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Schiff Base-Pd(II) Complexes Containing Pyridine and Thiophene Rings: Synthesis, Characterization, Suzuki-Miyaura C-C Coupling Reactions

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ABSTRACT

New Schiff base-palladium(II) complexes (1a-d) were synthesized with the reaction of Schiff base ligand and $[PdCl_2(CH_3CN)_2]$. Pd(II) complexes (1a-d) were solid and stable to moisture and air. Their structures were characterized through the spectroscopic techniques including microanalysis, FT-IR, UV-Vis, proton (^{1}H) and carbon (^{13}C) NMR, mass spectrometry. The data obtained from the spectroscopic techniques showed that Schiff base-Pd(II) complexes 1a, 1b and 1c were formed through the coordination of azomethine nitrogen and carbonyl/hydroxyl oxygen atoms, while 1d complex coordinated only azomethine nitrogen atom of the Schiff base. All complexes were found to have square plane. In addition, Pd(II) complexes (1a-d) were investigated in Suzuki-Miyaura coupling reactions of different aryl bromides using phenylboronic acid. The reactions were carried out in a mixed aqueous EtOH/H_2O (1/3) at room temperature with K_2CO_3 as the base for 1 hour. The Pd(II) complexes (1a-d) used as catalysts exhibited perfect catalytic activity in the Suzuki-Miyaura reaction.

Keywords:
Aryl bromides; Pd(II) complexes; Schiff base; Suzuki-Miyaura; Mass spectrometry.

INTRODUCTION

Schiff bases are a multinucleated ligand group that forms complexes with many metal atoms through donor atoms. They have been extensively studied due to their important properties such as easy accessibility, low cost, selectivity and sensitivity to central metal ions [1-6]. Schiff base complexes exhibit high catalytic activities for a wide range of chemical reactions (e.g., hydrosilylation, homogenous catalyst, transfer hydrogenation, C-C coupling reactions and epoxidation) [7-9]. The Suzuki-Miyaura reaction, which is one of the present C-C cross-coupling reactions, is the most important reaction between aryl halides and arylboronic acids and widely used to obtain biaryls. The Suzuki-Miyaura coupling reaction has been extensively studied in the field of synthesis owing to its ease of operation, perfect functional group compatibility, attainable and environmental friendly properties of the initial reagents. Due to their superior properties, biaryls is a widely preferred compound in numerous industrial applications such as pharmaceuticals, natural products, agrochemicals, advanced materials, cosmetics [10-17].

In the last decade, the synthesis of Schiff base-Pd(II) complexes has gained great attention since they are cost-effective and environmentally friendly. Particularly, the studies on the catalytic application of Schiff base-Pd(II) complexes, which do not contain phosphine ligands, have increased in Heck-Mizoroki and Suzuki-Miyaura coupling reactions. The research on the synthesis and catalytic activities of Schiff bases and their transition metal complexes has been ongoing[15, 18].

In this article, four Schiff base-palladium complexes **(1a-d)** were prepared using $[PdCl_2(CH_3CN)_2]$ metal salts, and their catalytic activities were investigated. The formation of Pd(II) complexes was determined through FT-IR, microanalysis, UV-Vis, ^{1}H and ^{13}C-NMR and mass spectrometer. The catalytic activity of **(1a-d)** complexes was examined in the Suzuki-Miyaura carbon-carbon coupling reaction. Pd(II) complexes showed excellent activity as catalysts in the Suzuki-Miyaura coupling reaction.

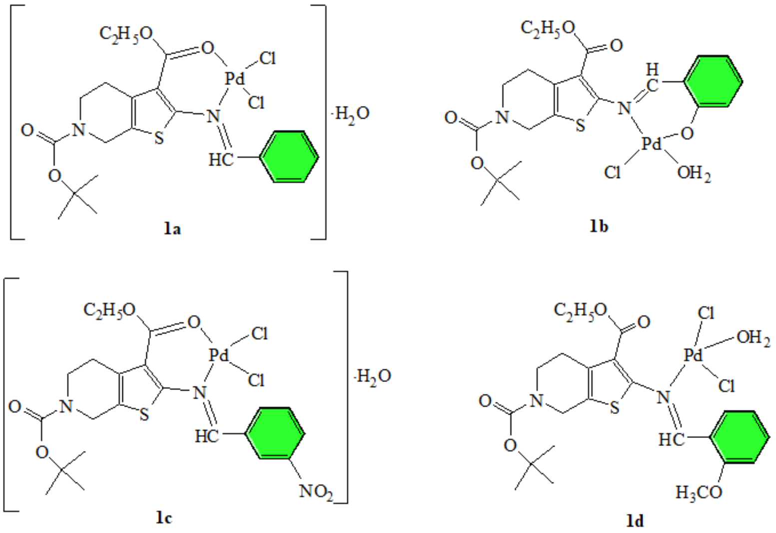

Figure 1. Synthesis of Schiff base-Pd(II) complexes **(1a-d)**

MATERIAL AND METHODS

Material and physical measurements

FT-IR spectra were performed in the range of 400-4000 cm^{-1} on a Perkin Elmer 65 spectrometer. UV-Vis spectra were performed in the range of 200-800 nm (in EtOH) on a Shimadzu-1800 spectrophotometer. Mass spectra of the complexes were measured on an Agilent 1100 MSD spectrophotometer. The NMR (^1H and ^{13}C) spectra were determined with a Brucker 300 MHz and 75 MHz. Microanalysis of C, H, N and S were determined on a CHNS-932 LECO instrument. The catalytic conversions were performed with GC-MS analysis by Agilent 7890B GC system and 5977MSD instrument. All reagents and solvents utilized were of commercial grade and without purified. All reactions were prepared under atmospheric condition.

Synthesis of Schiff base-Pd(II) Complexes (1a-d)

The **1a** complex was prepared in 20 mL of methanol through the reaction of (E)-6-tert-butyl 3-ethyl 2-(benzylideneamino)-4,5-dihydrothieno[2,3-c]pyridine-3,6(7H)-dicarboxylate (0.5 g; 1.2 mmol) and $[PdCl_2(CH_3CN)_2]$ (0.31 g; 1.2 mmol). The mixture was stirred at boiling temperature for one day, and the solvent was completely evaporated in a vacuum. The product was crystallized from a dichloromethane/diethyl ether (1:3) mixture. The proposed structure for the Pd(II) complex is given in Fig. 1. The other three complexes **1b**, **1c**, **1d** were prepared following the same procedure as for the complex **1a**.

Anal. cald. for $C_{22}H_{28}N_2O_5SPdCl_2$ (%): C, 43.35; H, 4.59; N, 4.59; S, 5.25. Found: C, 43.40; H, 4.66; N, 4.65; S, 5.32. IR (KBr, cm^{-1}): 3426 (-OH), 3043 (Ar-H), 2979 (Al-H), 1679 (C=O), 1578 (CH=N), 1577, 1491 (Ar-C), 781 (C-S-C), 554, 525 (M-O), 503, 491 (M-N). ^1H-NMR (300 MHz, DMSO-d_6, ppm): 8.10 (s, 1H, CH=N), 7.81-7.49 (m, 5H, ArH), 4.35, 4.20 (m, 4H, -2CH_2), 3.80-3.11 (m, 4H, CH_2 (pyridine)), 1.36, 1.20 (d, 12H, -CH_3). ^{13}C-NMR (75 MHz, DMSO-d_6, ppm): 158.00, 155.00 (2C=O), 163.00 (CH=N), 136.00-130.00 (thiophenyl), 134.00-127.00 (benzene ring), 79.80 (tert-butyl), 58.96, 14.00 (-OC_2H5), 43.00, 18.00 (pyridine), 24.01 ($C(CH_3)_3$). UV-Vis. (λ_{max}, nm): $\pi \rightarrow \pi^*$, 217, 225, 233; $n \rightarrow \pi^*$, 302, 384. LC-MS: m/z: 590.90 (cald.), 590.97 (found) $[M-H_2O]^+$.

Complex **1b**: Anal. cald. for ($C_{22}H_{27}N_2O_6SPdCl$) (%): C, 44.83; H, 4.58; N, 4.75; S, 5.43. Found: C, 44.81; H, 4.56; N, 4.62; S, 5.46. IR (KBr, cm^{-1}): 3506, 3442 (-OH), 3020 (Ar-H), 2977 (Al-H), 1685 (C=O), 1605 (CH=N), 1572, 1521 (Ar-C), 1151 (C-O), 760 (C-S-C), 565, 550 (M-O), 463 (M-N). ^1H-NMR (300 MHz, DMSO-d_6, ppm): 8.80 (s, ^1H, CH=N), 7.69-6.97 (m, 4H, ArH), 4.54, 4.40 (m, 4H, -2CH_2), 3.35-2.51 (m, 4H, CH_2 (pyridine)), 1.46-1.26 (d, 12H, -CH3). ^{13}C-NMR (75 MHz, DMSO-d_6, ppm): 162.70, 154.24 (2C=O), 161.61 (CH=N), 133.00-129.00 (thiophenyl), 136.00, 117.00 (benzene ring), 80.00 (tert-butyl), 61.07, 14.59 (-OC_2H_5), 44.02, 18.00 (pyridine), 28.51 ($C(CH_3)_3$). UV-Vis. (λ_{max}, nm): $\pi \rightarrow \pi^*$, 221; $n \rightarrow \pi^*$, 301, 391, 504. LC-MS: m/z: 553.42 (cald.), 553.17 (Found) $[M-Cl]^+$.

Complex **1c**: Anal. cald. for ($C_{22}H_{27}N_3O_7SPdCl_2$) (%): C, 40.34; H, 4.12; N, 6.42; S, 4.89. Found: C, 40.31; H, 4.17; N, 6.49; S, 5.01. IR (KBr, cm^{-1}): 3434 (-OH), 3051 (Ar-H), 2982 (Al-H), 1682 (C=O), 1580 (CH=N), 1531, 1467 (Ar-C), 1385 (C-NO_2), 781 (C-S-C), 556, 516 (M-O), 493, 467 (M-N). ^1H-NMR (300 MHz, DMSO-d_6, ppm): 8.32 (s, 1H, CH=N), 7.50-7.01 (m, 4H, Ar-H), 5.76, 4.19 (m, 4H, -2CH_2), 3.35-2.80

Table 1. Optimization of the reaction conditions for Suzuki coupling reaction.

Entry	Base	Time (h)	Yield (%)
1	NOH	8	64
2	Cs_2CO_3	8	51
3	KOH	8	67
4	K_2CO_3	8	95
5	$KOBu^t$	8	68
6	-	8	-
7	K_2CO_3	1	95

[1] Reaction conditions: Phenyl boronic acid (3.0 mmol), aryl bromide (2.0 mmol), base (4.0 mmol), catalyst (0.01 mmol), EtOH/H_2O (5 mL) and 25 °C.

(m, 4H, CH_2 (pyridine)), 1.69-1.21 (d, 12H, -CH_3). ^{13}C-NMR (75 MHz, DMSO-d_6, ppm): 165.00, 163.00 (2C=O), 159.30 (CH=N), 134.00-129.94 (thiophenyl), 129.00, 108.00 (benzene ring), 94.47 (tert-butyl), 59.50, 14.84 (-OC_2H_5), 47.00, 15.16 (pyridine), 29.16 ($C(CH_3)_3$). UV–Vis. (λ_{max}, nm): π→π*, 226; n→π*, 309, 343, 362. LC-MS: m/z: 652.90 (Cald.), 652.73 (Found) $[M-H]^-$.

Complex **1d**: Anal. cald. for ($C_{23}H_{30}N_2O_6SPdCl_2$) (%): C, 43.17; H, 4.69; N, 4.37; S, 5.00. Found: C, 43.13; H, 4.70; N, 4.38; S, 5.16. IR (KBr, cm^{-1}): 3445 (-OH), 3060 (Ar-H), 2978 (Al-H), 1683 (C=O), 1583 (CH=N), 1533 (Ar-C), 1247 (-OCH_3), 757 (C-S-C), 573, 510 (M-O), 487 (M-N). ^{1}H-NMR (300 MHz, DMSO-d_6, ppm): 8.68 (s, 1H, CH=N), 7.95-6.90 (m, 4H, ArH), 5.37, 4.32 (m, 4H, -2CH_2), 3.97-2.42 (m, 4H, CH_2 (pyridine)), 1.28-1.22 (d, 12H, -CH_3). ^{13}C-NMR (75 MHz, DMSO-d_6, ppm): 165.00, 161.00 (2C=O), 157.20 (CH=N), 134.00-120.10 (thiophenyl), 128.70-111.00 (benzene ring), 77.24 (tert-butyl), 55.60, 14.47 (-OC_2H_5), 53.44 (pyridine), 28.70 ($C(CH_3)_3$). UV–Vis. (λ_{max}, nm): π→π*, 205, 215, 251; n→π*, 315, 398. LC-MS: m/z: 550.89 (Cald.), 550.99 (Found) $[M-Cl2, H2O]^{2+}$.

Table 2. Optimization of the reaction conditions for Suzuki coupling reaction.

Entry	Solvent	Time (h)	Yield (%)
1	Ethanol	10	72
2	Methanol	10	60
3	Acetonitrile	10	35
4	Toluene	10	52
5	DMF	10	41
6	THF	10	39
7	Dioxane	10	41
8	i-PrOH+H_2O	10	76
9	EtOH +H_2O	1	95

[1] Reaction conditions: Phenyl boronic acid (3.0 mmol), aryl bromide (2.0 mmol), base (4.0 mmol), catalyst (0.01 mmol), solvent (5 mL) and 25 °C.

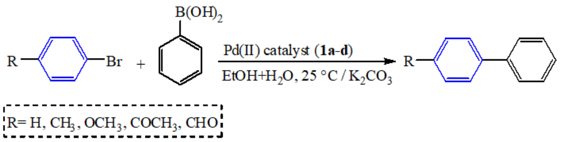

Figure 2. Suzuki-Miyaura cross-coupling reaction.

Catalytic Activity of Pd(II) Complexes for Suzuki-Miyaura Cross-Coupling Reaction

Suzuki-Miyaura cross-coupling is a reaction with palladium-catalyzed aryl and vinyl halide-containing aryl or vinyl boronic acid [19]. A reaction mixture of phenylboronic acid (3.0 mmol), aryl bromide (2.0 mmol), base (4.0 mmol) and catalyst (0.01 mmol) was mixed in (EtOH/H_2O) (1:3) (5 mL) at 25 °C for 1 h (Fig. 2). After the reaction completed, the mixture was extracted with ethyl acetate. The ethyl acetate solution was dried over $MgSO_4$ and filtered to remove the trace water content. The ethyl acetate solution was passed through a silica column (60-120 mesh), and there for impurities were removed. The yields were measured by GC-MS.

The optimization conditions were determined first during the experimental studies. In order to determine the appropriate base to be used in the Suzuki reaction, a reaction of p-bromoacetophenone and phenylboronic acid was performed in EtOH/H2O (1:3) mixture at 25 °C and atmospheric condition for 1 h. Optimum conditions were formed using **1a** complex as catalyst. Various bases such as KOH, NaOH, $KOBu^t$, K_2CO_3 and Cs_2CO_3 were checked. The obtained results are given in Table 1 and Table 2.

RESULTS AND DISCUSSION

IR spectra of Pd(II) complexes **(1a-d)** showed that azomethine peaks shifted to a high or low area, unlike ligands. This indicated that the Pd metal was coordinated with the N atom of the azomethine group of ligands. In the complexes **1a** and **1c**, it was connected with carbonyl oxygen and supported by band shifts [15]. In the **1b** complex, the absence of the peak of the OH group seen in the ligand and the shift in the (C-O) peak, unlike the ligand, indicated that there was coordination with the phenolic oxygen atom [16]. The new bands for all Pd(II) complexes (**1a-d**) in range of 573-510 cm^{-1} and 503-463 cm^{-1} suggested a coordination of the carbonyl/hydroxyl oxygen and azomethine nitrogen atom to the palladium, respectively [6, 17-19].

The ^{1}H-NMR spectra of Pd(II) complexes (**1a-d**) showed signals 8.10, 8.80, 8.32, 8.68 ppm for azomethine (-CH=N-) proton, respectively. For **1b** complex, the phenolic OH proton disappeared because of the deprotonation of

Table 3. Suzuki coupling reaction of various aryl bromide with phenylboronic acid.

Entry	*Pd (II)*	*Aryl Bromide*	*Temp (ºC)*	*Yield (%)*
1	**1a**		25	95
2	**1b**			96
3	**1c**			98
4	**1d**			98
5	**1a**		25	66
6	**1b**			97
7	**1c**			99
8	**1d**			98
9	**1a**		25	93
10	**1b**			95
11	**1c**			96
12	**1d**			96
13	**1a**		25	96
14	**1b**			75
15	**1c**			89
16	**1d**			94
17	**1a**		25	100
18	**1b**			100
19	**1c**			100
20	**1d**			73

[1]Reaction conditions: Phenyl boronic acid (3.0 mmol), aryl bromide (2.0 mmol), base (4.0 mmol), catalyst (0.01 mmol), solvent (5 mL) and 25 °C, 1h.

the ligand.

The ^{13}C-NMR spectra resonance of azomethine (-CH=N-) carbon atom of Pd(II) complexes (**1a-d**) observed at 163.01, 161.61, 159.30, 157.20 ppm, respectively. This signal indicated coordination of azomethine nitrogen Pd atom [18-20].

The UV-Vis spectrum of the complexes (**1a-d**) exhibited the bands 205-251 nm, which might be attributed to π→π* transition of the aromatic rings. The bands observed at 301-343 nm region might be attributed to n→π* transitions of the C=N moiety. Pd(II) complexes **(1a-d)** showed a low energy band in the range of 504-343 nm for MLCT (metal to ligand charge transfer) transitions. These results may suggest that Pd(II) complexes had a square-planar geometry [6, 22-26].

Table 3 showed that the palladium complexes (**1a-d**) had a good activity in Suzuki-Miyaura C-C coupling reaction. It was seen that there was no precise ranking between the activities of the complexes. The catalytic activities of some complexes were observed to vary between 66-100%, depending on the aryl bromides used (Table 3, entries 1-20). A yield of up to 100% was obtained with halogenated substrates bearing substituted groups attached to the aromatic ring. The activity of the bromoaryls used as the substrate appeared to vary from *p*-bromoacetophenone > *p*-bromoanisole > *p*-bromobenzene > *p*-bromoaldehyde > *p*-bromotoluene. This means that the catalytic activity of Pd(II) (**1a-d**) complexes were the highest in the case of p-bromoacetophenone, providing 95-98% conversion (Table 3, entries 1-4). It was found that both catalysts and aryl bromide were active in terms of structure.

In the present study, it was observed that the efficiency of catalytic transformations for all complexes did not change significantly depending on the substituting groups of Schiff base-Pd(II) complexes. The electronic parameters had no important effect on the catalytic conversion, but the steric effect of the groups present in the Schiff base structure significantly altered the activity of the complexes. In case of *p*-bromoaldehyde used as an aryl bromide, it provided a conversion between 66-98% (Table 3, entries 5-8). In this study, it was found that the efficiency of catalytic transformations for all complexes varied depending on the substituted groups of Schiff base-Pd(II) complexes. Electronic parameters of Schiff base ligands had a significant effect on the catalytic conversion. The absence of any groups attached to the benzene ring of the ligand resulted in a low yield of 66% in the **1a** complex. Therefore, it was concluded that the catalysts significantly affected the activity of the electronic structures.

When *p*-bromoanisole was used as an aryl bromide, catalytic conversion of the (**1a-d**) complexes ranging from 93-96% was obtained in the rate of catalytic conversion (Table 1, entries 9-12). When the catalytic transformations of the (**1a-d**) complexes were evaluated, it was seen that the activity of the catalysts did not change due to the substituted groups of Schiff base-Pd(II) complexes and gave close results to each other. Although the electronic parameters did not have an important influence on the catalytic conversion, the steric effect changed the activity of the complexes significantly. Therefore, it was found that the groups in the Schiff base structure had a significant influence on the activity of the catalysts.

When *p*-bromotoluene was used as an aryl bromide, catalytic transformations ranging from 75-96% were obtained in the catalysis of **1a-d** complexes (Table 1, entries 13-16). The assessment of the catalytic transformations of Pd(II) complexes (**1a-d**) showed that the activity of the catalysts did not change due to the substituting groups of Schiff base-Pd(II) complexes and gave similar results. The electronic parameters did not have an important impact on the catalytic conversion; however, the steric effect significantly changed the activity of the complexes. Pd(II) complex (**1b**) with *p*-bromotoluene was found to show the lowest rate of catalytic activity (Table 1, entry 14). This was due to the electron attracting –OH group in the structure of the **1b** complex.

When *p*-bromobenzene was used as aryl bromide, a good catalytic conversion of 73-100% was achieved (Table 1, entries 17-20). The catalytic transformations of **1a-d** complexes indicated that the activity of the catalysts did not change due to the substituted groups of Schiff base-Pd(II) complexes (**1a-d**) and gave similar results. Although the electronic parameters had no significant impact on the catalytic conversion, the steric effect significantly influenced the activity of the complexes. **1d** complex with p-bromobenzene was found to show a relatively low rate of catalytic activity (Table 1, entry 20). This was due to the electron-providing –OCH_3 group in the structure of the **1d** complex.

The Schiff base-Pd(II) complexes **(1a-d)** used in the Suzuki reaction yielded a good degree of catalytic conversion ranging from 66-100%. At the same time, the structure and position of the ligand-bound substituted groups increased their catalytic activity. It was found that the electronic structure of the Schiff base groups and the electronic effect of the substrate used did not significantly affect the activity of the catalysts. When the catalytic results of Pd(II) complexes were examined, it was observed that all complexes were active for Suzuki-Miyaura coupling reactions and formed active catalytic systems under optimum conditions. Pd(II) complexes (**1a-d**) were found to have a very good catalytic activity.

CONCLUSION

In this study, four new stable and easily soluble Schiff base-Pd(II) complexes (**1a-d**) containing thiophene and pyridine rings were synthesized. The structures were elucidated using different spectroscopic techniques. Spectroscopic data showed that the Pd(II) complexes had a square plane geometric structure. It was also found that Schiff base-Pd (II) complexes provided an excellent catalytic activity in the Suzuki-Miyaura coupling reaction. Pd(II) complexes (**1a-d**) occurred in the Suzuki coupling reactions in a short time and at low temperatures. Thus, it was concluded that the groups in the Schiff base structure had a significant effect on the activity of the catalysts.

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