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Communication

Application of polyethyleneglycol (PEG) functionalized ionic liquids for the rhodium-catalyzed hydrosilylation reaction of alkenes

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ABSTRACT

$Rh(PPh_3)_3Cl$–polyethyleneglycol (PEG) functionalized ionic liquids with various anions were used as a catalytic system for the hydrosilylation reaction of alkenes. The influence of the anion of the ionic liquid has been investigated. It was found that the anion has an impact on the catalytic activity and selectivity. $[PEG_{400}DIL][PF_6]$-$[Rh(PPh_3)_3Cl]$ shows an improved catalytic performance towards the hydrosilylation reaction of alkenes. The scope of alkenes and recycling of the catalytic system have been investigated.

1. Introduction

Hydrosilylation is one of the most powerful methods used for the synthesis of functionalized organosilicon compounds. Although a number of catalysts have been tested in the hydrosilylation process, most research and industrial syntheses have been carried out using platinum or rhodium complexes due to the fact that these complexes often exhibit high catalytic activity [1–3]. In addition, to promote the activity of the catalyst and selectivity of the adduct, a variety of ligands such as new phosphine ligands [4–8] and nitrogen-based ligands [9–11] have been used in the hydrosilylation process.

Ionic liquids as ligands or reaction media have been applied in many chemical fields [12–14]. Ionic liquids can promote the activity of a rhodium catalyst and the selectivity of the adducts in the hydrosilylation reaction of alkenes as well as improving there cyclability of the catalytic system [15–18]. Monomethoxy polyethylene glycol functionalized ionic liquids and transition metal complexes can form co-catalytic systems, which can increase the activity of the catalyst, promote the selectivity of the products and allow the recycling of the catalyst system [19]. Very recently, our group reported that a polyethylene glycol functionalized ionic liquid with $[PF_6]^-$ anion can be used as the ligand in the hydrosilylation reaction of alkenes catalyzed by a Rh catalyst [20]. Herein, we report the influence of the anions of novel polyethylene glycol functionalized ionic liquids (Fig. 1) on the hydrosilylation reaction of alkenes catalyzed using $Rh(PPh_3)_3Cl$.

2. Experimental

2.1. *Procedure for the synthesis of $[PEG_{400}DIL][X]$ [$X = PF_6$, BF_4, SO_3CF_3 and $N(SO_2CF_3)_2$]*

The chlorination of PEG_{400} and PEG_{400}-functionalized double imidazolium chloride ($PEG_{400}DI$) was accomplished using a literature procedure [20].

$PEG_{400}DIL[PF_6]$: $PEG_{400}DI$ (3.0 g, 5 mmol) was dissolved in deionized H_2O (10 mL) and added to a solution of NH_4PF_6 (1.6 g, 10 mmol) in H_2O (5 mL). The reaction mixture was stirred at room temperature for 6 h. After decantation, the oily crude product obtained was diluted with dichloromethane (30 mL), washed with H_2O (4 × 20 mL) and then dried over anhydrous magnesium sulfate. Removal of the solvent under vacuum afforded PEG_{400}-$DIL[PF_6]$ (3.8 g, 92%) as a yellow oil. ^1H NMR (400 MHz, CD_3OD), δ (ppm): 8.90 (s, 2H, 2NC*H*N), 7.63 (s, 4H, 2C*H*C*H*), 4.37 (t, $J = 13.4$ Hz, 4H, $2NCH_2$), 3.85 (s, 6H, NCH_3), 3.77–3.74 (m, 4H, OCH_2CH_2N), 3.66–3.60[m, $(OCH_2CH_2)_n$]. ^{13}C NMR (100.6 MHz, CD_3OD), δ (ppm): 137.2(NCHN), 123.1(NCHCHN), 122.7(NCHCHN), 70.1[$(CH_2CH_2O)_n$], 70.0[$(CH_2CH_2O)_n$], 69.9[$(CH_2CH_2O)_n$], 68.3(NCH_2CH_2O),

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49.4(N$CH_2$$CH_2$O), 35.2(N$CH_3$).

PEG_{400}DIL[BF_4]: The PEG_{400}DI (3.0 g, 5 mmol) obtained was dissolved in deionized H_2O (10 mL) and added to a solution of NH_4BF_4 (1.1 g, 10 mmol) in H_2O (5 mL). The mixture was stirred at room temperature for 6 h. After decantation, the oily crude product obtained was diluted with dichloromethane (30 mL), washed with H_2O (4 × 20 mL), and then dried over anhydrous magnesium sulfate. Removal of the solvent under vacuum afforded a yellow oil PEG_{400}-DIL[BF_4] (2.8 g, 78% yield). ^1H NMR (400 MHz, CD_3OD), δ (ppm): 8.90 (s, 2H, 2NC*H*N), 7.63 (s, 4H, 2C*H*C*H*), 4.37 (t, J = 13.4 Hz, 4H, 2NCH_2), 3.85(s, 6H, NCH_3), 3.77–3.74(m, 4H, OC$H_2$$CH_2$N), 3.66–3.60[m, (OC$H_2CH_2$)$_n$]. ^{13}C NMR (100.6 MHz, CD_3OD), δ (ppm): 137.2(N*C*HN), 123.1(N*C*HCHN), 122.7(NCH*C*HN), 70.1[(*C*H_2*C*H_2O)$_n$], 70.0[(*C*H_2*C*H_2O)$_n$], 69.9[(*C*H_2*C*H_2O)$_n$], 68.3(NCH_2*C*H_2O), 49.4(N*C*$H_2$$CH_2$O), 35.2(N*C*$H_3$).

PEG_{400}DIL[SO_3CF_3]: The PEG_{400}DI (3.0 g, 5 mmol) obtained was dissolved in deionized H_2O (10 mL) and added to a solution of $NH_4SO_3CF_3$ (1.7 g, 10 mmol) in H_2O (5 mL). The mixture was stirred at room temperature for 6 h. After decantation, the oily crude product obtained was diluted with dichloromethane (30 mL), washed with H_2O (4 × 20 mL), and then dried over anhydrous magnesium sulfate. Removal of the solvent under vacuum afforded a yellow oil PEG_{400}-DIL[SO_3CF_3] (3.3 g, 78% yield). ^1H NMR (400 MHz, DMSO), δ (ppm): 9.05 (s, 2H, 2NC*H*N), 7.68(s, 4H, 2C*H*C*H*), 4.34(t, J = 9.7 Hz, 4H, 2NCH_2), 3.87(s, 6H, NCH_3), 3.78–3.76(m, 4H, OC$H_2$$CH_2$N), 3.55–3.50[m, (OC$H_2CH_2$)$_n$]. ^{13}C NMR (100.6 MHz, CD_3OD), δ (ppm): 136.7(N*C*HN), 123.2(N*C*HCHN), 122.6(NCH*C*HN), 70.5[(*C*H_2*C*H_2O)$_n$], 69.7[(*C*H_2*C*H_2O)$_n$], 69.5 [(*C*H_2*C*H_2O)$_n$], 68.0(NCH_2*C*H_2O), 48.7(N*C*$H_2$$CH_2$O), 35.4(N*C*$H_3$).

PEG_{400}DIL[$N(SO_2CF_3)_2$]: The PEG_{400}DI (2.9 g, 5 mmol) obtained was dissolved in deionized H_2O (10 mL) and added to a solution of $LiN(SO_2CF_3)_2$ (2.9 g, 10 mmol) in H_2O (5 mL). The mixture was stirred at room temperature for 6 h. After decantation, the oily crude product obtained was diluted with dichloromethane (30 mL), washed with H_2O (4 × 20 mL), and then dried over anhydrous magnesium sulfate. Removal of the solvent under vacuum afforded a yellow oil PEG_{400}DIL[$N(SO_2CF_3)_2$] (3.2 g, 62% yield). ^1H NMR (400 MHz, CD_3OD), δ (ppm): 8.72(s, 2H, 2NC*H*N), 7.47(s, 4H, 2C*H*C*H*), 4.31(t, J = 9.1 Hz, 4H, 2NCH_2), 3.85(s, 6H, NCH_3), 3.79–3.77(m, 4H, OC$H_2$$CH_2$N), 3.70–3.64[m, (OC$H_2CH_2$)$_n$]. ^{13}C NMR (100.6 MHz, CD_3OD), δ (ppm): 136.8(N*C*HN), 123.4(N*C*HCHN), 121.6(NCH*C*HN), 71.4[(*C*H_2*C*H_2O)$_n$], 70.7[(*C*H_2*C*H_2O)$_n$], 70.4 [(*C*H_2*C*H_2O)$_n$], 68.5(NCH_2*C*H_2O), 49.8(N*C*$H_2$$CH_2$O), 36.3(N*C*$H_3$).

2.2. The catalytic hydrosilylation reaction

All catalytic reaction was performed in a 10 mL flat-bottomed tube without protection from air. The alkenes (4.0 mmol) and a requisite amount of catalyst and ionic liquid were placed in a dried tube and the reaction mixture stirred for 5 min. Thereafter, the silane (4.4 mmol) was added, the resulting reaction mixture heated with stirring for a requisite time and then allowed to cool to room temperature. The product phase was separated by decantation and the conversion of the alkenes and selectivity determined by GC-MS analysis using an Agilent 26890N/59731 apparatus equipped with a DB-5 column (30 m × 2.5 mm × 0.25 μm).

3. Results and discussion

3.1. The effect of the anion on the rhodium-catalyzed hydrosilylation reaction of styrene

Initially, the hydrosilylation reaction of styrene catalyzed using $Rh(PPh_3)_3Cl$ in the presence of various ionic liquids was conducted. The effect of the amount of ionic liquid on the hydrosilylation reaction catalyzed by $Rh(PPh_3)_3Cl$ was investigated and the results summarized in Table 1. A styrene conversion of 78.4% was achieved in the hydrosilylation reaction catalyzed using [$Rh(PPh_3)_3Cl$] and the selectivity for the β-adduct was only 64.9% (Table 1, entry 1). Under the same reaction conditions, the catalytic activity of [PEG_{400}DIL][PF_6]-Rh and the selectivity for the β-adduct were increased [20]. When [PEG_{400}DIL][BF_4]-[$Rh(PPh_3)_3Cl$] was used as the catalyst, the best selectivity for the β-adduct was 85.9% with a 90.7% conversion of styrene (Table 1, entry 9). Furthermore, using [PEG_{400}DIL][SO_3CF_3]-[$Rh(PPh_3)_3Cl$] and [PEG_{400}DIL][$N(SO_2CF_3)_2$]-[$Rh(PPh_3)_3Cl$] as the catalyst, the best selectivity was 80.4% and 76.4% with conversions of styrene of 91.0% and 86.6%, respectively (Table 1, entry 16 and 22). The presence of the PEG chain in the ionic liquids molecules suppressed the hydrogenation and dehydrogenative silylation side reactions [20]. The type of anion can also influence the activity of catalyst and the selectivity of adduct.

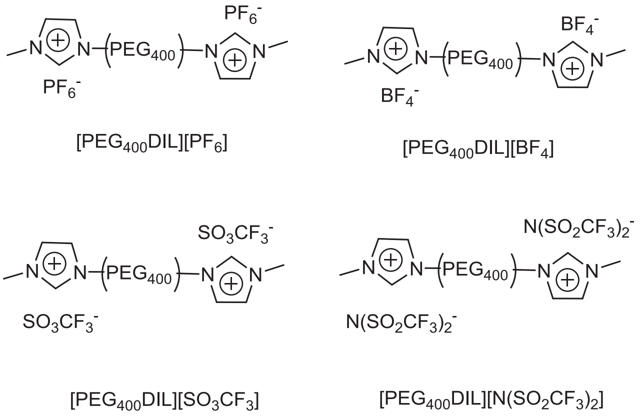


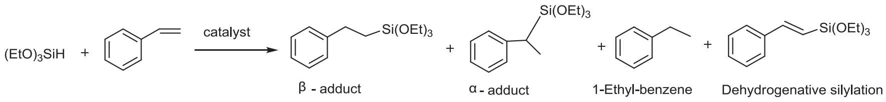
Fig. 1. Structure of ionic liquids.

The effect of the anion in the polyethylene glycol(400) functionalized ionic liquid on the reaction was investigated and the curve of reaction with time shown in Fig. 2. In the initial stage of the reaction, the three catalytic systems of [PEG_{400}DIL][X]-[$Rh(PPh_3)_3Cl$] except [PEG_{400}DIL][$N(SO_2CH_3)$]-[$Rh(PPh_3)_3Cl$] gave high initial turnover rates and the activity of [PEG_{400}DIL][SO_3CF_3]-[$Rh(PPh_3)_3Cl$] found to be highest. Meanwhile, the $Rh(PPh_3)_3Cl$ catalyst without an ionic liquid was lower than the other catalytic systems studied. During the intermediate stage of the reaction, [PEG_{400}DIL][PF_6]-[$Rh(PPh_3)_3Cl$] showed better activity than the other catalysts studied. However, both [PEG_{400}DIL][PF_6]-[$Rh(PPh_3)_3Cl$] and [PEG_{400}DIL][BF_4]-[$Rh(PPh_3)_3Cl$] displayed similar selectivity for the β-adduct. Though all the PEG functionalized ionic liquids tested can increase the activity of the rhodium catalyst and the selectivity towards the β-adduct in the hydrosilylation reaction of styrene using triethoxysilane, the [PEG_{400}DIL][SO_3CF_3]-[$Rh(PPh_3)_3Cl$] catalyst system showed the best activity, but the final yield of adduct was lower.

3.2. The effect of reaction temperature on the hydrosilylation reaction

With different anions, the ionic liquids have different properties including solubility, melting point and mobility. These properties have certain relationships with temperature. The effect of temperature on the hydrosilylation reaction of alkenes was investigated and the results shown in Fig. 3. Using an ionic liquid with the PF_6^- anion and rhodium catalytic system, the reaction yield reached its maximum at 80 °C. However, the [PEG_{400}DIL][$N(SO_2CF_3)_2$]-[$Rh(PPh_3)_3Cl$]system needed the highest reaction temperature for

Table 1
Effect of anions of ionic liquids on hydrosilylation catalyzed by $Rh(PPh_3)_3Cl$.



Entry	Ionic liquid	$Rh(PPh_3)_3Cl$/Ionic liquid	Conv. (%)	Selectivity (%)				β/α	TON
				β	α	1-Ethyl-benzene	Dehydrogenative silylation		
1	–	–	78.4	64.9	12.0	11.0	12.1	5.4	1568
2	[PEGDIL][PF_6]	1:2	98.5	77.7	8.4	6.7	7.2	9.2	1970
3		1:3	100	83.1	7.8	4.8	4.3	10.7	2000
4		1:5	98.9	83.6	5.4	5.4	5.6	15.5	1978
5		1:10	95.2	84.9	4.8	4.9	5.4	17.7	1904
6		1:30	89.6	78.9	7.6	6.6	6.8	10.3	1792
7		1:80	79.1	75.8	13.6	3.9	6.7	5.6	1582
8	[PEGDIL][BF_4]	1:2	88.6	76.2	9.0	7.7	7.0	8.5	1772
9		1:3	90.7	85.9	4.8	5.5	3.7	17.9	1814
10		1:5	83.9	79.7	12.6	3.3	4.4	6.3	1678
11		1:10	86.3	79.5	6.9	7.5	5.9	11.5	1726
12		1:30	67.7	84.5	5.0	5.8	4.7	16.9	1354
13		1:80	49.7	79.9	4.6	8.2	6.8	17.4	994
14	[PEGDIL][SO_3CF_3]	1:2	99.6	65.2	11.2	14.3	9.2	5.8	1992
15		1:3	99.7	65.5	7.8	19.6	6.9	8.4	1994
16		1:5	91.0	80.4	3.8	10.9	4.7	20.9	1820
17		1:10	97.5	80	4.4	12.2	3.5	18.1	1950
18		1:30	89.7	79.3	4.2	12.5	4.1	18.7	1794
19		1:80	65.3	73.5	5.7	27.8	5.5	13.0	1306
20	[PEGDIL][$N(SO_2CF_3)_2$]	1:1	87.7	69.8	7.4	14.7	8.1	9.4	1754
21		1:3	83.5	72.0	7.3	11.6	9.2	9.7	1670
22		1:5	86.6	76.4	6.6	8.9	8.1	11.6	1732
23		1:10	96.4	75.4	6.8	7.5	10.3	11.1	1928
24		1:30	89.7	65.6	5.6	19.1	9.7	11.7	1794
25		1:80	81.5	62.5	10.1	18.3	9.3	6.2	1630

Reaction conditions: styrene 4.0 mmol, $(EtO)_3SiH$ 4.4 mmol, 5 h, 90 °C, Rh is 0.05 mol% based on styrene.

attaining the maximum of yield in the hydrosilyaltion reaction of styrene.

3.3. The hydrosilylation reaction of alkenes using triethoxysilane

To investigate the effect of ionic liquids with various anions on hydrosilylation reaction of different alkenes, a series of alkenes in the presence of triethoxysilane were investigated in the hydrosilylation reaction catalyzed by $PEG_{400}DIL[X]$- $[Rh(PPh_3)_3Cl]$. The results indicated that different anions in the polyethylene glycol functionalized ionic liquid exhibited different effects on the activity and selectivity in the hydrosilylation reaction of various alkenes (Table 2).

For the hydrosilylation of 1-hexene, the $[PEG_{400}DIL][N(SO_2CF_3)_2]$ based catalyst system (Table 2, entry 4) was superior to the other ionic liquids studied. All the ionic liquids bearing different anions showed excellent catalytic performance for the hydrosilylation of 1-octene. When $[PEG_{400}DIL][SO_3CF_3]$-

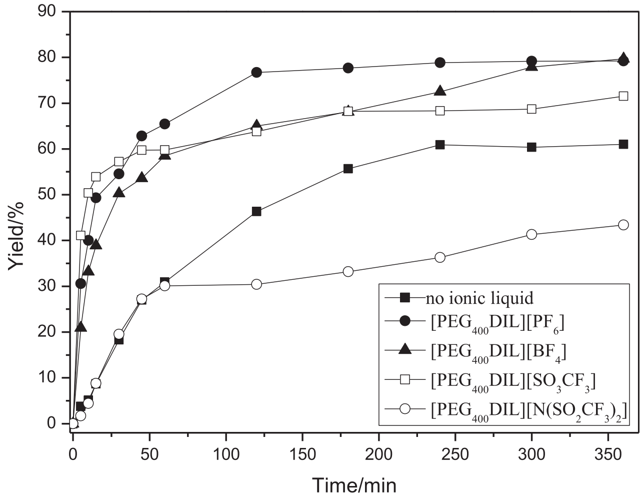


Fig. 2. Reaction curve with different ionic liquid-$[Rh(PPh_3)_3Cl]$. Reaction conditions: styrene 4.0 mmol, $(EtO)_3SiH$ 4.4 mmol, 5 h, 90 °C, Rh is 0.05 mol% based on styrene.

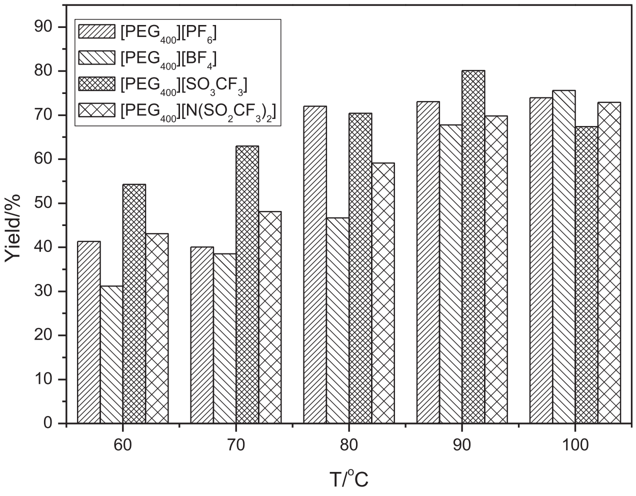


Fig. 3. Temperature-dependence curve for the hydrosilylation of styrene using different IL. Reaction conditions: styrene 4.0 mmol, $(EtO)_3SiH$ 4.4 mmol, 5 h, Rh is 0.05 mol% based on styrene.

Table 2
Hydrosilylation of different alkenes with triethoxysilane.

Entry	Alkenes	Ionic liquids	Conv. (%)	Selec. of β-adduct (%)	TON
1	1-Hexene	[PEGDIL][PF_6]	100	93.5	2000
2		[PEGDIL][BF_4]	100	95.8	2000
3		[PEGDIL][SO_3CF_3]	90.8	89.2	1816
4		[PEGDIL][$N(SO_2CF_3)_2$]	92.3	97.9	1846
5	1-Octene	[PEGDIL][PF_6]	92.6	98.2	1852
6		[PEGDIL][BF_4]	92.1	100	1824
7		[PEGDIL][SO_3CF_3]	96.2	99.5	1924
8		[PEGDIL][$N(SO_2CF_3)_2$]	91.1	96.4	1822
9	1-Decene	[PEGDIL][PF_6]	84.9	100	1698
10		[PEGDIL][BF_4]	100	98.7	2000
11		[PEGDIL][SO_3CF_3]	82.9	100	1628
12		[PEGDIL][$N(SO_2CF_3)_2$]	71.6	100	1432
13	3,3- Dimethyl-1-butene	[PEGDIL][PF_6]	95.4	100	1908
14		[PEGDIL][BF_4]	98.0	99.7	1916
15		[PEGDIL][SO_3CF_3]	99.9	99.9	1998
16		[PEGDIL][$N(SO_2CF_3)_2$]	100	99.6	2000
17	Styrene	[PEGDIL][PF_6]	100	82.7	2000
18		[PEGDIL][BF_4]	94.8	84.1	1896
19		[PEGDIL][SO_3CF_3]	98.5	86.3	1970
20		[PEGDIL][$N(SO_2CF_3)_2$]	96.4	75.4	1928
21	Allylbenzene	[PEGDIL][PF_6]	91.2	100	1824
22		[PEGDIL][BF_4]	97.0	100	1940
23		[PEGDIL][SO_3CF_3]	83.4	90.3	1638
24		[PEGDIL][$N(SO_2CF_3)_2$]	89.7	98.7	1794
25	Ethyl vinyl ether	[PEGDIL][PF_6]	97.9	99.4	1958
26		[PEGDIL][BF_4]	100	99.9	2000
27		[PEGDIL][SO_3CF_3]	99.5	99.5	1990
28		[PEGDIL][$N(SO_2CF_3)_2$]	99.9	99.9	1998
29	Isobutyl vinyl ether	[PEGDIL][PF_6]	94.2	100	1884
30		[PEGDIL][BF_4]	99.3	100	1986
31		[PEGDIL][SO_3CF_3]	100	96.4	2000
32		[PEGDIL][$N(SO_2CF_3)_2$]	92.4	99.0	1848

Reaction conditions: alkene 4.0 mmol, $(EtO)_3SiH$ 4.4 mmol, 5 h, 90 °C, $Rh(PPh_3)_3Cl$ is 0.05 mol% based on alkene, n(Rh)/n(PEG_{400}DIL[X]) = 1:10.

[$Rh(PPh_3)_3Cl$] was used as the catalyst system, a 96.2% conversion was obtained with 99.5% selectivity for the β-adduct. Meanwhile, a 92.1% conversion with 100% selectivity for the β-adduct was obtained using [PEG_{400}DIL][BF_4]-[$Rh(PPh_3)_3Cl$] as the catalyst. For 3,3-dimethyl-1-butene, a >99% selectivity and >98% conversion were attained by the catalyst system comprised of four kinds of ionic liquids and $Rh(PPh_3)_3Cl$, respectively. For aromatic alkenes, allylbenzene as substrate gave better selectivity for the β-adduct when compared to styrene, which was attributed to the electronic effects of the benzene ring and the conjugation effect of the allyl substituent that can influence the electron density of the olefinic bond to favor the anti-markovnikov addition reaction to give the β-adduct. Similar excellent results were obtained in the hydrosilylation reaction of vinyl ether catalyzed using different ionic liquids and $Rh(PPh_3)_3Cl$. The best conversion and selectivity was 100% and 99.9%, respectively, which was obtained in the hydrosilylation reaction of ethyl vinyl ether in the presence of triethoxysilane using [PEG_{400}DIL][BF_4]-[$Rh(PPh_3)_3Cl$] as the catalyst system.

3.4. Recycling of PEG_{400}DIL[X]–[$Rh(PPh_3)_3Cl$] in the hydrosilylation reaction of 1-octene

In recycling experiments, the ionic liquids and catalyst phase were allowed to separate from the reaction system by gravity after each run. The catalytic system without any further treatment was used for the next cycle. The results are listed in Fig. 4. The PEG_{400}DIL [PF_6]-[$Rh(PPh_3)_3Cl$] catalyst can be recycled 5 times with no significant loss in activity. However, PEG_{400}DIL[$N(SO_2CF_3)_2$]-[$Rh(PPh_3)_3Cl$] could only be reused 3 times and its catalytic activity declined rapidly. The other two systems can be recycled 3 and 4 times, respectively. The results showed that the PEG_{400}DIL[PF_6] ionic liquid was more capable of protecting the [$Rh(PPh_3)_3Cl$] catalyst from leaching out of the reaction system.

4. Conclusions

In summary, polyethylene glycol(400) functionalized imidazolium ionic liquids bearing different anions have been synthesized and used in the hydrosilylation reaction of alkenes catalyzed using a rhodium complex. All of the catalyst systems can be applied in the hydrosilylation reaction of different linear aliphatic and aromatic

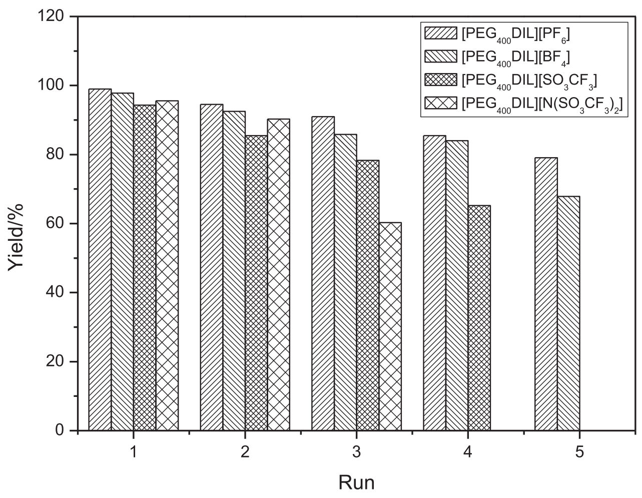


Fig. 4. Recyclability of catalyst systems.

alkenes in the presence of triethoxysilane. Of the four catalyst systems, $PEG_{400}DIL[PF_6]$-$[Rh(PPh_3)_3Cl]$ proved the best and showed excellent activity, selectivity (for the β-adduct) and recyclability in the hydrosilylation reaction.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jorganchem.2015.06.024.

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