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FULL PAPER

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A catalytic system with high efficiency and recyclability based on Suzuki and Heck reaction in aqueous admicellar medium

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A recyclable and non-phosphine solid palladium (II) catalyst was prepared and characterized by **HR-MS**, **FT-IR**, **XPS**, **EDS**, **TGA**, **SEM**, **TEM** and **ICP**. The Pd-catalyst exhibited high-performance catalytic activity in Suzuki and Heck C-C coupling reactions in an environmentally benign water medium. Further, the Pd-catalyst (**Z_4**) can be reused for 15 times with little decrease of activity through simple and efficient recovery. In addition, the turn-over number (TON) of Pd- catalyst can reach 380 at room temperature. These results proved that the Pd-catalyst has a stable structure and can be recycled many times, making the process sustainable.

KEYWORDS
aqueous admicellar medium, heck reaction, Pd-catalyst, Suzuki reaction

1 | INTRODUCTION

Pd-catalyzed C-C cross-coupling reactions have been emerged as a versatile tool in organic synthesis.[1–3] Such as Suzuki-Miyaura and Mizoroki-Heck cross-coupling reactions have been become essential methods in the synthesis of biaryl and aryl olefinic compounds.[4] In general, these reactions were usually carried out in the presence of Pd-phosphine complexes.[5–7] Despite their excellent catalytic performance, they suffer from high cost, toxic, unrecoverable and unstable phosphane ligands. Nowadays, the environmental awareness encourages more friendly chemical processes. Researchers have begun to implement green chemistry practices using of new recyclable, heterogeneous or phosphine-free catalysts. In detail, they have developed palladium catalysts using nitrogen containing,[8,9] N-heterocyclic carbenes, oximes,[10] and imines ligands,[11] and so on.

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Now, there is a necessary to design new catalysts that can retain the selectivity and activity of homogeneous catalysts and also can be removed from the reaction mixture, and reused following appropriate washing processes.[12] The usually used method was to immobilize palladium complexes on various supports.[13–17] So far, polymer-supported palladium catalyst and self-assembled imidazole-palladium catalysts have been developed for the Suzuki and Heck coupling reactions in water with high efficiency and recyclability.[18,19] Recently, we have developed a salen-type palladium (II) catalyst (Z_4) that exhibits excellent catalytic activity for Suzuki and Heck cross-coupling reactions under mild conditions (Figure 1). The steric environment adjacent to the nitrogen atom in the ligand parts enhanced the thermal stability of the palladium species.[20] On the other hand, increasing the donor capacity and steric bulk of the ligand should favor the oxidation addition and reduction elimination steps in the catalytic cycle, respectively.[21] Moreover, these two factors also promote the activation of the catalyst, which involves the formal reduction of the Pd (II) precatalyst to the Pd(0) active species.[22] Thus, these made the Pd-catalyst have an excellent catalytic activity with recyclability.

Meanwhile, the replacing dangerous, and expensive organic solvents with water are an important subject from a different perspective. In recent years, water has been applied to an array of organic reactions because it is a safe, abundant and a green reaction medium.[23] To realize the high catalytic performance in water, a method by adding phase-transfer agents have been reported.[24] Herein, we prepared a pd-catalyst (**Z_4**) (Figure 1) as a recyclable, heterogeneous and phosphine-free catalyst for Suzuki and Heck reactions under environmentally friendly conditions. Moreover, the reaction can be completed in aqueous admicellar medium in the presence of SDS (Sodium dodecyl sulfate)/K_2CO_3.

2 | EXPERIMENTAL

Experimental procedures, NMR spectra and other data for this study are available in Supporting Information.

3 | RESULT AND DISCUSSION

3.1 | Preparation and characterization of the catalyst

Firstly, the Pd-catalyst (**Z_4**) was prepared by a four-step procedure[25] (see **SI Scheme S_1**). The Pd-catalyst has a high melting point above 290 °C, and its poor solubility in organic solvents was observed, which enable the catalyst to be a solid catalyst for organic reactions. The structure of the pd-catalyst is characterized using different techniques such as HR-MS, FT-IR, XPS, EDS, TGA, FE-SEM, TEM and ICP. In the ^{1}H NMR spectrum of ligand (**Z_3**), a peak representing the hydroxyl proton resonated at δ 13.61 ppm, and some peaks corresponding to other protons are also shown. Unfortunately, NMR spectra were not recorded due to poor solubility of pd-catalyst (Z_4) in all accessible solvents (include $CDCl_3$, DMSO-*d*, DMF-*d*, THF-d_8, $(CD_3)_2CO$, CD_3CN, D_2O). A peak at m/z 1111.2957 in the HR-MS spectrum of Pd-catalyst (Z_4) corresponding to the $[M + H]^+$ ion further confirmed the structure of Pd-catalyst (Z_4). (See **SI Figure S1**).

3.2 | FT-IR spectroscopy and X-ray photoelectron spectroscopy (XPS) analysis

The FT-IR was employed to give a detailed investigation of the ligand (**Z_3**) and the pd-catalyst (**Z_4**). The Figure 2(A) exhibited a broadband at 3439 cm^{-1} correspond to the -OH group with the ligand (**Z_3**), and it was indicated from strong IR band appeared at 1620 cm^{-1} due to the C=N vibrations. In the FT-IR spectrum of Figure 2(B), the C=N peak was shifted to 1608 cm^{-1}, and the peak in the region of 3439 cm^{-1} (-OH stretch) disappeared.[26] These suggested that complete complexation with the palladium had occurred through the nitrogen of the C=N group and oxygen of the -OH group.

The XPS elemental survey scan of the surface of ligand (**Z_3**) and pd-catalyst (**Z_4**) were presented in Figure 2. As can be seen in Figure 2(C), three intense peaks at 300, 420 and 540 eV were corresponding to C1s, N1 s and O1s,[27] showing the existence of these three elements in ligand (**Z_3**). Besides, the peak with binding

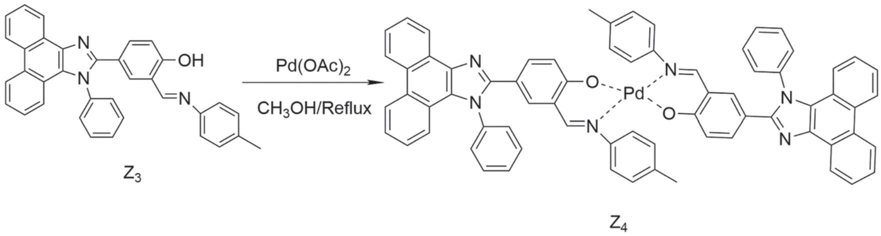


FIGURE 1 The Pd-catalyst (Z_4) structure

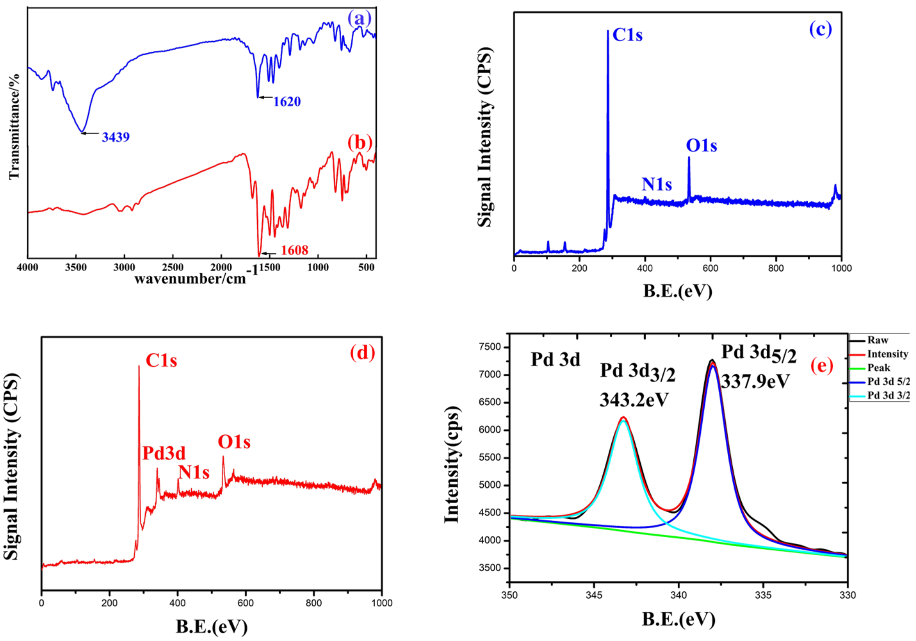


FIGURE 2 The comparison of FT-IR between the ligand (Z_3) (A) and its pd-catalyst (Z_4) (B); XPS between the ligand (Z_3) (C) and pd-catalyst (Z_4) (D), full range; XPS signals of the Pd3d core levels in the Pd-catalyst (Z_4) (E)

energy of 350 eV was related to Pd3d, as shown in Figure 2(D). Full XPS spectra of ligand (**Z_3**) and pd-catalyst (**Z_4**) contain signature peaks of different elements (Figure 2 **C, 2 D**). Additionally, to investigate the oxidation state of Pd in pd-catalyst, XPS analysis was conducted (Figure 2 **E**). The high-resolution Pd3d XPS scan of pd-catalyst revealed the doublet peaks at the binding energy of 337.9 eV and 343.2 eV, which can be indexed to electron transitions of 3d5/2 and 3d3/2 of Pd, respectively. This result confirmed that the chemical state of palladium is +2 in pd-catalyst and palladium (II) was not reduced during the preparation of the Pd-catalyst. The binding energy peak affirms the formation of Pd (II)-complex.

3.3 | Energy-dispersive spectroscopy (EDS) analysis

Furthermore, the ligand (**Z_3**) and pd-catalyst (**Z_4**) were examined using EDS analysis to determine the chemical composition (**SI Figure S_2**). The EDS mapping images in **Figure S_2(B)** revealed the presence of O, N, C and importantly Pd elements in the pd-catalyst (**Z_4**) structure, the amount of palladium obtained from EDS analysis is around 13.76 (wt %). However, EDS pattern in **Figure S_2(A)** indicated the presence of O, N and C elements in ligand (**Z_3**). Meanwhile, the amount of loaded Pd was determined to be 0.498 mmol/g^{-1} using inductively coupled plasma optical emission spectrometry (ICP-OES). Another catalytic experiment was further conducted to estimate the impact of Pd leaching. The amount of Pd remaining after the 15 cycles was 0.381 mmol/g^{-1} as determined using ICP-OES analysis (23% leaching).

3.4 | Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed to study the thermal stability of the ligand(**Z_3**) and Pd-catalyst(**Z_4**), using a heating rate of 10 °C/min under a nitrogen atmosphere between 30 °C and 600 °C (See **SI Figure S_3**). The loss of hydroxyl groups resulted in a weight loss of 5% between 126.3 °C for ligand (**Z_3**) (Figure S_3A), whereas loss of covalently bonded organic moieties at a temperature up to 312 °C resulted in a weight loss of 29%, whereas the pd-catalyst (**Z_4**) showed decomposition in two stages (Figure S_3B). The loss of 4% at a temperature up to 188.9 °C forms the first stage; loss of covalently bonded organic moieties at a temperature up to 350.6 °C resulted in a weight loss of 15%. Obviously, the pd-catalyst (**Z_4**) was stable at higher temperatures, which is also possible to be used as a catalyst in higher-temperature organic reactions.

3.5 | Field emission-scanning electron microscopic (FE-SEM) and transmission electron microscopy (TEM) analysis

To gain more insight into the Pd-complex catalyst, and survey the size, shape and surface morphology of ligand (**Z_3**) and pd-catalyst (**Z_4**), the FE-SEM and TEM (Figure 3) technology were used. The SEM images in Figure 3(A) showed that the ligand (**Z_3**) displayed a

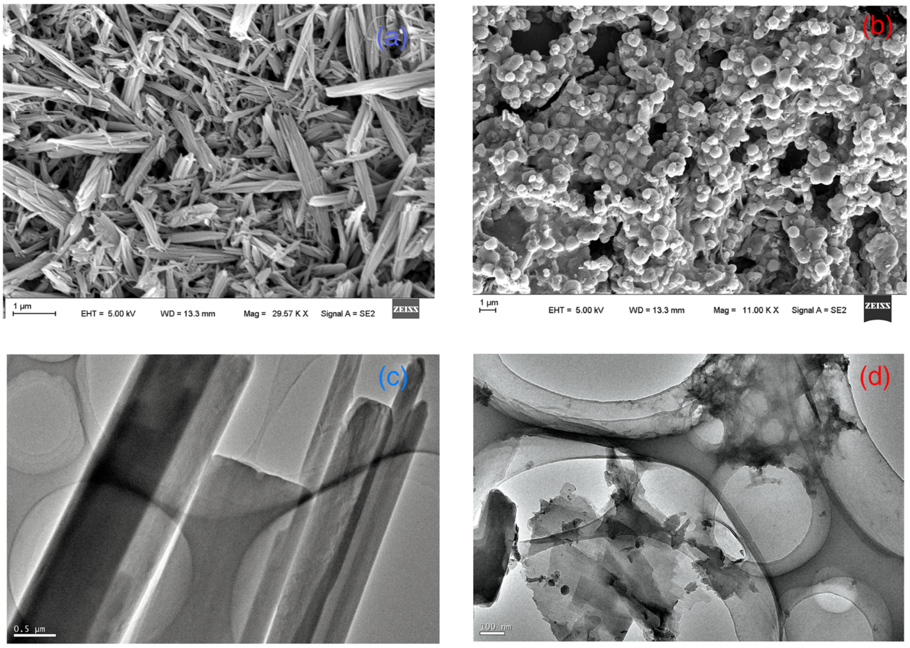


FIGURE 3 The SEM micrographs of ligand (Z_3) (A), pd-catalyst (Z_4) (B); TEM micrographs of ligand (Z_3) (C), Pd-catalyst (Z_4) (D)

uniform and nearly a fusiform shape. At the same time, Figure 3(B) displayed that fresh Pd- catalyst (**Z_4**) has a monodispersed with diameter of about 0.32–0.63 μm. The observed SEM images demonstrated the morphological and characteristic distinct vision between the ligand and the Pd-catalyst due to the change in the coordination of ligand (**Z_3**) with palladium by the coordination bond. The morphologies of the ligand (**Z_3**) and pd-catalyst (**Z_4**) were found out by transmission electron microscopy (TEM). TEM image Figure 3(C) indicated a uniform and nearly a fusiform shape of ligand (**Z_3**) was clearly observed in the TEM image. After ligand (**Z_3**) was coordinated with palladium through a coordination bond, and then in Figure 3 **(D)**, obvious Pd species on the surface of pd-catalyst (**Z_4**) was observed[26]. Furthermore, the presence of Pd elements was confirmed by EDS mapping images.

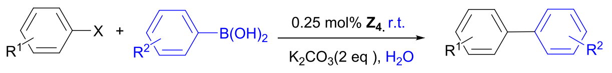
3.6 | The Suzuki–Miyaura cross-coupling reaction

On the successful preparation of the pd-catalyst (**Z_4**), it's catalytical-reactivity was tested in the Suzuki reaction. In the beginning, 4-Bromoanisole (**a_1**) and 4-Methylbenzeneboronic (**b_1**) were chosen as the model substrates for the reaction (see **SI Table S1**). From the initial screening, we found that the coupled product (**1**) was obtained in 85% isolated yield when K_2CO_3 was used as bases (Table S1, entries 1–6). Furthermore, the catalyst loading screening showed that increasing the catalyst loading could improve the reaction yield (Table S1, **entries 6–9**). It seemed that when the Pd-catalyst (**Z_4**) loading was 0.25 mol%, the corresponding product was obtained in 85% yield (Table S1, **entries 7**). We focused on replacing organic solvents with water. It seemed that the presence of water increases the solubility of the base and activates the boronic acid, thereby increasing the rate of reaction in aqueous medium.[28] To our delight, the reaction proceeded smoothly by adding PEG-400 and DMF to H_2O (Table S1, **entries 10–15**). This result was in accordance with our previous work.[23] Even if the amount of PEG-400 and DMF decreased from 1/1 to 1/10 the reaction was still proceeded smoothly. In water without other additive the reaction to give the desired products in 40% yields (Table S1, **entry 16**). It was worth noting that in the presence of SDS the yield of coupled product (**1**) was remarkably improved. (Table S1, **entries 17–22**). The SDS can play an important role as the phase transfer catalyst (PTC). The critical micelle concentration (CMC) of SDS as an efficient surfactant was around 8000–10000 μmol L^{-1}[29]. Next, we chose the CMC of the SDS. Notably, when the reaction was performed in the absence of pd-catalyst (**Z_4**), no formation of product coupled product **(1)** was observed (Table S1, **entry 9**). Meanwhile, Pd $(OAc)_2$, $PdCl_2(PPh_3)_2$ and pd-catalyst (**Z_6**)[30] (see **SI Scheme S_2**) as comparative catalysts gave a very poor yield forming product coupled product **(1)** under optimal conditions (Table S1, **entries 23, 24, 25**), and the structure of pd-catalyst (**Z_6**) was confirmed according to HR-MS and **FT-IR** Spectroscopy analysis (See **SI Figure S_4, Figure S_5**). This might be due to the steric environment adjacent to the nitrogen atom of the ligand (**Z_3**) enhanced the thermal stability of the palladium species. On the other hand, increasing the donor capacity and steric bulk of the ligand should facilitate the activation of the catalyst, which involved the formal reduction of the Pd (II) precatalyst to the Pd(0) active species. Thus, these results made that the Pd-catalyst (**Z_4**) has an excellent catalytic activity with recyclability. Finally, the yield was improved with the increasing of reaction time (Table S1, **entries 17–21**). It was also found that temperature has little effect on the yield of the product (Table S1, **entries 17 and 22**). Therefore, the best condition in this reaction was the combination of pd-catalyst (**Z_4**) as a catalyst and water as the solvent under room temperature in the presence of SDS/K_2CO_3.

The scope and generality of the optimized protocol was explored for Suzuki-Miyaura reaction by using a variety of aryl halide and phenylboronic acids in **Table** 1. The electronic nature of the coupling partner has little influence on the reaction efficiency. Notably, 2-naphthylboronic acid also reacted efficiently with aryl bromides to give the desired products (**23–25)** in good yields. Remarkably, ortho-substituted aryl halides were coupled with aryl boronic acids to give corresponding products in moderate yields (76%–80%) (**4, 11, 17, 18**). However, highly sterically hindered di-ortho substituted biphenyls cannot be synthesized under the same conditions (**7**), indicating that the catalyst was the low tolerance toward steric hindrance at the substrates. An increased of reaction time and catalyst loading did not significantly affect this behaviour. Finally, aryl chlorides were evaluated under room temperature, but none of them showed enough activity to give product. However, the reaction showed smooth reactivity at 80 °C and gave coupling product (**26**) and coupling product (**27**) in 50% and 51% isolated yields, respectively. Indicated that aryl chlorides were less active than aryl bromides, and their reluctance to perform the oxidative addition to palladium makes the coupling procedure very difficult.[31]

The recyclability of a catalyst is an important factor of catalysis reactions. We explored the reusability of pd-catalyst (**Z_4**) by a simple filtration from the reaction mixture. The catalyst showed excellent activity for 15 runs with no appreciable change in the product yield (95–80%) (See **SI Figure S_6**). **EDS** determined the palladium element of the Pd-catalyst (**Z_4**) after 15 consecutive runs, and no change was observed. Meanwhile, the amount of Pd remaining after the 15 cycles was 0.381 mmol g^{-1} as determined using ICP-OES analysis (23% leaching). These results proved that the Pd-catalyst (**Z_4**) has a stable structure and can be recycled many times with good sustainability. Further, we also studied the critical reaction parameters of catalyst (turnover numbers = TON; turnover frequency = TOF). **Table** 1 and **Table** 2 showed the results of these investigations. It was possible to achieve a TON of 380 for the reaction at room temperature (**1**). These results represented a significant improvement compared with refluxed conditions in the literature,[32] although vale of TON is equivalent. The results showed that the pd-catalyst (**Z_4**) has an excellent catalytic activity and high catalytic efficiency in the Suzuki reaction.

TABLE 1 Scope of the Suzuki reaction of aryl halogen with arylboronic acids[a]



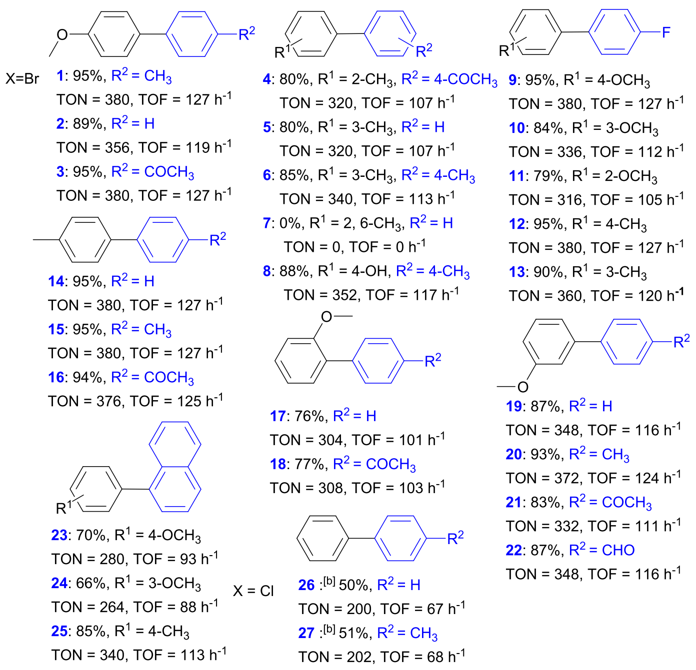
[a] Reaction conditions: r. t.; the Pd-catalyst (Z_4) 0.25 mol%; 0.3 mmol of aryl halide, 0.33 mmol of aryl boric acid, 0.6 mmol of K_2CO_3; 4 ml H_2O, 9.2 mg SDS; in air; 3 hr; Isolated yields; TON = (moles of product) / (moles of Pd in the catalyst), TOF = TON/reaction time, [b] 80 °C.

3.7 | The Mizoroki-heck cross-coupling reaction

Initially, 4-Iodoanisole (***c_1***) and Methyl acrylate (***d_1***) were chosen as the model substrates for Heck reaction. To our delight, it can be observed that the yield was significant enhanced to 86% at 80 °C. Meanwhile, as the reaction time was increased the yields are also improved to 88% (See **SI Table S_2**).

The scope and generality of the optimized protocol was explored for Heck reaction of various aryl iodide and bromides with olefins in **Table** 2. It can be observed that aryl iodine with electron withdrawing substituents such as -NO_2, -CN, and -Cl led to high yields of the corresponding products (**29–31**). Aryl iodines with electron-donating groups like -OMe underwent the reaction smoothly to give moderate yields of products (**28, 33, 39, 41, 44, 47**). Notably, the yields of products appeared to be influenced by the electronic nature of the aryl group, with electron-neutral or electron-donating aryl iodides giving lower yields than electron withdrawing aryl iodides (**28–31, 33, 39, 41, 44, 47**). Interestingly, the sterically hindered naphthyl aryl iodine, 4-tert-butyliodobenzene and 4-iodobiphenyl afforded the desired products in good yields (**32, 37, 38**). We further investigated the reaction of 2-bromothiophene with methyl acrylate, affording 89% yield of the desired product (**36**) in 36 hr. Excitingly, the reaction of aryl bromides with olefins generate products in over 62% yield (**49, 50**), highlighting the applicability of the reaction protocol in aryl bromide. We further investigated the substrate scope of various aryl chlorides with olefins. Unfortunately, the reaction does not work, which indicated that the activation of aryl chlorides was less than that of aryl iodides and bromides.[29]

TABLE 2 Scope of Mizoroki-Heck cross-coupling reaction of aryl halogen with olefins[a]



X=Br

1: 95%, R^2 = CH_3
TON = 380, TOF = 127 h^{-1}
2: 89%, R^2 = H
TON = 356, TOF = 119 h^{-1}
3: 95%, R^2 = $COCH_3$
TON = 380, TOF = 127 h^{-1}
14: 95%, R^2 = H
TON = 380, TOF = 127 h^{-1}
15: 95%, R^2 = CH_3
TON = 380, TOF = 127 h^{-1}
16: 94%, R^2 = $COCH_3$
TON = 376, TOF = 125 h^{-1}
23: 70%, R^1 = 4-OCH_3
TON = 280, TOF = 93 h^{-1}
24: 66%, R^1 = 3-OCH_3
TON = 264, TOF = 88 h^{-1}
25: 85%, R^1 = 4-CH_3
TON = 340, TOF = 113 h^{-1}

4: 80%, R^1 = 2-CH_3, R^2 = 4-$COCH_3$
TON = 320, TOF = 107 h^{-1}
5: 80%, R^1 = 3-CH_3, R^2 = H
TON = 320, TOF = 107 h^{-1}
6: 85%, R^1 = 3-CH_3, R^2 = 4-CH_3
TON = 340, TOF = 113 h^{-1}
7: 0%, R^1 = 2, 6-CH_3, R^2 = H
TON = 0, TOF = 0 h^{-1}
8: 88%, R^1 = 4-OH, R^2 = 4-CH_3
TON = 352, TOF = 117 h^{-1}
17: 76%, R^2 = H
TON = 304, TOF = 101 h^{-1}
18: 77%, R^2 = $COCH_3$
TON = 308, TOF = 103 h^{-1}

X = Cl

26 :[b] 50%, R^2 = H
TON = 200, TOF = 67 h^{-1}
27 :[b] 51%, R^2 = CH_3
TON = 202, TOF = 68 h^{-1}

9: 95%, R^1 = 4-OCH_3
TON = 380, TOF = 127 h^{-1}
10: 84%, R^1 = 3-OCH_3
TON = 336, TOF = 112 h^{-1}
11: 79%, R^1 = 2-OCH_3
TON = 316, TOF = 105 h^{-1}
12: 95%, R^1 = 4-CH_3
TON = 380, TOF = 127 h^{-1}
13: 90%, R^1 = 3-CH_3
TON = 360, TOF = 120 h^{-1}
19: 87%, R^2 = H
TON = 348, TOF = 116 h^{-1}
20: 93%, R^2 = CH_3
TON = 372, TOF = 124 h^{-1}
21: 83%, R^2 = $COCH_3$
TON = 332, TOF = 111 h^{-1}
22: 87%, R^2 = CHO
TON = 348, TOF = 116 h^{-1}

[a] Reaction conditions: the Pd-catalyst (Z_4) 1.5 mol%; 0.4 mmol of aryl halide, 0.6 mmol of olefins, 0.8 mmol of K_2CO_3; 4 ml H_2O, 9.2 mg SDS; in air; 80 °C; 36 hr; Isolated yields.

4 | CONCLUSIONS

In summary, a novel non-phosphine palladium (II) catalyst was successfully prepared. This Pd-catalyst represented an excellent catalytic activity with recyclability in catalyzing Suzuki and Heck coupling reactions in aqueous admicellar medium. In the current system, the catalyst loading can be as low as 0.25 mol%, and the TON value can be 380. Meanwhile, the water solvent endows the reaction with green and safe properties.

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CONFLICT OF INTEREST

There are no conflicts to declare.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

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