquids are often immiscible and can not be fully contacted each other, the catalytic efficiency of the catalyst is poor, resulting in a large amount of catalyst used. To successfully conquer these issues, the development of NHCsbased heterogeneous catalysts is considered as the most effective alternative solution. Furthermore, when immobilized on a suitable support having a large surface area, the welldispersed NHCs facilitate not only high catalytic activity but also facile separation of catalyst from reaction products [16–20]. Several groups have reported that supported imidazolium/thiazolium salt pre-catalysts have been prepared and used to catalyze various reactions, such as the benzoin reactions and alcohol etherification, and that the pre-catalysts could be readily recycled and reused many times [21–28]. Although these supported imidazolium salt pre-catalysts could catalyze benzoin reactions and be recycled, their wide application is limited due to the inherent shortcomings of organic compounds, such as poor thermal stability, low substrate adaptability, etc. On the other hand, silicon-based materials have become a research hotspot due to their large surface area and good thermal stability. Among numerous silicon-based materials, silica films have better structural stability and emerges as a promising candidate for the catalyst support [29]. When ionic liquid pre-catalysts were dispersed and immobilized on silica films, the surface area of ionic liquid pre-catalyst increases. This will enable the ionic liquid pre-catalyst to fully contact with the reactants and improve the catalytic efficiency. On account of the above reasons, we designed and prepared two easily prepared silica films immobilized imidazolium ionic liquids pre-catalysts and applied them to benzoin reaction of varied aromatic aldehydes. In order to compare the effects of different supports on the activity of catalysts, we also synthesized SBA-15 immobilized imidazolium salt pre catalyst according to the literature [20].

2 Results and Discussion



Scheme 1 Schematic illustrations of synthesis procedures for TMICl



Scheme 2 Schematic illustrations of synthesis procedures for TMICI@ silica films

was dissolved in the aqueous solution containing P123 and oxalic acid. The mixture was stirred at 40 °C for 1 h and a clear solution with partially hydrolyzed silica yielded. The reaction mixture was coated on glass slides, and then these glass plate was placed in an oven at 40 °C overnight to form a film. Silica films (Catalyst A) were obtained by removing P123 and oxalic acid and drying. 0.3TMICl @silica film (Catalysts B) and 0.7TMICl@ silica film (Catalyst C) were synthesized by using of TEOS, P123, oxalic acid and 1-methyl-3-trimethoxysilylpropylimidazol-ium chloride as raw materials according to the previous method of preparing catalyst A. The difference between catalyst B and catalyst C is that more ionic liquids are used to prepare catalyst C than catalyst B. At last, the synthesis of TMICl@ SBA-15 (Catalyst D) was carried out by first dispersing dehydrated SBA-15 in toluene, followed by adding TMICl to the SBA-15 suspension under inert (N₂ gas) atmosphere.

These as-prepared catalysts have been characterized by FT-IR, EA, BET and SEM. FT-IR spectra of silica films (catalyst A), 0.3TMICl@silica films (catalyst B) and 0.7TMICl@ silica films (catalyst C) are illustrated in Fig. 1. As shown in Fig. 1, the absorption bands at about 457, 797 and 1072 cm^{-1} were characteristic of SiO₂ [30, 31], whereas the bands at around 1640 and 3431 cm⁻¹ were attributed to the stretching frequency of Si-OH and physisorbed water [30, 32]. For 0.3TMICl @silica films and 0.7TMICl@ silica films, the characteristic peaks were found at around 1570 and 1456 cm^{-1} , which can be assigned to the C=N and C=C stretching vibrations of the imidazole ring [30, 33]. In comparison with silica films(A), 0.3TMICl@silica films (B) and 0.7TMICl@ silica films (C) exhibited the same characteristic bands such as the Si-O-Si vibrations (797 and 1072 cm^{-1}) and the stretching vibration of -OH (around 1640, 3440 cm^{-1}). In addition, the absorption peaks at 2985, 2924, 1456 and 1383 cm^{-1} are the stretching and bending vibration peaks of C-H bonds of methyl and methylene. The N2 adsorption-desorption isotherms and pore size distributions of silica films(A), 0.3TMICl@ silica films (B) and



Fig. 1 FT-IR spectra of Catalyst A, B and C



Fig. 2 N₂ Adsorption-desorption isotherms of catalyst A, B and C

0.7TMICl@ silica films (C) are illustrated in Figs. 2 and 3. The isotherms of 0.7TMICl@silica films (C) are typical type IV isotherms according to IUPAC classifications, and the hysteresis loops of H3 of 0.7TMICl@ silica films are the characteristics of mesoporous structure under the pressure of p/p0 = 0.45 - 1.0 [34]. The isotherms of silica films and 0.3TMICl@silica films show typical type I isotherms according to IUPAC classifications. However, the isotherm of 0.3TMICl@silica films has a very small H4 hysteresis loop under the pressure of p/p0=0.1-0.8, which indicates that silica films have microporous structure characteristics, while 0.3TMICl@silica films has mixed characteristics of microporous structure and mesoporous structure. These conclusions are also confirmed by the pore size distribution. The pore size distribution shows that the pore size of silica films is about 1.7 nm, that of 0.3TMICl@ silica films(B) is



Fig. 3 Pore size distribution of catalyst A, B and C

Table 1 BET surface properties of catalyst A, B and C

Sample	BET surface area (m ² /g)	Total pore volume (cm ³ /g)	Average pore width (nm)
A	630.96	0.3140	1.99
В	567.79	0.3866	2.28
<u>C</u>	537.90	0.6059	6.35

about 1.7 and 2.6 nm, and that of 0.7TMICl@silica films(C) is between 2.3 and 10. The reason for these differences is that the amount of TMICl ionic liquid used in the process of preparing these materials is different. The surface area, pore size and pore volume of the samples were calculated by the BET equation and shown in Table 1. According to the data in Table 1, it can be seen that the specific surface of the synthesized film material decreases slightly with the addition of TMICl and the increase of its dosage. On the contrary, the average pore size and the pore volume increase with the addition of TMICl and the increase of its dosage. The imidazolium salt loading levels of 0.7TMICl@ silica films(C) and 0.3TMICl@ silica films(B) were determined by elemental analysis, were 0.93 and 0.46 mmol g⁻¹, respectively. The morphology of catalyst A, B and C were also studied by use of SEM. As shown in Fig. 4a-c, the prepared solid catalysts displayed lamellar structure. Compared with the catalyst A, Catalysts B and C have porous structure due to the addition of ionic liquids. Finally, TMICl @ SBA-15(catalyst D) was characterized by IR, SEM and elemental analysis. FT-IR spectra of SBA-15 and catalyst D are showed in Fig. 5. Compared with the infrared spectra of SBA-15 and TMICl @ SBA-15, in the infrared spectra of TMICl @ SBA-15, the absorption peaks at 2980, 1574 and 1383 cm^{-1} are the characteristic absorption peaks of imidazolium salt and



Fig. 4 SEM images of catalyst A, B, C and D



Fig. 5 FT-IR spectra of catalyst D and SBA-15

methylene, which indicates that the imidazolium salt ionic liquid has been immobilized onto the SBA-15. The imidazolium salt loading level of TMICl @ SBA-15 was determined by elemental analysis, was 0.11 mmol g^{-1} . The morphology of TMICl@SBA-15 (catalyst D) was also studied by SEM. As shown in Fig. 4d, catalyst D showed a wheatlike morphology similar to the SBA-15, in agreement with the earlier literature [35], with the irregular surface and different sizes.

After the characterization of the catalysts, the catalytic activities of these catalysts were examined in the benzoin reaction. The investigation was initiated by using the benzoin reaction of benzaldehyde as a model reaction. Initially, we briefly evaluated the catalytic efficiency of different catalysts (A = silica film, B = 0.3TMICl@silica film, C = 0.7TMICl@ silica film and D = TMICl@SBA-15) for the benzoin reaction of benzaldehyde. As shown in Table 2 (entries 1–4), catalyst C showed better catalytic potency

Table 2 The benzoin reaction of benzaldehyde: optimization of the reaction conditions



Entry	Catalyst	Solvent	Base	Yield (%) ^a
1 ^b	A	CH ₃ OH	NaOH	0
2	В	CH ₃ OH	NaOH	64
3	С	CH ₃ OH	NaOH	88
4	D	CH ₃ OH	NaOH	70
5	С	THF	NaOH	21
6	С	CH ₃ CN	NaOH	0
7	С	Dioxane	NaOH	15
8	С	CH ₃ OH	DBU	9
9	С	CH ₃ OH	K ₂ CO ₃	0
10	С	CH ₃ OH	NaOAc	0
11 ^c	С	CH ₃ OH	NaOH	67
12 ^d	С	CH ₃ OH	NaOH	31
13 ^e	С	CH ₃ OH	NaOH	80
14^{f}	С	CH ₃ OH	NaOH	61
15 ^g	С	CH ₃ OH	NaOH	73
16 ^h	С	CH ₃ OH	NaOH	76
17 ⁱ	С	CH ₃ OH	NaOH	86

Unless otherwise stated, General reaction conditions:: 2.0 mmol of benzaldehyde, 0.1 mmol of imidazolium salt, 3 mL of solvent, 0.8 mmol of base, 130 °C oil bath under Ar atmosphere for 10 h ^aIsolated yield

- ^b0.1 g catalyst A, [imidazolium salt]=0 mol%
- °110 °C oil bath
- ^d80 °C oil bath
- ^e140 °C oil bath

^f0.5 mmol of NaOH was used in benzoin reactions

- ^g1 mmol of NaOH was used in benzoin reactions
- ^hReaction time was changed to 8 h

ⁱReaction time was changed to 14 h

than catalyst A, B and D for the benzoin reaction of benzaldehyde. This was related to the proper pore size of catalyst C. The surface pore size of catalyst C ranges from 2 to 10 nm, which facilitates the adsorption of substrate molecules and promotes the reaction. A subsequent solvent screen showed CH₃OH was proved to be a far superior solvent with respect to the rate of benzoin formation of those tested (Table 2, entries 3 vs. 5–7). This may be attributed to the good solubility of NaOH in methanol. To know the effect of bases on the outcome of the reaction, different bases were employed in the benzoin reaction and it was observed that NaOH was the most effective base (Table 2, entries 3 and 8-10). Therefore NaOH was used in subsequent reactions as the base. As described in Table 2, lower or higher temperature resulted in decrease of yields and 130 °C was the appropriate temperature (Table 2, entries 3 and 11-13). The experimental results also show that the amount of NaOH has a significant effect on the yield of the reaction. The optimal amount of base used here was 0.4 equiv., and more or less alkali led to decrease of yields (Table 2, entries 3 and 14-15). Finally, we investigate the effect of time on the reaction. The data in Table 2 indicated that the best yield was obtained when the reaction was extended to 10 h. Hower, if the reaction time is prolonged, the yield will not increase(Table 2, entries 3 and 16-17). It was that the benzoin reaction of benzaldehyde gave the best yield in the presence of 0.7TMICl@ silica film (Catalyst C, 5 mmol% imidazolium salt) and 40 mmol % of NaOH as base in CH₃OH at 130 °C under an Ar atmosphere for 10 h.

The scope of benzoin reaction was demonstrated by using various aromatic aldehyde under the optimized conditions. The results are listed in Table 3. We were delighted to find that our catalytic system was capable of tolerating relatively different aromatic aldehydes under the optimized conditions. As is evident from Table 3, the benzoin condensation of various substituted aldehydes including electron rich derivatives such as CH₃ or OCH₃ were accomplished with no complications, generally resulting in the desired product in good yields (56-80%). As it can be observed, substituent on the aromatic ring, such as CH₃, OCH₃ and Cl had a considerable effect on the yield. The best yield of 87% was obtained by using benzaldehyde as a substrate. On the other hand, no desired product was observed in the case of 4-nitro-benzaldehyde as previously recorded by Xia et al. [36]. As expected, alkyl aldehyde gave lower yield as compared to aryl aldehyde. For n-butylaldehyde, there are no acyloin products, but α,β -unsaturated aldehyde was obtained in moderate yield.

From environmental and economic points of view, the stability and sustained activity of the catalyst are of great importance. To explore the reusability of 0.7TMICl@ silica films (catalyst C), the benzoin reaction of benzaldehyde was chosen as a model reaction. As shown in Fig. 6, catalyst C can be recycled up to five times with no appreciable decrease in the yields. The slight decrease in the catalytic activity may be reasonably attributed to the negligible breakage of the supported ionic liquids from the surface of the silica films during the benzoin reaction processes. In each cycle, the catalyst was separated from the reaction mixture by filtration, washed with methanol (3–5 mL), then dried under

reduced pressure and reused in the next run under the same conditions without any further treatment.

3 Conclusions

To sum up, two silica films-supported imidazolium salts were successfully prepared, characterized and utilized in catalyzing the benzoin condensation reaction to give the α -hydroxyl ketone. On the basis of the characterization results, the ionic liquid (TMICl) were proved to be immobilized successfully onto the silica films. Moreover, silica films-supported imidazolium salt pre-catalysts showed good catalytic efficiency for the preparation of α -hydroxy ketones. Catalyst C (0.7TMICl @ silica films) displayed the best catalytic efficiency for benzoin reaction of aldehyde. In addition, the catalyst can simply be separated and reused for five times without significant decrease in its catalytic activity. Further applications of this catalyst to other transformations are currently under investigation.

3.1 Experimental Section

3.1.1 General Information

Reagents were obtained from commercial suppliers and used without further purification unless otherwise noted. All the reactions were carried out in reaction tube under argon atmosphere. Thin layer chromatography (TLC) was performed using Qingdao silica gel (silica gel GF254). TLC plates were analyzed by ultraviolet (UV) light. ¹H NMR and ¹³C NMR spectra were measured in Bruker AV 400 MHz using CDCl₃ or CD₃OD as solvent and TMS as internal standard. Melting points were measured with an X-5 micro-melting point apparatus and are uncorrected. Fourier transform infrared (FT-IR) spectra of all samples were recorded using a Bruker Vertex 80. The BET sorption of N₂ was determined using Micromeritics ASAP2010 equipment (Micromeritics Instrument Co., Ltd., Norcross, USA). The C, N and H amount contents of these catalysts was estimated by elemental microanalysis technique using a Vario EL cube elemental analyzer (Elementar Analysensysteme Co., Ltd., Hanau, Germany), and the loading amount of ionic liquids on the silica films was calculated by the content of N element. Morphologies and these catalysts were investigated by a Nova NanoSEM 450 scanning electron microscopy (SEM).

3.1.2 Preparation of Silica Films (A)

First, 1.224 g P123, 50 mL distilled water and 4.48 g oxalic acid were added to 250 mL round bottom flask, stirred for

O ,H 130°C, CH₃OH, NaOH Ar—́́́́́						
2 Ar—{ — 0	Ar, Catalyst C, 10h	→Ar HO				
Entry	Substrate	Product	Yield ^b , %			
1	CHO CH ₃	$\underset{H_3C}{\overset{O}{\longrightarrow}} \underset{HO}{\overset{O}{\longrightarrow}} \underset{HO}{\overset{CH_3}{\longleftarrow}}$	72			
2	CHO CH ₃	H ₃ C-CH ₃	78			
3	CHO CH ₃ CH ₃	H_3C H_3C H_0 H_0 H_3 $H_$	71			
4	СНО	CI HO	74			
5	CHO	Br	76			
6	CHO C(CH ₃) ₃	$(H_2C)_2C - (C) + (C)_{HO} - (C) + (C)_{HO} + (C)_{HO$	80			
7	СНО	HO HO	63			
8	CHO OCH3	H ₃ CO	56			
9	CHO CH(CH ₃) ₂	$(H_3C)_2HC$ $(H_3C)_2$ HO $(H_3C)_2$ HO $(H_3C)_2$	70			
10	CHO NO ₂	0 ₂ N-	0			
11	СНО	СНО	57			

Unless otherwise stated, general reaction conditions: 2.0 mmol of aldehyde, 0.1 mmol of imidazolium salt, 0.8 mmol of NaOH, 3 mL of CH_3OH at 130 °C under Ar atmosphere for 10 h

^bIsolated yield

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2 h at 40 °C, then 5 mL TEOS was added to flask, and stirred for 1 h. After stirring, the reaction mixture was coated on glass slides (20 mm \times 75 mm), and then these glass plate was placed in an oven at 40 °C overnight to form a film. The precipitate was scraped down and washed three times with a mixture of deionized water and ethanol (1:1) and ethanol to remove oxalic acid, and dried under reduced pressure to obtain a white solid. At last, silica films (catalyst A) were obtained by removing P123 from white solids by use of Soxhlet extraction using the 150 mL mixture of ethanol and hydrochloric acid (V_{CH2OH} : V_{HCI}:V_{HCI}=4:1) as solvent.

3.1.3 Synthesis of 1-Methyl-3-Trimeth-Oxyysilylpropyl-Imidazolium Chloride

The imidazolium-based ionic liquid was synthesized by mixing 18.6 mL of 3-chloropropyltrimethoxysilane with 8 mL of 1-methylimidazole in an inert (Ar gas) atmosphere at 120 °C for 24 h while under stirring condition [37]. The reaction mixture was then cooled to room temperature (25 °C) to obtain 1-methyl-3-trimethoxysilyl- propyl-imidazolium chloride (TMICl) ionic liquid, as illustrated in Scheme 1.

3.1.4 Preparation of 0.3TMICl@ Silica Films(B) and 0.7TMICl@ Silica Films (C)

Firstly, 1.224 g P123, 50 mL distilled water and 4.48 g oxalic acid were added to two 250 mL flasks, respectively, and stirred for 2 h at 40 °C. Then, 5 mL TEOS was added to the two flasks, respectively. At the same time, 0.30 g and 0.70 g TMIC1 were added to the two flasks respectively. After stirring for 1 h, the reaction mixture was coated on glass slides(20 mm \times 75 mm), and then these glass slides



Fig.6 Reusability of catalyst C in the benzoin reaction of benzaldehyde

was placed on in an oven at 40 °C for 12 h. Two kinds of white thin films were obtained. Subsequently, the thin films were scraped and washed three times with a mixture of water and ethanol (1:1) and ethanol to remove oxalic acid and excess ionic liquids, and then dried. Thereafter, two obtained solid powders were extracted in a Soxhlet apparatus using the 150 mL mixed solvent of ethanol and hydrochloric acid (V_{CH3OH}: V_{HCl}=4:1) as solvent to remove P123. 0.3TMICl@ silica films(B) and 0.7TMICl@silica films (C) were obtained, as showed in Scheme 2.

3.1.5 Preparation of TMICI@SBA-15 (Catalyst D)

Referring to the literature [20], the method of synthesizing TMICl@SBA-15 (catalyst D) is as follows: The synthesis of TMICl@ SBA-15 was carried out by first dispersing 1.7 g of dehydrated SBA-15 (pre-treated at 150 °C for 6 h under vacuum) in 100 mL of toluene, followed by adding 0.19 g of TMICl to the SBA-15 suspension under inert (N₂ gas) atmosphere. Subsequently, the mixture was stirred and refluxed at 80 °C for 24 h, followed by washing with toluene and water, filtered, then dried overnight to obtained the final solid product, which was denoted as TMICl@SBA-15 (catalyst D).

3.1.6 General Procedure for the Reduction Reaction

In a typical experiment, 0.331 g ([imidazolium salt] = 0.223 mmol) of catalyst C, 4 mL of CH₃OH and 0.455 mL (4.46 mmol) of benzaldehyde were added to a Schlenk tube. The reaction mixture was stirred for 10 h at 130 °C under argon. The mixture was allowed to cool down to room temperature, and the catalyst was obtained by filtration and washed with 5 mL CH₃OH. The organic layer was collected, then removed in vacuo, and the residue was purified by column chromatography on silica gel to afford the desired product. The recycled catalyst was cleaned with dry CH₃OH and dried at 40 °C twice.

3.1.7 Thermal Filtration Experiment

Thermal filtration experiment is usually an important test method to detect the heterogeneity of catalyst [38]. The heterogeneity of 0.7TMICl@ silica films(C) was studied by using the benzoin reaction of benzaldehyde as template. Firstly, the catalyst was removed by filtration after the reaction proceeded at 135 °C for 2 h, and the filtrate was further heated for 8 h under the same conditions. GC analysis showed that 31% yield was obtained after 2 h of reaction and 30% yield was obtained after 10 h of reaction. Clearly, no appreciable reaction took place after removal of the catalyst. The above experimental results distinctly showed that no active species leached into the liquid phase and provides direct evidence for the heterogeneity of the present catalyst system.

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Compliance with Ethical Standards

Conflict of interest The authors declare that they have no conflict of interest.

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