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N,*N*-Dimethylformamide-stabilized palladium nanoclusters as catalyst for Migita–Kosugi–Stille cross-coupling reactions



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1. Introduction

Transition-metal-catalyzed cross-coupling reactions are a significant area in the study of organic transformations for carboncarbon bond formation. In particular, Pd-catalyzed Migita-Kosugi-Stille cross-coupling reactions of organostannanes with organohalides are one of the most important cross-coupling reactions [1]. These reactions have been used in the syntheses of various biaryl compounds, which are important intermediates in the syntheses of natural products, functionalized polymers, and pharmaceuticals [2]. Pd-complex catalysts are generally used in cross-coupling reactions, not only in Migita-Kosugi-Stille crosscoupling reactions, but also in other relevant cross-couplings such as Suzuki-Miyaura [3], Mizoroki-Heck [4], and Hiyama-Hatanaka [5] cross-coupling reactions. However, these reactions are generally achieved using Pd complexes bearing phosphine ligands and using 1-5 mol% of catalyst. In addition, it is reported that the addition of a metal salt such as CuI and CsF to the Pd complex increased substantial catalytic activity in the Migita-Kosugi–Stille cross-coupling reaction [1,6]. Reducing the amount of rare metal resources used as catalysts in bulk-scale catalytic processes is desirable in terms of conserving resources. Furthermore, if the development of catalytic cross-coupling reactions that do not need the use of an inert gas (such as Ar) could be achieved

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ABSTRACT

N,*N*-Dimethylformamide-stabilized palladium nanoclusters showed high catalytic activity for Migita –Kosugi–Stille cross-coupling reactions. The present cross-coupling reaction proceeded efficiently using very small Pd catalyst loadings under ligandless, and even an open air, conditions. The reactions proceeded smoothly in good yields and with high turnover numbers of up to 3.5×10^4 .

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(*i.e.*, if the deactivation of metal catalysts under O_2 could be overcome), and if the reaction could be carried out *in an open air*, the well-established Migita–Kosugi–Stille cross-coupling would become a much more practical synthetic methodology for the construction of C–C bond formation biaryl moieties [7].

On the other hand, transition-metal nanoparticles (NPs) and nanoclusters (NCs) have also received a great deal of attention because of their inherently large surface areas, which are different from those of the bulk metals [8]. Generally, the more the particle size decreases, the more the surface area increases. NPs therefore have significantly different characteristics from those of complexes and bulk metals. To date, various methods for the synthesis of colloidal metal NPs have been developed, and these NPs have been used as catalysts in chemical transformations [9b,c]. In general, metal NPs are prone to losing reactivity in their bulk metal form, so various stabilizers such as dendrimers [9], functionalized polymers [10], inorganic solids [11], ligands [12], and ionic surfactants [7a,13] are prerequisites to prevent aggregation of the metal NPs in these preparations. However, NCs, which minimize aggregation by covering and protecting the surface area, are highly active catalysts for cross-coupling reactions.

In an investigation of highly active metal NC catalysts with minimal deactivation of the catalyst surface area as a result of use of a surfactant, we and other groups recently reported the solution synthesis of transition-metal NCs using an *N*,*N*-dimethylformamide (DMF) reduction method [14].

We previously reported that DMF-stabilized Pd NCs (diameter 1–1.5 nm) prepared using the DMF reduction method exhibited

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excellent catalytic activity in Mizoroki–Heck and Suzuki–Miyaura cross-couplings, and catalyst recycling and a high turnover number (TON), up to 6.0×10^8 , were achieved [14d]. Furthermore, similarly prepared DMF-stabilized Cu NPs (size: about 2 nm) showed high catalytic activity (with a TON of up to 2.2×10^4) in Ullmann-type cross-couplings of aryl halides with phenols under ligand-free conditions [14h].

2. Results and discussion

Inspired by these studies, we investigated further application to the DMF-stabilized Pd NCs for practical and highly active catalyst in cross-coupling reactions. In this study, we found that the catalyst system serves as an efficient and highly active catalyst in Migita–Kosugi–Stille cross-coupling reactions, which produce biaryls and vinylarenes by the reaction of aryl halides and aryl-/vinyl-stannanes; high TONs of up to 3.5×10^4 were attained (Table 1). The present cross-coupling reaction therefore proceeded efficiently using very small Pd catalyst loadings under ligandless, and even open-air, conditions.

The results for the Migita–Kosugi–Stille cross-coupling reactions are shown in Table 1. The reaction of 4-iodoanisole (**1a**: 0.5 mmol) with tributylphenylstannane (**2a**: 0.5 mmol) in the presence of Pd NCs (0.1 mol% based on **1a** used; prepared by the method previously reported by our group) in *N*-methylpyrrolidone (NMP)/DMF (3:1, 2 mL) at 120 °C for 15 h *in an open air*, was used as the model reaction, giving 4-methoxybiphenyl (**3a**) as crosscoupling product along with formation of a small amount of biphenyl (**4a**). We did not detect the formation of 4,4'-dimethoxybiphenyl (**5a**) under these conditions. The product **3a** was isolated by K₂CO₃-containing silica gel column chromatography [15].

When the reaction was carried out in DMF as the sole solvent, the products were obtained in moderate yields (entry 1, Table 1). On the other hand, when the reaction was performed using a mixed NMP/DMF (3:1) solvent, the best yields of the products were achieved (entry 2, Table 1).

It has been reported that the Pd-complex-catalyzed Migita-Kosugi-Stille cross-coupling reaction is promoted by the addition of CuI as an additive [6]. The addition of CuI (0.05 mmol, 2.5 mol%) was found to be effective in the present Pd–NCs-catalvzed reaction system, and the product was obtained in excellent yield (entry 3. Table 1). With respect to the solvent used, an NMP/DMF (3:1) mixed-solvent system gave the best results. Although the NMP solvent gave high reactivity (entry 3), other solvents such as acetonitrile and toluene were not good solvents and gave 3a in 10% and 16% yields, respectively (entries 5 and 6, Table 1). The reaction in H₂O was also tolerated and gave the cross-coupling products in moderate yields (entry 7, Table 1). Needless to say, the reaction did not proceed in the absence of Pd NCs (entry 9, Table 1). The optimized reaction temperature was 120 °C and the reaction at lower temperature (100 °C) gave the desired product 3a in 34% yield (entry 10, Table 1). The Pd NCs showed very high catalytic activity, and the highest TON (3.5×10^4) was achieved using a Pd NCs catalyst loading of 10^{-4} mol% (entry 13, Table 1). This reaction can proceed in an open air as well as under an inert gas (Ar). Pd NCs are stable in air, and high catalytic activity was retained even when the reaction was carried out under an O₂ (1 atm) atmosphere (entries 14 and 15, Table 1). Although a detailed reason and experimental evidence of this outcome is not confirmed, the existence of O₂ might promote the catalytic activity of Pd NCs, based on the experimental results (entries 2, 14 and 15).

We found that the use of chlorobenzene is totally inactive for the coupling under these reaction conditions.

To expand the scope of the reaction substrate, various organohalides and different organostannanes were used in the Migita– Kosugi–Stille cross-coupling reaction, under the same conditions as for entry 2 in Table 1; the results are shown in Table 2. The reactions of organohalides bearing both electron-donating and electron-withdrawing substituents, such as -OMe, $-CF_3$, and 1-

Table 1

Pd-NCs-catalyzed Migita-Kosugi-Stille cross-coupling reactions of 4-iodoanisole (1a) with tributylphenylstannane (2a).^a



Entry	Catalyst (mol%)	Solvent	Temp. (°C)	Total yield (%) ^b	Selectivity (3a:4a:5a)	TON ^c
1	10 ⁻¹	DMF	120	40	86:14:0	3.5×10^{2}
2	10^{-1}	NMP/DMF (3:1)	120	80 (68)	88:12:0	$7.0 imes 10^2$
3 ^d	10^{-1}	NMP/DMF (3:1)	120	>99 (89)	93:5:2	_
4	10^{-1}	NMP	120	68	76:24:0	5.2×10^2
5	10^{-1}	MeCN	120	10	100:0:0	1.0×10^2
6	10^{-1}	Toluene	120	16	81:19:0	$1.3 imes 10^2$
7	10^{-1}	H ₂ O	120	44	77:21:2	$3.4 imes 10^2$
8	10^{-1}	DMF/EG (1:1)	120	53	89:11:0	4.7×10^2
9	_	NMP/DMF (3:1)	120	0	_	_
10	10^{-1}	NMP/DMF (3:1)	100	34	90:10:0	3.1×10^2
11	10^{-1}	NMP/DMF (3:1)	140	88	77:19:4	$6.5 imes 10^2$
12	10 ⁻²	NMP/DMF (3:1)	120	60	88:12:0	5.3×10^3
13	10^{-4}	NMP/DMF (3:1)	120	48	72:28:0	$3.5 imes 10^4$
14 ^e	10^{-1}	NMP/DMF (3:1)	120	46	93:7:0	$4.1 imes 10^2$
15 ^f	10^{-1}	NMP/DMF (3:1)	120	88	76:24:0	$\textbf{6.4}\times 10^2$

^a Conditions: **1a** (0.5 mmol), **2b** (0.5 mmol), Pd NCs (10^{-1} mol%), solvent (2 mL), 120 °C, 15 h, open air.

^b GC yields based on limiting reagent used. The number in parentheses shows the isolated yield.

^c Turnover number (TON) = **3a** (mol)/Pd NCs (mol).

^d CuI (0.05 mmol) used.

^e Under Ar.

f Under O_{2.}

Table 2

Pd-catalyzed Migita-Kosugi-Stille cross-coupling reactions of various compounds.

R ¹ -SnBu₂	+	Ar-X	Pd NCs (10 ⁻ ' mol %)	R ¹ -Ar
1		2	NMP/DMF(1.5/0.5 mL)	3
0.5 mmol	0.75 mmol			
			open an	

Entry	R ¹	Ar-X	R ¹ –Ar	Yield (%) ^a
1	Ph	2b	3b	72 [86]
2	Ph	F ₃ C 2c	3c	53 [77]
3	Ph	2d	3d	66 [90]
4	Ph	MeO Br	3a	71 ^b
5	Ph	Br 2f	3b	83 ^b
6	Ph	F ₃ C Br	3c	28 ^b
7	Vinyl	2b	3e	65
8	Vinyl	Meo 2a	3f	44
9	Vinyl	F ₃ C Ja	3g	nd ^c

^a Isolated yields. The numbers in square brackets show the yields using CuI (0.05 mmol) as co-catalyst.

^b GC yields. Bromide (7.5 mmol) was used.

^c Not detected by GC.

iodonaphthalene, with tributylphenylstannane and tributylvinylstannane gave the corresponding products **3b–3f** in good yields (Table 2). On the basis of these experiments, substantial electronic effect on aryl ring was not observed for the coupling reaction.

The reusability of the Pd NCs catalyst was examined under the conditions given in entry 8, Table 1. The reuse experiment was performed as follows (see Supplementary data Fig. S1 for details). A mixture containing **1a**, **2a**, Pd NCs, DMF, and ethylene glycol (EG) was allowed to react Migita–Kosugi–Stille cross-coupling (first time) (Step A). After the reaction, *n*-hexane (8 mL) was added to the mixture (upper: hexane layer containing **1a**, **2a** and **3a**: polar (DMF/EG) layer containing Pd NCs) (Step B). Then the reaction mixture was extracted with hexane (8 mL) twice. After removal of the hexane layer (Step C), resulting polar (DMF/EG) layer containing Pd NCs can be used for the next catalytic sequence (Step D). The first

sequence gave 53% as the total yield of **3a** and **4a** (a small amount), as shown in entry 8, Table 1; 47% in the second cycle, and 30% in the third cycle. The ratio of the formation of **4a** to that of the main cross-coupling product **3** remained low (<10%) during the reuse sequences. These results indicate that the Pd NCs could be still reused as an active catalyst for the cross-coupling reaction at least three times. It is difficult to clearly elucidate the catalyst deactivation pathway, however, substantial amount of solid (such as salt) formation was observed during the reuse sequence (as shown in Fig. S1 in Supplementary data), which would be plausible factor to drop the catalytic activity for the coupling.

3. Conclusions

In conclusion, we found that DMF-stabilized Pd NCs show high activity for the Migita–Kosugi–Stille cross-coupling reaction. The DMF-stabilized metal NCs are very stable in air, so the reactions can be performed in the open air; excellent TONs of up to 3.5×10^4 was achieved.

4. Experimental section

4.1. General

GLC analysis was performed with a flame ionization detector using a 0.22 \times 25 m capillary column (BP-5). ¹H and ¹³C NMR were measured at 400 and 100 MHz, respectively, in CDCl₃ with Me₄Si as the internal standard. The products were characterized by ¹H NMR, ¹³C NMR. All reagents were commercially available and used without further purification.

Compound **3a** [6d], **3b** [6d], **3c** [16], **3d** [16], **3e** [17], **3f** [17] is known compound and reported previously.

4.2. Preparation of modified DMF-protected Pd NCs catalysts [14d]

4.2.1. Preparation of 0.1 M PdCl₂ solution in HCl aq. (A)

A mixture of $PdCl_2$ (purity 99.9%, 0.0177 g), concentrated hydrochloric acid (12 N, 300 μ L) and distilled water (700 μ L) was added to a vial bottle (3 mL), and allow the bottle to stand at room temperature overnight.

4.2.2. Preparation of 1 mM Pd NCs solution in DMF (B)

DMF (50 mL) was added to a 300 mL three-necked round bottom flask, and the solution was preheated to 140 °C (± 2 °C) and stirred 1300–1500 rpm for 5 min. Then the 0.1 M PdCl₂ solution (**A**) was added to the hot DMF solution, which allowed to react for 8– 10 h on stirring (1300–1500 rpm) at 140 °C (± 2 °C). The resulting clear red solution was used as 1 mM Pd NCs solution in DMF (**B**) for further reaction. After vacuum evaporation of the solvent, the residue was redissolved in selected solvents such as methanol, acetonitrile, toluene, H₂O, and *N*-methylpyrrolidone (NMP) for the dynamic light scattering (DLS) and the cross-coupling reactions.

4.3. A typical reaction procedure for Pd NCs catalyzed Migita– Kosugi–Stille cross coupling reaction of **1a** with **2a** (Table 1, entry 2)

A mixture of 4-iodoanisole **1a** (117 mg, 0.5 mmol), tributylphenyltin **2a** (183 mg, 0.5 mmol), and 1 mM Pd NCs in *N*,*N*methylformamide (DMF) (0.5 mL) as a catalyst in *N*-methyl-2pyrrolidone (1.5 mL) was stirred at 120 °C for 15 h under an open air. The conversions and yields of products were estimated from peak areas based on an internal standard (*n*-tridecane) using GC and the product **3a**, **4a** and **5a** was obtained in 80% total yield. The reaction mixture was extracted with concentrated aqueous solution of potassium iodide and *n*-hexane to separate products from the Pd NCs and copper iodide. The product **3a** was isolated by column chromatography (silica gel (230–400 mesh): $K_2CO_3 = 9:1$, *n*-hexane as eluent) [15] in 64% yield (59.0 mg).

4.4. A typical procedure for the catalyst-reusing sequence in the Pd NCs-catalyzed Migita–Kosugi–Stille cross-coupling reaction (Fig. S1)

After the reaction was performed under the conditions of Table 1, entry 8 (Step A), hexane (8 mL), internal standard (*n*-tridecane, 0.27 mmol) were added to the mixture (Step B). Then, the reaction mixture was extracted with hexane (8 mL) twice, and 4iodoanisole (1a), tributylphenyltin (2a), 4-methoxybiphenyl (3a), biphenyl (4a), 4,4'-dimethoxybiphenyl (5a), iodotributyltin and internal standard (*n*-tridecane) were extracted in hexane layer (Step C). Resulting polar layer containing Pd NCs dispersed in DMF/ ethylene glycol (EG), was used for next catalytic sequence under the same reaction conditions as entry 8, Table 1, by adding 4iodoanisole 1a (117 mg, 0.5 mmol) and tributylphenyltin 2a (183 mg, 0.5 mmol) or 4-iodotluene 1b (109 mg, 0.5 mmol) and tributylphenyltin 2a (183 mg, 0.5 mmol) (Step D).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jorganchem.2013.08.004.

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