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Selective alcohol oxidation catalysed BY $FeCl_3$ /novel glycine functionalised IONIC liquid

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ABSTRACT

An effective and eco-friendly technique were designated for quick alcohol oxidation by glycine functionalised imidazolium ionic liquids in presence of $FeCl_3$ at ambient-temperature. No over the primary alcohols oxidation to carbonyl compounds was observed in presence of this $FeCl_3$/[Gmim]Cl. These benefits of the catalyst resulted mainly from the circumstance with alcohols-H_2O_2, and the Fe^{3+} was coordinated by the immobilized IL to permitted both reactants to access the active sites of the catalyst effectively. The catalyst recycled nine times without loss of activity.

1. Introduction

Conversion Alcohol into carbonyl compounds is one of the vibrant functional group transformations in organic synthesis [1–4]. However, those compounds found a significant group of molecules in synthetic chemistry and also essential constituent of dyes, fragrances, pharmaceuticals, industrially important chemicals, and natural products [5–8]. There are several diverse schemes that accomplish this important functional group conversion [9–11].

Moreover, etiquettes based on O_2, air or H_2O_2 [12–14] are mainly attractive for cheap and eagerly existing oxidants. Accordingly, numerous studies have been reported for the alcohol oxidation with O_2 or H_2O_2 catalysed by metal complexes such as Ruthenium [15–17], Palladium [18–20], Cobalt [21–24], Osmium [25,26], Copper [27,28] and heterogeneous catalysts like metal catalysts besides supported catalysts, with mesoporous materials, zeolite, polymer-supported catalysts [29–31], tungsten systems like tungstic acid [32–35], quaternary ammoniumtetrakis (diperoxotungsto) phosphates [32–35] sodium tungstate [$(n\text{-}C_4H_9)_4N$] Cl, sodium tungstate and quaternary ammonium hydrogen sulfate [32–35].

However, in spite of this exhaustive research method, alcohol oxidation is still far being ideal from the green chemistry point of view and needs to improvement [36–38]. There is only a limited report of using cheap and less toxic iron catalysts for the alcohol oxidation to carbonyls so far [39–42]. Nowadays, Schiff-iron has established a substantial amount of care in the topical literature as a powerful transition metal catalysed reaction. Due to its low cost, ready abundance, and low toxicity, iron is an ideal metal catalyst for large scale synthesis. This growth of iron-catalysed methods, however, is still in its beginning. The current efforts to employ iron as a catalyst for Kumada couplings, hydrogenative reductions, friedel-crafts reactions and dihydroxylations provided a direct evaluation among state-of-the-art transition metal-catalysed reactions, environmentally friendly and sustainable metal [1–4,39–42,56].

This recent years, Caselli et al. [43] initiated non-heme complexes suitable for the selective oxidation of primary and secondary alcohols to the corresponding ketones. However, Iskra and Mozina [44] efficiently accomplished selective oxidation of secondary catalysed by iron (III) chloride. Sato and co-worker [45] testified an efficient catalytic oxidation of alcohols with hydrogen peroxide using mixed picolinate and quinaldinate iron (III) complexes. Liang et al. [46] developed a simple chemical model for aerobic oxidation of primary alcohols to the aldehydes catalysed by iron chloride/4-acetamido-TEMPO/sodium nitrite. Biswas et al. [47] studied primary and secondary alcohols to carbonyl compounds in the presence dinuclear iron complexes as catalysts using hydrogen peroxide as primary oxidant.

Over all the outlines afford good yield, but some have problematics issue such as extensive work-up method, severe reaction conditions (organic co-solvents) and require absolutely dry and inert media. To our best info, there is no report of with the $FeCl_3$/glycine functionalised ionic

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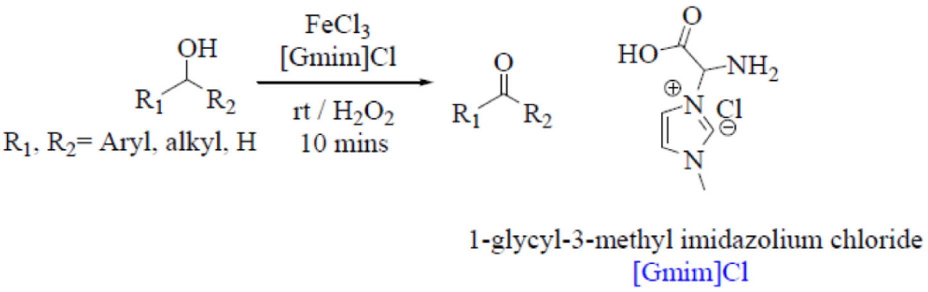


Scheme 1. Oxidation of alcohols catalysed by $FeCl_3$/[Gmim]Cl.

Table 1
Optimization of oxidation reaction.[a].

Entry	Different Solvent	Time (hrs)	Conv. (%)[b]	Yield (%)[c]
1	Blank[d]	24	3	2
2	Toluene	24	31	28
3	DCM	24	26	24
4	EtOAc	24	18	17
5	DME	24	22	20
6	THF	24	28	27
7	MeCN	24	34	33
8	$CHCl_3$	24	40	37
9	DMF	24	20	19
10	[Gmim]Cl	10[e]	96	94
11	[Gmim]Cl	10[e,f]	97	95
12	[Gmim]Cl	10[e,g]	96	94
13	[Gmim]Cl	15[e,h]	96	94
14	[Gmim]Cl	10[e,i]	96	94
15	[Gmim]Cl	10[e,j]	95	92

[a] Reaction condition: Benzyl alcohol (1 mmol), Hydrogen perxide (1.2 mmol), $FeCl_3$ (0.25 mol%), [Gmim]Cl (1 mL) at $25^{O}C$ for 10 min.
[b] Based on GC.
[c] Isolated yield by flash chromatography.
[d] Absence of catalyst.
[e] Time in mints.
[f] Increases of conc. of $FeCl_3$ from 0.25 to 0.5 mol%.
[g] Increases of conc. of H_2O_2 as the oxidant from 10% to 30%.
[h] Time to increase from 10 to 15 mints.
[i] Temperature (30 °C).
[j] Temperature (20 °C).

liquids, as catalyst for oxidation of alcohols. This prompted us to study the possibility of the oxidation of alcohols to carbonyl compounds in the presence of $FeCl_3$/[Gmim]Cl under mild conditions using H_2O_2 as oxidant (Scheme 1).

2. Experimental

2.1. Materials and methods

All solvents and chemicals were commercially available and used without further purification unless otherwise stated. The ^1H NMR spectra were recorded on a Bruker 500 MHz using $CDCl_3$ as the solvent and mass spectra were recorded on JEOL GC MATE II HRMS (EI) spectrometer. FT-IR was recorded on AVATRA 330 spectrometer with DTGS detector. Column chromatography was performed on silica gel (200–300 mesh). Analytical thin-layer chromatography (TLC) was carried out on pre-coated silica gel GF-254 plates.

2.2. Synthesis of [Gmim]Cl

The catalyst [Gmim]Cl was synthesized and reported [50,51]. First, chloroglycine (0.01 mol) reacted with N-Methylimidazole (0.11 mol) in 50 mL acetonitrile at $70^{O}C$ for 24 h to generate chloroglycine ligand modified by imidazole salt (3-(amino(carboxy)methyl)-1-methyl-1H-imidazole-3-ium chloride) [Gmim]Cl. The solvent (acetonitrile) was removed under reduced pressure at 80 ^{O}C. Then the residue was mixed with 50 mL water and extracted with ethyl acetate (3 × 5 mL). Further, the water phase was evaporated under reduced pressure at 80 ^{O}C until the mass of the residue did not change.

2.3. Typical oxidation procedure

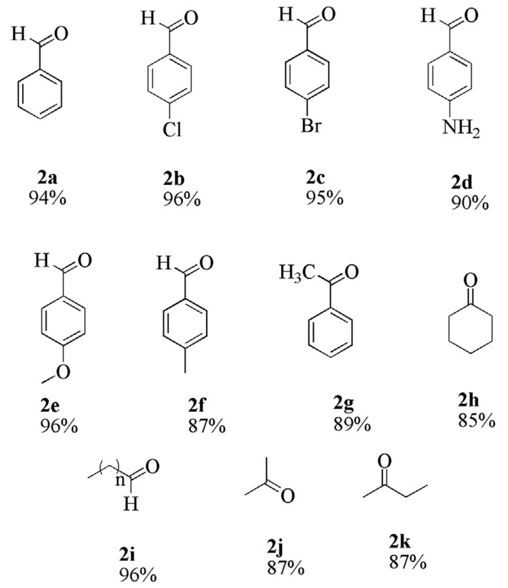
To a mixture of $FeCl_3$ (0.25 mol%), [Gmim]Cl (1 mL), benzyl alcohol (10 mmol) and H_2O_2 (1.2 mmol) was slowly added. The resulting reaction mixture was stirred at room (25 °C) temperature for 10 min. After completion of the reaction, ether was added (3 × 5 mL) to separate the product from catalyst. The organic layer was concentrated and purified by column chromatography to give the benzaldehyde.

3. Results and discussion

To get information on the optimal catalyst conditions, we carried out intensive examinations to define the suitable solvent for this benzyl alcohol oxidation reaction. According to publications from Rani and Bhat [52], Hergovich and Speier [53] and Renhua Liu [54] polar, non-polar solvents tend to give the best results for the oxidation reaction, while Ma [55] obtained high-activity of catalysts in DCE solvent. Among the previous reports, alcohol oxidation in absence polar and non-polar solvents was the most productive (Table 1, entries 2–9). This may be due to the easy coordination of $FeCl_3$ with organic co-solvents. Our goal was to employ $FeCl_3$ under ligand, organic solvent-free conditions to effect the alcohol oxidation reaction on a recyclable basis. Amino acid functionalised ionic liquids were chosen because of their recyclability and reusability. The products can be easily separated from the $FeCl_3$ catalysts dissolved in ionic liquids by simple extraction with normal organic solvents. We reasoned that if the ligand has an imidazole moiety, it will have high solubility in ionic liquids having the imidazole skeleton.

Subsequent, the combination of glycine functionalised ionic liquids with $FeCl_3$ was tried as the catalyst for the oxidation reaction. As shown in (Table 1, entry 10) the catalytic oxidation continued with mild conversion of alcohol to aldehyde with satisfactory yield. Furthermore, according to literature, Renhua Liu and co-workers obtained good yield in the oxidation reaction of primary alcohols to the aldehydes using iron chloride (0.25 mmol) stirred at $50^{O}C$ and oxygen pressure of 0.4 MPa for 03–23 h [54]. Using $Fe(NO_3)_3{\cdot}9H_2O$ in the 10 mol%, Ma et al. observed acceptable rate in the oxidation [55]. Among the previous reports, increasing the quantity of the catalyst can improve the reaction yield and shorten reaction time. However, the influences of the amount of $FeCl_3$ and H_2O_2 were also examined. It is renowned that increasing increase from 0.25 to 0.5 mol% the quantity of $FeCl_3$ led to improved yield (Table 1, entry 11). However, the quantity of H_2O_2 increased from 10% to 30%, the reaction was not influenced to greater extent (Table 1, entry 12). It is found that the alcohol oxidation was very fast in 10 min and nearly approached the chemical equilibrium subsequently 15 min (Table 1, entry 13). However, temperature also plays a significant role in the model reaction. When we conducted the oxidation reaction at $30^{O}C$,

Table 2
Catalytic oxidation of primary alcohols Oxidation in the presence of $FeCl_3$/[Gmim]Cl[a].



[a] Reaction condition: Benzyl alcohol (1 mmol), H_2O_2 (1.2 mmol), $FeCl_3$ (0.25 mol%), [Gmim]Cl (1 mL) at 25°C for 10 min. [b] Conversion by GC. [c] Isolated yield by flash chromatography. [n] Number of carbons-1-5.

Table 3
Recycling of $FeCl_3$/[Gmim]Cl.[a].

Run	Conversion (%)[b]	Yield (%)[c]
1	96	94
2	96	94
3	96	93
4	96	93
5	95	93
6	95	93
7	94	92
8	94	92
9	94	91

[a] Reaction condition: benzyl alcohol (10 mmol), H_2O_2 (12 mmol), $FeCl_3$ (0.25 mol%), [Gmim]Cl (1 mL) at room temperature for 10 min.
[b] Conversion by GC.
[c] Isolated yield by flash chromatography.

there was no change in the yield (Table 1, entry 14) but in the case of 20°C decrease in the carbonyl compound (Table 1, entry 15).

Under the optimized conditions, the other alcohols oxidation was studied (Table 2). The primary aromatic alcohols explored and provided first-rate yields (Table 2, entries a-g). However, parallelly the oxidation of benzyl alcohol gave good conversion and yield (85–96%) were found over the alcohols attached electron-withdrawing and donating groups in the aromatic ring, i.e., *p*-chloro, p-bromo, *p*-methoxyl, p-amino and p-methyl benzyl alcohols (Table 2, entries b-h).

Interestingly, in contrast to B. Andrioletti, work [48] aliphatic alcohols were also oxidized to carbonyl compounds, with outstanding yield in short duration (Table 2, entries i-k). Thus, we could achieve oxidation of various aliphatic, alicyclic and aromatic alcohols to corresponding carbonyls without over oxidation under solvent free condition, in contrast to Shannon S. Stahl co-works [49] which showed organic solvents were necessary during the oxidation. The system gave well to quantitative yields on oxidation of alcohol due to the effect of cation that facilitated for the formation of catalytic complex with the Fe^{3+} which resulted into a very high activity of the system [39–42].

One of the main objectives of green chemistry is to increase the life of the resulting catalyst. We have completed an introductory study of the recycling productivity of $FeCl_3$/[Gmim]Cl using benzyl alcohol as model substrate. The $FeCl_3$/[Gmim]Cl was disconnected from the reaction after each trial by extraction with diethyl ether followed by ethyl acetate to recover [Gmim]Cl. The excess solvent was removed by vacuum distillation sensibly before using it in the next cycle. The catalyst was recycled nine times without conspicuous loss and activity (Table 3, entries 1–9).

4. Conclusions

In summary, we have established the effective examples of $FeCl_3$/[Gmim]Cl procedure that have catalysed oxidation of various alcohols to the ketones and aldehydes in outstanding yields. The activity of several alcohol oxidation in the currently testified catalytic systems is much higher than that of other aerobic oxidations in ILs/ILs-transition metals, even with lesser catalyst loading and devoid of any additive. Additionally, Easy technique, broad substrate applicability, high yields, short reaction times can be mentioned as advantages of this method. No solvent vapors released and smallest wastage of reagents during the reaction makes this as ‘green’ process.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jics.2021.100099.

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