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Palladium schiff base complex immobilized on magnetic nanoparticles: An efficient and recyclable catalyst for Mizoroki and Matsuda-Heck coupling

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Introduction

Palladium catalyzed Heck cross-coupling represent versatile tool for the carbon-carbon bond formation [1-3]. Heck cross-coupling has witnessed a widespread applications in organic synthesis and pharmaceuticals over last two decades and recognized the nobel prize to Heck, Suzuki and Nigeshi in 2010. Depending upon the coupling partner of alkenes with aryl halides or arenediazonium salts, two types of Heck coupling viz Mizoroki-Heck and Matsuda-Heck coupling exist. These are well established reactions useful in modern organic synthesis. Heck coupling plays a pivotal role in organic synthesis and has great industrial potential for the syntheses of fine chemicals, natural products, bioactive compounds, and advanced materials [4]. Heck coupling is catalyzed by Pd, and ligand as co-catalyst in basic conditions. The nature of ligand has an influence on the stability and efficiency of Pd catalyst. If ligand is not coordinated to the Pd, the process becomes costly, retains toxic ligand and its derivatives, thus causing the separation of products complicated [5]. In light of these facts scientists are engaged in development of either ligand-free methods or methods involving reusable co-ordinated ligand Pd complex for Heck coupling [6].

ABSTRACT

The present work elucidates the catalytic efficiency of palladium Schiff base complex immobilized on amine functionalized magnetic nanoparticles for Heck coupling of structurally different aryl halide/ arenediazonium tetrafluoroborate with styrene/acrylate/acrylonitrile. Matsuda-Heck coupling proceeds in aqueous media at room temperature whereas Mizoroki-Heck coupling was carried out at 80 °C. Both reactions were successfully furnished with low catalyst loading. The catalyst was easily separated from reaction mixture and reused up to six times without significant loss of catalytic activity.

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Over the decades, various homogeneous catalytic systems, with special ligands have been used for Heck coupling [7]. However, most of them suffer from drawbacks such as high ligand sensitivity towards air and moisture, tedious multistep synthesis, high cost of the ligand and use of various additives and harmful organic solvents. Because of the high cost of Pd, its recycling and recovery has became more necessary. To circumvent these problems, a variety of inorganic and organic solid-supported catalytic systems have been developed [8-10]. However, compared with their homogeneous counterparts, the solid-supported catalysts often suffer from lower activity and selectivity [11,12]. Thus, it is highly desirable to develop new heterogeneous catalytic systems comparable to homogeneous catalysts in catalytic performance. Recently, our group has designed magnetically separable catalyst, Pd-AcAc-Am-Fe₃O₄@SiO₂ (Fig. 1) and explored its catalytic activity in Suzuki-Miyaura cross coupling [13].

In continuation with our research interest in coupling reactions [14–18] and Pd-AcAc-Am-Fe₃O₄@SiO₂, herein we explored catalytic efficiency of Pd-AcAc-Am-Fe₃O₄@SiO₂ for the Mizoroki as well as Matsuda-Heck coupling.

Result and discussion

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https://doi.org/10.1016/j.tetlet.2020.151801 0040-4039/© 2020 Published by Elsevier Ltd. Initially, we have focused our attention on standardization of the procedure for Mizoroki-Heck coupling of bromobenzene and

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Fig. 1. Catalyst: Pd-AcAc-Am-Fe₃O₄@SiO₂.

styrene as model reaction to evaluate the influence of solvent, base and amount of catalyst. The results are summarized in Table 1.

When the reaction was performed in the absence of the catalyst, no product was detected. However, when the reaction was performed with Pd-AcAc-Am-Fe₃O₄@SiO₂ in DMF (Table 1, entry 3), the product was obtained in 82% yield after 5 h, which indicates that palladium plays a crucial role of catalyst in the Mizoroki-Heck coupling.

It was also observed that the yield of product improved to 92% in presence of DMF solvent and triethylamine (TEA) as a base (Table 1, entry 6). The acid formed in the reaction gets neutralized with triethyl amine forming a highly hydrophobic quaternary salt and this step is essential for breaking the activated complex, leading to product formation which generates again the catalytic species for fresh reactant. The amount of catalyst was also varied and we observed that even though with increasing the amount of catalyst to 0.5 mol %, no significant change in the yield or reaction time was found (Table 1, entry 8), while decreasing the amount of catalyst (0.2 mol %) reduced the yield of product to 88% (Table 1, entry 10). The reaction was conducted with various bases such as NaOH, K₂CO₃, and TEA. Among them, TEA exhibited the best result. Moreover, with the aim of checking the influence of solvent and temperature on the reaction yield, various organic solvents such as CH₃CN, EtOH, toluene, and DMSO were examined at wide range of temperature 25-120 °C. Gratifyingly, it is found that the entry 9 of Table 1 is the best conditions for Pd-AcAc-Am-Fe₃O₄@SiO₂ catalyzed Mizoroki-Heck coupling.

In order to check the catalytic activity of Pd-AcAc-Am-Fe₃O₄@-SiO₂ towards other reactants under optimized conditions, various types of iodo-, bromo-, and chloroaryl derivatives with electron-donating and electron-withdrawing groups were coupled with different vinylic substrates like acrylates, styrene and acrylonitrile

(Scheme 1). The *trans*-products were selectively obtained in all cases. The results exhibited that the above catalytic system is remarkably active and tolerates a range of functional groups. It was observed that the reactions of aryl iodides with terminal olefins, proceeds smoothly compared to aryl bromides and chlorides due to the lower bond energy of C-I in comparison with C-Br and C-Cl. The reaction of chlorobenzene with butyl acrylate, styrene and acrylonitrile afforded the product in lower yield than the iodobenzene (Table 2, entries 3e, 3j & 3p). The reaction of aryl halides bearing electron withdrawing groups such as -NO₂, -COCH₃, and -CN are faster than those with electron-donating groups such as -CH₃ and -OCH₃. Also, the Heck coupling of sterically hindered 1-iodonaphthalene afforded a good yield of the corresponding product (Table 2, entry 3k).

Changing the quantity from 1 equiv. to 1.5 equiv. of diazonium salt has no remarkable change in the yield of cross coupling product (Table 3, entry 8). Furthermore, amount of non-ionic surfactant, Triton X-100 was varied from 1 mol % to 10 mol % and observed that, 5 mol % of the surfactant gives better yield of the product (Table 3, entry 8). Therefore, a solution of arenediazonium salt in water was allowed to react with 1.2 equiv. of butyl acrylate, 5 mol % of surfactant, Triton X-100 in the presence of 20 mg (0.2 mol % Pd) of catalyst at room temperature and obtained 98% yield (Table 3, entry 10) of Matsuda-Heck coupling.

To evaluate the scope of the protocol, we applied these optimum conditions to screen different olefins and arenediazonium tetrafluoroborate salts (Scheme 2). The results are summarized in Table 4. We have studied the coupling of styrene with arenediazonium salts under the optimized reaction conditions. Due to the poor reactivity of the styrene substrate, the moderate yields of the products were obtained (Table 4, entries 6a–6e). In all cases, the coupling between arenediazonium tetrafluoroborate salts bearing electron donating as well as electron withdrawing groups with acrylate substrates, proceeded smoothly to afford the corresponding products in 92–98% yield (Table 4, entries 6f–6m). However, high yields were obtained for the arenediazonium salts, bearing strong electron withdrawing groups with acrylonitrile (Table 4, entry 6p).

A plausible mechanism for the Pd-AcAc-Am-Fe₃O₄@SiO₂ catalyzed Mizoroki–Heck and Matsuda-Heck cross-couplings is proposed in Scheme 3. The mechanism begins with the reduction of Pd(II) to the active Pd(O) species in the presence of alkene. Then, oxidative addition of the C—X bond of an aryl halide (X = I, Br or $-N_2^+BF_4$) to the palladium atom generates the σ -arylpalladium intermediate (A). In the next step, Pd (II) forms a π -complex

Table 1

Effect of solvents, base and amount of catalyst on Mizoroki-Heck coupling.



| Entry | Solvent | Base | Amount of Catalyst (Mol % Pd) | Time (h) | Yield (%) ^b |
|-------|------------|--------------------------------|-------------------------------|----------|------------------------|
| 1 | Water | NaOH | - | 12 | NR |
| 2 | Water | K ₂ CO ₃ | 0.4 | 12 | Trace |
| 3 | DMF | NaOH | 0.4 | 5 | 82 |
| 4 | DMF | K ₂ CO ₃ | 0.4 | 5 | 86 |
| 5 | DMF | K ₂ CO ₃ | 0.4 | 4 | 86 |
| 6 | DMF | TEA | 0.4 | 4 | 92 |
| 7 | DMF: Water | TEA | 0.4 | 5 | 72 |
| 8 | DMF | TEA | 0.5 | 4 | 92 |
| 9 | DMF | TEA | 0.3 | 4 | 92 |
| 10 | DMF | TEA | 0.2 | 4 | 88 |

^a Reaction conditions: Bromobenzene (1 mmol), styrene (1.1 mmol), base (2 mmol), temp. = 80 °C, solvent = DMF 5 mL; ^bIsolated yield.

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Scheme 1. Mizoroki-Heck coupling of different aryl halides with alkenes.

Table 2 Pd-AcAc-Am-Fe₃O₄@SiO₂ catalyzed Mizoroki-Heck coupling.



Reaction conditions: Aryl halide (1 mmol), alkene (1.1 mmol), Pd-AcAc-Am-Fe₃O₄@SiO₂ (0.3 mol % Pd), triethylamine (2 mmol), temp. = 80 °C solvent = DMF (5 mL). Yields are isolated.

followed by the migratory insertion of acrylate, results in the formation of palladium–carbon bond via a *syn*-addition, which gives complex (B). In the last step, cycle completed with formation of coupling product by β -hydride elimination and regeneration of Pd (0) occurs *via* reductive elimination of Pd (1).

The reusability of the catalyst was investigated for both Mizoroki-Heck and Matsuda-Heck coupling. (Fig. 2) After the first cycle of the reaction, nanocatalyst was recovered with help of an external magnet and then washed thoroughly with ethyl acetate $(2 \times 10 \text{ mL})$ and ether. The recovered nanocatalyst was dried under vacuum at 50 °C. Then, model reaction of Mizoroki-Heck and Matsuda-Heck was carried out under optimized reaction conditions. The results indicated that, at the end of six successive runs, there is no significant loss in the catalytic activity of the catalyst. The high catalytic efficiency of the Pd-AcAc-Am-Fe₃O₄@SiO₂ for Mizoroki-Heck coupling elucidated by comparison study with different catalysts from the literature (Table 5). The heterogenized Pd/ MnBDC in DMF offered heck product at very high reaction time (Table 5, entry 1). Some of the Pd catalytic systems were furnished at very high temperature (120 °C) (Table 5, entries 2–6). Ferrite supported Schiff base immobilized palladium (Fe₃O₄@Schiff-base-Pd) catalyzed heck reaction at 100 °C resulted in 3 h with low yield (Table 5, entry 7). Overall Pd-AcAc-Am-Fe₃O₄@SiO₂ is found to be catalyst of choice in terms of stability, non-toxicity, low loading of Pd in DMF in 3 h with very high yield (Table 5, entry 9).

We examined the Matsuda-Heck Coupling in the presence of several homogeneous and heterogeneous catalysts in comparison with Pd-AcAc-Am-Fe₃O₄@SiO₂ (Table 6). The catalytic system

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4 Table 3

Effect of solvents, amount of surfactant and catalyst on Matsuda-Heck coupling.^a



| Entry | Solvent | Equiv. of salt | Triton X-100 (mol %) | Amount of catalyst (Mol % Pd) | Time (h) | Yield (%) ^b |
|-------|--------------------|----------------|----------------------|-------------------------------|----------|------------------------|
| 1 | DMF | 1.2 | 10 | 0.4 | 2 | 82 |
| 2 | Ethanol | 1.2 | 10 | 0.4 | 12 | 95 |
| 3 | H ₂ O | 1.2 | 10 | 0.4 | 0.5 | 98 |
| 4 | THF | 1.2 | 10 | 0.4 | 5 | 62 |
| 5 | CH ₃ CN | 1.2 | 10 | 0.4 | 4 | 85 |
| 6 | H ₂ O | 1.2 | 1 | 0.4 | 4 | 89 |
| 7 | H ₂ O | 1.2 | 5 | 0.4 | 0.5 | 98 |
| 8 | H ₂ O | 1.5 | 5 | 0.5 | 1.0 | 98 ^c |
| 9 | H_2O | 1.2 | 5 | 0.3 | 0.5 | 98 |
| 10 | H ₂ O | 1.2 | 5 | 0.2 | 0.5 | 98 |
| 11 | H ₂ O | 1.2 | 5 | 0.1 | 2.0 | 91 |

Reaction conditions: Benzenediazodium salt (1.2 mmol), butyl acrylate (1 mmol), temp = r.t, solvent = 3 mL.

^b Isolated yield. ^c temp. 50 °C.



Scheme 2. Matsuda-Heck coupling of different arenediazonium salt with alkenes under the optimized conditions.

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Table 4





Reaction conditions: arenediazonium salt (1 mmol), olefin/acrylate/acrylonitrile (1.2 mmol), Catalyst (0.2 mol % Pd), Triton X-100 (5.0 mol %), H₂O (3 mL). Yields are isolated.



Scheme 3. Plausible mechanism for Heck cross coupling.



Fig. 2. Reusability of Pd-AcAc-Am-Fe₃O₄@SiO₂.

Agarose supported nano Pd comprised good yield at optimum reaction conditions, but accompanied with more reaction time (Table 6, entry 1). In some reports, the reaction was carried out

in organic solvent with 1 mol% of Pd loading (Table 6, entries 2 & 4). The Pd(OAc)₂ homogeneously catalyzed heck coupling in ionic liquid as solvent and water with Triton X-100 furnished high yield but with high loading of Pd (Table 6, entries 3 & 5). From this study it reveals that the catalyst, Pd-AcAc-Am-Fe₃O₄@-SiO₂ in comparison with reported catalyst is superior with marks of eco-friendly reaction conditions and high yield in short reaction time (Table 6, entry 6).

Conclusion

In summary, we have disclosed modified protocol for Heck coupling. The experimental procedure is simple, avoided the use of organic solvents for Matsuda-Heck coupling which is in agreement with green chemistry principles, utilization of an inexpensive and readily available catalyst, good reactivity to generate the corresponding products in good to excellent yields for all reactions are the advantages of the present method. In addition, the catalyst was easily separable from the reaction mixture by external magnet and can be reused for six runs without any significant loss of stability and activity.

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| Comparison of the | nresent methodology | with other reported | n caraivers for th | e Milzoroki-Heck | (α) |
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| Entry [Ref.] | Catalyst | Reaction conditions | Time (h) | Yield (%) ^a | TON ^b | $TOF^{c}(h^{-1})$ |
|---------------|---|--|----------|------------------------|------------------|-------------------|
| 1 [19] | Pd/MnBDC | Et ₃ N, DMF, 90 °C, 0.12 mol% Pd | 9-12 | 88 | 880 | 73 |
| 2 [20] | PFG-Pd | K ₂ CO ₃ , DMF, 120 °C, 1.7 mol% Pd | 3 | 90 | 53 | 18 |
| 3 [21] | Schiff-base-Pd@MNPs | K ₂ CO ₃ , DMF, 120 °C, 2.62 mol% Pd | 6 | 97 | 37 | 6 |
| 4 [22] | Pd-Py- MCM-41 | K ₂ CO ₃ , DMF, 120 °C, 3.12 mol% Pd | 4 | 94 | 30 | 7.5 |
| 5 [23] | Palladium(II) + SBA-16 | K ₂ CO ₃ , DMF, 80 °C, 1 mol% Pd | 1 | 98 | 98 | 98 |
| 6 [24] | Fe ₃ O ₄ @MCM-41@Pd-SPATB | K ₂ CO ₃ , PEG, 120 °C 0.94 mol % Pd | 0.5 | 92 | 98 | 196 |
| 7 [25] | Fe ₃ O ₄ @Schiff-base-Pd | Cs ₂ CO ₃ , H ₂ O, 100 °C, 0.3 mol % Pd | 3 | 76 | 253 | 84 |
| 8 [26] | Fe ₃ O ₄ @SiO ₂ -TCT-PVA-Pd(0) | Piperidine, reflux, 0.25 mol % Pd | 6 | 94 | 376 | 63 |
| 9 [This Work] | Pd-AcAc-Am-Fe ₃ O ₄ @SiO ₂ | TEA, DMF, 80 °C, 0.3 mol% Pd | 4 | 96 | 320 | 40 |

^aIsolated yield, ^bTON = mol product/mol cat, ^cTOF = TON/reaction time (h⁻¹).

Table 6

Comparison of the present methodology with other reported catalysts for the Matsuda-Heck Coupling.

| Entry [Ref.] | Catalyst | Reaction conditions | Time (h) | Yield (%) ^a | TON ^b | $TOF^{c}(h^{-1})$ |
|---------------|---|---|----------|------------------------|------------------|-------------------|
| 1 [27] | Agarose supported nano Pd | H ₂ O, 40 °C, 0.26 mol % Pd | 3 | 83 | 319 | 106 |
| 2 [28] | PS-NHC-Pd(II) | EtOH, r.t., 0.9 mol% Pd | 1 | 94 | 104 | 104 |
| 3 [29] | $Pd(OAc)_2$ | BMIMBF ₆ , 70–80 °C, 10–20 mol % Pd(OAc) ₂ | 6.5 | 88 | - | - |
| 4 [30] | Pd/Al ₂ O ₃ | MeOH, 25 °C, 1 mol% Pd | 0.5 | 95 | - | - |
| 5 [16] | $Pd(OAc)_2$ | H ₂ O, Triton x-100, r.t. 2 mol % Pd(OAc) ₂ | 1 | 100 | - | - |
| 6 [This work] | Pd-AcAc-Am-Fe ₃ O ₄ @SiO ₂ | H ₂ O, Triton X-100, r.t., 0.2 mol% Pd | 0.5 | 98 | 490 | 980 |

^a Isolated yield.

^b TON = mol product/mol cat.

^c TOF = TON/reaction time (h^{-1}) .

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.151801.

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