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Polyamine Anchored Palladium Catalyst for Suzuki–Miyaura and One-Pot *O*-Alkylation-Suzuki Reactions

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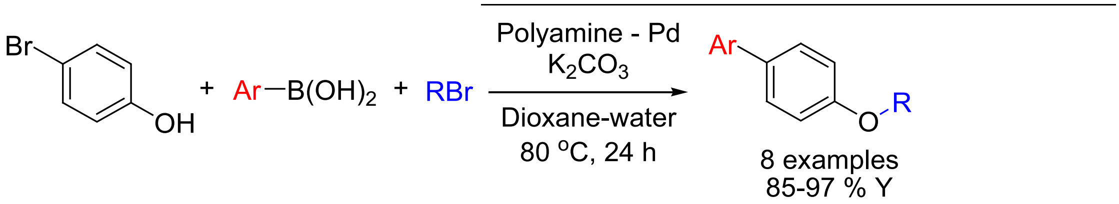
Abstract Polymer anchored amine–palladium complexes were screened as catalysts for Suzuki–Miyaura coupling reactions of aryl halides. The robust, recyclable catalyst was effective in this reaction of aryl bromides and iodides. Aryl bromide was found to undergo selective Suzuki–Miyaura reaction with phenyl boronic acid, even in presence of styrene. The catalyst system was further employed for one-pot *O*-alkylation-Suzuki reaction to prepare alkoxy biaryl systems in good conversions.

Graphical Abstract



Keywords Polyamine · Palladium · Heterogeneous catalysis · Suzuki · One-pot reaction

1 Introduction

Synthetic transformations induced by transition metal assisted activations are important reactions in the armory of modern chemists. In general, the catalyst is either homogeneous or heterogeneous, each having its own advantage. Even though the former category of catalyst is believed to be more efficient due to its efficient interaction with reactants, there are still some distinct advantages of the latter type. The advantages of heterogeneous metal catalysts include easy separation from the reaction mixture, reusability of the catalysts, less contamination of the toxic metal ions in the products, cost benefits and some environmental considerations. Processes using the heterogeneous catalysts can be modified into continuous flow procedures for the practical applications.

The key aspect of the development of heterogeneous catalyst involves loading of transition metal ion onto the solid surface. This concept of loading catalyst on solid support involves attaching a suitable ligand on the appropriate surface and then exposing it to metal ions to form anchored complex for heterogeneous catalysis [1–5]. Adequate care needs to be taken during the process of making the heterogeneous catalysts. The activity of the metal complex needs to be retained in order to perform the role of the catalyst. It therefore offers an access to the reacting species and prevents leaching of the metal ions during the reaction as well as the work-up steps. It also maintains the reusability for subsequent catalytic cycles. Organic polymers are often chosen as supports to anchor metal ions to prepare heterogeneous catalysts. The development in the field of polymer chemistry has lead to the availability of polymeric materials which are

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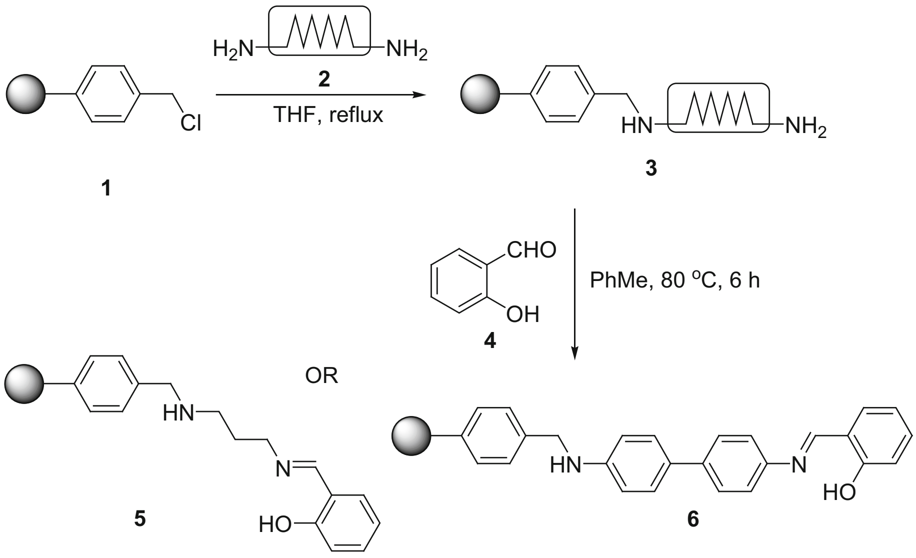
Scheme 1 Synthesis of polymer anchored amino ligands

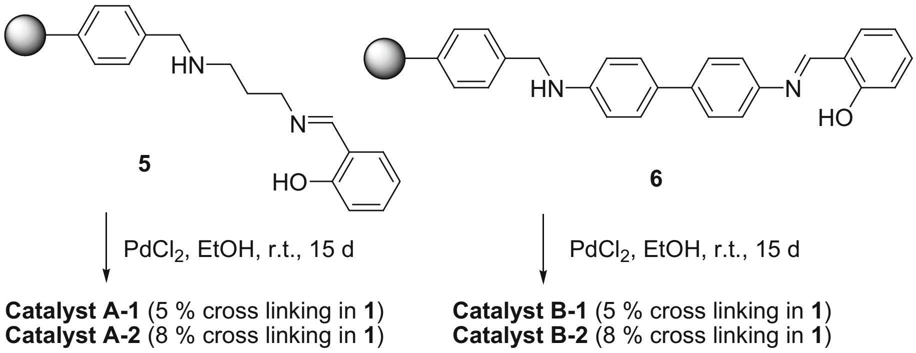


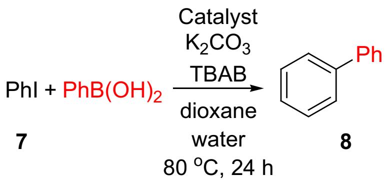
insoluble in many organic solvents. The materials can be easily functionalized and its porosity can be tuned to modify permeability. The area of polymer-anchored metal complexes and their applications as heterogeneous catalysts for many useful organic transformations is widely investigated [6–14]. Since the pioneering efforts by Merrifield for the polymer supported peptide synthesis [15], several noteworthy developments have been reported on the use of different polymeric materials as support for the synthesis and catalysis [16–19]. These include the use of soluble polymer support [20, 21], dendrimers [22–24], polysiloxanes [25, 26], self-supported polymeric catalysis [27–29], nano-particles [30, 31], metal ions anchored on clays and zeolites [32, 33], metal oxides and mesoporous materials [34, 35] etc. More recently new supports such as graphine [36] and metal organic frameworks [37] have been utilized to embed the palladium nanoparticles and used as catalysts for organic transformations. Development of supported palladium catalysts for coupling reactions with an effective combination of good activity, stability and reusability has always been a challenge.

Cross-linking polymerization of styrene and divinylbenzene gives material which is easily available and a well studied support for facile functionalization [6]. Previously we have reported modification of this material by attaching Schiff base ligands and then studied their role in palladium catalyzed Mizoroki–Heck reaction [38]. Chloromethylated poly(styrene-divinyl benzene) copolymer spherical beads with suitable crosslink **1** were attached with two sets of diamines, 1,3-diaminopropane **2a** or 4,4′-diaminobiphenyl **2b** to afford polymer supported primary amines **3**. These were then treated with salicylaldehyde **4** to get appropriate polymer-anchored Schiff base complexes **5** or **6** (Scheme 1).

The two sets of polymer attached Schiff base materials were treated with palladium chloride till the metal forms complex with the ligand. Polymers **5** with different cross link (5 or 8 %) afforded two polymer-supported palladium catalysts **A-1** and **A-2** (Scheme 2). Similar procedure was adopted for **6** to afford another set of palladium catalysts **B-1** and **B-2**. The two polymer supported Schiff bases **5** and **6** as well as the two sets of metal loaded catalysts **A-1/A-2** and **B-1/B-2** were fully characterized [38].

Scheme 2 Loading the polyamines with palladium





Scheme 3 Synthesis of biphenyl by Suzuki–Miyaura reaction with the prepared catalysts

Synthesis of biaryls is efficiently achieved by palladium mediated Suzuki–Miyaura reaction [39–46] of aryl halide and arylboronic acid, as against Ullmann coupling reaction [47, 48] which requires drastic conditions. In this paper we will present our findings to utilize the present series of polyamine supported palladium catalysts for the Suzuki–Miyaura reaction. There are few reports on the use of different heterogeneous palladium based catalysts for Suzuki–Miyaura coupling reaction with varying degree of success [49–58].

A strategy of performing a combination of different but complementary reactions in a single flask or one-pot reactions is gaining popularity in recent years [34, 59–64]. Such reaction protocol, also referred as cascade, tandem or domino reactions, involves a number of sequential steps until a stable product is formed. These methods often reduce the consumption of reagents; decrease the number of purifications and cuts on reaction time contributing to overall efficiency of the chemical process. Recent developments pertaining to the involvement of catalytic reactions in such cascade reactions have been nicely summarized [65–67]. In our ongoing interest in the area of one-pot synthesis and syntheses of conjugated molecules, we have explored several combination of one-pot reactions [68–72]. In the present work we will also present a combination of *O*-alkylation-Suzuki reaction catalyzed by the present polymer-supported palladium catalysts to synthesize alkoxylated biaryls.

2 Experimental

2.1 Standard Procedure for Suzuki Reaction

2.1.1 Synthesis of Biphenyl (9)

A round bottom flask was charged with iodobenzene (0.2 g, 0.980 mmol), catalyst **B-2** (0.061 g, 0.000098 mmol Pd, 0.01 mol %), phenyl boronic acid (0.143 g, 1.176 mmol), dry potassium carbonate (0.270 g, 1.960 mmol), TBAB (0.063 g, 0.196 mmol) and dioxane-water (1:1; 10 mL) as solvent. This mixture was heated to 80 °C and continued for 24 h. The reaction mixture was filtered to remove the catalyst and was quenched with water and extracted with ethyl acetate (3 × 25 mL). The combined organic phase was washed with water and dried over anhydrous sodium sulfate. Solvent was removed in vacuum and the crude product was purified by column chromatography on silica gel to afford biphenyl (0.148 g, 98.7 %) as white solid (m.p. 66–67 °C, lit [73] 68–70 °C).

2.1.2 Synthesis of 9-Butyl-3-phenyl-9H-carbazole (30)

A round bottom flask was charged with 3-iodo-9-butyl carbazole (0.15 g, 0.429 mmol), catalyst **B-2** (0.027 g, 0.000043 mmol Pd, 0.01 mol %), phenyl boronic acid (0.063 g, 0.515 mmol), dry potassium carbonate (0.118 g, 0.859 mmol), TBAB (0.027 g, 0.086 mmol) and dioxane-water (1:1; 10 mL) as solvent. This mixture was heated to 80 °C and continued for 24 h. The reaction mixture was filtered to removed catalyst and was quenched with water and extracted with ethyl acetate (3 × 25 mL). The combined organic phase was washed with water and dried over anhydrous sodium sulfate. Solvent was removed in vacuum and the crude product was purified by column chromatography on silica gel to afford 9-butyl-3-phenyl-9*H*-carbazole (0.098 g, 76.6 %) as colourless liquid.

^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 1.6 Hz, 1H, Ar-C4*H*), 8.18 (dd, *J* = 7.2, 0.8 Hz, 1H, Ar-C2*H*), 7.77–7.74 (m, 3H, Ar–*H*), 7.54–7.45 (m, 5H, C6*H*5), 7.40–7.36 (m, 1H, Ar–C6*H*), 7.31- 7.27 (m, 1H, Ar–C7*H*), 4.36 (t, *J* = 7.2 Hz, 2H, $–N–CH_2–$), 1.96–1.88 (m, 2H, $–N–CH_2–CH_2–$), 1.49–1.43 (m, 2H, $–N–CH_2–CH_2–CH_2–$), 0.99 (t, *J* = 7.2 Hz, 3H, $–CH_3$).

^{13}C-NMR (100 MHz, $CDCl_3$): δ 142.22, 140.93, 139.98, 132.28, 128.80, 127.34, 126.42, 125.81, 125.19, 123.34, 122.99, 120.45, 118.92, 108.94, 108.87, 42.98 ($NCH_2–$), 31.21 ($NCH_2CH_2–$), 20.64 ($NCH_2CH_2CH_2–$), 13.96 ($–CH_3$) (one signal is missing).

IR (KBr): υ 3055, 2958, 2930, 2872, 1627, 1599, 1472, 1349, 1257, 1212, 802 cm.$^{-1}$

MS (EI) (*m*/*z*): 299 (98) [M^+], 255 (100).

Table 1 Scanning of catalysts for Suzuki–Miyaura reaction of PhI with $PhB(OH)_2$

No	Catalyst	Pd content (g/g of resin)	Crosslink (%)	Isolated yield (%) (TON)
1	**A-1**	1.0×10^{-4}	5	93.0 (7398)
2	**A-2**	2.0×10^{-4}	5	98.0 (9737)
3	**B-1**	2.5×10^{-4}	8	96.7 (9607)
4	**B-2**	1.7×10^{-4}	8	98.7 (9803)

Table 2 Examples of Suzuki–Miyaura coupling with catalysts **B-2** $\mathrm{ArX} + \mathrm{Ar'\text{-}B(OH)_2} \xrightarrow[\text{Base}]{\text{Pd-Catalyst}} \mathrm{Ar{-}Ar'}$

No	ArX	Boronic acid	Biaryl product	Isolated yield (%) (TON)
1	C_6H_5Br (**9**)	**BA-1**	C_6H_5–C_6H_5 (**8**)	93.9 (9379)
2	4-$NO_2C_6H_4Br$ (**10**)	**BA-1**	4-$NO_2C_6H_4$–C_6H_5 (**11**)	91.2 (9074)[a]
3	1,3-Dibromo-5-methylbenzene (**12**)	**BA-1**	5′-Methyl-1,1′:3′,1″-terphenyl (**13**)	90.5 (4500)
4	1,2-$Br_2C_6H_4$ (**14**)	**BA-2**	1,2-$Ar_2C_6H_4$ (**15**) Ar = 2-$CH_3C_6H_4$	86.0 (4300)
5	4,4′-Dibromo biphenyl (**16**)	**BA-1**	*p*-Quaterphenyl (**17**)	76.9 (3840)[a]
6	1,4-Dibromo benzene (**18**)	**BA-1**	*p*-Terphenyl (**19**)	94.5 (4724)[a]
7	4-$BrC_6H_4NHCOCH_3$ (**20**)	**BA-1**	4-$PhC_6H_4NHCOCH_3$ (**21**)	95.2 (9466)[a]
8	2-BrC_6H_4CN (**22**)	**BA-1**	2-PhC_6H_4CN (**23**)	94.6 (9424)[a]
9	4-$NO_2C_6H_4Br$ (**10**)	**BA-2**	2-Methyl-4′-nitro biphenyl (**24**)	94.3 (9421)[a]
10	4-$BrC_6H_4NHCOCH_3$ (**20**)	**BA-2**	4-(*o*-tolyl)acetanilide (**25**)	94.9 (9448)
11	1,3,5-Tribromobenzene (**26**)	**BA-1**	1,3,5-Triphenylbenzene (**27**)	96.6 (3202)
12	1,3,5-Tribromobenzene (**26**)	**BA-2**	1,3,5-Tri(*o*-tolyl)benzene (**28**)	84.2 (2810)

With Pd (0.01 mol %) and K_2CO_3 (2.0 eq) for one Br

BA-1 (phenylboronic acid) and BA-2 (*o*-tolylboronic acid); *TON* turn over number

[a] With recovered catalyst **B-2**

Table 3 Recycle study of catalyst **A-1** for the synthesis of biphenyl from **7** and $PhB(OH)_2$

No of cycle	Isolated yield (%)
1	92.7
2	91.7
3	91.2
4	90.0

HRMS (ESI$^+$): Calculated for $C_{22}H_{21}N$ $[M + H]^+$ 300.1752, found 300.1744.

2.2 Standard Procedure for *O*-alkylation-Suzuki Reaction

2.2.1 Synthesis of 2-(Octyloxy)-7-phenylnaphthalene (44)

A round bottom flask was charged with 7-bromo-2-hydroxy naphthalene (0.15 g, 0.672 mmol), catalyst **B-2** (0.042 g, 0.0000672 mmol, 0.01 mol %), n-octyl bromide(0.155 g, 0.807 mmol), phenyl boronic acid (0.098 g, 0.807 mmol), dry potassium carbonate (0.371 g, 2.69 mmol), TBAB (0.043 g, 0.134 mmol) and dioxane-water (1:1, 10 mL) as solvent. This mixture was heated to 80 °C and continued for 24 h. The reaction mixture was filtered to removed catalyst and was quenched with water and extracted with ethyl acetate (3 × 25 mL). The combined organic phase was washed with water and dried over anhydrous sodium sulphate. Solvent was removed in vacuum and the crude product was purified by column chromatography on silica gel to afford 2-(octyloxy)-7-phenylnaphthalene (0.192 g, 86.0 %) as white solid [m.p. 56–58 °C].

^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.95 (s, 1H, Ar–C8*H*), 7.85 (d, *J* = 8.4 Hz, 1H, Ar–C5*H*), 7.90–7.73 (m, 3H, Ar–*H*), 7.61 (dd, *J* = 8.4, 1.6 Hz, 1H, Ar–C3*H*), 7.50 (t, *J* = 7.6 Hz, 2H, Ph Ar–*H*), 7.40 (t, *J* = 7.2 Hz, 1H, Ph Ar–*H*), 7.21 (d, *J* = 2.4 Hz, 1H, Ar–C1*H*), 7.17 (dd, *J* = 8.8, 2.4 Hz, 1H, Ar–C3*H*), 4.12 (t, 6.4 Hz, 2H, –O–CH_2–), 1.90–1.87 (m, 2H, –O–CH_2–CH_2–), 1.59–1.52 (m, 2H, –O–CH_2–CH_2–CH_2–), 1.41–1.33 (m, 8H, n-(CH_2)$_4$–), 0.92 (t, *J* = 6.8 Hz, 3H, –CH_3).

^{13}C-NMR (100 MHz, $CDCl_3$): δ 157.54 (Ar*C*-OR), 141.37, 139.04, 134.95, 129.09, 128.85, 128.19, 128.09, 127.46, 127.32, 124.76, 123.27, 119.19, 106.84, 68.09 (–O*C*H$_2$–), 31.91, 29.47, 29.34, 29.32, 26.19, 22.75, 14.20 (–*C*H$_3$).

IR (KBr): υ 3059, 3032, 2924, 2851, 1625, 1604, 1460, 1391, 1211, 888, 840, 750, 690 cm.$^{-1}$

MS (EI): (*m/z*) 322 (31) [M$^+$], 331 (20), 219 (100).

HRMS (ESI$^+$): Calculated for $C_{24}H_{29}O$ $[M+H]^+$ 333.2218, found 333.2213.

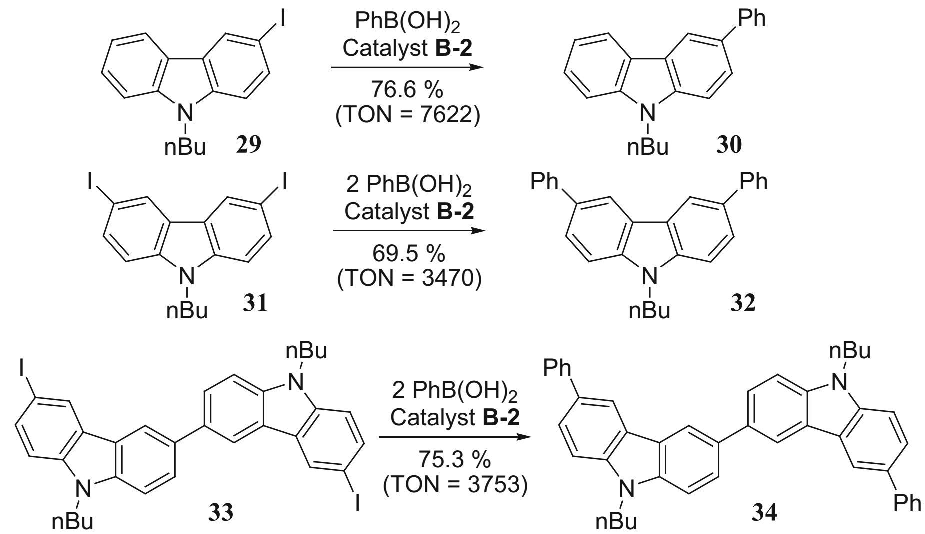
2.3 Spectral Data of New Compounds

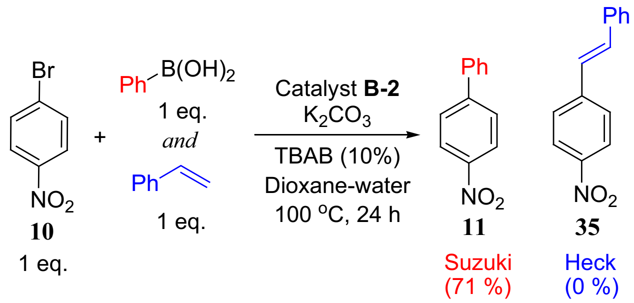
2.3.1 6,6′-Diphenyl-3,3′-di(N-butyl Carbazole) (34)

Pale yellow solid (0.128 g, 75.3 %).

^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.52–8.39 (m, 4H, Ar–C4/C5 and C4′/C5′*H*), 7.90–7.84 (m, 2H, Ar–C2/C2′*H*),

Scheme 4 Synthesis of aryl carbozoles by Suzuki–Miyaura reaction





Scheme 5 Selectivity of Suzuki–Miyaura reaction over Mizoroki–Heck reaction with catalyst **B-2**

7.79–7.76 (m, 6H, Ar–*H*), 7.56–7.49 (m, 8H, Ph*H*), 7.39–7.37 (m, 2H, Ph*H*), 4.41 (t, *J* = 7.2 Hz, 4H, –N–C*H*$_2$–), 1.99–1.90 (m, 4H, –N–CH$_2$–C*H*$_2$–), 1.52–1.45 (m, 4H, –N–CH$_2$–CH$_2$–C*H*$_2$–), 1.02 (t, *J* = 7.2 Hz, 6H, –C*H*$_3$).

^{13}C-NMR (100 MHz, CDCl$_3$): δ 142.19, 140.45, 140.05, 133.41, 132.29, 128.81, 127.33, 126.43, 125. 69, 125.24, 123.61, 119.02, 118.99, 109.12, 109.07, 43.12 (N*C*H$_2$–), 31.30 (NCH$_2$*C*H$_2$–), 20.67 (NCH$_2$CH$_2$*C*H$_2$–), 14.00 (–*C*H$_3$) (one signal is missing).

IR (KBr): υ 3035, 2954, 2871, 1600, 1475, 1379, 1349, 1268, 1210, 1130, 874, 798, 757 cm.$^{-1}$

MS (EI): (*m*/*z*) 596 (3) [M$^+$], 576 (10), 520 (22), 451 (38), 407 (35), 312 (30).

HRMS (ESI$^+$): Calculated for $C_{44}H_{40}N_2$ [M+K]$^+$ 635.2829, found 635.2828.

2.3.2 2′-(Octyloxy)-5′-Phenyl-1,1′:3′,1″-Terphenyl (46)

Colourless liquid (0.166 g, 84.7 %).

^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.70–7.65 (m, 5H, 5-Ph*H*), 7.60 (s, 2H, Ar–C4/C6*H*), 7.49–7.44 (m, 6H, 1,3-Ph*H*), 7.41–7.36 (m, 4H, 1,3-Ph *H*), 3.26 (t, *J* = 6.4 Hz, 2H, –O–C*H*$_2$–), 1.27–0.93 (m, 12H, n-(C*H*$_2$)$_6$-), 0.88 (t, *J* = 6.8 Hz, 3H, –C*H*$_3$).

^{13}C-NMR (100 MHz, CDCl$_3$): δ 153.76 (Ar*C*-OR), 140.56, 138.87, 136.95, 136.46, 129.58, 128.96, 128.90, 128.80, 128.09, 127.17, 127.05, 73.40 (–O*C*H$_2$–), 31.78, 29.79, 29.13, 29.10, 25.73, 22.67, 14.17 (–*C*H$_3$ of octyl).

IR (KBr): υ 3058, 3032, 2925, 2855, 1689, 1599, 1576, 1495, 1461, 1426, 1380, 1221, 1074, 963, 887, 753, 697 cm.$^{-1}$

MS (EI): (*m*/*z*) 434 (18) [M$^+$], 322 (100), 321 (98).

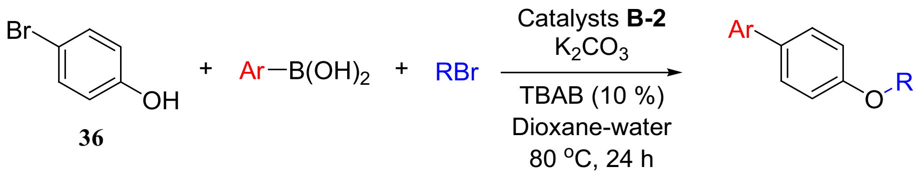
HRMS (ESI$^+$): Calculated for $C_{32}H_{34}O$ [M+Na]$^+$ 457.2507, found 457.2505.

3 Result and Discussion

The catalysts of the two sets with known amount of cross link and palladium loading were screened for the standard Suzuki–Miyaura reaction with phenyl iodide **7** and phenyl boronic acid (Scheme 3, Table 1). All the catalysts were found to be very effective as evident from the high isolated yields and turn over numbers (TON). From the observation we conclude that the catalysts **A-2** and **B-2** showed slightly higher efficiency, probably due to higher cross linking resulting in better permeability of reagents. The surface area of **B-2** was found to be 45.4 m^{2}g^{-1} compared to 34.6 m^{2}g^{-1} for **B-1**, this marginally higher value may also be responsible for this observation [38].

The catalyst **B-2** was then scanned for a series of aryl bromides as they are more readily accessible and are stable substrates compared to their iodo derivatives. The catalyst works consistently well for many examples (Table 2) and can tolerate several functional groups for the Suzuki–

Scheme 6 One-pot *O*-alkylation and Suzuki–Miyaura reaction



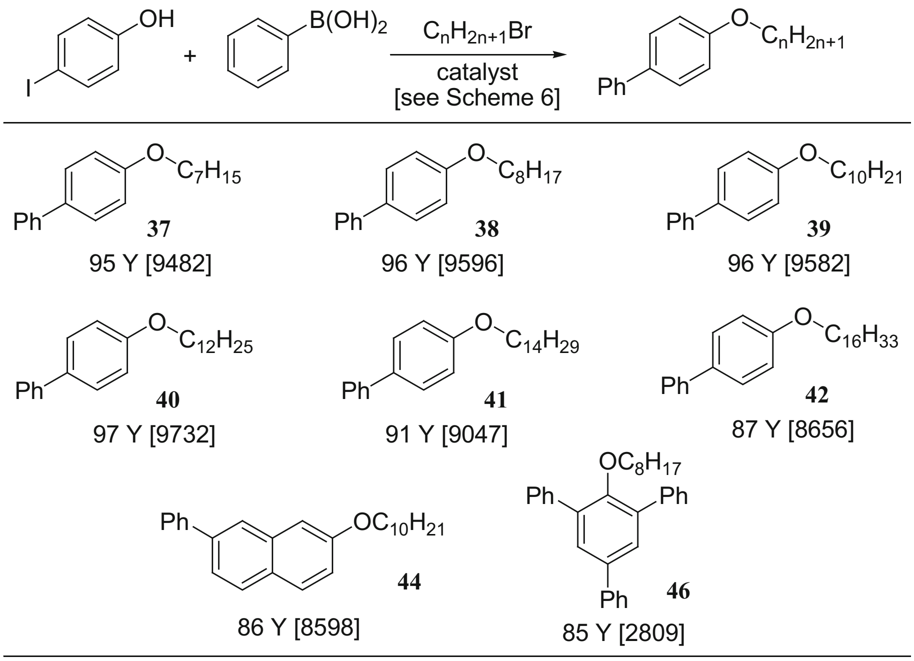
Miyaura coupling reaction with very low palladium loading (0.01 mol%). The present catalysts with lower amount of palladium, compared to some of the similar imine based heterogeneous catalysts, establishes good catalytic activity for Suzuki–Miyaura reaction [74–76]. The effect of solvent on Suzuki–Miyaura reaction was found to be similar to what we have observed for Mizoroki–Heck reaction, polar solvents were more suitable due to better swelling of the resin beads facilitating the access of reagents to the active sites. Moreover, we have already demonstrated thermal stability of the catalysts up to 300–325 °C by thermogravimetric analysis [38], which much higher than the reaction condition of present Suzuki–Miyaura reaction. Two derivatives of aryl boronic acids were scanned for the reaction where the products were isolated in good yields.

The efficiency of heterogeneous catalyst depends on their easy ability to separate and reusability for subsequent catalytic cycles. This was tested by conducting four reactions under identical conditions with recycled catalyst **A-1** (Table 3). This indicates that the catalyst loses very little activity even after these many cycles confirming its reusability.

In this brief study we have also prepared aryl derivatives of carbazoles by Suzuki–Miyaura coupling with the corresponding iodo analogues (Scheme 4). Structurally similar arylated carbazoles have been studied as hole-transport materials [77–79], as fluorophores [80], light emitting diode [81–83], photovoltaic devices [84], and for other electron transfer applications [85–89] etc. The required iodo derivatives of the carbazole **29** and **31** or bis-carbazole **33** were prepared according to the literature procedures [81, 90–92] and then subjected to the Suzuki–Miyaura reaction with the present catalyst. The products **30**, **32** and **34** were isolated in good yields and characterized by usual spectral techniques and by comparison with the known analytical data.

In the present study we found the Suzuki–Miyaura reaction to be more facile compared to the Mizoroki–Heck reaction on aryl bromide with our catalysts. This was established by performing a controlled experiment of 4-nitro bromobenzene **10** with equivalent amount of phenyl boronic acid and styrene in presence of catalyst **A-1** (0.01 % Pd), potassium carbonate (2.0 eq.), TBAB (10 %) in aqueous dioxane. The careful analysis of the product confirms the formation of 4-nitro biphenyl **11** with good yield along with small quantity of biphenyl **8**. It is noteworthy to observe the absence of Heck product, 4-nitro stilbene **35** during the reaction which confirmed the

Chart 1 Examples of one-pot *O*-alkylation-Suzuki reaction. With catalyst **B-2**. Compound **44** with 7-bromo-2-hydroxy naphthalene and **46** from 2,4,6-tribromo phenol



selectivity in favor of Suzuki–Miyaura reaction with active aryl bromide (Scheme 5).

Many alkoxy substituted derivatives of biphenyl or biaryl compounds have been studied for their specific properties. Compounds containing alkoxy functional groups have been studied as materials with thermal properties [93–96]. Several compounds possessing alkoxy substituents and showing liquid crystalline properties [97–100] are required for appropriate crystal packing and hence are responsible for the specific thermal behavior. Usually the introduction of alkoxy substituent is carried out as a separate specific chemical operation but we have recently developed a couple of one-pot *O*-alkylation-Wittig and *O*-alkylation-Wittig-Heck reactions to synthesize alkoxy stilbenes [72]. In this work we have extended the strategy to synthesize *O*-alkoxy biphenyls and *O*-alkoxy biaryls by simultaneously conducting Suzuki–Miyaura and *O*-alkylation reaction on bromo phenols (Scheme 6). As outlined the three components of this version include bromo phenol, arylboronic acid and the alkyl halide treated with catalyst **B-2** in presence of suitable base, PTC in aqueous dioxane.

A series of examples were tested to establish this one-pot reaction and the results are presented in Chart 1. Consistently good conversions and isolated yields were observed for the examples studied in this work.

4 Conclusion

In conclusion we wish to report application of polymer anchored amines as heterogeneous ligands for palladium catalysts for Suzuki–Miyaura coupling reactions of aryl halides. The yields and TON of our catalytic system for this reaction were comparable with other heterogeneous palladium catalysts. The catalyst system was also scanned for the one-pot *O*-alkylation-Suzuki combination to synthesize series of alkoxy biaryl systems.

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