Journal of Organometallic Chemistry 940 (2021) 121787

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Self-assembled ionic liquid based organosilica-titania: A novel and efficient catalyst for green epoxidation of alkenes

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ARTICLE INFO

Article history:
Received 6 December 2020
Revised 5 March 2021
Accepted 15 March 2021
Available online 20 March 2021

Keywords:
Ionic liquid based organosilica-titania
Supported titanium catalyst
Alkene epoxidation
Recoverable catalyst

ABSTRACT

A novel titanium-containing self-assembled ionic liquid based hybrid organic-inorganic organosilica (Ti-ILOS) was prepared, characterized and applied as highly effective catalyst for the green epoxidation of alkenes in the presence of hydrogen peroxide as oxidant. The Ti-ILOS catalyst was characterized using FT-IR, SEM, XPS and EDX analyses. This catalyst was recovered and reused several times without significant loss of performance. The leaching test was also performed to investigate the stability and nature of designed catalyst under applied conditions.

1. Introduction

Since epoxides are key intermediates in the organic chemistry and drug synthesis for production of fine chemicals, the epoxidation of alkenes is a significant and attractive process between chemists [1,2]. This process has been performed using many homogeneous and heterogeneous catalytic systems in the presence of several oxidants such as hydrogen peroxide, in which the use of heterogeneous catalysts is more interested in green chemistry due to their easy recoverability, reusability and stability [3-7]. One of effective and well-known catalysts is titanium based ones that considered to be an environmentally friendly, excellent and inexpensive heterogeneous catalyst with good properties under most conditions [8-11]. An effort to enhance these properties, expand applications and grow catalytic activity of Ti-based catalysts, is preparation of titanium containing composites [12-14]. One of the most important composites is silica-titania materials that can be prepared *via* both simultaneous hydrolysis and condensation of titanium and silicon precursors and also chemical attachment of tetraaloxy titaniums onto silica materials [15-22]. Titanium species have also been well incorporated in several mesoporous silicas such as MCM-41, MCM-48 and SBAs *via* the grafting of titanium precursors on the pore surface of these nanomaterials [23-27]. The Ti-containing materials have been applied as effective catalyst in oxidation of several organic compounds. Some of recently developed systems are Co-SiO_2@Ti-Si for the aerobic oxidation of diphenyl sulfide, [15] Ti−Fe_3O_4@MCM-41 for selective epoxidation of olefins, [28] TMS-TS4 for cyclohexene oxidation reaction, [18] Pd/SiO_2@Ti-MS for one-pot oxidation of methyl phenyl sulfide and diphenyl sulfide, [29] Ti-SBA-15 for selective oxidation of aromatic sulfur compounds, [30] Au/TS-1@meso-SiO_2 for selective epoxidation of propylene, [31] PdAu@Ti-MHSS for one-pot oxidation of methyl phenyl sulfide [32] and ILNOS-Ti for green oxidation of alcohols [33].

Ionic liquids with unique properties such as low melting point, high thermal and chemical stability, non-flammability, high electrical conductivity, low vapor pressure and excellent solubility have found many applications in the synthesis of organic compounds as catalysts and solvents. Especially, the supported ionic liquids simultaneously have the benefits of ionic liquids and recoverable heterogeneous solids. Ionic liquids are often stabilized on solid surfaces through both chemical and physical methods. Among these, organosilica materials containing ionic liquids have wide applications in adsorbents, chromatography, catalyst, electrochemistry, solid phase extraction, sensor technology and gas storage due to their unique properties such as high lipophilicity, thermal and mechanical stability. In particular, the incorporation/immobilization of metals such as Cu, Pd, Au, Ni, Fe, Mn, and Ti in the framework of ionic liquids led to the development of their applications in the catalytic field [26,33-42].

Although significant studies have been reported on the use of Ti-containing silica catalysts in oxidation processes, however, most of these processes have been performed in the presence of toxic organic solvents. In view of the above and in continuation of our recent studies in the development of heterogeneous catalysts containing ionic liquid, [26,33,35,43-45] herein, we have reported the

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https://doi.org/10.1016/j.jorganchem.2021.121787
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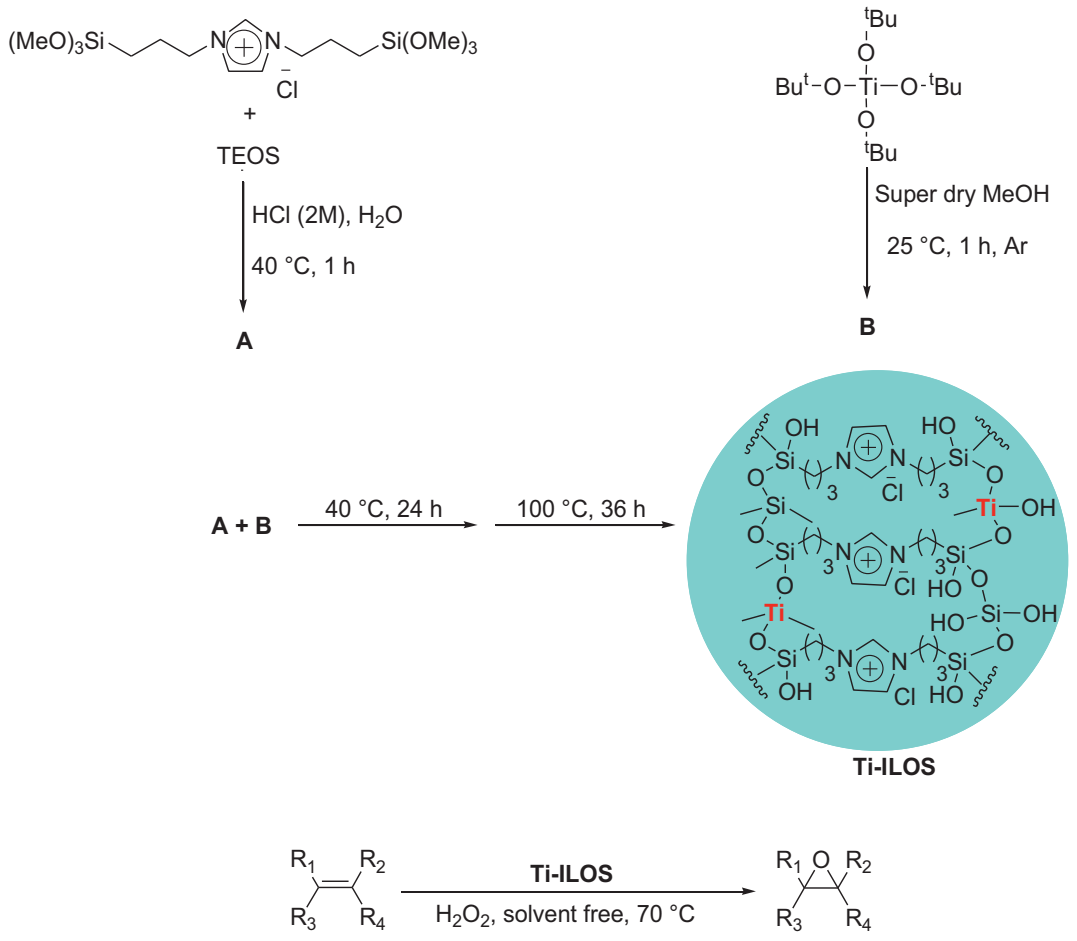


Scheme 1. Synthesis of Ti-ILOS and its catalytic application in the epoxidation of alkenes

synthesis of an organic-inorganic hybrid xerogel material in which ionic liquids and titanium are dispersed very well in the silica framework (Ti-ILOS, Scheme 1). In fact, our motivation in this study is the design and preparation of a novel catalyst for green oxidation of alcohols involving the advantages of both supported ionic liquids and chemically immobilized catalysts. The Ti-ILOS was characterized using FT-IR, EDX, SEM and XPS techniques.

2. Experimental section

2.1. Procedure for the preparation of Ti-ILOS

The titanium-containing self-assembled ionic liquid based hybrid organic-inorganic organosilica (Ti-ILOS) was prepared through simultaneous hydrolysis and condensation of propyl-imidazolium ionic liquid, TBOT and TEOS by sol-gel method. The molar ratios of the precursors were as follows: 6Si:1IL:3Ti. The sols were prepared following this procedure: The IL (2 mmol) and TEOS (6 mmol) were hydrolyzed for 1 h at 40°C in HCl (2 M, 30 mL) and H_2O (20 mL) to give a mixture called A. The TBOT (3 mmol) was also dissolved in super dry methanol while stirring for 1 h at 25°C under Argon atmosphere to give a mixture called B. The A was added to B and this mixture was firstly stirred for 24 h at 40°C and then statically heated at 100°C for 36 h. The obtained solid material was filtered and washed completely with deionized water and ethanol. The final sample was dried at 70°C for 12 h and designated as Ti-ILOS.

2.2. General procedure for the epoxidation of alkenes using Ti-ILOS catalyst

For this purpose, the Ti-ILOS catalyst (0.014 g) and alkene (1 mmol) were added into a reaction vessel. Then, H_2O_2 (3 mL) was added slowly and drop by drop during the reaction time. The resulted mixture was stirred at 70°C and the reaction progress was monitored by studying oxidized products at different times. The oxidized products were analyzed by using a gas chromatograph (Agilent 6890N) equipped with SE-30 capillary column and a FID detector. To ensure the reproducibility of the results, the experiments were repeated under identical reaction conditions.

2.3. Procedure for the recovery of the Ti-ILOS catalyst in the epoxidation of alkenes

For this, the epoxidation of styrene was selected as a test model. The styrene (1 mmol) and catalyst (0.014 g) were added in a reaction vessel and H_2O_2 (3 mL) was added slowly and drop by drop during the reaction time. Then, this mixture was magnetically stirred at 70°C under solvent free conditions and the reaction progress was monitored using TLC and GC. After completion of the reaction, the catalyst was collected and separated using centrifuge, washed with EtOH and dried under vacuum. The recovered catalyst was reused in the next run under the same conditions as the first run. These steps were repeated several times and the results showed that the catalyst could be recovered and reused at least five times without significant decrease in efficiency.

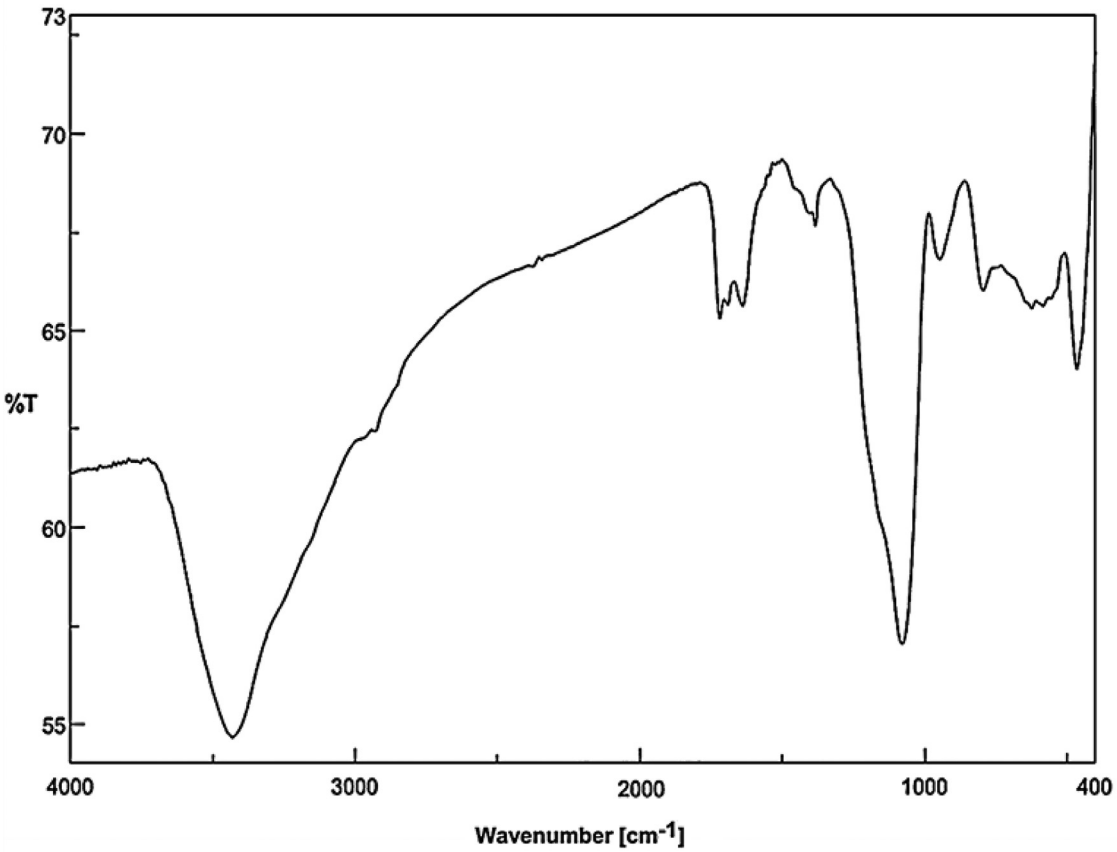


Fig. 1. FT-IR spectrum of the Ti-ILOS catalyst

2.4. Procedure for hot filtration test

This test was also performed on the epoxidation of styrene under optimized conditions. For this, after about 45% of the reaction was completed, it was stopped and the catalyst was separated using centrifuge. The catalyst-free residue was allowed to continue under optimum conditions as above. After about 10 h, no noticeable conversion was observed confirming no leaching of active titanium centers under applied media.

3. Results and discussion

3.1. Characterization of Ti-ILOS

The FT-IR spectroscopy (Fig. 1) of the Ti-ILOS showed the C-Si stretching vibrations at 700-800 cm^{-1}. This analysis also showed absorption bands at 946.8 cm^{-1} (for Si-O-Ti stretching), 1079, 1185 and 962 cm^{-1} (for Si-O-Si stretching), 1384 cm^{-1} (for C=C stretching) 1634 cm^{-1} (for C=N stretching), 2900-3000 cm^{-1} (for aliphatic C-H stretching), 3100 cm^{-1} (for unsaturated C-H stretching) and 3300 cm^{-1} (for O-H stretching).

The EDX analysis also showed the signals of Si, C, Cl, Ti, N and O elements for the material (Fig. 2). The results of FT-IR and EDX analyses confirmed the successful incorporation/immobilization of ionic liquid, silica and titania moieties into/onto the material framework.

The SEM image showed the presence of spherical particles with an average size of about 80 nm for the material (Fig. 3).

The XPS spectrum showed two sharp peaks at 460.0 eV and 464.6 eV that are, respectively, attributed to Ti $2p_{3/2}$ and Ti $2p_{1/2}$ (Fig. 4). It is well documented in the literature that the BE of the Ti $2P_{3/2}$ in the titanosilicate materials is deconvoluted into two peaks.

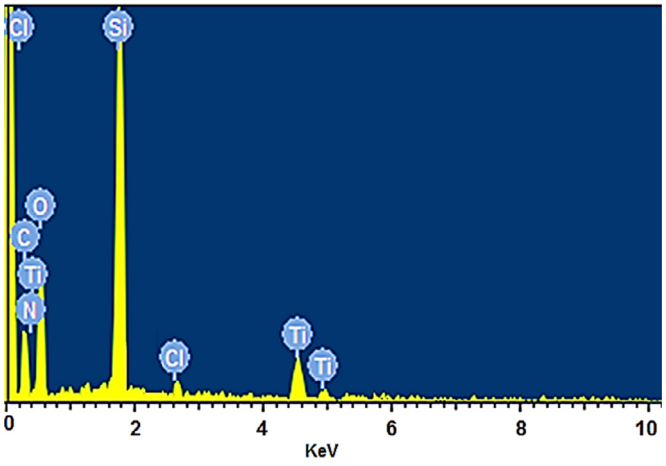


Fig. 2. EDX analysis of the Ti-ILOS catalyst

One at 458.5 eV, that is assigned to Ti (VI) ion in octahedral coordination. Another peak at 460.0 eV corresponding to Ti (IV) ion in tetrahedral coordination [31,46-49]. As shown in Fig. 4, only one peak around 460.0 eV related to Ti $2p_{3/2}$ component in tetrahedral coordination is seen for the Ti-ILOS catalyst.

3.2. Catalytic activity of the Ti-ILOS

Due to epoxides are important intermediates in the chemical industry and laboratory, in the next study, the catalytic efficiency of Ti-ILOS was studied in the epoxidation of alkenes to give their corresponding epoxides (Scheme 1). To obtain an optimized pro-

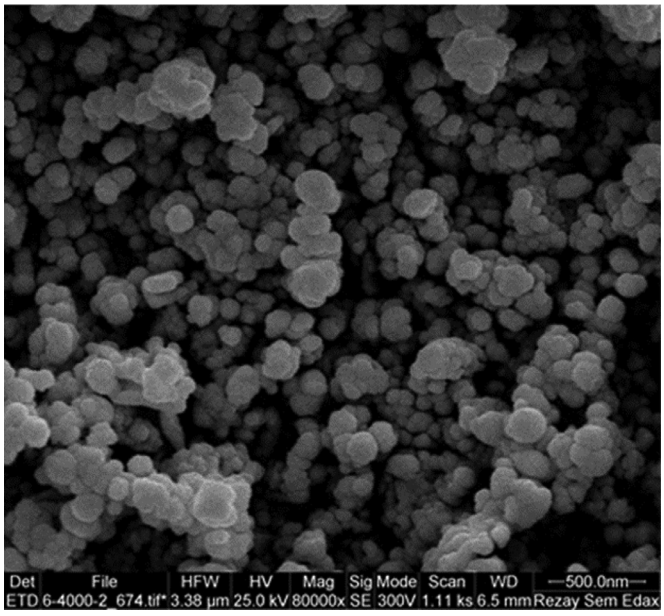


Fig. 3. SEM image of the Ti-ILOS catalyst

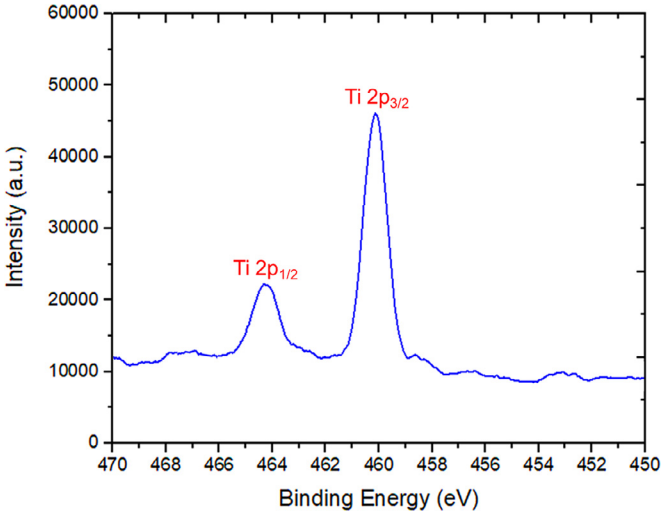


Fig. 4. XPS spectrum in the Ti2p region of the Ti-ILOS catalyst

tocol for performing the aforementioned process, the oxidation of styrene was selected as a reaction model and the effect of catalyst loading, solvent, oxidant and temperature was investigated (Table 1). As can be seen, the investigations showed that increasing the amount of catalyst has a significant effect on increasing the product yield (Table 1, entries 1-4). As shown in the presence of 0.005, 0.01, 0.014 and 0.02 g of the designed catalyst, respectively, 15, 28, 35 and 35% yield of desired epoxide are obtained. Therefore, the use of 0.014 g of catalyst was selected as optimum catalyst loading for next studies. The effect of solvent study also showed between water, THF, toluene, methanol and solvent-free media, under solvent-free conditions the best result is delivered (Table 1, entry 3 *versus* entries 5-8). Importantly, between several oxidants in hand, molecular oxygen and hydrogen peroxide were used due to their low cost, environmentally-friendly and also produce water as the sole by-product. The study showed that the H_2O_2 oxidant compared to oxygen is more effective in the epoxidation process (Table 1, entry 8 *versus* entry 9). It was also found that the reaction temperature considerably impresses the reaction rate. As shown, between 25, 40, 60, 70 and 80 ºC, the best result is delivered at 70 ºC (Table 1, entries 9-13). In the following, the epoxidation of the model substrate was investigated in the absence of the Ti-ILOS in which no oxidation product was obtained indicating the presence of the Ti-ILOS is required to perform the reaction (Table 1, entry 12 *versus* entry 14). Also, it was found that the reaction is not performed in the absence of oxidant (Table 1, entry 12 *versus* entry 15). In the next, to show the exact role of Ti species in the catalytic process, the activity of titanium-free ILOS material was studied and the result was compared with that of Ti-ILOS (Table 1, entry 12 *versus* entry 16). Interestingly, the Ti-free ILOS delivered no epoxide product under the same conditions as Ti-ILOS, confirming that the epoxidation process is completely catalyzed by immobilized-Ti sites. Accordingly, the use of 0.014 g of Ti-ILOS at 70 ºC and solvent-free media in the presence of H_2O_2 were chosen as optimal conditions.

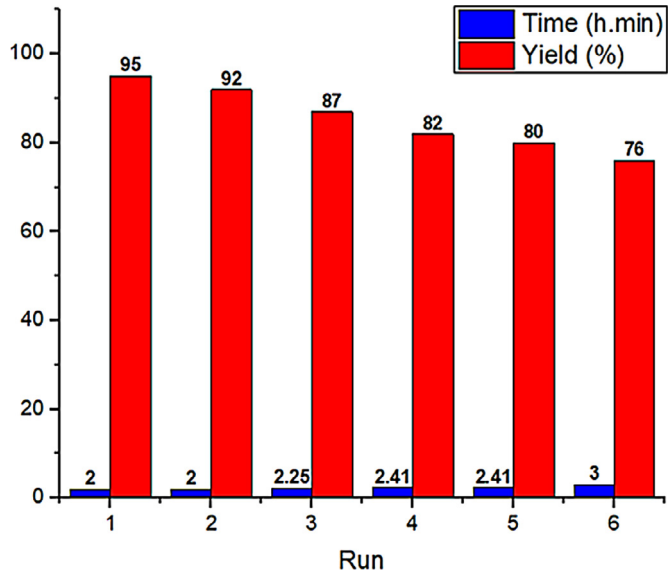


Fig. 5. Recyclability of Ti-ILOS catalyst

To show the generality and substrate scope of designed catalytic system, several alkenes were applied as substrate under optimal conditions. The results showed that Ti-ILOS catalyst can epoxidize styrene and α-methylstyrene efficiently and selectively. Also, cyclohexene, cyclooctene, (Z)-stilbene and 1-octene were converted to their corresponding epoxides with excellent selectivity by using Ti-ILOS catalyst. It should be noted that the by-products synthesized during the epoxidation of each substrate are shown in Table 2.

In the next study, the recyclability of the catalyst was investigated. For this, after reaction was completed in the epoxidation of styrene as a test model, the catalyst was separated and washed with EtOH. The recycled catalyst was used in the next reaction after being dried under vacuum. It was found that the Ti-ILOS catalyst can be recovered and reused for at least five runs with no significant decrease in its efficiency (Fig. 5).

Next, a leaching test was also performed to determine the true nature of the catalyst under applied conditions. For this, after the epoxidation was completed about 45%, the catalyst was separated from reaction mixture and the catalyst-free residue was allowed to continue under optimal condition. This showed no further progress in the epoxidation process indicating the high stability and no leaching of active titanium centers under applied media.

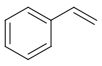
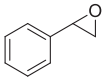
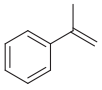
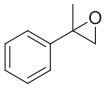
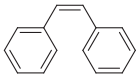
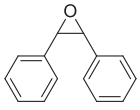
Finally, the efficiency of Ti-ILOS was compared with some of the catalysts used recently in the epoxidation of alkenes. According to Table 3, our catalyst is better than and/or comparable with former catalysts in terms of reaction conditions and recycling times. The high efficiency of our designed catalyst is attributed to the valu-

Table 1
The effect of solvent, temperature, oxidant and catalyst loading in the epoxidation of styrene

Entry	Catalyst (g)	Oxidant	Solvent	Temperature (°C)	Time (h)	Yield (%) [a]
1	0.005	O_2	Water	60	12	15
2	0.010	O_2	Water	60	12	28
3	0.014	O_2	Water	60	12	35
4	0.020	O_2	Water	60	12	35
5	0.014	O_2	Toluene	60	12	–
6	0.014	O_2	THF	60	12	–
7	0.014	O_2	Methanol	60	12	18
8	0.014	O_2	Solvent free	60	12	38
9	0.014	H_2O_2	Solvent free	60	3	76
10	0.014	H_2O_2	Solvent free	25	3	20
11	0.014	H_2O_2	Solvent free	40	3	55
12	**0.014**	**H_2O_2**	**Solvent free**	**70**	**2**	**95**
13	0.014	H_2O_2	Solvent free	80	2	92
14	–	H_2O_2	Solvent free	70	12	–
15	0.014	–	Solvent free	70	12	–
16[b]	0.014	H_2O_2	Solvent free	70	12	–

[a] GC yields. [b] ILOS was used as catalyst.

Table 2
Epoxidation of alkenes using Ti-ILOS[a]

Entry	Alkene	Product	Time (h)	Yield (%)[b]	Conversion (%)	Selectivity (%)	The main by-products
1			2	95	99	96	Benzaldehyde
2			2.5	81	98	83	Acetophenone,
3			3.5	58	62	94	Benzaldehyde
4			3	65	70	93	1,2-Cyclohexanediol and cyclohexanone
5			3	58	68	85	1,2-Cyclooctanediol
6			6	36	36	100	Unknown

[a] Conditions: alkene (1 mmol), H_2O_2 (3 mL), Ti-ILOS catalyst (0.014 g) and 70 °C. [b] GC yields.

Table 3
The comparison study between efficiency of the present catalyst with that of former catalysts

Catalyst	Conditions	Oxidant	Time (h)	Recovery times	Ref.
5MoCT	Cat. 100 mg, dichloroethane, 80 °C	t-BuOOH	10	5	[50]
PTA/Si–imid@ Si–MNPs	Cat. 0.1 g, 1,2-dichloroethane, 70 °C	t-BuOOH	6	5	[51]
Ti–Fe_3O_4@MCM-41	Cat. 0.1 g, toluene, 80 °C	t-BuOOH	6	8	[28]
Co-250	Cat. 10 mg, DMF, 100 °C	Air	4	4	[52]
Ti-SBA-15	Cat. 0.2 g, decane, 368 °K	t-BuOOH	5	2	[53]
Ti–SiO_2	Cat. 0.4 g, CH_3CN, 338 °K	H_2O_2	3	5	[54]
60Si/Ti-MCM-41	Cat. 50 mg, decane, 333 °K	t-BuOOH	5	2	[55]
Ti/S9460iO_2-Dav	Cat. 150 mg, TFT, 363 °K	t-BuOOH	24	4	[56]
Ti-ILOS	Cat. 0.014 g, Solvent free, 70 °C	H_2O_2	2	5	This work

able roles of ionic liquid moieties (for good lipophilicity) and also chemically immobilized Ti species.

4. Conclusion

In summary, in the present study, a self-assemble ionic liquid based organosilica-titania (Ti-ILOS) was prepared, characterized and its efficiency was developed in the green epoxidation of alkenes. The FT-IR and EDX analyses showed well incorporation and high stability of desired organic and inorganic moieties into the material network. The Ti-ILOS demonstrated excellent activity in the green catalytic epoxidation of alkenes and delivered the epoxide products in good to high yield. This catalyst was also recovered and reused a number of times without a significant decrease in efficiency.

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

Acknowledgments

The authors thank Yasouj University and the Iran National Science Foundation (INSF) for supporting this work.

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