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DOI: 10.1002/adsc.201500529

# Integrated Process of Aerobic Oxidation–Olefination–Asymmetric C–C Bond Formation Catalyzed by Robust Heterogeneous Gold/Palladium and Chirally Modified Rhodium Nanoparticles

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Received: June 1, 2015; Revised: July 6, 2015; Published online: September 25, 2015

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/adsc.201500529.

Abstract: We have achieved an integrated process of oxidation-olefination-asymmetric 1,4-addition reactions under aerobic and aqueous conditions using metal nanoparticles as robust heterogeneous catalysts. A tandem process of aerobic oxidation and Horner-Wadsworth-Emmons olefination was successfully catalyzed by gold/palladium bimetallic nanoparticles. A wide range of substrates gave the desired products in high yields and excellent selectivities. This tandem process was further connected with an asymmetric 1,4-addition reaction catalyzed by chiral rhodium nanoparticles to achieve a challenging three-step, one-pot sequential reaction. Various types of esters with a chiral center at the  $\beta$ -position were obtained from readily available and stable alcohols and arylboronic acids in high yields with high enantioselectivities in a one-pot sequential reaction integrating three transformations.

**Keywords:** aerobic oxidation; heterogeneous asymmetric catalysis; metal nanoparticles; olefination; one-pot process

The olefination reaction is one of the most important transformations in organic synthesis.<sup>[1]</sup> In particular,  $\alpha,\beta$ -unsaturated esters are useful intermediates for various asymmetric 1,4-addition reactions, such as addition of arylboronic acids,<sup>[2]</sup> to provide key precursors of pharmaceuticals and bioactive compounds.<sup>[2e,3]</sup> In general, those compounds can be synthesized from carbonyl compounds using olefination reagents.<sup>[4]</sup> Direct olefination of alcohols with such reagents is more attractive because readily available and more stable alcohols can be used as starting materials.<sup>[5]</sup> Although most  $\alpha,\beta$ -unsaturated ester formations *via* direct olefination of alcohols required stoichiometric amounts of oxidants, several aerobic oxidative pro-

cesses were successfully achieved using homogeneous catalysts with Wittig reagents or *in situ* generated diazocarbonyl compounds using  $PPh_3$ .<sup>[5d, f-i]</sup>

Integration of reactions such as tandem or one-pot reactions is not only a powerful but also a green sustainable method, because readily available and stable substrates can be used directly as starting materials without preactivation, and because the decreased number of tedious purification procedures of intermediates results in efficiency and elimination of wastes.<sup>[5c,6]</sup> In this context, we envisioned a challenging three-step integrated process of asymmetric 1,4-addition of anylboronic acids to  $\alpha,\beta$ -unsaturated esters (3), which were provided by direct olefination of alcohols (1) under aerobic oxidative conditions in a one-pot sequential manner, affording chiral esters (4) as useful compounds for synthesis of various pharmaceuticals and bioactive compounds (Scheme 1). For this purpose, olefinations using Wittig reagents and in situ generated diazocarbonyl compounds are inappropriate because phosphine-based coproducts would interfere with the catalysts for asymmetric 1,4-addition reactions to diminish selectivity and reactivity.<sup>[5c-h]</sup> We considered that Horner-Wadsworth-Emmons (HWE) olefination under a biphasic system of aqueous and organic layers might be compatible because an aqueous layer could easily remove cogenerated phosphites (5) in an olefination step. However, very robust catalysts that are compatible with both aerobic and aqueous conditions are needed for this purpose. In addition, the use of heterogeneous catalysts is more desirable from the viewpoint of environmentally benign organic synthesis and the chemical industry.<sup>[7]</sup>

Herein, we present an integrated process of oxidation-olefination under aerobic and aqueous conditions using metal nanoparticles as robust heterogeneous catalysts. This is the first direct olefination of alcohols through aerobic oxidation and HWE reaction. This tandem process can be further connected to a highly enantioselective C–C bond formation reac-



Scheme 1. Integrated three continuous transformations.

tion catalyzed by another heterogeneous catalyst to achieve a three-step asymmetric tandem oxidation process in a one-pot sequential manner that has never been achieved, using distinct heterogeneous metal nanoparticle catalysts.

We have developed various metal nanoparticle catalysts, including bimetallic nanoparticles, that were immobilized in polystyrene-derived polymer with cross-linking moieties, as reusable heterogeneous catalysts, namely polymer incarcerated metal nanoparticles (PI-Ms).<sup>[8]</sup> Catalysts using a nanocomposite of polymer and carbon black with high specific surface area (PI/CB-M) increase the total surface area of catalysts to enhance catalytic activity.<sup>[9]</sup> Among them, gold-based nanoparticles catalysts showed excellent reactivity for aerobic oxidation of alcohols and tandem oxidation processes.<sup>[10]</sup>

The investigation of tandem aerobic oxidation-HWE reactions was initiated with 4-methylbenzyl alcohol (1a) and ethyl diethylphosphonoacetate (2a) in the presence of potassium carbonate and PI/CB-Au in water inspired by previous reports about the HWE reaction in basic water (Table 1).<sup>[11]</sup> The aerobic oxidation proceeded even in the presence of 2a and the desired (E)-isomer of  $\alpha,\beta$ -unsaturated ester (3aa) was successfully obtained with excellent selectivity; however, the yield was moderate because of the low activity of the catalyst under the reaction conditions (entry 1). The yield was improved with PI/CB-Au/Pd (0.262 mmol/g Au, 0.251 mmol/g Pd), indicating higher oxidation ability and the more robust nature of PI/CB-Au/Pd than PI/CB-Au (entry 2). However, overoxidation of a generated aldehyde to a carboxylic acid proceeded because of the slower HWE reaction than the aerobic oxidation. To suppress this undesired reaction and to realize the desired green process, the reaction was examined with a smaller amount of PI/ CB-Au/Pd. Consequently, the yield was improved even with only 0.5 mol% of the catalyst in the presence of 3 equiv. of base (entry 3). Base screening was further conducted. In the cases of carbonates containing alkali metals, Cs<sub>2</sub>CO<sub>3</sub> afforded a comparable or

Table 1. Investigations of the amounts and kinds of bases.



<sup>[a]</sup> Determined by GC analysis.

<sup>[b]</sup> Determined by <sup>1</sup>H NMR analysis.

<sup>[c]</sup> PI/CB-Au(/Pd) (1 mol%) was used.

<sup>[d]</sup> Toluene/H<sub>2</sub>O (1/2) was used as a solvent.

slightly lower yield than that obtained with  $K_2CO_3$ , while Li<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub> showed poor yields (entries 4–6). In the case of potassium phosphates, no improvement of the yield was observed, probably because deprotonation from phosphate might not proceed smoothly because of the too weak a basicity and an *in situ* generated aldehyde might be converted to the carboxylic acid before the HWE reaction proceeded (entry 7). Finally, a biphasic system of water and toluene was found to be the best for this reaction system using  $K_2CO_3$  as a base (entry 8).

Recovery and reuse of PI/CB-Au/Pd were investigated. The used catalyst was washed with a basic solution, heated at 170 °C and used in the next run. Con-



Scheme 2. Recovery and reuse of PI/CB-Au/Pd.

sequently, the catalyst was successfully reused, providing more than 80% yield (Scheme 2).

With the optimized reaction conditions in hand, substrate scope for the direct olefination of alcohols was investigated. Initially, various primary alcohols were reacted with triethyl phosphonoacetate (2a). Benzyl alcohol (1b), benzyl alcohol substituted with an electron-donating or an electron-withdrawing group (1c or 1d, respectively), gave the desired products in high yields with excellent selectivities (Table 2, entries 2-4). Heteroaryl-substituted alcohols were applicable, also. Slightly lower yield was obtained in the case of furfuryl alcohol (1e) (entry 5); however, 2-thiophenemethanol (1f) and 2-pyridinemethanol (1g) gave satisfactory yields with excellent selectivities (entries 6 and 7). In addition, an aliphatic alcohol (1h), which is a more difficult substrate, gave the desired product in good yield (entry 8). Furthermore, a tertbutyl unsaturated ester (3i) could be obtained from *tert*-butyl diethylphosphonoacetate (**2b**).

Table 2. Substrate scope for tandem aerobic oxidation/HWE reactions.

R<sup>1</sup> OH

<b>1a–h</b> 0.4 mm	iol PI/C	PI/CB-Au/Pd (0.5 mol% Au) $K_2CO_3$ (3.0 equiv.)			∧ CO-R <sup>2</sup>				
+ toluene/H <sub>2</sub> O (1/2), 60 °C, EtO $\stackrel{\text{H}}{\xrightarrow{\text{H}}}$ CO <sub>2</sub> R <sup>2</sup> O <sub>2</sub> , 16–24 h EtO $2\mathbf{a},\mathbf{b}$ 1.5 equiv.									
Entry	$R^1$	R <sup>2</sup>	Product	( <i>E</i> ) Yield [%] <sup>[a]</sup>	$E/Z^{[b]}$				
1	4-MeC <sub>6</sub> H <sub>4</sub>	Et	3a	83	98/2				
2	Ph	Et	<b>3</b> b	76	98/2				
3	$4-MeOC_6H_4$	Et	3c	78	99/1				
4	$4-ClC_6H_4$	Et	3d	77	97/3				
5 <sup>[c]</sup>	2-furyl	Et	3e	72	98/2				
6	2-thienyl	Et	3f	82	98/2				
7	2-pyridyl	Et	3g	63	> 99/ < 1				
8 <sup>[d]</sup>	$Ph(CH_2)_2$	Et	3h	58	95/5				
9	$4-MeC_6H_4$	t-Bu	3i	82	97/3				

[a] Isolated yield.

<sup>[b]</sup> Determined by <sup>1</sup>H NMR analysis.

[c] Toluene/ $H_2O(2/1)$  was used as a solvent.

<sup>[d]</sup> PI/CB-Au/Pd (1 mol%) was used.

Adv. Synth. Catal. 2015, 357, 3815-3819

We next attempted a more difficult sequential onepot process integrating three continuous transformations including asymmetric C-C bond formation catatwo different metal nanoparticles lvzed bv (Scheme 1). The developed tandem aerobic oxidation-HWE reaction was further connected with an asymmetric 1,4-addition reaction catalyzed by chiral Rh nanoparticles.<sup>[12]</sup> To avoid an increased volume of solid catalysts that caused lower stirring efficiency and to prevent partial decomposition of the arylboronic acid in the presence of Au/Pd nanoparticles in the asymmetric 1,4-addition step, 0.25 mol% of PI/ CB-Au/Pd was employed in the tandem aerobic oxidation-HWE reaction. After completion of the twostep tandem reaction of 1a and 2a, PI/CB-Rh, a chiral diene ligand and 3-methoxyphenylboronic acid were added. Pleasingly, the desired chiral ester 4a was obtained in good yield with excellent enantioselectivity via a three-step, one-pot reaction (Table 3, entry 1). The substrate scope of the three-step reaction was investigated. Both electron-rich and electron-poor arylboronic acids gave the products in moderate to good yields with outstanding enantioselectivities (Table 3, entries 1–5). Benzyl alcohol (1b) and 4-chlorobenzyl alcohol (1d) gave the desired products in high yields with excellent enantioselectivities (entries 6 and 7); however, only a moderate yield was obtained in the case of 4-methoxybenzyl alcohol (1c) (entry 8). Heteroaryl-substituted alcohols such as furfuryl alcohol (1e) or thienylmethanol (1f) were employed in the reaction, and both substrates gave outstanding enantioselectivities (entries 9 and 10). Furthermore, an aliphatic alcohol, 3-phenyl-1-propanol (1h), could also be employed and the desired product also was obtained with excellent enantioselectivity (entry 11). This wide scope of the challenging three-step, one-pot reaction catalyzed by two heterogeneous catalysts showed the power of our metal nanoparticles catalysts. Valuable optically active chiral esters were easily prepared from primary alcohols, HWE reagents, and arylboronic acids using molecular oxygen as an oxidant.

In summary, we have achieved the first direct olefination of alcohols through oxidation and HWE reaction using heterogeneous bimetallic nanoparticles catalysts under aqueous and aerobic conditions. A wide range of substrates, including benzyl alcohols substituted bv electron-withdrawing/electron-donating groups, heteroaryl-substituted alcohols, and aliphatic Table 3. Substrate scope for the sequential one-pot process integrating three continuous transformations.

	$R \longrightarrow OH + EtO = CO_2Et$ $\frac{1}{1} \qquad 2a$ 0.4 mmol 1.5 equiv.	$\begin{array}{c} PI/CB-Au/Pd (0.25 \text{ mol}\% \text{ Au}) \\ K_2CO_3 (3.0 \text{ equiv.}) \\ \hline \\ toluene/H_2O (1/2) \\ 60 \ ^\circ\text{C}, \ O_2, \ 24 \ h \\ \hline \\ \hline \\ PI/CB-Rh (1.5 \text{ mol}\% \text{ Rh}), \text{ chiral diene } (0.1 \text{ mol}\%) \\ \hline \\ \hline \\ toluene/H_2O (1/2), \ 100 \ ^\circ\text{C}, \ \text{Ar}, \ 16 \ h \\ \hline \\ \hline \\ \end{array}$				
Entry	R	Ar	Chiral diene Product	( <i>E</i> ) Yield [%] <sup>[a]</sup>	ee [%] <sup>[b]</sup>	
1	$4-MeC_6H_4$	3-MeOC <sub>6</sub> H <sub>4</sub>	<b>4</b> a	81	98	
2	$4-\text{MeC}_6\text{H}_4$	$4-FC_6H_4$	<b>4b</b>	70	99	
3 <sup>[c]</sup>	$4-MeC_6H_4$	$4-MeOC_6H_4$	<b>4</b> c	84	98	
4 <sup>[d,e]</sup>	$4-MeC_6H_4$	$2-MeOC_6H_4$	<b>4d</b>	36	>99	
5	$4-MeC_6H_4$	Ph	<b>4e</b>	79	99	
6	Ph	$3-MeOC_6H_4$	<b>4f</b>	81	99	
7 <sup>[1]</sup>	$4-\text{ClC}_6\text{H}_4$	$3-MeOC_6H_4$	<b>4</b> g	72	99	
8 <sup>[c]</sup>	$4-MeOC_6H_4$	$3-MeOC_6H_4$	4h	50	>99	
9	2-furyl	$3-MeOC_6H_4$	<b>4i</b>	69	>99	
10 <sup>[c,d,g]</sup>	2-thienyl	$3-\text{MeOC}_6\text{H}_4$	4j 41-	34	93	
11. ,	$\operatorname{PII}(\operatorname{CH}_2)_2$	$5$ -MeOC <sub>6</sub> $\Pi_4$	4K	30	94	

[a] Isolated yield.

[b] Enantiomeric excess (ee) was determined by chiral HPLC analysis.

[c] Reverse enantiomer of the ligand was used.

[d] 0.2 mol% of the ligand was used.

[e]  $K_2CO_3$  (1.0 equiv.) was added in the third step.

<sup>[f]</sup> PI/CB-Au/Pd (0.5 mol% Au) was used.

<sup>[g]</sup> Toluene/H<sub>2</sub>O (2/1) was used as a solvent. PI/CB-Rh/Ag was used instead of PI/CB-Rh.

<sup>[h]</sup> PI/CB-Au/Pd (1 mol% Au) was used.

alcohols gave the desired products in 58-83% yields with excellent selectivities. The catalyst was successfully reused with retention of its activity. In addition, this tandem process was further connected with an asymmetric 1,4-addition reaction catalyzed by chiral Rh nanoparticles to achieve a challenging three-step, one-pot sequential reaction including highly selective asymmetric catalysis (93-99% ee) that is also catalyzed by metal nanoparticles. These results also verify the robustness of our heterogeneous metal nanoparticles catalysts. To the best of our knowledge, this is also the first example of integration of oxidation of alcohols, C-C unsaturated bond formation, and asymmetric 1,4-addition reaction in a one-pot tandem manner. Various types of esters with a chiral center at the  $\beta$ -position were obtained from readily available and stable alcohols and arylboronic acids. Further development of useful tandem reactions using multiple heterogeneous catalysts is under investigation.

### **Experimental Section**

#### Procedure for One-Pot Aerobic Oxidation-HWE-Asymmetric 1,4-Addition Reactions (Table 3, entry 1)

p-Tolylmethanol 1a (48.9 mg, 0.4 mmol), ethyl 2-(diethoxyphosphoryl)acetate 2a (134.5 mg, 0.6 mmol), PI/CB-Au/Pd (Au: 0.25 mol%), potassium carbonate (165.8 mg, 1.2 mmol), toluene (0.1 mL) and water (0.2 mL) were combined in a Carousel<sup>TM</sup> tube. After the mixture had been stirred for 10 h under a molecular oxygen atmosphere at 60°C, phenylboronic acid (the mixture of boroxine and boronic acid, 86.5 mg, 0.8 mmol as B), PI/CB-Rh (1.5 mol% as Rh), chiral diene (0.0267 mL of 3.9 mg/mL solution in toluene), toluene (0.375 mL) and water (0.8 mL) were added in this reaction mixture. The mixture was stirred for 16 h under an argon atmosphere at 100 °C. Diethyl ether was added to the mixture and the solid catalyst was removed by filtration. The mixture was transferred to separatory funnel and the aqueous layer was extracted with diethyl ether. The collected organic layers were washed with brine and dried with sodium sulfate. The sodium sulfate was filtered off and the solvent was removed under vacuum, then the residue was

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<sup>3818</sup> 

purified by preparative TLC to afford (S)-ethyl 3-phenyl-3-(p-tolyl)propanoate **4a**; yield: 97.1 mg (81%). The *ee* value of the product was determined by chiral HPLC analysis (98% *ee*).

## Acknowledgements

This work was partially supported by a Grant-in-Aid for Science Research from the Japan Society for the Promotion of Science (JSPS), Global COE Program, The University of Tokyo, MEXT, Japan, the Japan Science and Technology Agency (JST), and NEDO. We also thank Mr. Noriaki Kuramitsu (The University of Tokyo) for STEM analysis.

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