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COMMUNICATION

Regio- and Stereoselective Syntheses of Chiral α-Quaternary (*Z*)-Trisubstituted Allylic Amino Acids *via* Synergistic Pd/Cu Catalysis

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A synergistic palladium/copper catalysis for asymmetric allylic alkylation of vinylethylene carbonates with aldimine esters has been developed for synthesis of α-quaternary (*Z*)-trisubstituted allylic amino acids under mild conditions. This methodology features broad substrate compatibilities in yields of up to 87% and up to 94% *ee*. A facile scale-up and straightforward conversion to 1,2,3,5-tetrasubstituted pyrrole and 1,2,5,6-tetrahydropyridine bearing chiral quaternary carbon centers verify the synthetic utility of this method.

Optical α, α-disubstituted amino acids are a class of important organic molecules that exist in many natural products, medicinal chemistry, and synthetic intermediates (Fig. 1),[1] such as a highly potent inhibitor of the chymotrypsin-like proteolytic activity of the 20S proteasome (**Marizomib**),[2] a selective Ah receptor modulator (**Carbidopa**),[3] a building block of the peptidoglycan (**DAP**),[4] a new orally active iron chelator (**Deferitazole**),[5] a class of hepatitis C virus NS3 protease inhibitors (**BILN 2061**).[6] Consequently, the development of direct and efficient methods for the enantioselective syntheses of allylic amino acid derivatives remains highly desired and sought after.

Despite the known biological potency of these skeletons, rarely asymmetric methodologies were developed to construct related multisubstituted allylic amino acids bearing a quaternary carbon center.[7] Various methods to access disubstituted allylic amino acid derivatives including organocatalysis and metal catalysis have been well established and applied to approach these scaffolds.[7a-c, 8] However, poor enantioselectivities and low reactivity were obtained in the presence of the single catalysis style. Notably, synergistic catalysis has become a hot research topic owing to directly decreasing the active energy and forming chiral species by interacting with feedstocks in recent years, which would resolve this challenge.[9] Zhang's and Wang's group independently reported regioselective and enantioselective construction of linear α, α-disubstituted amino acids via asymmetric allylic alkylation of the amino aicds derived aldimine or ketimine esters by Pd/Cu synergistic catalysis under base conditions. [10] Despite these great improvements, their reported methods are limited to preparing chiral disubstituted allylic amino acid scaffolds with *E*-configuration. Therefore, a general asymmetric catalytic methodology for the regio- and stereoselective construction of highly functionalized multisubstituted (*Z*)-configurational allylic amino acids from simple and variable substrates remains an enormous challenge and will be particularly appealing.

Inspired by the fact that vinylethylene carbonates were not only applied in cycloaddition but also employed in nucleophilic substitution.[11, 12, 13, 13c, 14, 15, 16] To the best of our knowledge, the method by attacking the terminal position of intermediate **C** for the construction of chiral functionalized products with the (*Z*)-configuration is still scarce and yet underdeveloped. [17] A single catalytic style mode was difficult to control the chiral stereo center owning to the long distance of prochiral center with π-allylpalladium intermediate. Another point probably worthy of note is that this substrate-dependent selectivity has hampered the development of the regioselectivity of allylic reaction by the single-catalyst system. Recently, our group reported an allylic alkylation of ketimines with vinylethylene carbonates in the presence of palladium and copper synergistic catalysis,[18a] in

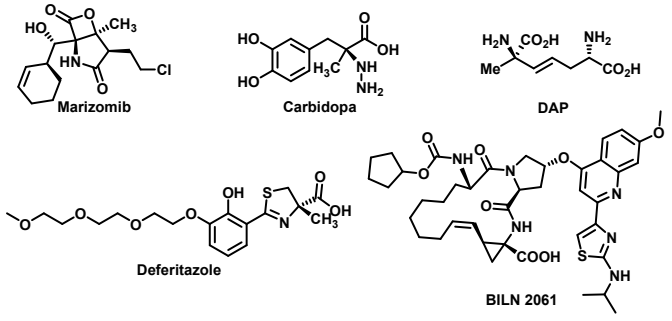

Figure 1. Significant functionalized allylic amino acid scaffolds.

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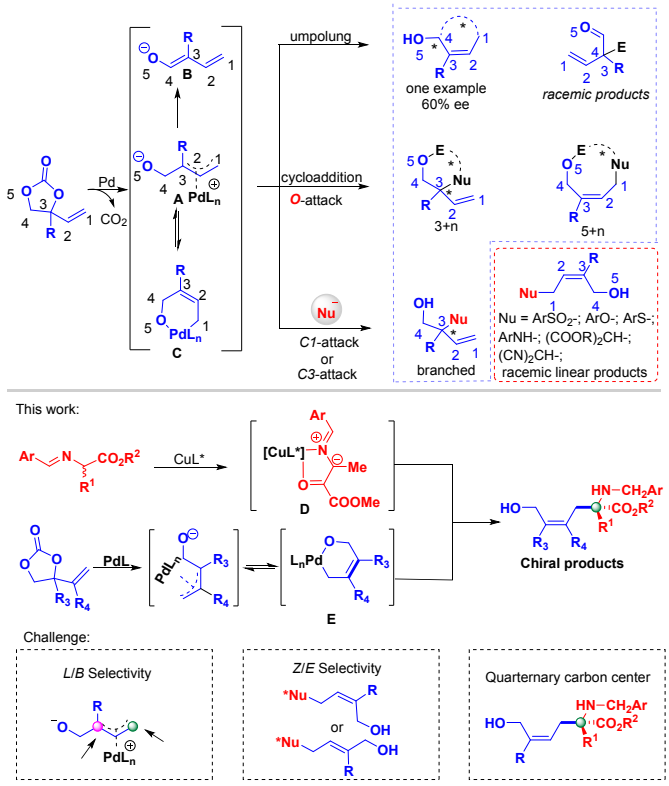


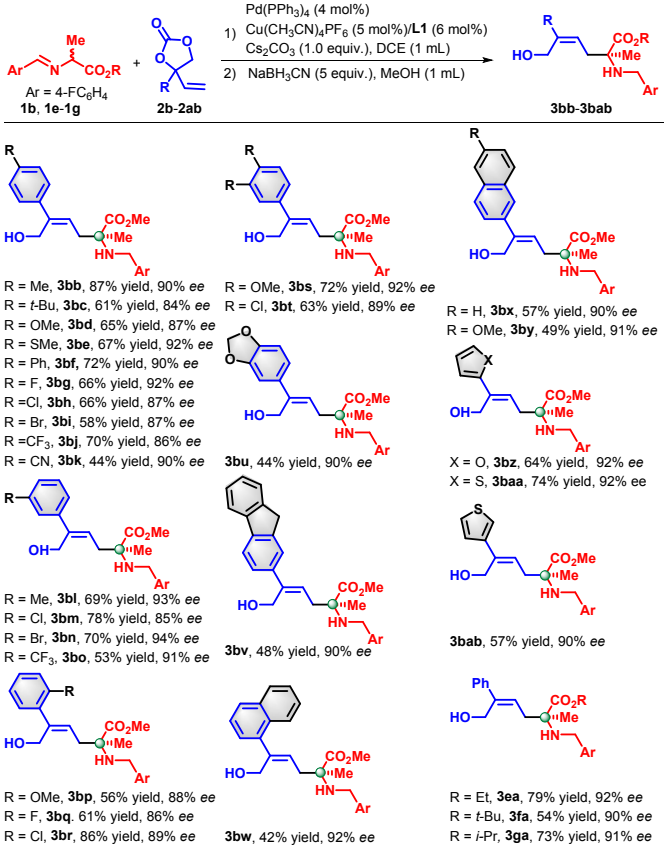
Scheme 1. Construction of chiral trisubstituted allylic amino acid derivatives

which various chiral trisubstituted allylic amino acids were achieved in good yield with excellent enantioselectivities and stereoselectivities. However, when the alkyl groups were introduced at the α-position, chiral trisubstituted amino acid derivatives bearing a quaternary carbon center failed to be obtained in current reaction conditions. As a part of our ongoing interest in the chemistry of vinylethylene carbonates (VCEs), we envisioned that the chiral α-substituted metallized azomethine ylide **D** *in-situ*-formed from aldimine esters could attack the reactive achiral cyclopalladated complex **E** to form linear α-quaternary trisubstituted allylic amino acids.

To probe our hypothesis, treatment of aldimine ester **1a** with 1.2 equivalent of phenyl vinylethylene carbonate **2a** and 1.5 equivalent of Cs_2CO_3 in the presence of 5 mol% of $Pd(PPh_3)_4$, 10 mol% of $Cu(CH_3CN)_4BF_4$ and 12 mol% of (*S*, *S*)-Ph-Phosferrox (**L1**) in 1,2-dichloroethane at room temperature for 8 hours gave (*Z*)-α-quaternary trisubstituted allylic acid **3aa** in 66% yield with 92% *ee* and good *Z*/*E* ratio (Table S1, entry 1, see supporting information). The reactivity and enantioselectivity dramatically hinged on the nature of the catalyst employed. Various copper salts including $Cu(CH_3CN)_4PF_6$, CuI, and CuCl were screened (Table S1, entries 2-4), and $Cu(CH_3CN)_4PF_6$ was the best choice for the asymmetric allylic alkylation to provide **3aa** in 94% *ee*.[19] It was found that the substituents on the oxazolinyl ring had much effect on the enantioselectivity. The results suggested that a bulkier group was good for *ee* value (Table S1 entries 7 and 8). Furthermore, the different bases were surveyed, while the enantioselectivities and yields were not improved (Table S1, entries 10 and 11). Afterward, various solvents (toluene, DCM, THF, DCE) were also investigated (entries 12-14), but the yields dramatically decreased. It was found that $NaBH_4$ could treat with compound **3aa** to form a complex, which was confirmed by HRMS (see SI, Table S1). Therefore, we screened different reductants including $NaBH(OAc)_3$ and $LiAlH_4$, and the decreased yields were obtained (entries 15 and 16). The use of $NaBH_3CN$ as a reductant could achieve the best yield and enantioselectivity (Table S1, entry 17). The temperature for the reaction was also very of importance. The best yield and enantioselectivity were given at 40 ℃ (Table S1, entry 18). The electronic nature of aldimine esters were also crucial for the reaction (Table S1, entries 19-21). To our delight, chiral trisubstituted allylic amino acid **3ba** was furnished in 86% yield with 92% *ee* when aldimine ester bearing electron-withdrawing group was employed. The results showed that the loading of the precatalyst and ligand were the key factors for this reaction (see SI, Table S1). Finally, The optimized reaction conditions were achieved in presence of 4 mol% of $Pd(PPh_3)_4$, 5 mol% of $Cu(CH_3CN)_4PF_6$ and 6 mol% of **L1** (Table S1, entry 22).

With a set of conditions of asymmetric allylic alkylation of vinylethylene carbonates with aldimine **1b** established, we then explored the substrate scope concerning allylic reagents **2**. The results were summarized in Scheme 2. An array of vinylethylene carbonates could be well-tolerated in the reaction with aldimine **1b**, and desired products **3bb**-**3bab** were generally obtained in

Scheme 2. Scope of aldimine esters and γ-vinylethylene carbonates [a]



[a] Reaction conditions: **1b**, and **1e-1g** (0.1 mmol), **2b-2ab** (0.13 mmol), $Pd(PPh_3)_4$ (4 mol%), $Cu(CH_3CN)_4PF_6$ (5 mol%), **L1** (6

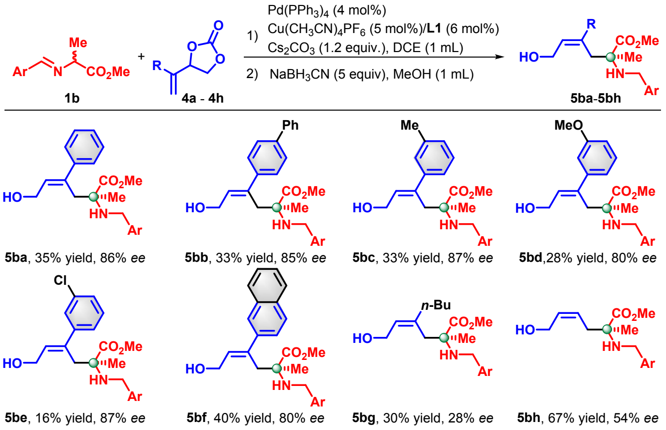
mol%), Cs_2CO_3 (1 equiv), DCE (1 mL), $NaBH_3CN$ (5 equiv.), MeOH (1.0 mL), N_2, 6 h, 40 °C.

moderate to good yields with good enantioselectivities. All the tested *para*-substituted aryl-substituted *γ*-monosubstituted vinylethylene carbonates possessing electron-donating group (methyl, *t*-butyl, methoxyl, methylthioyl, and phenyl) and electron-withdrawing group (-F, -Cl, - Br, $-CF_3$, -CN)) exhibited similar reactivities leading to access products **3bb**-**3bk** in 44-87% yields with 84-92% *ee*. The *meta*-substituted vinylethylene carbonates bearing different substituents were also well tolerated to furnish **3bl**-**3bo** in 53-78% yields with 85-94% *ee*. Notably, the more hindered *ortho*-substituted VECs were tested. To our delight, the corresponding trisubstituted allylic amino acid derivatives **3bp**-**3br** could be obtained in 56-86% yields with 86-89% *ee*. Multisubstituted VECs were well-compatible and provided the corresponding products **3bs**-**3bu** in 44-72% yields with good enantioselectivities. When the phenyl ring was replaced with a fused aromatic ring system (carbazolyl-, *α*- or *β*-naphthyl, 2- or 3-thienyl, or 2-furanyl group), the reaction also proceeded smoothly and afforded products **3bv**-**3bab** in good yields with good enantioselectivities, albeit with slightly low yield for **3bv**, **3bw**, and **3by**. In addition, other aldimine esterswere also suitable for this transformation as well, and the corresponding products **3ea**-**3ga** were prepared in 54-79% yields with 90-92% *ee*.

Next, reactions of various *β*-monosubstituted vinylethylene carbonates with **1b** were examined to further explore the generality of this allylation. Compared to *α*-monosubstituted vinylethylene carbonates, *β*-monosubstituted vinylethylene carbonates gave a lower reactivity. All results were shown in Scheme 3. *β*-monosubstituted vinylethylene carbonates bearing electron-donating (Me, Ph, OMe) and withdrawing group could be tolerated, resulting in the generation of the corresponding products **5ba**-**5be** in 16-35% yield with 80-87% *ee*. 2-Naph-thyl substituted *β*- monosubstituted vinylethylene carbonate could transform to the target product **5bf** in 40% yield with 80% *ee*. When the R group was *n*-Butyl or H, the corresponding products **5bg** and **5bh** were obtained in 30% and67% yield, respectively.

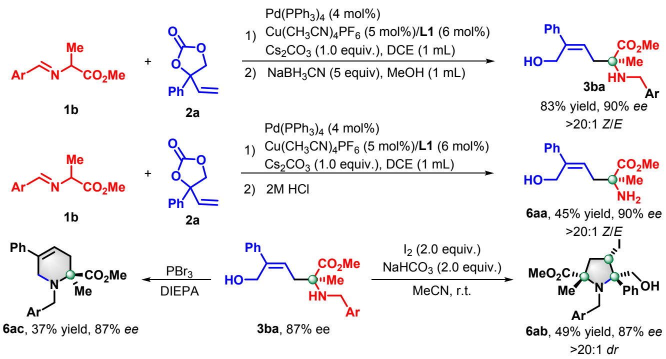
To insight into the reaction practicability and application, a gram-scale reaction was accomplished to furnish trisubstituted

Scheme 3. Scope of *β*-vinylethylene carbonates



[a] Reaction conditions: **1b** (0.1mmol), **4a-4h** (0.12 mmol), $Pd(PPh_3)_4$ (4 mol%), $Cu(CH_3CN)_4PF_6$ (5 mol%), **L1** (10 mol%), Cs_2CO_3 (1.2 equiv.), DCE (1 mL), $NaBH_3CN$ (5 equiv.), N_2, 7 h, 40 °C.

Scheme 4. Gram-scale reaction and derivatization of compound **3aa**



allylic amino acid **3ba** in 83% yield with 90% *ee* under standard conditions. Further transformations of allylic amino acid **3ba** were subsequently performed. Acidic hydrolysis of compound **3ba** with 2 mol/L aqueous HCl, the unprotected allylic amino acid **6aa** was obtained in 45% yield with 87% *ee*. The 1,2,3,5-trisubstituted pyrrole **6ab** with two quaternary carbon centers was obtained in 49% yield with 87 % *ee* and >20:1 *dr via* the I_2-mediated intramolecular cyclization. The hydroxyl substituted compound **3ba** could successfully transform to tetrahydropyridine **6ac** through a bromination reaction and S_N2 substitution cascade.

We next carried out several stereocontrol experiments to further explore the reaction (seeing SI, Scheme S1). The product (*S*)-**3ga** was furnished in 73% yield with 91% *ee* under standard conditions. And the product (*R*)-**3ga** was obtained in 52% yield with 93% *ee* in the presence of (*R*)-**L1** ligand. The optical (*S*) and (*R*)-**1g** were used for this transformation. Relatively lower enantioselectivity (46% *ee*) was obtained using (*S*)-**1g** (100% *ee*) as the reaction partner, and the rest of (*S*)-**1g** was racemized *in situ.* Excellent enantioselectivity (95% *ee*) and good yield (87%) were performed using (*R*)-**1g** as the substrate. All these experiment results suggested that this transformation might proceed through dynamic kinetic resolution (DKR).

Based on previous reports and control experiments,[10, 18] a plausible mechanism was proposed in Scheme S2 (seeing SI). The decarboxylation of vinylethylene carbonate **2a** under Pd(0) catalysis would generate *π*-allylpalladium complex **F**, which isomerize to six-membered palladacycle intermediate **G**,[17a, 17b] which was able to react with complexed azomethine ylide chiral species **D** to form linear *α*-quaternary trisubstituted allylic amino acids **3ba**, PdL(0) complex, and CuL*.

Conclusions

In conclusion, we report an asymmetric allylic alkylation of vinylethylene carbonates with aldimine esters through Pd/Cu synergistic catalysis, providing a series of chiral trisubstituted allylic amino acids in good yields with good enantioselectivities and exclusive regioselectivity under mild conditions. The gram-scale reaction was well-performed to achieve the target

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product in good yield with excellent enantioselectivity. Furthermore, the chiral 1,2,3,5-tetrasubstituted pyrrole bearing two quaternary carbon centers were synthesized *via* I_2-mediated intramolecular cyclization. The Chiral 1,2,5,6-tetrahydropyridine was also prepared through bromination and S_N2 substitution cascade.

Author Contributions

M. Ke, X. Xiao and F. Chen designed the project. M. Ke carried out the experiments. Y. Yu, L. Sun, X. Li, and Q. Cao contributed to part experiments. M. Ke, X. Xiao and F. Chen co- wrote the paper. All authors have approved the final version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

Acknowledgment

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