Pyridine-derived ruthenium and platinum complexes immobilized on ordered mesoporous silica as catalysts for Heck vinylation

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Abstract

A comparative study of Heck vinylation of various aryl iodides with methyl acrylate, by either Ru(III) and Pt(IV) complexes immobilized on quinoline–carboimine functionalized FSM-16 (Ru-2QC, Pt-2QC) or Ru and Pt chlorides loaded impregnated on the same support (Ru-FSM, Pt-FSM) has been performed. The catalysts exhibit high activity and selectivity towards Heck vinylation of aryl iodides with methyl acrylate, even at low concentrations of metals (0.05–0.1 mol% with respect to aryl halide). Conversion of various aryl iodides of 100% within 3–8 h reaction and selectivity for *trans*-stereoisomer higher than 99% has been achieved over ruthenium containing catalysts (Ru-FSM, Ru-2QC). Platinum catalysts (Pt-FSM, Pt-2QC) were less active, resulting in the conversion of aryl iodides of 50–70%. The method of the modification influenced on heterogeneous character of reaction. The leaching was negligible in the case of Ru(III) and Pt(IV) complexes immobilized on quinoline–carboimine functionalized FSM-16, while metal species of transition metals supported on FSM-16 were leached to the solution.
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1. Introduction

Palladium catalyzed carbon–carbon bond formation reactions are powerful tools in organic synthesis, providing mild methods for the synthesis of valuable chemicals. An important example of such reaction is Heck vinylation of aryl halides with olefins. The most frequently used catalysts for Heck reactions are palladium complexes with phosphine or nitrogen chelating ligands; these catalysts have been widely studied, and several reviews describing the state of the art are available in literature [1–5]. Moreover, palladium supported on various supports [6–9] has been shown to catalyze efficiently Heck vinylation of aryl iodides and activated aryl bromides.

In contrast, there are very few reports on the use of metals other than palladium for Heck reactions. Platinum complex catalysts such as PtCl₂(COD)/PPh₃ (COD: cyclooctadiene) [10] and platinum triphenylphosphine trisulfonate sodium salt complex (Pt–TPPTS) as well as Rh–TPPTS, Ru–TPPTS, Ni–TPPTS, and Co–TPPTS complexes have been used in homogeneous catalysis for the vinylation of aryl iodides. The dehalogenation occurred rather than the vinylation over platinum complexes, especially in the presence of organic base, and rhodium complexes only catalyzed dehalogenation [11]. A similar effect of the base on the competition between the vinylation and the reduction has been published for Heck vinylation of 4-bromo-4′-hydroxybiphenyl using PPh₃/Pd(OAc)₂ [12]. Nickel was found to be quite active for Heck vinylation and a conversion of 89% was achieved for the reaction between bromobenzene and ethyl acrylate using a NiCl₂/PPh₃ catalytic system [13].

A number of supported metallic catalysts, such as Ni/HY zeolite, Ni/Al₂O₃, Co/Al₂O₃, and Cu/Al₂O₃, was also

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prepared [14,15]. Nevertheless, catalytic active species were extensively leached from these catalysts, and the catalysts were not recyclable [15]. Generally, the leaching of active metal species under reaction conditions in Heck vinylation is a serious drawback of metal-supported catalysts. There are several reports describing leaching phenomena of palladium-supported catalysts [6,7,16,17]. Careful combination of solvent, base, and thermal treatment of the Pd–zeolite was reported to prevent Pd leaching to the solution [17].

The immobilization of active palladium complexes on the support with nitrogen deriving ligands has been found to be an effective method for preventing palladium leaching [18–21]. Recently, we reported high efficiency of pyridine-derived palladium complexes immobilized on ordered silica for Heck vinylation of aryl halides with negligible leaching of palladium species to the solution during reaction [22]. They were prepared by building up a suitable ligand structure on the modified surface of FSM-16 mesoporous silica followed by complexation with palladium chloride.

In this work, we have examined the catalytic performance of Pt and Ru complexes immobilized on quinoline–carboimine functionalized FSM-16 mesoporous silica. In addition, supported Pt and Ru on FSM-16 were also prepared in order to compare catalytic behavior of immobilized and supported metal catalysts in Heck vinylation.

2. Experimental

2.1. Materials

FSM-16 was supplied from Toyota Central Laboratory. 3-Aminopropyltrimethoxysilane was purchased from Aldrich Japan. $RuCl_3 \cdot xH_2O$ and $H_2PtCl_6 \cdot 6H_2O$ were purchased from Wako Pure Chemical Industries Ltd. and Nacalai Tesque, Japan, respectively. All chemicals were used as received without further purification.

2.2. Preparation of Ru and Pt chloride catalysts supported on FSM-16

Ru^{3+}- and Pt^{4+}-containing catalysts were prepared by incipient wetness impregnation method. The required amounts of $RuCl_3 \cdot xH_2O$ and $H_2PtCl_6 \cdot 6H_2O$ for weight percent loading of metal on the support were dissolved in a minimal amount of acetone. Then, the solution was added drop-wise to dry FSM-16 and whole wet solid was stirred for 6 h. Finally, the catalysts were dried over night at 40 °C and calcined using a heating program as follows: RT-500 °C for 475 min and 500 °C for 420 min. To compare catalytic performance of Ru(III)-FSM and Ru(0)–FSM catalysts, Ru(0)-FSM catalyst was prepared by the reduction of cationic Ru(III) catalyst at 450 °C for 6 h in a flow of hydrogen. Similarly, Pt (IV)-FSM was reduced at 350 °C for 3 h in a flow of hydrogen to obtain Pt(0)-FSM.

2.3. Preparation of Ru(III) and Pt(IV) complexes on quinoline–carboimine functionalized FSM-16

Surface modification of FSM-16 with 3-aminopropyl-trimethoxysilane and subsequent preparation of a quinoline–carboimine ligand on FSM-16 by the reaction of 2-quinolinecarboaldehyde with 3-aminopropylated FSM-16 was performed according to standard methods, described in detail in our previous paper [22].

Appropriate amounts of $RuCl_3 \cdot xH_2O$ and $H_2PtCl_6 \cdot 6H_2O$ for the weight percent loading of metal into quinoline–carboimine functionalized FSM-16 were dissolved in acetone. Then, the functionalized FSM-16 was added to the prepared solution, and the suspension was stirred at room temperature for 6 h. Physisorbed metal species were removed by extraction with refluxing dichloromethane for 5 h.

2.4. Heck vinylation

In a typical Heck vinylation, aryl iodide (1 mmol), methyl acrylate (2 mmol), triethylamine (NEt_3, 2 mmol), *N*-methylpyrolidone (NMP, 5 ml), and the catalyst (0.1 mol% of metal complex against halide) were taken in a round bottom flask. The mixture was degassed by an argon flow followed by heating at 130 °C for required time. After the reaction, the reaction mixture was cooled to room temperature, and allowed to stand for about 10 min until the solid materials precipitated. Then, the liquid phase containing NMP, NEt_3, triethylammonium halide, substrates, and products was separated by decantation, and analyzed by GC using dodecane as an internal standard. Products were characterized by GC-MS analysis. The conversion was determined from the amount of aryl halide consumed in the reaction.

2.5. Leaching experiments

Leaching experiments were performed using 4-iodoanisole and methyl acrylate as reactants in Heck vinylation. A 1 ml of liquid phase was taken from the reaction mixture under hot conditions at the early stage of reaction (1–3 h) at low conversion of 4-iodoanisole. Fresh methyl acrylate was added to the filtrate and the reaction was continued for another 8 h and then, the product was analyzed by using GC. In addition, the amount of Ru as well as Pt in fresh and used catalysts were determined using ICP analysis.

2.6. Characterization of catalysts

X-ray diffractograms were recorded on a Shimadzu XRD-6000 diffractometer with Cu Kα radiation ($\lambda = 0.15418$ nm). Nitrogen adsorption isotherms were obtained at 77 K using a Belsorp 28SA apparatus (Bel Japan). TG/DTA measurements were performed on a Shimadzu DTG-50 apparatus. FT-IR spectra of Ru(III) and Pt(IV) complexes were recorded on

a Nexus 470 FT-IR spectrometer (Thermo Nicolet) by KBr technique. Elemental analysis by ICP was performed using a JICP-PS-1000UV analyzer (Leeman Labs Inc.).

3. Results and discussion

3.1. Characterization of catalysts

The XRD pattern of calcined FSM-16 shows characteristic low angle peaks attributed to d_{100}, d_{110}, and d_{200} reflections assuming a hexagonal lattice [23] (not shown). Both methods, the impregnation and the immobilization of transition metals on FSM-16, did not significantly affect the structure of ordered mesoporous silica and the periodicity of FSM-16 was maintained well in good agreement with previously reported results [22]. Relevant analytical and physicochemical properties of the catalysts are given in Table 1.

Decreasing surface area and pore volume of the catalysts compared with calcined FSM-16 are due to surface modification. Differences between Ru(III) and Pt(IV) complexes immobilized on quinoline–carboimine functionalized FSM-16 and Ru and Pt chlorides supported on FSM-16 are related to the method of the modification. However, BET surface area and pore volume of immobilized transition metal complexes (Ru-2QC, Pt-2QC) were approximately 60 and 30% of parent FSM, respectively. These results indicate that the pores of FSM-16 are occupied by the complex after the immobilization of pyridine derived Ru and Pt complexes. Nevertheless, the accessibility of substrates and products to active center in the mesopores is maintained after modification. BET surface area and pore volume of Ru- and Pt-
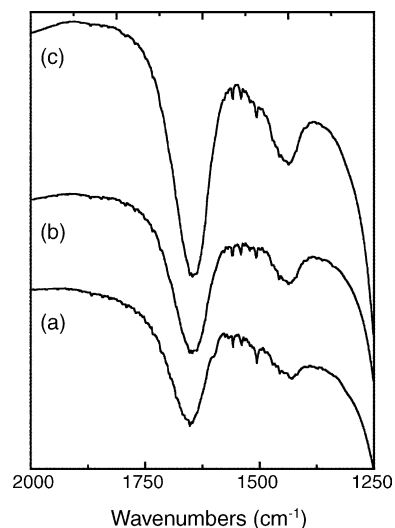

Fig. 1. FT-IR spectra in the region of 2300–1250 cm^{-1} for 2QC-FSM (a) (peaks at: 1650, 1598, 1563, 1506, and 1430 cm^{-1}), Ru-2QC (b) (peaks at 1635, 1563, 1505, and 1430 cm^{-1}) and Pt-2QC (c) (peaks at: 1636, 1598, 1563, 1506, and 1430 cm^{-1}).

FSM catalysts were not changed due to low metal loading to FSM.

The formation of the complexes by the reaction of $RuCl_3 \cdot xH_2O$ and $H_2PtCl_6 \cdot 6H_2O$ with quinoline–carboimine functionalized FSM-16 according to Scheme 1 was confirmed by FT-IR spectroscopy. Fig. 1 shows FT-IR spectra of quinoline–carboimine functionalized FSM-16 (2QC) in the range of 2300–1250 cm^{-1}. The infrared spectrum of functionalized FSM-16 with quinoline–carboimine shows a characteristic band at 1650 cm^{-1} (Fig. 1a) assignable the C=N

Table 1
Physicochemical properties of the catalysts

| Catalyst	Surface area[a] (m^2/g)	V_p[b] (cm^3/g)	APD[b] (nm)	Amount of ligand[c] (mmol/g catalyst)	M content[d] (wt.%)
FSM-16	997	0.472	3.0	–	–
Ru-2QC	397	0.336	2.87	0.99	0.59
Pt-2QC	429	0.360	2.88	0.99	0.48
Ru-FSM	1017	0.470	3.0	–	0.25
Pt-FSM	929	0.458	3.0	–	1.5
Pt-FSM$_{red}$[e]	935	0.460	3.0	–	1.5
Ru-FSM$_{red}$[f]	1000	0.468	3.0	–	0.25

[a] BET method.
[b] Kelvin equation (APD, average pore diameter).
[c] TG analysis.
[d] ICP analysis.
[e] Reduction with H_2 at 350 °C, 3 h.
[f] Reduction with H_2 at 450 °C, 6 h.
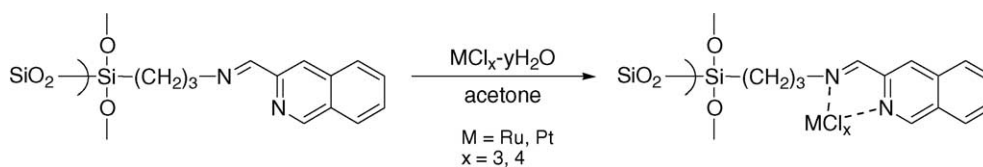
Scheme 1. Formation of transition metal complexes by the reaction of $MCl_x \cdot yH_2O$ (M = Ru and Pt, x = 3, 4) with quinoline–carboimine functionalized FSM-16.

double bond. After the complexation with $RuCl_3 \cdot xH_2O$ and $H_2PtCl_6 \cdot 6H_2O$, this band is shifted to $1635\,cm^{-1}$ (Fig. 1b and c); this is due to bond formation between metal and ligand. Similar shifting of the band to lower wavenumbers has been reported for palladium complex [20].

3.2. Heck vinylation

The activity of transition metal-complexes immobilized on FSM-16 (Ru-2QC, Pt-2QC) as well as Ru and Pt chlorides supported on FSM-16 (Ru-FSM and Pt-FSM) was examined in Heck reaction of aryl iodides with methyl acrylate in NMP as solvent using NEt_3 as a base. We found that NMP was effective as a solvent in Heck reaction by Pd-2QC supported on FSM-16 in our previous paper [22] as reported by Kaneda and co workers [24].

Table 2 summarizes typical results of Heck vinylation of aryl iodides with methyl acrylate over Ru-2QC and Ru-FSM catalysts. Various aryl iodides reacted with methyl acrylate to provide corresponding methyl cinnamates. 4-Iodoanisole gave 100% conversion over Ru-2QC and Ru-FSM catalysts (0.1 mol%) in 3 h and only methyl p-methoxycinnamate was formed with the regioselectivity of 100%. More hindered 1-iodonaphthalene was also coupled in 100% conversion over Ru containing catalysts (Ru-2QC and Ru-FSM) at molar concentration of 0.1% of ruthenium. Decrease in Ru concentration to 0.05 mol% against aryl iodide decreased the rate of reaction; however, the conversion of 4-iodotoluene and 4-iodoanisole in the range of 70–80% was achieved after 8 h reaction over both Ru catalysts (Ru-2QC and Ru-FSM). 1-Bromo-4-iodobenzene gave methyl 4-bromocinnamate in 70–80% yield over both Ru catalysts. Pd-2QC on FSM-16 afforded quantitative conversion to methyl 4-acetylcinnamate within 1.5 h at 130 °C in the case of 4-bromoactophenone [22]; however, no cinnamate was formed with both Pt-2QC and Ru-2QC catalysts. These results show that catalytic activity of Pt-2QC and Ru-2QC is much lower than that of Pd-2QC on FSM-16.

Pt catalysts (Pt-2QC and Pt-FSM) were less active than Ru catalysts (Table 2). Moderate activity was observed at molar

concentration of 0.1% of platinum. The activity of Pt-2QC was higher compared to Pt-FSM catalyst. The conversion of aryl iodides achieved over Pt-2QC was ca. 10–18% higher than that obtained over Pt-FSM. The activity of Ru-2QC was at the similar level as Ru-FSM as described above.

This difference can be most probably related to the influence of the metal and quinoline–carboimine ligand of the functionalized FSM-16. Nevertheless, it should be pointed out that the formation of dehalogenation products in yield around 5% was observed over Pt-FSM, whereas Pt-2QC gave only methyl cinnamates.

Turnover numbers (TON) in Table 2 were calculated typically as 24 h for Ru-2QC and 27 h for Pt-2QC. These values are smaller than the value 200 for Pd-2QC reported in our previous paper [22].

Two types of reaction mechanism were proposed in Heck reaction using Pd catalysts. One is a traditional Pd(0)/Pd(II) cycled mechanism. The other is recently suggested: the catalysis proceeds via a Pd(II)/Pd(IV) cycle [25–27]. Heck reaction catalyzed by Pd-2QC is assumed to proceed via the latter reaction mechanism due to the robust monomeric Pd(II) complex structure surrounded by strong N-ligands [24]. This is supported by leaching results reported previously [22].

We cannot have a conclusive discussion on the mechanism for Ru-2QC and Pt-2QC catalysis from our results. However, we can propose a possible mechanism with the electron rich Ru(III) and Pt(IV) complexes in the case of Ru-2QC and Pt-2QC catalysts: oxidative addition of aryl iodide to the complex, and the migration of aryl group to coordinated alkene followed by β-elimination gives the vinylation product.

Table 3 summarizes the influence of various bases on Heck vinylation of 4-iodoanisole for the most active Ru-2QC and Pt-2QC catalysts. The type of base used significantly influenced the catalytic behavior of both Ru-2QC and Pt-2QC catalysts. NEt_3 and sodium acetate were the best among used bases with respect to high activity of catalysts. Sodium carbonate was less effective than NEt_3: the conversion of 4-iodoanisole with sodium carbonate was only 16–19%. Other organic bases such as 1,2,3,4-tetrahydroquinoline and indoline were not effective for Heck vinylation using M-2QC

Table 2
Heck vinylation of aryl halides with methyl acrylate over M-2QC and M-FSM (M = Ru and Pt) catalysts[a]

Substrate	M (mol%)	Time (h)	Conversion (%)[b]			
			Ru-FSM	Pt-FSM	Ru-2QC	Pt-2QC
4-Iodoanisole	0.1	3	100	56	100	72
	0.05	8	60	13	68	30
4-Iodotoluene	0.05	8	80	33	84	45
1-Iodonaphtalene	0.10	8	100	15	100	28
	0.05	8	34	0	42	0.5
1-Bromo-4-iodobenzene	0.10	8	71	42	77	52
4-Bromoacetophenone	0.10	24	–	–	–	–

[a] Reaction conditions: aryl halide, 1 mmol; methyl acrylate, 2 mmol; catalyst, 0.1 mol% (M against aryl iodide); NEt_3, 4.0 mmol; NMP (solvent), 5 ml; temperature, 130 °C; period, 8 h.

[b] Liquid phase was analyzed by GC using biphenyl as an internal standard.

Table 3
Influence of base on catalytic activity of M-2QC (M = Ru and Pt) catalysts in Heck vinylation of 4-iodoanisole[a]

Base	Conversion (%)[b]	
	Ru-2QC	Pt-2QC
NEt$_3$	68	72
K$_2$CO$_3$	1.9	0
NaOAc	51	61
Na$_2$CO$_3$	16	19
Indoline	8.2	1.5
1,2,3,4-tetrahydroquinoline	0	0

[a] Reaction conditions: aryl halide 2 mmol; methyl acrylate 4.0 mmol; catalyst: Ru-2QC 0.010 g (0.05 mol% against 4-iodoanisole) or Pt-2QC 0.020 g (0.10 mol% against 4-iodoanisole); base 4.0 mmol; NMP(solvent) 5 ml; temperature 130 °C, period: 8 h for Ru-2QC or 3 h for Pt-2QC.
[b] Liquid phase was analyzed by GC using biphenyl as an internal standard.

catalysts. The results suggest that mild bases such as NEt$_3$ and sodium acetate are the best choice to obtain good activity of catalysts.

Similar influences of base as absorber of hydrogen halide have been described in the homogeneous Heck vinylation of aryl iodides catalyzed by platinum (Pt(COD)Cl$_2$/PPh$_3$) [10], ruthenium (Ru–TPPTS) [11] and palladium (Pd(OAc)$_2$/PPh$_3$) [12] complexes.

To ensure heterogeneous character of the catalysis, leaching experiments were performed with M-2QC and M-FSM (M = Ru and Pt) catalysts. Results observed for the Heck vinylation of the 4-iodoanisole with methyl acrylate together with ICP analysis of fresh and used catalysts are presented in Table 4.

Only slight differences in the conversion between suspension and filtrate were found over Ru-2QC and Pt-2QC catalysts (5.6% for Ru-2QC and 9.2% for Pt-2QC, respectively). As for metal supported on FSM-16 catalysts, strong leaching of active metal species into solution has been observed. Differences in the conversions of Ru- and Pt-FSM between the suspension at split time and the filtrate after 8 h reaction (41 and 32%, respectively) indicate that the reaction over these catalysts occurs mainly in homogeneous phase. Reduction of cationic Ru(III) and Pt(IV) catalysts did not

suppress the leaching of metal species into solution, either. Differences of the conversion between the suspension and the filtrate for cationic and reduced catalysts were almost in the same level. In addition, the amount of remaining metal (Ru and Pt) on the catalysts after reaction was determined by ICP analysis to confirm the results of leaching experiments. Almost no differences in metal content have been found for M-2QC (M = Ru and Pt) catalysts recovered after the catalysis, whereas Ru- and Pt-FSM catalysts recovered contained lower metal amounts than fresh catalysts. Nevertheless, such a decrease is not always proportional to high activity of the filtrate. This is due to the fact that dissolved metal species are re-adsorbed at the surface of the support through a dissolution-readsorption equilibrium upon cooling of the reaction mixture. Similar phenomena have been observed for palladium, nickel and cobalt supported catalysts [6,14,16]. These results show quite different features of immobilized and metal supported catalysts; the immobilization of the active metal species (Ru-2QC and Pt-2QC) on modified support seems to be an efficient method for avoiding this undesirable loss of active species.

We consider the activity of impregnated M-FSM (M = Ru and Pt) catalysts for Heck vinylation is mainly due to the activity of leached active species and the reaction occurred in homogeneous phase. The leaching of metal species in NMP solvents was described in some papers [16,28]. Fine Ru and Pt particles on FSM-16 were stabilized by the solvation of NMP, and leached as "cluster" from the support. These clusters work as the catalysts in homogeneous phase.

The activity of Ru-2QC and Ru-FSM catalysts in Heck vinylation of 4-iodoanisole with methyl acrylate in recycling experiments is shown in Fig. 2. The activity of Ru-2QC was nearly constant during three recyclings, whereas the activity of impregnated Ru-FSM and reduced Ru(0)-FSM dropped after second recycling. For all catalysts, the conversion of 4-iodoanisole of 100% has been achieved with fresh catalyst, whereas the conversion of only 35% has been observed in second recycling using Ru(0)-FSM. Similar decrease in the activity was also observed in the case of Pt(0)-FSM. The significant decrease in metal amount was found for the recovered

Table 4
Comparison of the leaching level between M-2QC and M-FSM (M = Ru and Pt) catalysts in Heck vinylation of 4-iodoanisole[a]

Catalyst	Conversion (%)			M (mmol)10^{-2}/g catalyst[c]	
	Suspension (split time (h))	Filtrate (time (h))	Difference	Fresh catalyst	Used catalyst
Ru-2QC	34 (2)	40(8)	6	5.9	5.8
Pt-2QC[b]	29 (1)	39(8)	10	2.5	2.4
Ru-FSM	9.6 (3)	56(8)	46	2.4	1.4
Pt-FSM[b]	9.6(1)	41(8)	31	8.2	7.8
Pt-FSM$_{red}$[d]	10 (1)	39(8)	29	8.2	7.7
Ru-FSM$_{red}$[e]	25 (3)	49(8)	24	2.4	1.3

[a] Reaction conditions: aryl halide 2 mmol; methyl acrylate 2.5 mmol; catalyst 0.05 mol%(M against 4-iodoanisole); Et$_3$N 2 mmol; NMP (solvent) 5 ml; temperature 130 °C.
[b] Pt 0.1 mol%.
[c] Determined by ICP.
[d] Reduction with H$_2$ at 350 °C for 3 h.
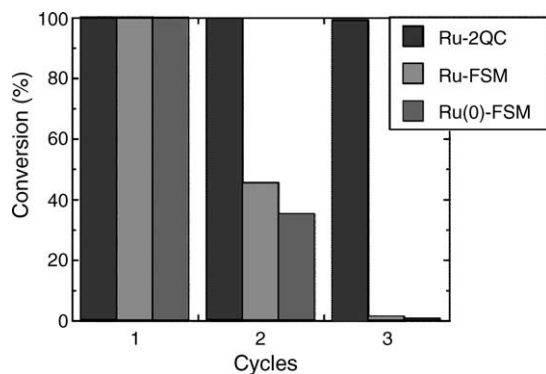[e] Reduction with H$_2$ at 450 °C for 6 h.

Fig. 2. Recycling of Ru-2QC, Ru-FSM, and Ru(0)-FSM catalysts in the reaction of 4-iodoanisole with methyl acrylate. Reaction conditions: 4-iodoanisole, 2 mmol; methyl acrylate, 4 mmol; Et$_3$N, 4 mmol; catalyst, 0.1 g; solvent, NMP 5 ml; temperature, 130 °C; period, 3 h.

catalysts from third recycling (0.046×10^{-2} mmol of Ru/g-catalyst for Ru(0)-FSM and 0.1×10^{-2} mmol of Pt/g-catalyst for Pt(0)-FSM). These results obtained from recycling experiments also support the efficiency and the advantage of the immobilization of active metal species as a complex on modified support over impregnation method.

4. Conclusions

Ru- and Pt-quinoline–carboimine complexes immobilized on FSM-16 mesoporous silica (Ru-2QC and Pt-2QC) have been found to be active catalysts for Heck vinylation of aryl iodides. Various aryl iodides can be coupled efficiently with methyl acrylate to corresponding cinnamates in 100% conversion with complete stereoselectivity over Ru-2QC catalyst within 3–8 h reaction at low molar concentration of Ru (0.1 mol%). Pt-2QC catalysts showed moderate activity for studied reaction with the conversion of aryl iodides of 50–70%. Only negligible leaching has been observed for M-2QC (M = Ru and Pt) catalysts.

Similar high activity was observed using Ru and Pt supported on FSM-16. However, the leaching of active metal species into the solution has been observed despite low loading of metal onto the support, and the reaction occurs mainly in homogeneous phase.

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