Article

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Efficient and Reusable Sn(II)-containing Imidazolium-based Ionic Liquid as a Catalyst for the Oxidation of Benzyl Alcohol

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A simple and efficient catalytic system [BBIM]Br–$SnCl_2$ for the oxidation of benzyl alcohol using hydrogen peroxide as the oxidant has been developed. Reaction conditions such as the catalyst dose, the solvents, reaction temperature, reaction time, and the amount of hydrogen peroxide were investigated. The optimum reaction conditions identified were 0.11 g of catalyst, no solvent, 65°C, 15 min, and 2 mmol of hydrogen peroxide. Oxidation of various alcohols was also investigated under the optimized conditions. The catalyst [BBIM]Br–$SnCl_2$ can be easily recovered and reused for six reaction runs without significant loss of catalytic activity, because the Sn species of the catalyst can be coordinated with the imidazole ring of the ionic liquid. The reused catalyst was further characterized by Fourier transform infrared spectroscopy to evaluate its chemical properties. The results proved that the [BBIM]Br–$SnCl_2$ catalyst was stable and reusable for the oxidation reactions. A possible mechanism for the oxidation of benzyl alcohol to benzaldehyde is proposed.

Keywords: Ionic liquid; Tin(II) chloride dihydrate; Benzyl alcohol; Solvent-free oxidation.

INTRODUCTION

The selective oxidation of alcohols into aldehydes or ketones is one of the fundamental transformations in laboratory synthesis and industrial production,[1–4] because aldehydes and ketones can serve as important chemicals for the synthesis of perfumes, pharmaceuticals, dyes, fragrances, and organic intermediates in fine chemicals industries. Therefore, investigations on the liquid-phase oxidation of benzyl alcohol to benzaldehyde, used as a versatile intermediate in synthetic chemistry, have been widely pursued.[5] It is generally known that a suitable catalyst plays an essential role in these oxidation reactions. Various chemical reagents used as the catalysts have been applied for this transformation efficiently and selectively, including inorganic oxidants,[1,6–8] supported metals,[9–15] copper(II) complexes,[16–19] bipyridine–heteropolyacid hybrid,[20] $Pd(OAc)_2$/ionic liquid,[21] heteropoly acid/ionic copolymer,[22] $Au_{25}(SPh\text{-}p NH_2)_{17}$/SBA-15,[23] CoMgAl hydrotalcites,[24] and a photocatalyst.[25] From the viewpoint of green chemistry, a promising oxidation process should take place at atmospheric pressure under solvent-free and base-free conditions. On this account, there exists still opportunity for researchers to investigate and prepare cheap, abundant, and eco-friendly heterogeneous or homogeneous catalysts with promising catalytic performance.

Ionic liquids (ILs) have attracted considerable attention since the discovery of air- and water-stable ILs by Wilkes and Zaworotko in 1992.[26] They are promising candidates for developing green chemical processes for fine chemicals syntheses, and exhibit favorable properties such as very low volatility, a wide liquid temperature range, thermal stability, good electrical conductivity, excellent electrochemical properties, ease of recyclability, and catalytic ability.[27–35] ILs possess the significant ability to solubilize organic and inorganic reagents and form the homogeneous or biphasic reaction systems.[36] Based on this point, they usually serve as reaction media in a wide range of organic reactions. From the viewpoints of practical and fundamental research, functionalization of ILs with various groups has been attempted with particular interest in catalysis. Recently, the metal-containing ILs were found to be promising catalytic materials, which have the textural porosities of ionic liquids and simultaneously the advantages of the catalytic properties of metal

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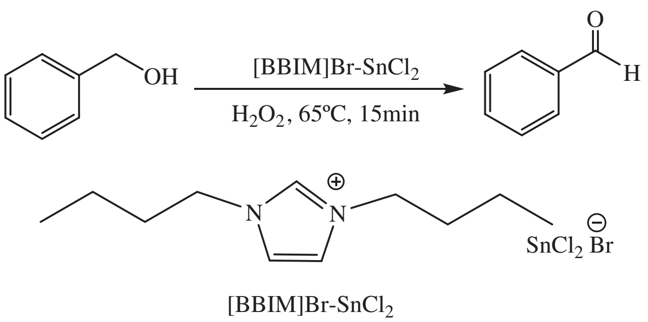
*Corresponding author. Email: xingrr@126.com salts/oxides.[37,38] Among the metal-containing ionic liquids, chlorometallate ILs are often employed as Lewis-acidic catalysts.[39,40] Very recently, the iron chloride ionic liquid/SBA-15 was applied as a catalyst for the oxidation of benzyl alcohol with hydrogen peroxide (H_2O_2).[41]

H_2O_2 is a clean and environmentally friendly oxidant because it can produce highly active oxygen species, and is widely applied in liquid-phase oxidation reactions.[42] Moreover, this oxidant is also readily available and is much cheaper and safer than most other organic and inorganic oxidants owing to production of only water during the oxidation processes.[6] Compared to other transition- and noble metal salts, tin(II) dichloride is commercially inexpensive and easily obtainable, and can be applied directly as a catalyst in various organic transformations or modified on the various carriers used as the heterogeneous catalysts.[43–48] To the best of our knowledge, little work have been reported on the tin-containing 1,3-di-*n*-butylimidazolium bromide ([BBIM]Br) IL and its application to the oxidation of benzyl alcohol to benzaldehyde. In this work, we report a simple and inexpensive protocol for the efficient oxidation of benzyl alcohol with H_2O_2 in the presence of the [BBIM]Br–$SnCl_2$ as a separable and reusable catalyst under solvent-free conditions (Scheme 1). The Sn species of the catalyst can be coordinated with the imidazole ring of the [BBIM]Br. All the preparation methods used are simple, cost-effective, and less time consuming.



Scheme 1. Catalytic oxidation of benzyl alcohol to benzaldehyde.

RESULTS AND DISCUSSION

Catalytic performance of various catalysts

We first investigated the catalytic performance of various metal-containing ILs in the oxidation of benzyl alcohol using H_2O_2 as the oxidant under solvent-free conditions. The results are summarized in Table 1. The oxidation reaction did not occur over [BBIM]Br (Table 1, entry 1), indicating that it was catalytically inactive. $SnCl_2$ was also found to be of very low activity with a conversion of 2.6% (Table 1, entry 2). The oxidation of benzyl alcohol was catalyzed by the introduction of [BBIM]Br followed by $SnCl_2$ or by the introduction of $SnCl_2$ followed by [BBIM]Br, respectively (Table 1, entries 3 and 4), and both gave low conversion of benzyl alcohol and poor selectivity of benzaldehyde. However, the [BBIM]Br–$SnCl_2$, with the advantages of both the IL and the metal salt, significantly enhanced the catalytic activity and led to complete conversion and a selectivity of 95.1% (Table 1, entry 5), indicating a clear synergic effect in comparison with a single species. The oxidation of benzyl alcohol did not effectively take place using the [BBIM]Br–$FeCl_2$ or [BBIM]Br–CuCl as the catalyst at the temperature range 0–65°C (Table 1, entries 6 and 7). At the beginning of the reaction, the rapid self-decomposition of H_2O_2 was observed. It is speculated that isolated iron(II) or copper(I) species was responsible this phenomenon. That means that the oxygen released during the H_2O_2 decomposition process played no role in the oxidation of benzyl alcohol, leading to the extremely low conversion of benzyl alcohol. The [BBIM]Br–AgCl displayed very low activity with a conversion of 2.1% (Table 1, entry 8). In this reaction, there was no obvious self-decomposition of H_2O_2. So the very low conversion meant that there was no synergic effect between [BBIM]Br–AgCl and H_2O_2. These findings implied that the metal species of the catalyst played a key role in the oxidation reactions using H_2O_2 as an oxidant under solvent-free conditions. On the basis of the above results, [BBIM]Br–$SnCl_2$ was chosen as the model catalyst for further investigation.

Optimization of oxidation conditions

The nature of the solvents can have a dramatic effect on the catalytic activity. So the effect of the solvents on the catalytic performance of [BBIM]Br–$SnCl_2$ in the oxidation of benzyl alcohol with H_2O_2 was investigated. In Table 2, the effect of no solvent, organic solvents, and

Table 1. Comparison of the catalytic activity of various metal-containing ionic liquid catalysts[1]

Entry	Catalyst	T/°C	Conv. /(%)[2]	Sel. /(%)[2]
1	[BBIM]Br	65	4.6	Trace
2	$SnCl_2$	65	2.6	Trace
3	[BBIM]Br + $SnCl_2$[3]	65	31.5	67.1
4	$SnCl_2$ + [BBIM]Br[4]	65	36.4	55.2
5	[BBIM]Br–$SnCl_2$	65	100	95.1
6	[BBIM]Br–$FeCl_2$	0–65	5	Trace
7	[BBIM]Br–CuCl	0–65	Trace	Trace
8	[BBIM]Br–AgCl	65	2	Trace

[1] Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol), 65°C, 15 min.
[2] GC analysis.
[3] By introduction of [BBIM]Br followed by $SnCl_2$.
[4] By introduction of $SnCl_2$ followed by [BBIM]Br.

water on the conversion and selectivity is summarized. The reaction system exhibited the lowest catalytic activity with a conversion of 16% in water. Organic solvents including dimethyl sulfoxide (DMSO), ethanol, acetonitrile (CH_3CN), dichloromethane (CH_2Cl_2), and dimethylformamide (DMF) used in the oxidation reaction did not show any appreciable performance in comparison with the solvent-free system, which gave a complete conversion and a selectivity of 95.1%. It is speculated that the high catalytic performance of [BBIM]Br–$SnCl_2$ under solvent-free condition is due to the significant ability to solubilize H_2O_2 and then form easily Sn peroxo complex (I) species (see the following mechanism). This is beneficial to the reaction of the substrates.

The catalyst dose can significantly affect both the conversion and selectivity in the oxidation reaction of benzyl alcohol to benzaldehyde. As Figure 1 shows, when 0.06 g of the catalyst was used for the oxidation of benzyl alcohol at the same conditions, the catalyst gave a conversion of 69% and a selectivity of 90%, respectively, which is due to the fewer catalytic sites within a lower amount of the catalyst. On increasing the catalyst dose from 0.06 to 0.11 g, the complete conversion of benzyl alcohol and the highest selectivity of 95.1% were obtained. On increasing the catalyst dose further in the range 0.11–0.6 g, complete conversion of benzyl alcohol was still obtained, but the selectivity of benzaldehyde decreased slightly. This indicates that an excess amount of the catalyst led to further oxidation of benzaldehyde to benzoic acid.[49] The results led to the optimum catalyst dose as 0.11 g.

Table 2. Effects of various solvents on the oxidation of benzyl alcohol[1]

Entry	Catalyst	Solvent	Conv. /(%)[2]	Sel. /(%)[2]
1	[BBIM]Br–$SnCl_2$	–	100	95.1
2		H_2O	16	84
3		CH_2Cl_2	46	81
4		Ethanol	34	50
5		DMF	18.8	80
6		DMSO	67.5	85
7		CH_3CN	38	62

[1] Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol), 65°C, 15 min, solvent (1 mL).
[2] GC analysis.

It is well known that the reaction temperature influences greatly the reaction rate and the catalytic properties of the catalysts. Therefore, the effect of the reaction temperature ranging from 45 to 75°C was investigated for the oxidation benzyl alcohol to benzaldehyde with H_2O_2. The results are shown clearly in Figure 2. On increasing the reaction temperature from 45 to 65°C, the conversion of benzyl alcohol increased significantly from 20.5 to 100%, and the selectivity of benzaldehyde increased from 84.7 to 95.1%. However, the selectivity of benzaldehyde gradually reduced to be 87.1% at complete conversion when the reaction temperature was increased from 65 to 75°C. The result shows that a higher temperature is disadvantageous to a higher selectivity of benzaldehyde, which could be further oxidized to benzoic acid.

The amount of H_2O_2 applied in the reaction is a vital factor influencing the progress of benzyl alcohol

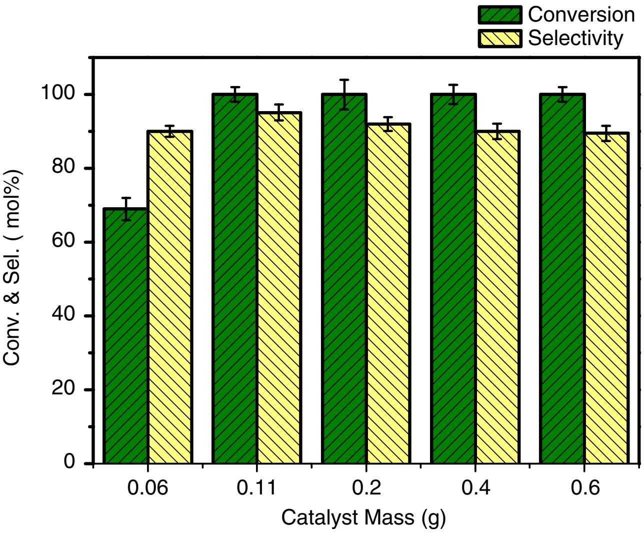


Fig. 1. Effect of the catalyst dose on the oxidation reaction of benzyl alcohol to benzaldehyde. Reaction conditions: benzyl alcohol (1.5 mmol), H_2O_2 (30%, 2 mmol), 65°C.

oxidation. As is shown in Figure 3, the influence of the amount of H_2O_2 was investigated by varying the amount of H_2O_2 from 1 to 6 mmol. As expected, as the amount of H_2O_2 was increased from 1 to 2 mmol, the conversion of benzyl alcohol increased from 48.5 to 100%, and the selectivity of benzaldehyde increased from 89.3 to 95.1%. With further increase of the amount of H_2O_2 from 2 to 6 mmol, the conversion of benzyl alcohol was unchanged, but the selectivity of benzaldehyde decreased slightly. This implied that a small amount of benzaldehyde may be further oxidized to benzoic acid by the excess amount of H_2O_2 and catalyst. The above results suggested that 2 mmol of H_2O_2 was optimal for the oxidation reaction.

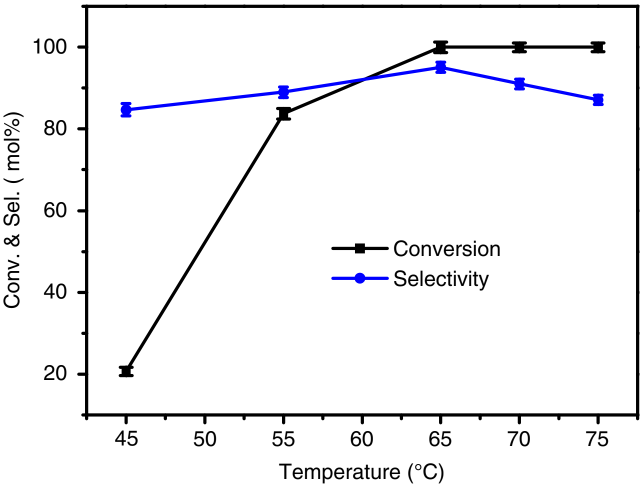


Fig. 2. Effect of the temperature on the oxidation reaction of benzyl alcohol to benzaldehyde. Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol).

The effect of reaction time was also investigated. As seen in Figure 4, benzyl alcohol conversion increased from 75 to 100%, and the selectivity of benzaldehyde increased from 86 to 95.1% as the reaction time was increased from 10 to 15 min. However, when the reaction time was further increased from 15 to 30, 45, and 60 min, respectively, a slight decrease of the selectivity of benzaldehyde was observed. The results showed that the optimum reaction time was 15 min.

Study of various alcohols

To investigate the generality of the catalytic activity of [BBIM]Br–$SnCl_2$, it was further applied to the oxidation reactions of a range of benzyl alcohols and their derivatives under the optimized conditions. As seen from Table 3, benzyl alcohols with the electron-donating or -withdrawing substituents were converted to the corresponding aldehydes with high conversion and selectivity by prolonging the reaction time or increasing the reaction temperature, indicating that the catalyst [BBIM]Br–$SnCl_2$ shows good catalytic activity for substituted aromatic alcohols. It is worth noting that the inactive benzhydrol would be also be suitable to the system and gave excellent conversion (>99%) and selectivity (99%), suggesting that the steric hindrance of the substituent groups has no effect on the catalytic activity. Unfortunately, the inactive phenethyl alcohol and cyclohexanol showed a low reaction activity and gave lower selectivity even when the reaction time was increased.

Reusability of the catalyst

From the viewpoint of practical application, recyclability of the catalyst is very important. Recycling experiments of the oxidation reaction of benzyl alcohol with H_2O_2 under solvent-free conditions were performed. As shown in Figure 5, even after the sixth cycle of the oxidation reaction, [BBIM]Br–$SnCl_2$ showed similar catalytic activity and gave 100% conversion and 93.5% selectivity. These results confirmed that [BBIM] Br–$SnCl_2$ was a robust and reusable material and can

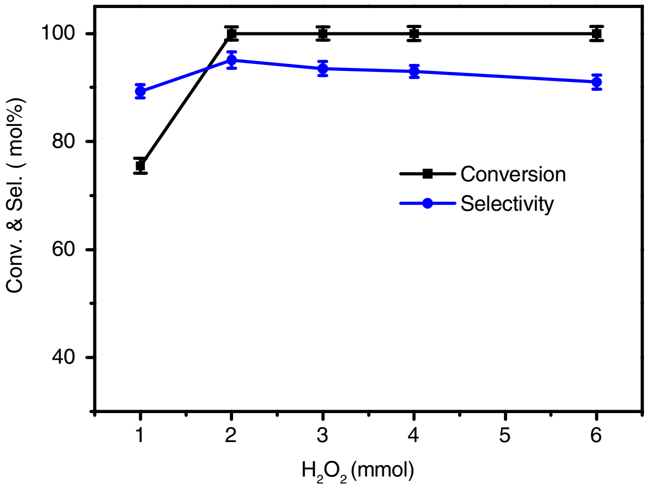


Fig. 3. Effect of the amount of H_2O_2 on the oxidation reaction of benzyl alcohol to benzaldehyde. Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), 65°C.

maintain excellent conversion and selectivity even after the six cycles of the oxidation reactions.

The reused catalyst was further characterized by Fourier transform infrared (FT-IR) spectroscopy to evaluate its chemical properties. Figure 6 shows the FT-IR spectra of fresh and used catalysts. The FT-IR spectrum of the reused catalyst was completely consistent with that of fresh one. The band appearing at 3430 cm^{-1} in the spectra of fresh and used samples is assigned to the O—H stretching vibration of the adsorbed water. The absorption bands at around 3137, 2931, and 2874 cm^{-1} are associated with the stretching vibration of the C—H bonds. The bands at 3080, 2966, 1331, and 1168 cm^{-1} are assigned to the stretching vibration of the C—N bonds. The bands at 1632 and 1563 cm^{-1} are attributed to the C=N stretching. The bands at 1464 and 1378 cm^{-1} correspond to CH_2 and CH_3 bending vibration, respectively. The bands ranging from 883 to 1111 cm^{-1} are attributed to C—H bending vibration. The results show that no structural transformation or change in chemical performance had taken place in the catalyst after the oxidation reactions. The results further proved that [BBIM]Br–$SnCl_2$ was a stable and reusable material for oxidation reactions.

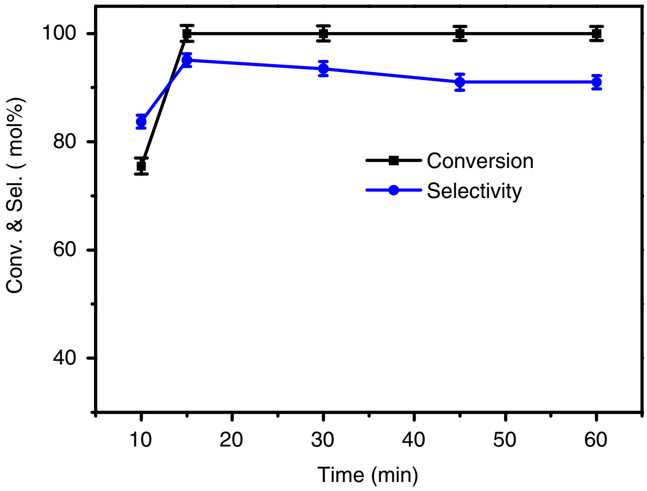


Fig. 4. Effect of the reaction time on the oxidation reaction of benzyl alcohol to benzaldehyde. Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol), 65°C.

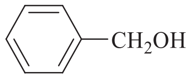
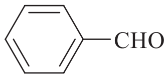
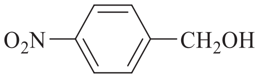
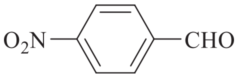
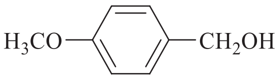
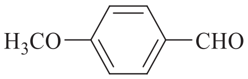
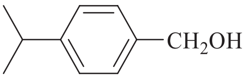
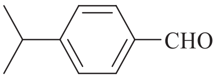
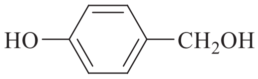
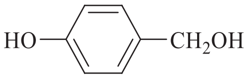
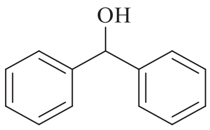
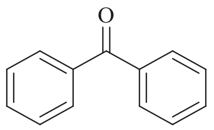
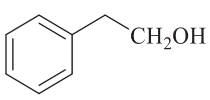
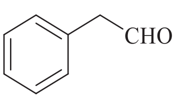
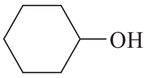
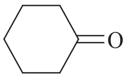
Possible mechanism of the oxidation reaction

Based on the above experimental results and previous literature,[48,50] a possible mechanism for the oxidation of benzyl alcohol to benzaldehyde is proposed (Scheme 2). In the oxidation reaction, the Sn species of the catalyst coordinate with the imidazole ring of the ionic liquid and react with the oxidant H_2O_2 via coordinating to oxygen to form Sn peroxo complex (I) species, which then react with the benzyl alcohol to form the transition state (II). The transition state (II) further removes a molecule of H_2O to form the transition state (III), which would be transformed into the desired product of benzaldehyde, and complete the catalytic cycle, simultaneously. During the oxidation reaction, [BBIM]Br–$SnCl_2$ transferred successfully the H_2O_2 from an aqueous solution into an organic reaction phase. It helped proceeding with the oxidation reaction. The [BBIM]Br–$SnCl_2$ catalyst showed high catalytic activity and stability due to the Sn species coordinating to the imidazole ring of [BBIM]Br, which prevented it from leaching from the catalyst.

CONCLUSION

In summary, a simple, durable, economical, recyclable, and highly efficient catalytic oxidation system with [BBIM]Br–$SnCl_2$ and H_2O_2 has been developed, which could effectively oxidize benzyl alcohol and its derivatives to the corresponding aldehydes under mild reaction conditions, such as atmospheric pressure, as

Table 3. Catalytic properties of $[BBIM]Br–SnCl_2$ in the oxidation of various alcohols with H_2O_2[1]

Entry	Alcohols	Products	Time/min	Conv. /(%)[2]	Sel. /(%)[2]
1			15	100	95.1
2			60	71	99
3			15[3]	99	90
4			160	87	91
5			15	99	50
6			15	99	98
7			15	99	>99
8			60[4]	20	40
9			60[4]	32	30

[1] Reaction conditions: alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol), 65°C.
[2] GC analysis.
[3] The reaction was carried out using H_2O_2 (30%, 3.5 mmol).
[4] The reaction was carried out at 85°C.

well as solvent-free and base-free conditions. Moreover, the catalytic system may offer many advantages, such as simplicity of operation, high yields, stability, excellent recyclability, and easy isolation of products.

EXPERIMENTAL

All chemicals used in the experiments were purchased from commercial sources and used without further purification. All reagents were of analytical grade. ^1H NMR spectra were recorded on a Bruker 400 MHz spectrometer using DMSO-d_6 as the solvent with tetramethylsilane (TMS) as the internal standard. FT-IR spectra were recorded on a Bruker VERTEX 80 FT-IR spectrometer using the KBr pellets. All products were identified using gas chromatography-mass spectrometry (GC-MS) using an Agilent 7890B-7000C instrument with an HP-5-MS capillary column. Their amounts were quantified on an Agilent 7890B GC equipped with a DB-1701 (30 m × 0.32 mm × 0.25 μm) capillary column and a flame ionization detector (FID), using dodecane as the internal standard to calculate the conversion and selectivity of the reaction. Elemental analysis was performed on a Perkin-Elmer Series-II 2400 CHNS/O analyzer, inductively coupled plasma (ICP) analysis on a Perkin-Elmer Optima 8000DV, and ion chromatography on a Thermo-Fisher ICS-5000$^+$, respectively.

Synthesis of $[BBIM]Br–SnCl_2$

First, the synthesis of [BBIM]Br was carried out by modifying the procedure reported previously.[51,52]

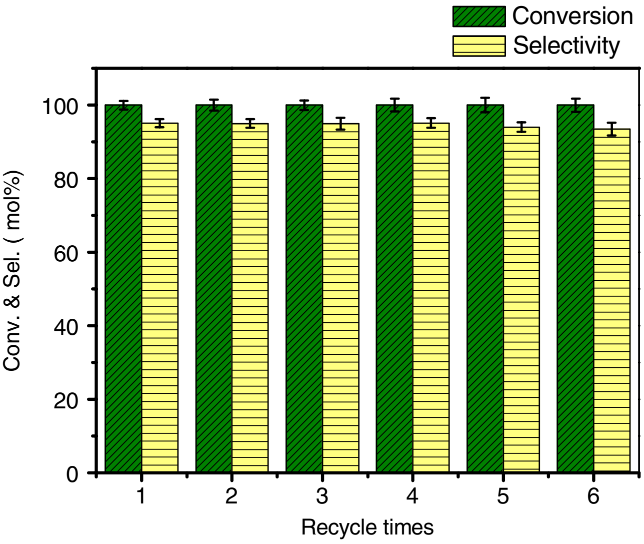


Fig. 5. Reusability of the catalyst. Reaction conditions: benzyl alcohol (1.5 mmol), catalyst (0.11 g), H_2O_2 (30%, 2 mmol), 15 min, 65°C.

Fifty mmol of 1-bromobutane was dissolved in 30 mL of toluene, and 50 mmol of 1-butylimidazole was slowly added to the above mixture solution, which was stirred and refluxed at 80°C for 24 h. The resulting viscous liquid was cooled to room temperature, and then washed three times with ethyl acetate (50 mL). After the removal of the solvent by heating of the solution under vacuum at 80°C, the product [BBIM]Br was obtained. The tin-containing ionic liquid was synthesized by reaction with a certain amount of the [BBIM]-Br and $SnCl_2$ in toluene at 95°C for 24 h. The obtained tin-containing ionic liquid, denoted as [BBIM]Br–$SnCl_2$, was dried in vacuum at 80°C for 24 h. The molar ratios of [BBIM], Br, Cl, and Sn of [BBIM]Br–$SnCl_2$ were 1:1:1:0.5. ^{1}H NMR: δ 0.90 (t, J = 7.2 Hz, 6H, CH_3), 1.25 (sept, J = 7.2 Hz, 4H, $CH_3CH_2(CH_2)_2N$), 1.79 (pent, J = 7.2 Hz, 4H, NCH_2CH_2), 4.19 (t, J = 7.2 Hz, 4H, NCH_2), 7.84 (s, 2H, NCHCHN), 9.32 (s, 1H, NCHN).

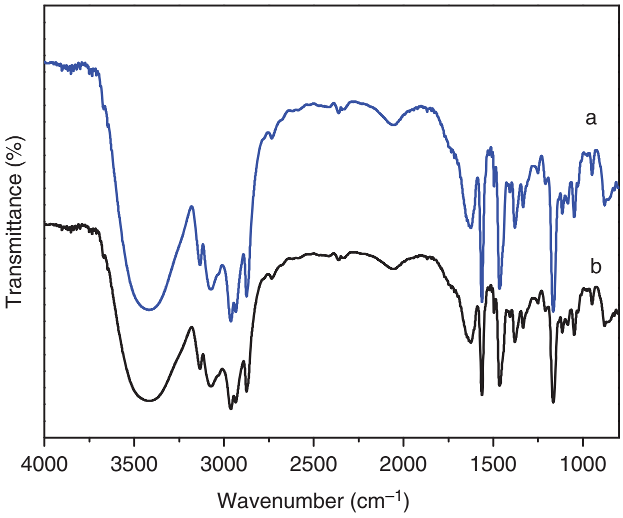
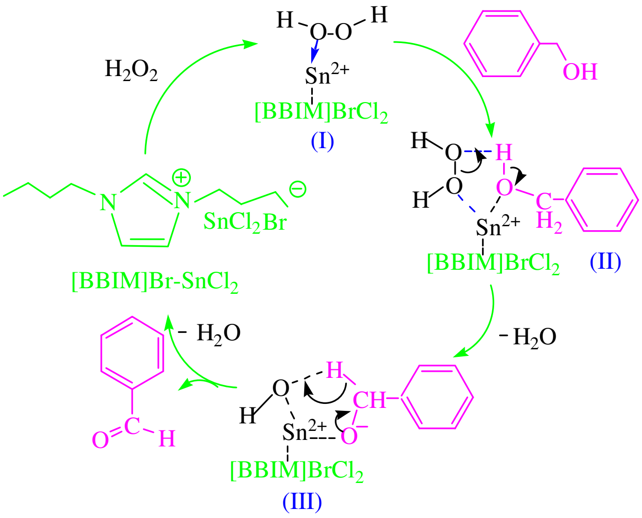


Fig. 6. FT-IR spectra of (a) fresh and (b) reused catalysts.



Scheme 2. Possible mechanism for the oxidation of benzyl alcohol to benzaldehyde.

General procedure for the oxidation reactions

The liquid-phase oxidation of alcohol reactions was performed in a glass flask with a reflux condenser and a magnetic stirrer. In a typical experiment, 1.5 mmol of benzyl alcohol and 0.11 g of [BBIM]Br–$SnCl_2$ were stirred at 65°C, and then 2 mmol of the aqueous solution of H_2O_2 (30%) was slowly added dropwise into the reaction system. After the reaction at 65°C for 15 min, the products were extracted with ether or ethyl acetate (3 × 10 mL), and then identified using GC-MS. The remaining [BBIM]Br–$SnCl_2$ catalyst was recovered by the decantation of water produced in the reaction and concentrating under vacuum. The next run was carried out under optimized reaction conditions.

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Supporting information

Supporting information for this article is available on the www under 10.1002/jccs.201600286.

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