



1-Butyl-3-methyl-2-(diphenylphosphino)imidazolium hexafluorophosphate as an efficient ligand for recoverable palladium-catalyzed Suzuki-Miyaura reaction in neat water

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ABSTRACT

The 1-butyl-3-methyl-2-(diphenylphosphino)imidazolium hexafluorophosphate is prepared and used as a water-soluble ligand in the Suzuki-Miyaura reaction. This ligand and palladium chloride generates a new active catalyst for the Suzuki coupling reaction between aryl iodides or bromides with arylboronic acids in neat water. This catalyst can be recycled and reused for the coupling reaction of 4-bromobenzonitrile with phenylboronic acid for at least six runs with low decreasing in catalytic activity.

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1. Introduction

The Suzuki reaction is a suitable method for the formation of different type of compounds especially biaryls [1–20]. The resulting products of Suzuki reaction have different application in the preparation of natural products, nucleoside analogues, biologically active compounds, liquid crystals and pharmaceuticals [21–23]. In spite of abundant reports on performing the Suzuki-Miyaura coupling reactions, developments to improve it are currently done. For example, conducting the Suzuki-Miyaura coupling reaction in environmentally friendly solvents such as water have particular importance [24–42]. Water is a great alternative to organic solvents which disposal is a main problem in the industry. Water is cheap, green, nonflammable, nontoxic, renewable,

ecofriendly and has a high thermal capacity. Performing the reactions in water leads to high selectivity which is not achieved with organic solvents [2,8,43–47]. Additionally, organic products can be easily separated from the hydrophilic catalyst and the aqueous solution recycled because catalyst remained on it saving time consuming process. Phosphorylated ligands are crucial for the formation of complexes with different transition metals that produced active catalysts in organic synthesis [48–58]. In recent decades, great attention has been paid to the use of catalysts containing water-soluble phosphine ligands in aqueous media for Suzuki [59], Heck [60], Buchwald-Hartwig [61] and Sonogashira [62] coupling reactions. In fact, water-soluble phosphine ligands, that can be combined with Pd atoms, create hydrophilic catalyst, and leads to an increase in the activity in the aqueous media, is highly attractive.

In this work, we have successfully used an imidazolium-based phosphine-ligated in the presence of suitable amounts of PdCl₂ as the recyclable and efficient catalyst in the Suzuki cross-coupling reaction of substituted aryl iodides, bromides and chlorides with various arylboronic acids in water or PEG 200 solvent.

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2. Results and discussion

Initially, compound **2** was synthesized by adding *n*-BuLi and DCM solvent to 1-butyl-1H-imidazole-3-ium hexafluorophosphate **1** and placing the mixture at a temperature of $-78\text{ }^{\circ}\text{C}$ and then **3**, in orange solid form, was obtained by adding PPh_2Cl to it under the same conditions which was characterized by ^1H NMR, ^{13}C NMR and ^{31}P NMR (Scheme 1) [63]. Desired ligand **3** is air- and moisture-stable in solid state.

The formation of active Pd(0) species was achieved by reacting of PdCl_2 with **3** in water. The appearance of dark brown color after 1 h indicated the formation of active Pd(0) species [64] (Fig. 1).

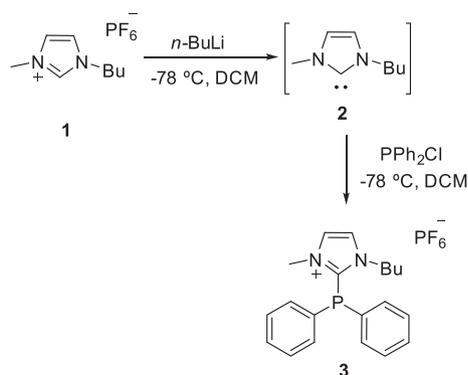
Furthermore, the formation of the Pd(0) catalyst was supported by the analysis of the solid state UV–Vis spectra of the powder obtained by drying the aqueous mixture of PdCl_2 and the phosphorous ligand (Fig. 1c). Results indicated that the related peak at 280 nm corresponding to palladium(II) disappeared in the presence of **3** (Fig. 2) [65].

The XRD patterns of this powder, resulted from the evaporation of the aqueous solution of PdCl_2 and IL **3**, showed that the characteristic bands of the formation of Pd^0 species at (111), (200), (220) and (311) crystallographic planes were detected at 40.2° , 46.7° and 68.2° (Fig. 3a) [66]. The XRD patterns for the Pd nanoparticles showed a wide peak centered at around 40.35° . The average size of the Pd nanoparticles were calculated from the full width at half maximum of the strongest diffraction peak [111] obtained by the Scherer equation (Eq. (1)):

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where B is the full width at half maximum, θ is the angle of Bragg diffraction and k is the X-ray wavelength that K is a constant taken as 0.89 [67,68]. The average size of the Pd nanoparticles obtained at around 4.65 nm. The presence of the palladium(0) also was confirmed by XPS analysis (Fig. 3b) the binding energies had shifted with respect to palladium(II) and the maximum intensities corresponded to Pd^0 $3d_{3/2}$ and $3d_{5/2}$ peaks.

To find the optimal conditions, the reaction of 4-iodoanisole with phenylboronic acid was selected as the model reaction and the effect of various factors such as temperature, solvents were investigated (Table 1). Initially the reaction was conducted in different solvents such as H_2O , DMF, THF, CH_3CN , and toluene by using K_2CO_3 as a base at $50\text{ }^{\circ}\text{C}$ (Table 1, entries 1–5). Results demonstrated that the best yield (90%) was obtained in water solvent during 24 h (Table 1, entry 6). By increasing reaction temperature to $60\text{ }^{\circ}\text{C}$ and using water as the solvent 90% yield was achieved. It should be noted by reducing the reaction temperature



Scheme 1. Synthesis of ligand **3**.

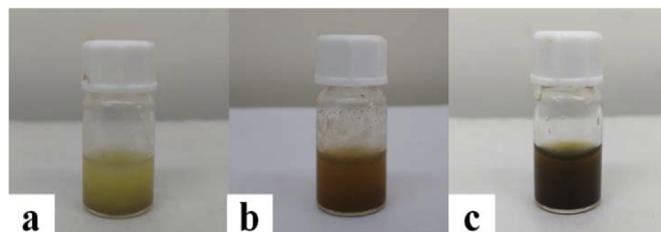


Fig. 1. Photograph presentation of the mixture of **3** with PdCl_2 a) after 0 min, b) 30 min c) 60 min.

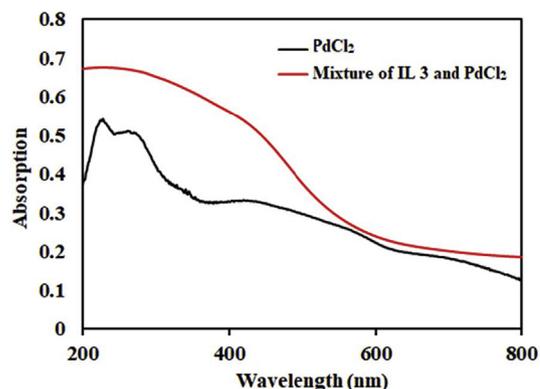


Fig. 2. UV–Vis spectra of **3**- PdCl_2 (red) and PdCl_2 (blue). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

to the room temperature, the product yield was diminished to 40% (Table 1, entry 7). Furthermore, performing reaction in the absence of **3** afforded low yield indicating important role of ligand in the efficiency of the reaction (Table 1, entry 8).

Having optimized condition in hand, Suzuki coupling reaction of variety of aryl iodides with different organoboronic acids were studied. Reactions of aryl iodides having electron donating groups such as $-\text{Me}$, $-\text{OMe}$ as well as iodobenzene proceeded in 24 h and the desired products were isolated in 90–92% yields (Table 2, entries 1–3). Furthermore, reactions of aryl iodides having electron withdrawing groups were satisfactorily performed and afforded biaryls in 96–98% yields (entries 4–9). It should be noted that reaction of 2,5-diiodothiophene as heterocyclic with phenylboronic acid and the reaction of sterically hindered methyl 4-amino-3,5-diiodobenzoate with phenylboronic acid were proceeded well and 82 and 80% yields were obtained respectively (Table 2, entries 4 and 10). We have also studied the Suzuki reaction of aryl bromides using these optimal conditions. For this purpose, reaction of 4-bromoanisole with phenylboronic acid was studied and 50% yield was achieved after 24 h at $50\text{ }^{\circ}\text{C}$. However, when the reaction temperature was increased to $80\text{ }^{\circ}\text{C}$, 92% isolated yield was obtained. Therefore, we studied the coupling reaction of different aryl bromides with arylboronic acid at $80\text{ }^{\circ}\text{C}$ in water solvent. Generally, the results showed that aryl bromides having both electron-donating or electron-withdrawing groups afforded excellent yields (Table 2, entries 11–20). We further extend application of the present catalyst for the Suzuki reaction of aryl chlorides. However, initial results indicated that reactions of 4-chlorobenzaldehyde under the optimized reaction conditions ($80\text{ }^{\circ}\text{C}$) was sluggish and 21% yield was obtained. Therefore, different reaction conditions were studied. It has been found that using PEG200 at $120\text{ }^{\circ}\text{C}$ afforded 80–87% yields for activated aryl chlorides (Table 2, entries 21–23).

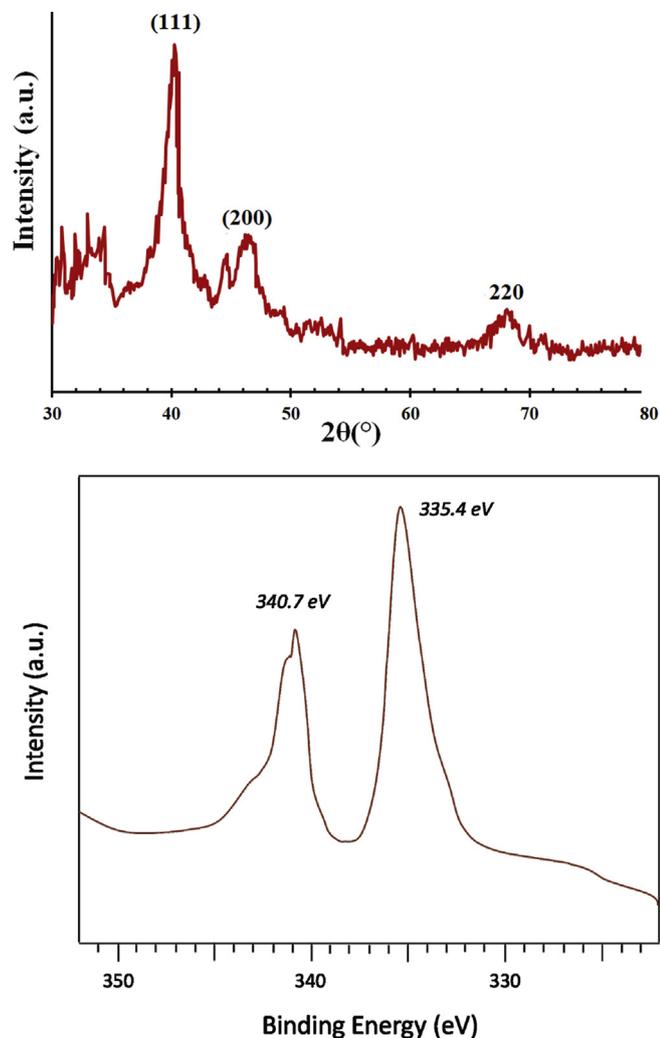
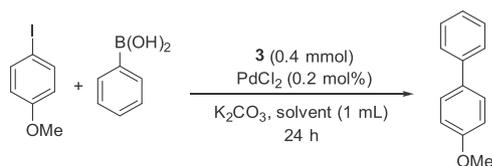


Fig. 3. a) XRD pattern of the mixture of **3** with PdCl₂; b) XPS analysis of the mixture of **3** with PdCl₂.

Table 1

Optimization of the reaction conditions for the coupling of 4-iodoanisole and phenylboronic acid.^a.



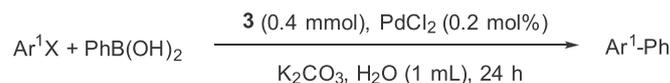
Entry	Solvent	T (°C)	Yield (%)
1	H ₂ O	50	80
2	DMF	50	2
3	THF	50	5
4	CH ₃ CN	50	11
5	Toluene	50	23
6	H ₂ O	60	90
7	H ₂ O	r.t	40
8	H ₂ O	60	45 ^b

^a 4-iodoanisole (0.5 mmol), phenylboronic acid (0.75 mmol), K₂CO₃ (0.75 mmol), **3** (0.4 mmol), PdCl₂ (0.2 mol%), solvent (1 mL).

^b Reaction was performed in the absence of **3**.

Table 2

Effect of the ligand **3** in combination with PdCl₂ as catalyst for the Suzuki coupling reactions of various aryl iodides, bromides and chlorides with arylboronic acids.^a.



Entry	Ar ¹ X	T (°C)	Yield (%) ^b
1	Ph-I	60	>99 ^c
2	4-(MeO)C ₆ H ₄ I	60	90
3	4-(Me)C ₆ H ₄ I	60	92
4		60	82
5	3-(CN)C ₆ H ₄ I	60	98 ^c
6	3-(CHO)C ₆ H ₄ I	60	95
7	4-(NO ₂)C ₆ H ₄ I ^d	60	90
8	4-(F)C ₆ H ₄ I	60	93
9	4-(Cl)C ₆ H ₄ I	60	93
10		60	80
11	4-(MeO)C ₆ H ₄ Br	80	92 ^c (50 ^e)
12	4-(HO)C ₆ H ₄ Br	80	91
13	4-(CN)C ₆ H ₄ Br	80	98 ^c
14	4-(CN)C ₆ H ₄ Br ^d	80	91
15	3-(CHO)C ₆ H ₄ Br	80	82
16	4-(CHO)C ₆ H ₄ Br	80	97 ^c
17	4-(NO ₂)C ₆ H ₄ Br	80	>99 ^c
18	4-(Ac)C ₆ H ₄ Br	80	93
19	4-(F)C ₆ H ₄ Br	80	96 ^c
20		80	94
21	4-(CHO)C ₆ H ₄ Cl	120	80 ^f (31 ^e)
22	4-(CN)C ₆ H ₄ Cl	120	87 ^f
23	4-(NO ₂)C ₆ H ₄ Cl	120	85 ^f

^a Reaction conditions: Aryl bromide, chloride or iodide (0.5 mmol), arylboronic acid (0.75 mmol), K₂CO₃ (0.75 mmol), **3** (0.4 mmol), PdCl₂ (0.2 mol%), and H₂O (1 mL).

^b Isolated yields.

^c Yields determined by GC.

^d Naphthalene-1-boronic acid was used instead.

^e Reaction was performed in the absence of **3**.

^f Reaction conditions: Aryl chlorides (0.5 mmol), arylboronic acid (0.75 mmol), K₂CO₃ (0.75 mmol), **3** (0.4 mmol), PdCl₂ (0.2 mol%), and PEG 200 (1 mL).

We also investigated the recycling of catalyst for coupling of 4-bromobenzonitrile with phenylboronic acid at 80 °C in water. After completion of the reaction, organic phase was extracted with ethyl acetate and the aqueous phase, which included the catalyst was used in next reaction. Results indicated that catalyst was recycled successfully for six consecutive runs with small decrease in the activity (Fig. 4).

3. Conclusions

In this work, we have prepared novel water-soluble phosphine as a ligand for Suzuki coupling reaction in water without the use of an organic co-solvent. This ligand is easily prepared in two steps which in combination with PdCl₂ as an efficient catalyst has been applied in Suzuki coupling reaction of aryl iodides and bromides in water solvent. Additionally, this catalyst successfully has been used

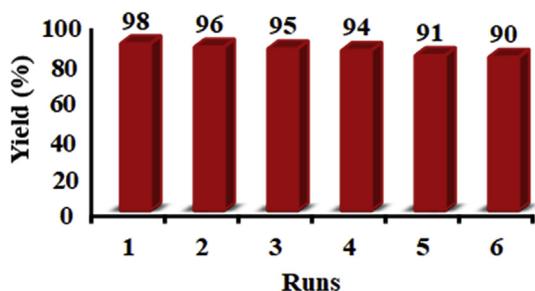


Fig. 4. Recycling of the catalyst for the coupling reaction of 4-bromobenzonitrile with phenylboronic acid at 80 °C.

in the Suzuki coupling reactions of wide range of substrates of aryl iodides and bromides including electron-donating and electron withdrawing groups with various arylboronic acids. This catalyst simply recycled and reused for six runs with slight decreasing in catalytic activity.

4. Experimental section

4.1. General

Palladium(II) chloride (PdCl_2), arylboronic acids ($\text{ArB}(\text{OH})_2$), aryl halides (ArX), potassium carbonate (K_2CO_3) and chlorodiphenylphosphine (PPh_2Cl) from sigma Aldrich and 1-Butyl-3-methylimidazolium hexafluorophosphate and *n*-butyllithium (*n*-BuLi) from ChemSpider were purchased respectively. The crystallographic structures of **3** with PdCl_2 were characterized by X-ray diffractometer (XRD) with Cu $K\alpha$ radiation. UV–Vis spectra were investigated on a UV–Vis spectrophotometer (JASCO, UV-550).

4.2. General procedure for performing of Suzuki coupling reaction

PdCl_2 (0.01 mmol, 2 mg) was dissolved in 1 mL distilled water by sonication and 0.1 mL of resulting solution was added to a 5 mL flask containing **3** (0.4 mmol, 129 mg) and H_2O (1 mL), and the mixture stirred for 1 h at room temperature. Then, aryl halide (0.5 mmol), arylboronic acid (0.75 mmol), K_2CO_3 (0.75 mmol) were added and the reaction mixture was stirred at 60 °C, for ArI, and 80 °C for aryl bromides. Progress of the reactions were monitored by GC. Afterwards, crude product was extracted using ethyl acetate or *n*-hexane (3x5 mL) and further purified by column or plate chromatography.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jorgchem.2019.120941>.

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