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 [PdCl₂(CH₃CN)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 (¹H) and carbon (¹³C) 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/H₂O (1/3) at room temperature with K₂CO₃ as the base for 1 hour. The Pd(II) complexes (1a-d) used as catalysts exhibited perfect catalytic activity in the Suzuki-Miyaura reaction.

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 accessiblity, 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 aryloboronic acids and widely used to obtain biaryl. 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, biaryl 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 [PdCl₃(CH₃CN)₂] metal salts, and their catalytic activities were investigated. The formation of Pd(II) complexes was determined through FT-IR, microanalysis, UV-Vis, ¹H and ¹³C-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.
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 ¹³C) 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 [PdCl₂(CH₃CN)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 C₂₂H₂₈N₂O₅SPdCl₂ (%): 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-d₆, ppm): 8.10 (s, 1H, CH=N), 7.81-7.49 (m, 5H, ArH), 4.35, 4.20 (m, 4H, -2CH₂), 3.80-3.11 (m, 4H, CH₂ (pyridine)), 1.36, 1.20 (d, 12H, -CH₃). ¹³C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 43.00, 18.00 (pyridine), 24.01 (C(CH₃)₃). UV-Vis. (λmax, nm): π→π*, 217, 225, 233; n→π*, 302, 384. LC-MS: m/z: 590.90 (cald.), 590.97 (found) [M-H₂O]+.

Complex 1b: Anal. cald. for (C₂₂H₃₁N₂O₆SPdCl) (%): 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-d₆, ppm): 8.80 (s, 1H, CH=N), 7.69-6.97 (m, 4H, ArH), 4.54, 4.40 (m, 4H, -2CH₂), 3.35-2.51 (m, 4H, CH₂ (pyridine)), 1.46-1.26 (d, 12H, -CH₃). ¹³C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 44.02, 18.00 (pyridine), 28.51 (C(CH₃)₃). UV-Vis. (λmax, nm): π→π*, 221; n→π*, 301, 391, 504. LC-MS: m/z: 590.90 (cald.), 590.97 (found) [M-H₂O]+.

Complex 1c: Anal. cald. for (C₂₂H₂₃N₃O₇SPdCl₂) (%): 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=NO₂), 781 (C-S-C), 556, 516 (M-O), 493, 467 (M-N). ¹H-NMR (300 MHz, DMSO-d₆, ppm): 8.32 (s, 1H, CH=N), 7.50-7.01 (m, 4H, Ar-H), 5.76, 4.19 (m, 4H, -2CH₂), 3.35-2.80 (m, 4H, CH₂ (pyridine)), 1.46-1.26 (d, 12H, -CH₃). ¹³C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 44.02, 18.00 (pyridine), 28.51 (C(CH₃)₃). UV-Vis. (λmax, nm): π→π*, 221; n→π*, 301, 391, 504. LC-MS: m/z: 553.42 (cald.), 553.17 (found) [M-Cl]-.

Complex 1d: Anal. cald. for (C₂₅H₃₇N₃O₆SPdCl₂) (%): 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-d₆, ppm): 8.10 (s, 1H, CH=N), 7.81-7.49 (m, 5H, ArH), 4.35, 4.20 (m, 4H, -2CH₂), 3.80-3.11 (m, 4H, CH₂ (pyridine)), 1.36, 1.20 (d, 12H, -CH₃). ¹³C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 43.00, 18.00 (pyridine), 24.01 (C(CH₃)₃). UV-Vis. (λmax, nm): π→π*, 217, 225, 233; n→π*, 302, 384. LC-MS: m/z: 590.90 (cald.), 590.97 (found) [M-H₂O]+.
(m, 4H, CH₂ (pyridine)), 1.69-1.21 (d, 12H, -CH₃). 13C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 47.00, 15.16 (pyridine), 29.16 (C(CH₃)₃).

UV–Vis. (λmax, 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 (C₂₃H₃₀N₂O₆SPdCl₂) (%): 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 (-OCH₃), 757 (C-S-C), 573, 510 (M-O), 487 (M-N). 1H-NMR (300 MHz, DMSO-d₆, ppm): 8.68 (s, 1H, CH=N), 7.95-6.90 (m, 4H, ArH), 5.37, 4.32 (m, 4H, -2CH₂), 3.97-2.42 (m, 4H, CH₂ (pyridine)), 1.28-1.22 (d, 12H, -CH₃). 13C-NMR (75 MHz, DMSO-d₆, 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 (-OC₂H₅), 53.44 (pyridine), 28.70 (C(CH₃)₃). UV–Vis. (λmax, 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 1. Optimization of the reaction conditions for Suzuki coupling reaction.

| Entry | Base       | Time (h) | Yield (%) |
|-------|------------|----------|-----------|
| 1     | NOH        | 8        | 64        |
| 2     | Cs₂CO₃    | 8        | 51        |
| 3     | KOH        | 8        | 67        |
| 4     | K₂CO₃     | 8        | 95        |
| 5     | KOBu⁺     | 8        | 68        |
| 6     | -          | 8        | -         |
| 7     | K₂CO₃     | 1        | 95        |

*Reaction conditions: Phenyl boronic acid (3.0 mmol), aryl bromide (2.0 mmol), base (4.0 mmol), catalyst (0.05 mmol), solvent (5 mL) and 25 °C.

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/H₂O (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 MgSO₄ and filtered to remove the trace water content. The ethyl acetate solution was passed through a silica column (60-120 mesh), and therefore 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⁺, K₂CO₃ and Cs₂CO₃ 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 the 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 ¹H-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
The 13C-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.

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

| Entry | Pd (II) | Aryl Bromide | Temp (ºC) | Yield (%) |
|-------|---------|--------------|-----------|-----------|
| 1     | 1a      | Br-\(\text{COCH}_2\) | 25        | 95        |
| 2     | 1b      | \(\text{Br-CHO}\) | 25        | 96        |
| 3     | 1c      | \(\text{Br-OC}_3\) | 25        | 98        |
| 4     | 1d      | \(\text{Br-CH}_3\) | 25        | 98        |
| 5     | 1a      | \(\text{Br-H}\)   | 25        | 66        |
| 6     | 1b      | \(\text{Br-H}\)   | 25        | 97        |
| 7     | 1c      | \(\text{Br-H}\)   | 25        | 99        |
| 8     | 1d      | \(\text{Br-H}\)   | 25        | 98        |
| 9     | 1a      | \(\text{Br-H}\)   | 25        | 93        |
| 10    | 1b      | \(\text{Br-H}\)   | 25        | 95        |
| 11    | 1c      | \(\text{Br-H}\)   | 25        | 96        |
| 12    | 1d      | \(\text{Br-H}\)   | 25        | 96        |
| 13    | 1a      | \(\text{Br-H}\)   | 25        | 96        |
| 14    | 1b      | \(\text{Br-H}\)   | 25        | 75        |
| 15    | 1c      | \(\text{Br-H}\)   | 25        | 89        |
| 16    | 1d      | \(\text{Br-H}\)   | 25        | 94        |
| 17    | 1a      | \(\text{Br-H}\)   | 25        | 100       |
| 18    | 1b      | \(\text{Br-H}\)   | 25        | 100       |
| 19    | 1c      | \(\text{Br-H}\)   | 25        | 100       |
| 20    | 1d      | \(\text{Br-H}\)   | 25        | 73        |

*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 ºC, 1h.*
ted 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 13-16). 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 –OCH3 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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