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Short Communication

C-3 alkylation of oxindole with alcohols catalyzed by an indene-functionalized mesoporous iridium catalyst

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ABSTRACT

A heterogeneous indene-based iridium catalyst with a highly ordered dimensional-hexagonal mesostructure was prepared through complexation of $IrCl_3$ with the indene-functionalized SBA-15 silica materials. During C-3 alkylation of oxindole with various alcohols, this heterogeneous catalyst exhibited highly catalytic activity (up to 93%). Such a catalyst could be recovered easily and used repetitively eight times without significantly affecting its catalytic activity.

1. Introduction

Oxindole as an important motif of many natural products has a wide variety of biological activity [1–6]. In particular, oxindole derivatives with C-3 functionalization, such as the anti-inflammatory Tenidap [7] and the anti-cancer kinase inhibitor Sunitinib [8], are well known for treatment of disease in medicinal chemistry. Generally, main strategies to synthesize these biologically active molecules involve conventional alkylation with alkyl halides. However, the inherent disadvantages of alkyl halides, such as toxicity and expensiveness, limit their application in industrial process. Furthermore, alkylations with alkyl halides often suffer from bad regioselectivity due to formation of bis-alkylated by-products [9]. Alcohols as a kind of alkylative reagents are desirable economic and environmentally friendly because only water is by-product. More importantly, alcohols as relatively weak alkylative reagents possess high regioselectivity. Recently, the catalytic reactions using alcohols as alkylative reagents for C-3 alkylation of oxindole have appeared in the literature [10–16]. Grigg group [10] reported a catalytic reaction employing alcohols as alkylative reagents, in which organometallic iridium showed a high catalytic activity in C-3 alkylation of oxindole. A similar strategy reported by Madsen group [11] also showed a high catalytic efficiency for C-3 alkylation of oxindole when $RuCl_3$ worked as a catalyst. Although these catalysts had showed a high catalytic activity in C-3 alkylation of oxindole, their practical applications in the industrial process were hindered due to difficulty in recovery and reuse of these expensive metal catalysts, as well as product contamination from catalysts during work-up stage. Thus, besides employing economic and environmentally friendly alcohols as alkylative reagents, giving consideration to catalyst recycling for C-3 alkylation of oxindole is a promising way in the industry.

Immobilization is well-known to solve the problem of transition-metal catalyst recycling [17,18]. In particular, the immobilization of homogeneous catalysts onto the mesoporous materials has showed some salient features [19–22]. Besides general features of easy separation and efficient recycling, these heterogeneous mesoporous catalysts possess relatively large surface area and pore volume, and tunable pore dimension and well-defined pore arrangement [23–25]. These features are not only beneficial to control the dispensability of catalytically active species, but also to adjust the catalytic microenvironment of catalytically active centers. Furthermore, they also have a facile preparation, remarkable thermal and mechanical stability. Recently, we have reported a series of mesoporous catalysts and their applications in catalytic processes [26–28]. Especially, microwave-assisted Ti-catalyzed allylation of aldehydes [26] and microwave-assisted tandem Ti/Ru-catalyzed allylation–isomerization reaction [27] of benzaldehyde have showed a highly catalytic activity. Herein, we develop a facile preparation of indene-functionalized mesoporous iridium catalyst **5** and apply it to C-3 alkylation of oxindole with various alcohols. The key feature is that an indene-functionalized mesoporous material is prepared by a co-condensation method, which the potential coordination-multifunctionilty of indenyl group to various transit metals may result in an extensively application in catalytic reaction. This research focuses on construction of highly ordered indene-functionalized mesoporous iridium catalyst and investigation of its catalytic performance in C-3 alkylation of oxindole.

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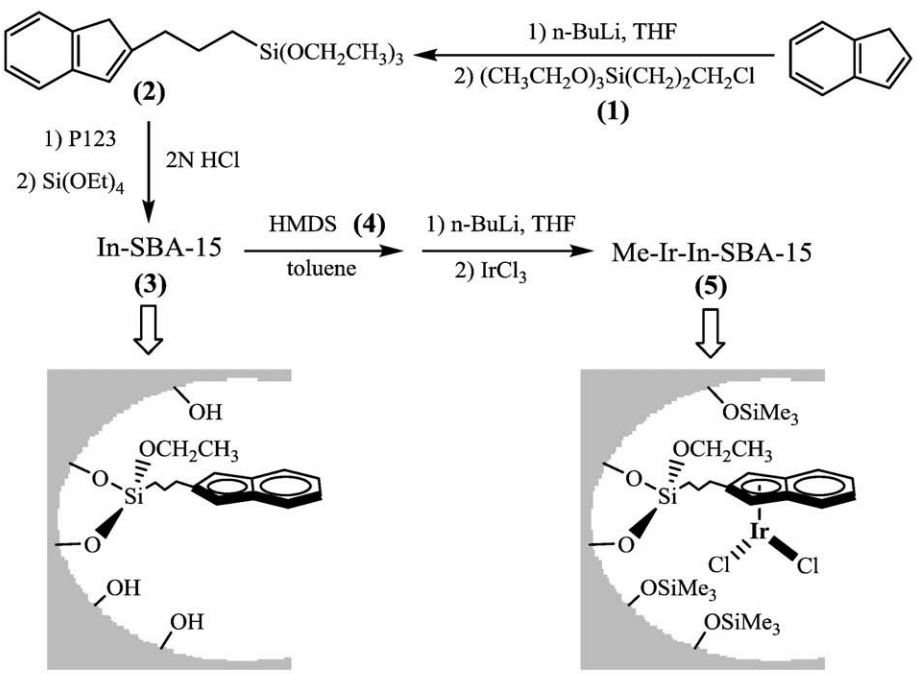


Scheme 1. Preparation of the indene-functionalized mesoporous iridium catalyst **5**.

2. Experimental

2.1. Synthesis of **2**

Under argon atmosphere, to a stirred solution of indene (1.0 mL, 1.00 g, 15.15 mmol) in dry THF (20 mL) was added n-BuLi (6.50 mL, 16.25 mmol, 2.50 M in hexane) at −60 °C. The resulting mixture was then allowed to warm to room temperature slowly and stirred for another 1 h. After cooling down to 0 °C, a solution of **1** (3.64 g, 15.15 mmol) in 3 mL dry THF was added dropwise over 15 min. The mixture was then warmed up to room temperature and stirred for further 3 h. After the solvent was removed in vacuo, saturated NH_4Cl was added to quench the reaction. The reaction mixture then was extracted with ethyl acetate several times. The combined organic layer was washed subsequently with water and brine, and dried over anhydrous Na_2SO_4. Evaporation of solvent gave the crude product, which was fast passed through a short column (silica gel, eluent: Et_3N/ethyl acetate/petroleum ether = 1:10:500) and concentrated in vacuo to afford **2** (3.79 g, 11.85 mmol) as beige glass. Yield: 78.2%; IR (KBr) cm^{-1}: 3068 (w), 2975 (s), 2923 (s), 2885 (s), 1607 (w), 1483 (m), 1457 (m), 1441 (m), 1394 (m), 1270 (w), 1167 (m), 1104 (s), 1078 (s), 955 (s), 788 (s), 767 (s), 720 (m), 690 (w), 648 (w), 549 (w), 482 (w), 422 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 7.55–7.28 (m, 4H), 6.30 (s, 1H), 3.94–3.89 (q, J = 6.8 Hz, 6H), 3.41 (s, 2H), 2.70–2.67 (t, J = 5.4 Hz, 2H), 1.94–1.90 (m, J = 5.4 Hz, 2H), 1.39–1.25 (t, J = 6.8 Hz, 9H), 0.87–0.83 (t, J = 5.4 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 13.5, 17.0, 26.2, 33.0, 54.2, 73.0, 124.4, 127.1, 128.0, 129.2, 130.5, 134.1, 141; HPLC-MS (100 eV) m/z (%): Anal. Calcd. for $C_{18}H_{28}O_3Si$: 320.18; Found: 319.20 $[M-H]^-$; Anal. Calcd. for $C_{18}H_{28}O_3Si$: C, 67.46; H, 8.81. Found: C, 67.28; H, 8.76%.

2.2. Preparation of In-SBA-15 (**3**)

In a typical synthesis, 2.0 g of the structure-directing agent, pluronic P123, was fully dissolved in a mixture of 60 mL hydrochloric acid (2.0 N) and 15 mL deionized water. Then, 4.05 mL (18.00 mmol) of tetraethyl orthosilicate (TEOS) was added as the silica precursor at 40 °C. After a TEOS pre-hydrolysis period of one hour, 0.64 g (2.00 mmol) of **2** was added. The reaction mixture was stirred at 40 °C for 24 h and aged at 100 °C for 24 h. The resulting solid was filtered and rinsed with excess ethanol before being dried overnight on a filter. The surfactant template was removed by refluxing in acidic ethanol (400 mL per gram) for 24 h. The solid was filtered and rinsed with ethanol again, and dried at 60 °C under reduced pressure overnight to afford In-SBA-15 (**3**) (1.02 g) in the form of a white powder. IR (KBr) cm^{-1}: 3440 (s), 2981 (w), 2879 (w), 1643 (m), 1544 (w), 1459 (w), 1400 (w), 1081 (s), 960 (w), 795 (w), 557 (w), 464 (m); Elemental analysis (%): C 15.47, H 3.09; d_{pore}: 5.453 nm; S_{BET}: 471.2 m^2/g; ^{29}Si MAS NMR (79.5 MHz): Q^4 (δ = −111.7 ppm), Q^3

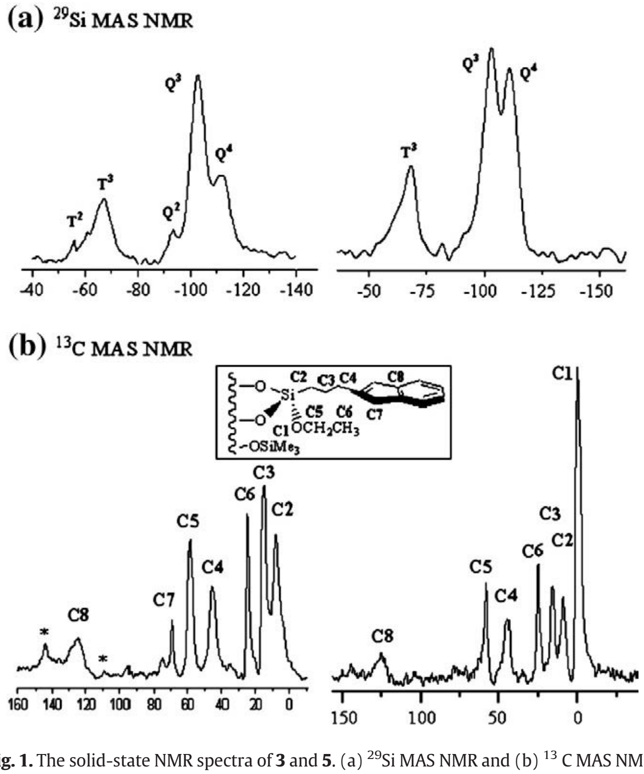


Fig. 1. The solid-state NMR spectra of **3** and **5**. (a) ^{29}Si MAS NMR and (b) ^{13}C MAS NMR.

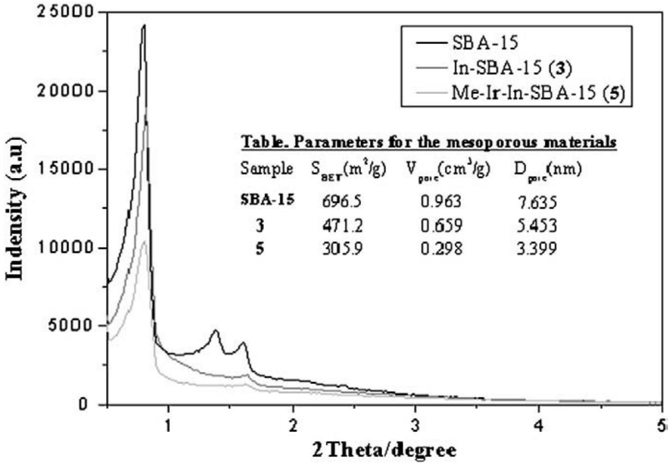


Table. Parameters for the mesoporous materials

Sample	S_{BET}(m^2/g)	V_{pore}(cm^3/g)	D_{pore}(nm)
SBA-15	696.5	0.963	7.635
3	471.2	0.659	5.453
5	305.9	0.298	3.399

Fig. 2. The powder XRD patterns of SBA-15, **3** and **5**.

($\delta = -102.9$ ppm), T^3 ($\delta = -67.3$ ppm), T^2 ($\delta = -60.8$ ppm); ^{13}C CP MAS NMR (100.6 MHz): 8.1, 14.9, 24.9, 45.3, 58.4, 69.1, 124.3 ppm.

*2.3. Preparation of the indene-functionalized mesoporous iridium catalyst (**5**)*

Under argon atmosphere, a suspension of **3** (1.00 g) and HMDS [$(CH_3)_3Si)_2N$] (10 mL, 0.050 mol) in 25 mL dry toluene was stirred overnight. The volatiles were stripped on a rotary evaporator and the dry powder was washed five times with excess dry CH_2Cl_2 and finally dried under vacuum at 60 °C for 6 h to afford end-capping of **3** (1.05 g) in the form of a white powder. Then it was suspended in 20 mL dry THF again. Under argon atmosphere, to this stirred suspension was added n-BuLi (2.00 mL, 5.00 mmol, 2.50 M in hexane) at −60 °C. The resulting mixture was then allowed to warm to room temperature slowly and stirred for another 1 h. After cooling down to 0 °C, a solution of $IrCl_3$ (0.60 g, 2.00 mmol) in 5 mL dry THF was added dropwise over 15 min. The mixture was then warmed up to room temperature and stirred for further 5 h. After the solvent was removed in vacuo, saturated NH_4Cl was added to quench the reaction. The mixture was then filtered through a filter paper and rinsed with excess water and CH_2Cl_2, dry toluene. After Soxlet extraction in THF solvent to remove unreacted start materials for 48 h, the solid was dried at room temperature under vacuum overnight to afford catalyst **5** (0.78 g) as a light yellow powder. ICP analysis showed that the Ir loading-amount is 53.52 mg per gram catalyst. IR (KBr) cm^{-1}: 2964 (w), 2879 (w), 1643 (m), 1543 (w), 1454 (w), 1398 (w), 1257 (m), 1159 (m), 1081 (s), 848 (w), 802 (w), 755 (w), 457 (m); Elemental analysis (%): C 14.42, H 2.96; d_{pore}: 3.399 nm; S_{BET}: 305.9 m^2/g. ^{29}Si MAS NMR (300 MHz): Q^4 ($\delta = -111.0$ ppm), Q^3 ($\delta = -102.8$ ppm), T^3 ($\delta = -68.2$ ppm); ^{13}C CP MAS NMR (161.9 MHz): 0.1, 9.3, 15.6, 25.1, 44.5, 58.3, 124.9 ppm.

2.4. Catalytic reaction

A typical procedure is as follows: The solid catalyst (48.22 mg, 0.013 mmol based on Ir from ICP), KOH (22.0 mg, 0.40 mmol), oxindole (0.266 mg, 2.0 mmol), and alcohols (2.2 mmol) and 2 mL toluene were placed in a 25-mL thick-walled screw-cap vial. The vial was purged with Ar and sealed with a screw-cap. The mixture was placed in an aluminum block preheated to 110 °C and stirred for 20 h. After completion of the reaction, the reaction mixture was allowed to cool to room temperature followed by dilution with 2 mL of ethyl acetate. The mixture was then stirred for 1 min, and the reactor was centrifuged (10000 r/min) for 3–5 min. The solid was reused for recycling experiment. The solution was purified by column chromatography (2 in × 12 in silica column) using *n*-hexane/EtOAc (4:1) as eluent to afford the corresponding products.

3. Results and discussion

*3.1. Syntheses and structural characterizations of the catalyst **5***

The indene-functionalized mesoporous materials and iridium catalyst, abbreviated as In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**), were prepared according to the procedures illustrated in Scheme 1. Firstly, the key intermediate, indene-based silica resource **2**, was synthesized by the reaction of indene with n-BuLi followed by coupling with 3-chloropropyltriethoxysilane (**1**) (NMR and IR Spectra refer to the supporting information). The condensation **2** with Si$(OEt)_4$ was then carried out to afford **3** as a white powder following a similar procedure reported in literature [29]. Finally, after the protection of silicon-hydroxyl groups in **3** using trimethysilyl (-$SiMe_3$) group as a protection reagent [30], the resulting mixture was reacted with n-BuLi followed by complexation with $IrCl_3$ to afford crude product. After removal of the unreacted start materials and homogeneous omplexes *via* Soxlet extraction thoroughly in THF, Me-Ir-In-SBA-15 (**5**) was obtained as a light yellow powder. The inductively coupled plasma (ICP) optical emission spectrometer analysis showed that Ir loading amount in the **5** was 53.52 mg (0.28 mmol) per gram of catalyst.

The incorporation of indenyl groups and the complexation of iridium onto the indene-functionalized mesoporous silica materials could be further confirmed by FT-IR spectra (see the supporting information) and solid-state NMR spectra. As shown in Fig. 1(a), the ^{29}Si MAS NMR spectra of In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**)

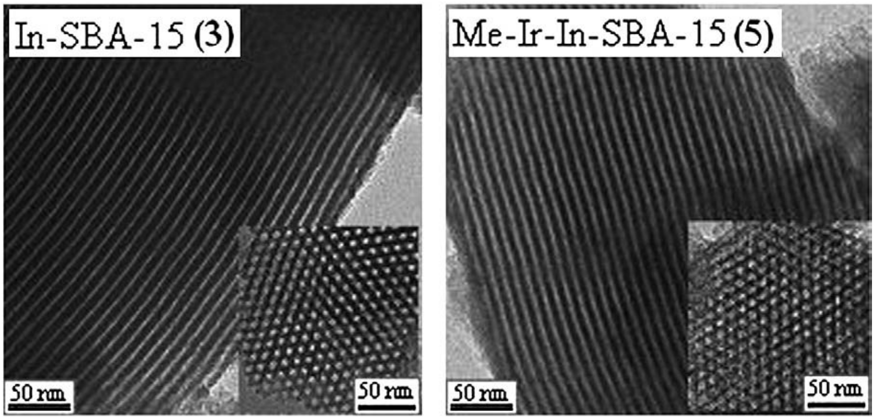


Fig. 3. The TEM images of **3** and **5** viewed along [100] and [001] directions.

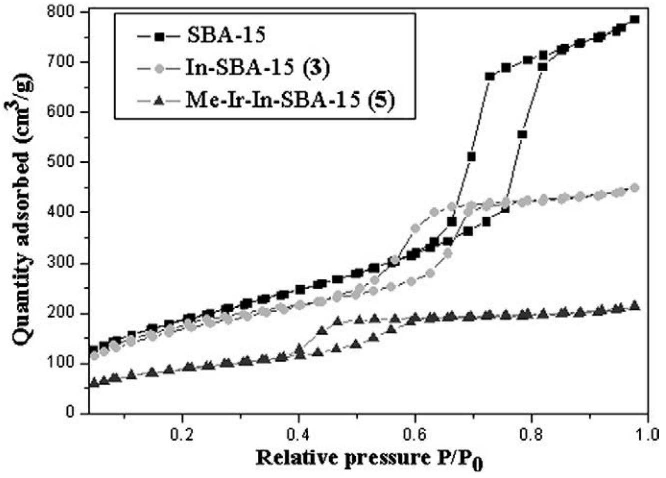
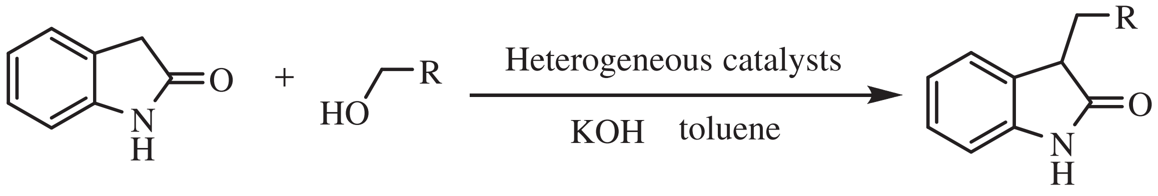


Fig. 4. The Nitrogen adsorption–desorption isotherms of SBA-15, **3** and **5**.

showed two groups of signals with four oxygen neighbors (Q-type species) originated from TEOS and with three oxygen neighbors (T-type species) derived from silylether groups. Typical isomer shift values were −91.5/−101.5/−110 ppm for $Q^2/Q^3/Q^4$ signals (Q^2{$(HO)_2Si(OSi)_2$}, Q^3 {$(HO)Si(OSi)_3$}, Q^4 {$Si(OSi)_4$}) and −48.5/−58.5/−67.5 ppm for $T^1/T^2/T^3$ signals (T^1{$R(HO)_2SiOSi$}, T^2 {$R(HO)Si(OSi)_2$}, T^3 {$RSi(OSi)_3$}) [31]. In-SBA-15 (**3**) showed three Q signals (Q^4: −111.7 ppm, Q^3: −102.9 ppm, Q^2: −94.3 ppm) and two T signals (T^2: −60.8 ppm and T^3: −67.3 ppm), while the catalyst Me-Ir-In-SBA-15 (**5**) only showed two Q signals (Q^3: −102.8 ppm and Q^4: −111.0 ppm) and one T signal (T^3: −68.2 ppm). In comparison with **3**, the enhanced Q^3–Q^4 signals and disappeared Q^2 in Me-Ir-In-SBA-15 (**5**) suggested that **5** possessed mainly network structures of {$(HO)Si(OSi)_3$} and {$Si(OSi)_4$}, while the enhanced T^3 and disappeared T^2 signals in Me-Ir-In-SBA-15 (**5**) indicated the formation of {$RSi(OSi)_3$} (R = iridium complexes) as a part of wall in mesoporous silica structure [32,33]. As marked in Fig. 1(b), the ^{13}C CP/MAS NMR spectra clearly displayed the peaks at 8.1, 14.9, 24.9, 45.3, 58.4, 69.1, 124.3 ppm for In-SBA-15 (**3**) and at 0.1, 9.3, 15.6, 25.1, 44.5, 58.3, 124.9 ppm for Me-Ir-In-SBA-15 (**5**), corresponding to indenyl and aliphatic carbon atoms, respectively.

As shown in Fig. 2, the small-angle XRD patterns revealed that In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**) showed one similar intense d_{100} diffraction peak along with two similar weak diffraction peaks (d_{110}, d_{200}), suggesting that the dimensional-hexagonal pore structure (*p*6*mm*) observed in pure SBA-15 [26] could be preserved after the co-condensation and the complexation [34]. Compared to pure SBA-15, the decreases of peak intensity in In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**) implied that the incorporation of indenyl groups and the complexation of iridium onto the indene-functionalized mesoporous silica materials might disturb the highly ordered mesoporous structure to a certain degree. The TEM morphologies further confirmed that In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**) had well-ordered mesostructures with the dimensional-hexagonal arrangement as shown in Fig. 3. Nitrogen adsorption–desorption isotherms in In-SBA-15 (**3**) and Me-Ir-In-SBA-15 (**5**) (Fig. 4) exhibited typical IV type isotherms with H_1 hysteresis loop and a visible step at $P/P_0 = 0.35–0.90$, corresponding to capillary condensation of nitrogen in mesopores. As the structural parameters inserted in Fig. 2, the protection of silicon-hydroxyl groups and the complexation of iridium complexes onto the indene-functionalized mesoporous silica materials caused a decrease in mesopore size, surface area, and pore

Table 1
The catalytic C-3 alkylation of oxindole with various alcohols.[a]



6: R = Ph; **7**:R = Me; **8**: R = n-C_5H_{11}; **9**: R = n-C_7H_{15}
10: R = 4-MePh: **11**: R = 4-MeOPh; **12**: R = 4-C1Ph; **13**: R= 2-C1Ph.

Entry	Catalyst	R	Time	Yield (%)[b]
1	5	6	1	93.5
2	$IrCl_3$	6	1	72.0[c]
3	Me-SBA-15 + $InIrCl_2$	6	1	91.7[c]
4	Me-In-SBA-15 + $IrCl_3$	6	1	69.6[c]
5	5	7	1	90.3
6	5	8	1	90.5
7	5	9	1	86.7
8	5	10	1	86.9
9	5	11	1	82.0
10	5	12	1	91.9
11	5	6	2	93.2[d]
12	5	6	3	92.8[d]
13	5	6	4	91.8[d]
14	5	6	5	91.5[d]
15	5	6	6	91.2[d]
16	5	6	7	91.0[d]
17	5	6	8	89.2[d]

[a] Reaction conditions: solid catalyst (48.22 mg, 0.013 mmol based on Ir from ICP), KOH (22.0 mg, 0.40 mmol), oxindole (0.266 mg, 2.0 mmol), alcohols (2.2 mmol), toluene (2 mL), reaction time 20 h., and reaction temperature 110 °C.

[b] Isolated yields (NMR Spectra of products refer to the supporting information).

[c] The amounts of solid catalysts [($IrCl_3$ (4.00 mg, 0.013 mmol) in entry 2, Me-SBA-15 (43.16 mg) plus $InIrCl_2$ (5.06 mg, 0.013 mmol) in entry 3 and Me-In-SBA-15 (44.22 mg) plus $IrCl_3$ (4.00 mg, 0.013 mmol) in entry 4].

[d] Recovered catalyst was used.

volume, obviously due to coverage of pore surface with trimethysilyl (-SiMe$_3$) groups and/or iridium complexes, leading to an increase of the wall thickness [32,33].

3.2. Catalytic property of the indene-functionalized mesoporous catalyst **5**

With the indene-functionalized mesoporous iridium catalyst Me-Ir-In-SBA-15 (**5**) in hand, we examined its C-3 alkylation of oxindole with alcohols according to the reported method in the literature [11]. It was found that oxindole was reacted with 1.1 equiv. of benzalcohol to give a C-3 alkylation product in 93.5% reaction yield, which was the nearly same as that of 94% isolated yield using [Cp*IrCl$_2$]$_2$ as a catalyst [11], and obvious higher than that of the parent catalyst IrCl$_3$ (entry 1 versus entry 2). On the basis of the about excellent result, the catalyst **5** was further investigated using a variety of alcohols as substrates (entries 5–10). In general, a variety of alcohols were smoothly reacted with oxindole to afford the corresponding desired products in high yields under similar conditions. Neither the significant effect of reactant using aromatic/aliphatic as substrates nor the obviously electronic effect of substituent on the aryl ring were observed for this catalytic reaction.

In order to explore the catalytic nature of the indene-functionalized mesoporous iridium catalyst and to eliminate the effect of non-covalent adsorption on catalytic process, two control experiments were carried out using Me-In-SBA-15 plus IrCl$_3$ and SBA-15 plus InIrCl$_2$ as catalysts under similar reaction conditions. It was found that the former afforded the corresponding product in 69.6% isolated yield, while the latter gave the corresponding product in 91.7% isolated yield (entries 3 and 4). Low activity in the former suggested that most of the postmodified IrCl$_3$ had not been coordinated on catalytic process, resulting in a low catalytic efficiency. High activity in the latter indicated that the catalyst *via* non-covalent adsorption on catalytic process also gave desired product in a high yield, suggesting the catalytic microenvironment had not been changed on catalytic process. However, both catalytic activities disappeared completely when employed both above catalysts after Soxlet extracting in toluene, indicating that the non-covalent adsorption of InIrCl$_2$ and unreacted starting materials of IrCl$_3$ on catalytic process could be eliminated through Soxlet extracting. This was also further proved by ICP analysis (both nearly same amounts of Ir were detected in solution after Soxlet extracting).

An important feature of design of the indene-functionalized mesoporous iridium catalyst is the easy and reliable separation *via* simple filtration. Taking benzalcohol as an example, upon completion of the reaction, Me-Ir-In-SBA-15 (**5**) was quantitatively recovered *via* filtration. As shown in entries 11–17 in Table 1, the recycling catalyst still afforded 89.2% yield after the eighth recycle, suggesting that the catalyst with highly ordered dimensional-hexagonal mesostructure is beneficial to uniform distribution catalytically active species and can avoid the loss of catalyst. The gradual decrease of catalytic activity as the catalyst was recycled was due to the slight loss of iridium catalyst, which was confirmed by ICP analyses.

4. Conclusion

In conclusion, we supplied a co-condensation approach to prepare an indene-functionalized mesoporous materials. The catalyst **5** synthesized through direct complexation of IrCl$_3$ with this materials exhibited high catalytic activity in C-3 alkylation of oxindole with various alcohols. More importantly, such a catalyst could be recovered and reused eight times without affecting obviously its reactivity, showing good potential in industrial application.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data to this article can be found online at doi:10.1016/j.catcom.2010.12.021.

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