

Solvent-free selective oxidation of alcohols with *tert*-butyl hydroperoxide catalyzed by palladium(II) isatin Schiff base complex supported into three-dimensional mesoporous silica KIT-6

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Abstract

In this work, the catalytic activity of a palladium(II) isatin Schiff base complex immobilized into mesoporous silica KIT-6 (Pd-isatin Schiff base@KIT-6) was studied for the oxidation of different alcohols with *tert*-butyl hydroperoxide (TBHP, 70% aqueous solution) as an oxidant under solvent-free conditions. To find the suitable reaction conditions, the effect of essential factors including the solvent, temperature, catalyst amount and kind of oxidant on the oxidation of benzyl alcohol was explored. The results showed that in this catalytic system, the corresponding aldehydes and ketones were obtained with high to excellent yields at 50 °C without the formation of carboxylic acids as by-products. The catalyst was easily recovered by simple filtration and reused in five subsequent reaction cycles without any significant loss in the catalytic activity. Moreover, the comparison of the Fourier transform infrared (FT-IR) spectrum, X-ray diffraction (XRD) pattern, scanning and transmission electron microscopy (SEM and TEM) images of the used catalyst with a fresh one showed that the structure of the Pd-isatin Schiff base@KIT-6 catalyst remained intact after five times of reuse.

Keywords Palladium · Isatin · Schiff base · KIT-6 · Alcohol oxidation · Solvent-free

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Introduction

The products of the oxidation of alcohols like aldehydes and ketones are widely used for organic synthesis of fine chemicals in laboratories and chemical industries [1]. Aldehydes and ketones exist in many biologically active molecules and functional intermediates for the pharmaceuticals, agricultural chemicals, drugs and fine chemicals [2–4]. The most common procedures for the oxidation of alcohols may use some toxic, unsafe, unstable and expensive reagents such as persulfate and chromate which are not environmentally friendly and economically justified [5, 6]. Therefore, the development of environmentally friendly catalytic systems for the oxidation of alcohols with high activity and selectivity has been considered by researchers. Recently, Guðmundsson et al. reported the first Fe(II) catalyzed biomimetic aerobic oxidation of alcohols along with an electron-rich quinone and an oxygen-activating Co(salen)-type complex as electron transfer mediators (ETMs). These electron transfer mediators are part of what is called the electron transport chain (ETC), where O_2 is typically used as the terminal oxidant and lead to a low energy pathway via stepwise electron transfer [7].

Azomethines or Schiff base ligands have played the main role in developing the transition metal coordination chemistry due to their facile synthesis, versatility, chemical and thermal stabilities [8]. In recent years, special attention has been paid to the transition metal Schiff base complexes owing to their outstanding structural features, catalytic applications, biological roles in biochemical processes and designing of chemosensors [9-11]. Schiff base transition metal complexes are also interesting oxidation catalysts because of their ease of preparation, low cost, chemical and thermal stabilities, along with their selective performance [12]. Recently, Reddy et al. reported new copper Schiff base complexes based pyrene and anthracene with high fluorescence property for the very fast detection of pyric acid (PA) at different pH ranges [13, 14]. They also designed a new Schiff base ligand by condensation of 1,3-diaminopropane with anthracene-9-carboxaldehyde, which gave a nickel complex as an efficient fluorescence chemosensor for the selective detection of 2,4,6-trinitrophenol (TNP) [15]. TNP is released into the environment as industrial wastage and is one of the main reasons for the pollution. It creates health hazards and chronic, skin and eye diseases and is used as a powerful explosive similar to TNT [16].

In catalysis science, the separation and recycling of catalysts are one of the important issue. For this purpose, the homogeneous catalysts are usually supported on organic substrates such as polystyrene and polyethylene glycol or inorganic supports like clay, carbon nanotubes and silica mesoporous materials [17]. The mesoporous silica KIT-6 has an *Ia3d* cubic structure with high thermal stability, high surface area and large pore size [18]. KIT-6 is superior because of its mesoporous structure with two-dimensional channels like MCM-41 and SBA-15 owing to its better dispersion of catalyst and more rapid diffusion of reactants and products during the reactions [19].

Nowadays, extensive research efforts have been devoted to the eco-friendly solvent-free conditions. Discarding hazardous organic solvents are the main

problem in chemical industries accounting about 80% of their wastes [20]. The solvent-free reactions are not interesting just from an ecological point of view, but also as they take place more efficiently and selectively with respect to their solution counterpart [21, 22].

The homogeneous catalysts undergoing problems related to their separation and recovery may also bring some unwanted metal impurity in the products [23]. Hence, for efficient recovery and recycling of the homogeneous catalysts, many investigations have been focused on their immobilization onto different solid supports [24]. Recently, Kumar and co-workers have developed different supported Au nano-structures (nanorods, nanohexagons and nanostars) on amorphous titanium dioxide (TiO₂), and the prepared nanocomposites were utilized as efficient photocatalysts in the oxidation of methanol to formaldehyde under natural sunlight irradiation [25].

The supported palladium complexes have been widely used as heterogeneous catalysts for the oxidation of alcohols [26], epoxidation of olefins [27, 28] and Suzuki and Heck coupling reactions [29–34]. For example, Chirra and co-workers have synthesized a novel Pd-KIT-6 catalyst by a room temperature sol–gel method and utilized this material in Suzuki–Miyaura cross-coupling reactions in the solventfree conditions under microwave irradiation [35]. Also, Zhang et al. have reported a proline–palladium catalyst supported on magnetic nanoparticles for the oxidation of alcohols into the corresponding carbonyl compounds in excellent yields under atmospheric pressure of molecular oxygen [36].

Recently, we have reported the synthesis, characterization and use of palladium isatin Schiff base complex immobilized into mesoporous silica KIT-6 (Pd-isatin Schiff base@KIT-6) in the selective oxidation of sulfides to sulfoxides and oxidative coupling of thiols to disulfides [37]. In this work, we investigated the potential of Pd-isatin Schiff base@KIT-6 in the oxidation of alcohols with *tert*-BuOOH (70% aqueous solution) under the solvent-free conditions. In addition, the reusability and stability of the involved catalyst were studied in this work.

Experimental

Materials and methods

All the used chemical materials were of commercial reagent grade. The alcohols were obtained from Merck and Fluka. The Pd-isatin Schiff base@KIT-6 catalyst was prepared and characterized based on the procedure previously described in detail [37]. The Fourier transform infrared (FT-IR) spectra were obtained as KBr pellets in the wavenumber range of 400–4000 cm⁻¹ with a Thermo SCIENTIFIC NICOLET iS10 spectrometer. An ICP-SpectroCiros CCD instrument was used for the inductively coupled plasma spectroscopy to determine the metal content of the catalyst. A Panalytical X pert Pro diffractometer with Cu-K α radiation was employed to obtain the X-ray powder diffraction (XRD) patterns of the powder samples at room temperature. A Zeiss Sigma VP-500 FE-SEM instrument was used to take scanning electron micrographs. TEM images were recorded using a Zeiss-EM10C transmission electron microscope. The progress of the reaction was studied by thin-layer

chromatography (TLC) on silica-gel polygram SILG/UV 254 plates. The gas chromatography (GC) was carried out by a Philips GC-PU 4600 instrument armed with a flame ionization detector (FID) and a 2 m column packed with silicon DC-200.

General procedure for the oxidation of alcohols with tert-BuOOH catalyzed by Pd-isatin Schiff base@KIT-6 under solvent-free conditions

In a test tube equipped with a condenser, a mixture of alcohol (1 mmol), Pd-isatin Schiff base@KIT-6 (20 mg, 0.0048 mmol), as the catalyst, and *tert*-BuOOH (70% aqueous solution), as the oxidant, was prepared. The test tube was put in an oil bath at 50 °C, and the mixture was stirred for the appropriate times. The reaction progress was checked by thin-layer chromatography (TLC) using eluent *n*-hexane: ethyl acetate with various ratios. After the completion of reaction, the solid catalyst was filtered and washed thoroughly with diethyl ether, and the combined washings and filtrates were purified on a silica-gel column to achieve the pure product. The products were identified by comparing their physical data with the authentic materials.

Results and discussion

Catalytic oxidation of alcohols

Scheme 1 shows the preparation route of Pd-isatin Schiff base@KIT-6 catalyst. At first, the mesoporous silica KIT-6 was prepared according to the procedure as reported by Kleitz et al. [38]. Then, the prepared KIT-6 was reacted with 3-amino-propyltriethoxysilane (3-APTES) to obtain amino-functionalized KIT-6 (KIT-6-pr-NH₂). Subsequently, the condensation reaction between the amine group of KIT-6-pr-NH₂ and the keto group at the third position of isatin resulted in isatin Schiff base@KIT-6. Finally, the Pd-isatin Schiff base@KIT-6 catalyst was synthesized by the reaction of dissolving palladium(II) acetate in methanol with the above prepared isatin Schiff base@KIT-6. The Pd content of the prepared catalyst was determined by ICP, showed a value about 0.24 mmol/g of supported catalyst.

The high catalytic activity of Pd-isatin Schiff base@KIT-6 in the selective oxidation of sulfides and oxidative coupling of thiols [37] encouraged us to study the ability of this heterogeneous catalyst in other organic transformation reactions such as oxidation of alcohols to carbonyl compounds. In this regard, to achieve the optimized reaction conditions, the effect of reaction parameters including kind of solvent and oxidant, temperature and catalyst amount, was investigated in the oxidation of benzyl alcohol as a model reaction.

Firstly, different solvents such as acetonitrile, methanol, ethanol, water, toluene, chloroform, dichloromethane, as well as the solvent-free conditions were used for the oxidation of benzyl alcohol with *tert*-BuOOH in the presence of Pd-isatin Schiff base@KIT-6. The obtained results (Table 1) showed that the higher amount of benzaldehyde was obtained under the solvent-free conditions (Table 1, entry 8). Hence, the effect of reaction parameters was studied on the solvent-free conditions.



Pd-isatin Schiff base@KIT-6

Scheme 1 Preparation route of Pd-isatin Schiff base@KIT-6 catalyst

e effect of solvent on n of benzyl alcohol hyde with <i>tert</i> - alyzed by Pd-isatin	Entry	Solvent	Time (h)	Isolated yield (%)
@KIT-6 ^a	1	CH ₃ CN	3	65
	2	CH ₃ OH	3	53
	3	C ₂ H ₅ OH	3	70
	4	H ₂ O	3	47
	5	Toluene	3	38
	6	CHCl ₃	3	25
	7	CH ₂ Cl ₂	3	30
	8	Solvent-free	3	90

^aReaction conditions: benzyl alcohol (1 mmol), tert-BuOOH (3 mmol), catalyst (20 mg), solvent (1 mL), T = 50 °C

To investigate the effect of temperature on the reaction, the oxidation of benzyl alcohol was carried out in the range of 25-60 °C, while the other parameters were kept fixed. The results (Table 2) showed that the benzaldehyde yield enhanced with increasing temperature, and the highest yield was observed at 50 °C. Therefore, in this catalytic system, 50 °C was chosen as the optimum temperature.

Also, the catalyst amount in the oxidation of benzyl alcohol was optimized. The results given in Table 3 reveal a trace amount of product in the absence of a catalyst, while increasing the catalyst amount from 5 to 20 mg led to more conversion of alcohol. By using 25 mg of the catalyst, no noticeable increase in the yield of the product was observed. Therefore, 20 mg (0.0048 mmol) of the catalyst was selected as the optimum catalyst amount.

Another important factor in the oxidation of alcohol is the nature of oxidant. Different oxygen donors such as 30% H₂O₂, urea hydrogen peroxide (UHP), tert-BuOOH (70% aqueous solution) and NaIO₄ were tested for the oxidation of benzyl alcohol in the presence of Pd-isatin Schiff base@KIT-6 catalyst. The results (Table 4) showed that among them, tert-BuOOH (70% aqueous solution) provides the best benzaldehyde yield.

Table 2 The effect of temperature on the oxidation of benzyl alcohol to benzaldehyde with <i>tert</i> -BuOOH catalyzed by	Entry	Temperature (°C)	Time (h)	Isolated yield (%)
Pd-isatin Schiff base@KIT-6	1	25	3	30
under solvent-free conditions"	2	30	3	38
	3	40	3	63
	4	50	3	90
	5	60	3	85

^aReaction conditions: benzyl alcohol (1 mmol), tert-BuOOH (3 mmol), catalyst (20 mg)

Entry	catalyst amount (mg)	Time (h)	Isolated yield (%)
1	0	3	Trace
2	5	3	35
3	10	3	48
4	15	3	67
5	20	3	90
6	25	3	87

^aReaction conditions: benzyl alcohol (1 mmol), *tert*-BuOOH (3 mmol), catalyst. T=50 °C

Entry	Oxidant	Time (h)	Isolated yield (%)
1	30% H ₂ O ₂	3	70
2	H ₂ O ₂ /urea (UHP)	3	53
3	<i>tert</i> -BuOOH (TBHP, 70% aqueous solution)	3	90
4	Anhydrous <i>tert</i> - BuOOH (26% TBHP in benzene)	3	85
5	NaIO ₄	3	18
6	No oxidant	3	Trace

Table 4The effect of variousoxidants on the oxidation ofbenzyl alcohol to benzaldehydecatalyzed by Pd-isatin Schiffbase@KIT-6 under solvent-freeconditions^a

 Table 3
 The effect of catalyst amount on the oxidation of benzyl alcohol to benzaldehyde with *tert*-BuOOH catalyzed by Pd-isatin Schiff base@KIT-6 under solvent-free conditions^a

^aReaction conditions: benzyl alcohol (1 mmol), oxidant (3 mmol), catalyst (20 mg), T=50 °C



Scheme 2 Oxidation of alcohols with *tert*-butyl hydroperoxide (TBHP) catalyzed by Pd-isatin Schiff base@KIT-6 under solvent-free conditions

Afterward, the ability of this heterogeneous catalyst in the oxidation of various types of primary and secondary alcohols was studied under the optimum reaction conditions (Scheme 2). The results are presented in Table 5. As seen, different benzylic alcohols including electron-donating (such as $-CH_3$ and $-C(CH_3)_3$) and electron-withdrawing substitutes (e.g. $-NO_2$ and -Cl) on benzene ring, were selectively oxidized into the corresponding carbonyl compounds with good to excellent yields, without any overoxidation to the corresponding carboxylic acid (Table 5, entries 1–7). Also, the benzylic alcohols containing electron-withdrawing groups

Entry	Alcohol	Product	Time (h)	Yield (%)
1	СН2ОН	CHO	3	87
2	CH2OH Me Me	CHO	3	91
3	MeO Me	СНО	3	90
4	(Me) ₂ CH ₂ OH (Me)	CHO	3	93
5	CI CH2OH	CI	3	71
6	CI	CHO	3	58
7	O ₂ N CH ₂ OH	D ₂ N CHO	3	63
8	H ₂ N F	CHO L _{2N}	3	82
9	ОН	СНО	3	78
10	ОН	СНО	3	71
11	ОН	CHC	3	72
12	OH		4	85

Table 5 Oxidation of various alcohols with *tert*-BuOOH catalyzed by Pd-isatin Schiff base@KIT-6 under solvent-free conditions^a





^aReaction conditions: alcohol (1 mmol), tert-BuOOH (3 mmol), catalyst (20 mg), T=50 °C

in *para*-position exhibit less activity than the electron-donating counterparts. The involved catalytic system was found to be effective in oxidizing the primary aliphatic alcohols. The results showed that these aliphatic alcohols were converted to carbonyl compounds with a good yield (Table 5, entries 9 and 10). Moreover, this catalyst was effective for the oxidation of aliphatic and aromatic secondary alcohols, and the corresponding ketones were obtained in good yields (Table 5, entries 11–15). The present oxidizing system could be used for the oxidation of sterically hindered alcohols such as diphenylmethanol. In this oxidation process, the corresponding ketone was obtained with the good yield (Table 5, entry 15). Furthermore, in this catalytic system, furfuryl alcohol as a heteroaromatic alcohol was oxidized to furfural with a good yield without any decomposition or polymerization (Table 5, entry 8).

The advantages of this method such as high yields of products, environment benignity and recyclability make it amenable to scalability readily. As an example, a scaled up procedure (10 mmol) for oxidation of benzyl alcohol led to isolation of the benzaldehyde in 85% yield at the same time and conditions mentioned in Table 5.

The proposed mechanism for oxidation of alcohols with *tert*-BuOOH catalyzed by Pd-isatin Schiff base@KIT-6 is given in Scheme 3. The catalytic cycle begins with binding of the alcohol to the Pd(II) catalyst (**A**) to form alcohol bound intermediate **B**. Deprotonation of **B** then occurs to form a Pd alkoxide (**C**), which undergoes β -hydride elimination to release the carbonyl product and form a Pd hydride species (**D**). The Pd hydride can reductively eliminate an equivalent of acid to form Pd(0), which is then reoxidized to Pd(II) catalyst by *tert*-BuOOH and 2 equivalent of acid



Scheme 3. Proposed mechanism for oxidation of alcohols with *tert*-BuOOH catalyzed by Pd-isatin Schiff base@KIT-6

[39–41]. Hence, any steric hinderance around the hydroxyl group of alcohols such as *ortho*-substituted benzylic alcohols (Table 5, entry 6) or more hindered secondary alcohols such as diphenylmethanol (Table 5, entry 16) causes longer reaction time and lower yields. Also, the oxidation of benzyl alcohol was controlled in the presence of Ph_2NH as the radical trap and results showed that there was no change in the catalyst activity.

A comparison of the Pd-isatin Schiff base@KIT-6 catalyst with other reported ones, presented in Table 6. As seen, our system has some advantages including short reaction time and high catalytic performance such as selectivity, TON and TOF under the solvent-free conditions.

Catalyst reusability

To explore the reusability and stability of the Pd-isatin Schiff base@KIT-6 catalyst, its activity in the oxidation of benzyl alcohol was tested for five subsequent cycles. At the end of each cycle, the solid catalyst was simply filtered from the reaction mixture, washed carefully with methanol and dried in vacuum. Then, the recycled catalyst was used for the subsequent run under the optimum reaction conditions. The results show that it is possible to reuse the catalyst several times without noticeable loss of its initial activity (Table 7). After each run, the filtrate was analyzed by the

Table 6 hyde	Comparison of catalytic activity	of Pd-isatin Schiff base@KIT-6 catalyst w	vith some ot	her catalyst r	eported for	selective oxida	tion of be	nzyl alcohol t	o benzalde-
Entry	Catalyst	Conditions	Time (h)	Con. (%)	Sel. (%)	Benzalde- hyde yield (%)	TON	TOF (h ⁻¹)	Ref
1	Mn(TPP)OAc@SMNP	$ m H_2O,~air,~60~^\circ C$	4.5	06	> 99	90	1765	392.1	[42]
5	Polymer anchored Cu(II)	H ₂ O, TBHP, 50 °C	9	95	96	91	271.2	45.2	[43]
3	$SiO_2@Fe_3O_4$ -Pro-Pd	Toluene, K_2O_3 , O_2 , $90 \circ C$	10	76	> 99	96	192	19.2	[36]
	$CoFe_2O_4$ @ APTES @Ni(OH)_2	CH_3CN , H_2O_2 , $80 \circ C$	L	87	> 99	87	174	24.8	[44]
	SBA-15-pr-NH ₂ -Mn(Salen)	CH ₃ CN, TBHP, 90 °C	8	73.5	100	73.5	143.2	17.9	[45]
9	MIL-101-SO ₃ -Cu	CH ₃ CN, TEMPO, NaHCO ₃ , O ₂ , 60 °C	9	> 99	< 99	66	99.8	16.6	[46]
7	Pd-isatin Schiff base@KIT-6	Solvent-free, TBHP, 50 °C	3	87	100	87	181.2	60.4	This work

Table 7Reusability and stability of Pd-isatin Schiff base@KIT-6 catalyst in the oxidation of benzyl alcohol to benzaldehyde with <i>tert</i> -BuOOH under solvent-free conditions ^a	Run	Time (h)	Yield (%) ^b	Pd leached (%) ^c
	1	3	90	2.1
	2	3	87	1.5
	3	3	85	0
	4	3	85	0
	5	3	83	0

^aReaction conditions: benzyl alcohol (1 mmol), *tert*-BuOOH (3 mmol), catalyst (20 mg), T=50 °C

^bIsolated yield

^cDetermined by ICP

inductively coupled plasma (ICP) technique to determine the amount of leached Pd. It was found that just a small amount of palladium had been leached in the first two runs (Table 7).

The recovered catalyst was also characterized by FT-IR, XRD, SEM and TEM techniques. The FT-IR spectrum of the recycled catalyst presented in Fig. 1b was found to be similar to that of fresh catalyst (Fig. 1a). For the recycled catalyst, the FT-IR bands at 1085, 805 and 464 cm⁻¹ can be assigned to Si–O-Si vibrations and the band at 2932 cm⁻¹ is associated with vibrations of aliphatic CH₂ groups. Moreover, the characteristic frequencies of C=N and C=O stretching modes were observed at 1624 and 1728 cm⁻¹, respectively. All of these FT-IR bands appeared for the fresh catalyst (Fig. 1a), as well. The XRD pattern of reused catalyst is shown in Fig. 2b. As shown, XRD pattern is in good agreement with XRD pattern of fresh catalyst (Fig. 2a). Also, the Debye–Scherrer formula was used to determine the crystal size of fresh and reused catalyst. The average crystallite



Fig.1 FT-IR spectrum of a fresh and b recycled Pd-isatin Schiff base@KIT-6 catalyst



Fig.2 XRD pattern of a fresh and b reused Pd-isatin Schiff base@KIT-6 catalyst

size was estimated about 43.5 and 43.7 nm for fresh and reused Pd-isatin Schiff base@KIT-6, respectively. Moreover, the SEM and TEM images of the recovered Pd-isatin Schiff base@KIT-6 compared with the fresh one (Figs. 3 and 4, respectively) showed that the morphology remained intact after recovering. These observations demonstrated the preservation of the catalyst's structure after recycling.

Conclusions

In summary, palladium(II) isatin Schiff base complex supported into the mesoporous silica KIT-6, Pd-isatin Schiff base@KIT-6, was used as a heterogeneous catalyst for the oxidation of a wide range of alcohols with *tert*-butyl hydroperoxide (TBHP, 70% aqueous solution) as an oxidant under the solvent-free conditions. The good to excellent yields, eco-friendly and selective oxidation of alcohols without by-product formation can be mentioned as the benefits of this protocol for the oxidation of alcohols. Furthermore, the involved catalyst could be conveniently recovered by simple filtration and reused five times with no significant loss of efficiency.



Fig.3 SEM images of **a** fresh and **b** recovered Pd-isatin Schiff base@KIT-6 catalyst



Fig.4 TEM images of a fresh and b recovered Pd-isatin Schiff base@KIT-6 catalyst

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