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Electrocatalytic linear coupling of alkenes *via* radical anion under mild conditions†

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The reductive coupling of alkenes is an efficient strategy for directly constructing C–C bonds from readily available bulk chemical feedstocks. Herein, a one-step electrocatalytic protocol is proposed for the direct linear coupling of alkenes. Under electrochemical conditions, the key intermediate, a highly unstable radical anion, is generated without the need for stoichiometric amounts of dangerous organolithium reagents. The radical anion undergoes a subsequent radical addition reaction, and the target compound is formed through proton capture and/or hydrogen atom transfer processes. This strategy supports both homo-coupling and cross-coupling reactions of alkenes and has been successfully applied to the synthesis of bioactive molecules. Electroredox catalysis provides a straightforward and efficient method for generating radical anions from alkenes, paving the way for the wide application of these highly reactive intermediates in chemical synthesis under mild conditions in the absence of metal and oxidant/reductant.

Green foundation

(1) This work utilizes an electrochemical reduction strategy to generate olefin radical anion intermediates under mild conditions at room temperature in air, offering a greener alternative to conventional methods that typically rely on highly reactive alkali metals or their organometallic salts for inducing such species.
(2) The reaction exhibits high atom economy due to its simple system, eliminating the need for additional oxidants or reductants.
(3) Furthermore, the transition-metal-free nature of this reaction provides a favorable pathway for its future application in synthesizing pharmaceuticals and other bioactive molecules, effectively avoiding concerns over transition metal residues.

Introduction

Alkenes are among the most readily available and abundant feedstocks in the chemical industry. They have been widely utilized as substrates in numerous powerful synthetic strategies in organic chemistry.[1,2] The coupling reaction between alkenes is one of the most important strategies to construct C–C bonds.[3–6] For instance, the remarkable olefin metathesis reaction enables the construction of chain olefins or cycloolefins by removing ethylene;[7–11] hydroalkenylation reactions between alkenes produce internal alkenes;[12–17] oxidative coupling of alkenes generates 1,3-diene moieties with excellent *E*/*Z* selectivity;[18–24] and in reductive coupling reactions, unsaturated olefin sites in substrates are linked together to form fully saturated carbon skeletons (Fig. 1A).

Catalytic strategies are conventionally employed in reductive coupling reactions of alkenes (Fig. 1B). For example, Markovnikov products can be obtained from electron-deficient olefin substrates *via* metal-hydride-catalyzed hydrogen atom transfer processes.[25–29] In contrast, strategies for constructing linear C–C bonds through reductive coupling of alkenes are still limited. Jiang and coworkers developed a photocatalytic coupling reaction between vinyl ketones and vinyl azaarenes to achieve chiral linear products.[30] In this work, dicyanopyrazine (DPZ) served as the photocatalyst, and stoichiometric Hantzsch ester was used as the reductant. Recently, Lu's group reported a photocatalytic coupling of electron-deficient alkenes using oxalic acid as a traceless linchpin.[31] In this reaction, DPAIPN catalyzed the hydrocarboxylation process, while Ir served as the catalyst for the subsequent decarboxylative cross-coupling procedure. Gong's team developed a method for dimerization of unactivated terminal olefins using a nickel–copper synergistic catalytic reduction system.[32] Here, $(MeO)_3SiH$ was used as the reductant, and stoichiometric

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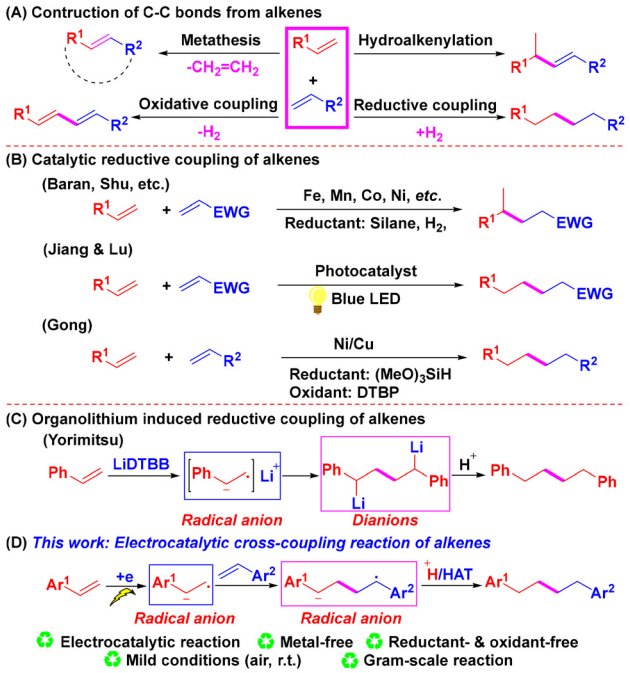


Fig. 1 C–C bond formation *via* alkene coupling: reaction design and overview.

DTBP acted as the oxidant. Yorimitsu reported an interesting homo-coupling reaction of alkenes induced by organolithium reagents in flow microreactors (Fig. 1C).[33] In this strategy, the dangerously high-reactivity lithium 4,4′-di-*tert*-butylbiphenylide (LiDTBB) was consumed stoichiometrically to generate highly unstable radical anions, which subsequently dimerized to form key dianion intermediates. Inspired by these elegant studies and the electrochemical catalysis of olefin functionalization,[34–37] we aimed to explore whether highly unstable radical anions could be generated *via* the electrochemical reduction of alkenes, thus avoiding the use of dangerous and expensive organolithium reagents (Fig. 1D). In addition, electrochemical reactions offer several advantages, including being metal-free, reductant- and oxidant-free, and operating under mild reaction conditions, making them an attractive alternative.

1,4-Diphenylbutane derivatives, synthesized through the reductive coupling of simple alkenes, are ubiquitous in lignin extracted from plants. They are also essential structural moieties found in various bioactive molecules, including pesticides, lipoxygenase inhibitors, anti-tumor agents, antibacterial compounds, and anti-inflammatory agents (Fig. 2).[38–42] Therefore, the synthesis of 1,4-diphenylbutane derivatives remains an attractive and significant research focus in pharmaceutical and synthetic chemistry.

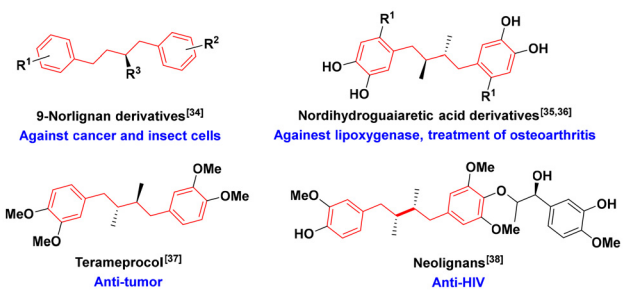
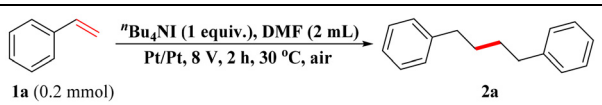


Fig. 2 Bioactive molecules containing 1,4-diphenylbutane moieties.

Results and discussion

To begin the investigation, the reductive homo-coupling of styrene (**1a**) was chosen as a template reaction to optimize the conditions (Table 1). At room temperature, the reaction proceeded smoothly in an undivided electrolytic cell containing tetrabutylammonium iodide as the electrolyte in DMF. Under constant-voltage electrolysis (8 V) using platinum electrodes as both the anode and cathode, 1,4-diphenylbutane (**2a**) was obtained in 68% yield within 2 h (entry 1). In contrast, no product was detected in the absence of an electrical current (entry 2). Adjusting the reaction voltage to 7 V reduced the yield to 42% (entry 3), while increasing the voltage to 9 V did not improve the yield (entry 4). Changing from constant-voltage mode to constant-current mode (I = 15 mA) decreased the yield to 49% (entry 5). Replacing tetrabutylammonium iodide with tetramethylammonium iodide or tetrabutylammonium bromide significantly reduced the yield to 41% and 18%, respectively (entries 6 and 7). Using DMSO instead of DMF as the solvent resulted in a lower yield of 29% (entry 8). Changing the platinum electrodes to other combinations, such as Pt(+)/Fe(−), Pt(+)/C(−), Pt(+)/Ag(−), Mg(+)/Pt(−),

Table 1 Control experiments[a]



Entry	Variation from standard conditions	Yield[b] (%)
1	—	68
2	No electricity	N.D.
3	7 V	42
4	9 V	68
5	15 mA	49
6	Me_4NI instead of $^nBu_4NI$	41
7	$^nBu_4NBr$ instead of $^nBu_4NI$	18
8	DMSO instead of DMF	29
9	Pt(+)/Fe(−) instead of Pt(+)/Pt(−)	17
10	Pt(+)/C(−) instead of Pt(+)/Pt(−)	13
11	Pt(+)/Ag(−) instead of Pt(+)/Pt(−)	20
12	Mg(+)/Pt(−) instead of Pt(+)/Pt(−)	30
13	Al(+)/Pt(−) instead of Pt(+)/Pt(−)	23
14	Zn(+)/Pt(−) instead of Pt(+)/Pt(−)	24
15	Ni(+)/Pt(−) instead of Pt(+)/Pt(−)	11

[a] Standard conditions: platinum anode (10 mm × 10 mm × 0.1 mm), platinum cathode (10 mm × 10 mm × 0.1 mm), **1a** (0.2 mmol), $^nBu_4NI$ (0.2 mmol), DMF (2 mL), 10 mL reaction tube, undivided cell, constant voltage = 8 V, 30 °C, 2 h. [b] NMR yield (nitromethane as an internal standard).

Al(+)/Pt(−), Zn(+)/Pt(−), and Ni(+)/Pt(−), led to various degrees of yield reduction (entries 9–15).

With the optimized reaction conditions in hand, the scope of substrates for the reductive homo-coupling reaction of styrene and its derivatives was investigated (Table 2A). The reaction of styrene and alkyl-substituted styrenes proceeded smoothly, affording 1,4-diphenylbutane derivatives in good yields (**2a–e**). Notably, when halogenated styrenes were used as substrates, dehalogenative homo-coupling products were obtained in moderate yields (**2a**). This procedure might be the elimination of halogen anion after the SET reaction of halogenated styrene at the cathode.[43,44] The reductive homo-coupling of phenylacetylene also produced 1,4-diphenylbutane (**2a**). When 4-phenyl-substituted styrene was used as a substrate, the designed product was obtained with an excellent yield of 97% (**2f**). However, when the benzene ring of styrene was substituted with groups exhibiting strong electronic effects, whether electron-donating or electron-withdrawing, the target products were obtained in near-moderate yields (**2g**, **2h**). Importantly, borate-substituted styrene was smoothly converted into the electroreductive homo-coupling product with a satisfactory yield of 55% (**2i**). This product can be readily transformed into more complex compounds *via* Suzuki coupling reactions.[45–48] Sterically hindered 1,1-stilbene also underwent reductive homo-coupling, providing the designed product in a good yield of 66% (**2j**). Additionally, internal olefins were successfully converted into coupling products with satisfactory yields (**2k**, **2l**). Both α-naphthalene and β-naphthalene derivatives delivered the target products with high yields of 74% (**2m**) and 98% (**2n**), respectively. The reductive coupling of pyridine-substituted ethylenes also proceeded smoothly (**2o**, **2p**).

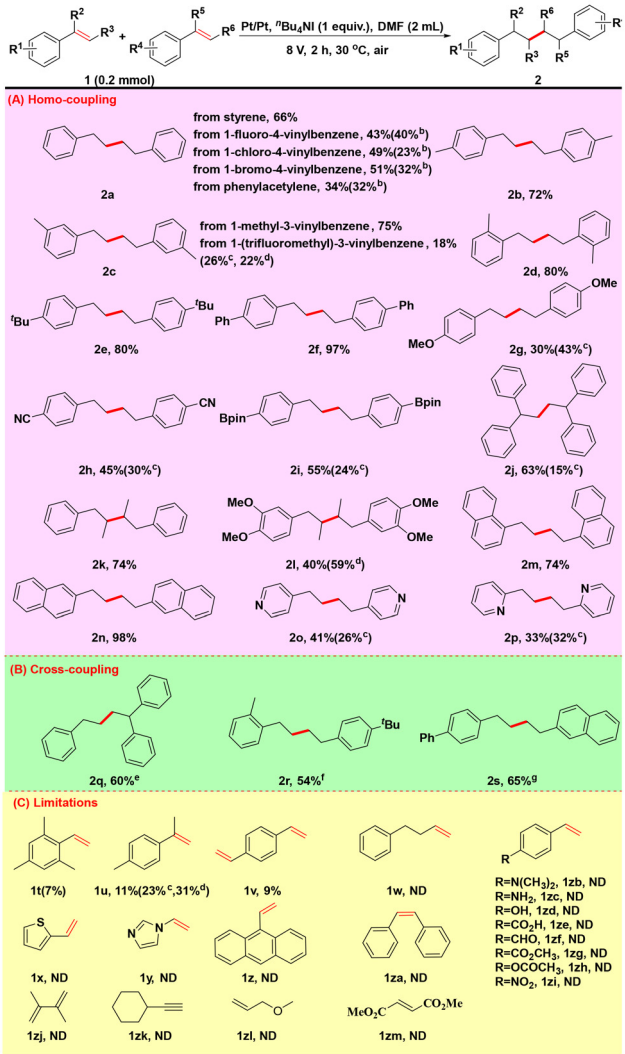
Subsequently, the cross-coupling reaction was explored to further broaden the applicability of this method. Good yields of cross-coupling products were obtained even when two substrates were used in equimolar amounts (Table 2B, **2q–2s**).

However, poor yields of the designed products were observed when o-dimethyl-substituted styrene, α-methyl-substituted styrene, or 1,4-divinylbenzene were used as substrates (Table 2C, **1t–1v**). In addition, the reaction proved incompatible with non-conjugated olefins, substrates containing active hydrogens, or those bearing groups intolerant to reducing conditions (**1w–1zi**).

The 1,4-diphenylbutane structural motif is frequently found in bioactive molecules. As proof-of-concept, the synthesized compounds **2g** and **2l** exhibit anti-inflammatory and anti-HIV activities, respectively (Fig. 3A).[41] In addition, a valuable bioactive molecule **3** with anti-cancer activity was successfully synthesized in one step using **2a** as the starting material (Fig. 3B).[49] To demonstrate the scalability of this method, a scaled-up homo-coupling reaction of substrate **1n** was performed, affording the designed product **2n** in a good yield of 78% (Fig. 3C).

To explore the reaction mechanism, a series of control experiments were performed (Fig. 4). First, radical-blocking

Table 2 Substrate scope[a]



[a] Isolated yield. [b] Recovery rate of styrene. [c] Recovery rate of starting material. [d] Reduction yield of olefin to alkane. [e] **1a** (0.2 mmol), **1j** (0.2 mmol). [f] **1d** (0.4 mmol), **1e** (0.2 mmol). [g] **1g** (0.2 mmol), **1n** (0.2 mmol).

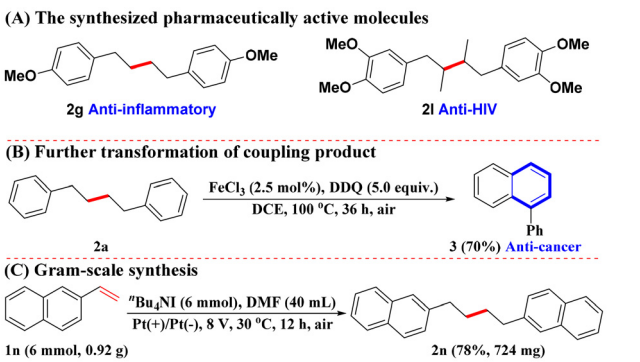


Fig. 3 Practical verification experiments.

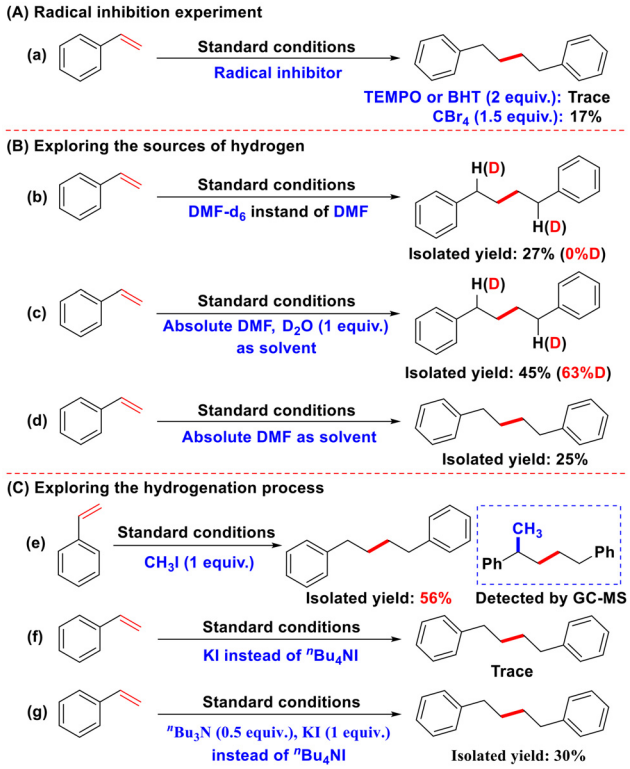


Fig. 4 Control experiments.

experiments were carried out, and the reaction was found to be almost completely terminated in the presence of TEMPO or BHT, indicating that a radical process was inevitable (Fig. 4A). CBr_4 could also block the experiment well, which further supports the radical process. Next, the source of hydrogen in the product was examined (Fig. 4B). When DMF-d6 was used as the solvent instead of DMF, almost no deuterium-containing product was detected, indicating that the hydrogen atoms did not originate from DMF (**Eq. b**). However, when absolute DMF with 1 equivalent of D_2O was used as the solvent, the deuteration rate of the benzyl hydrogen reached 63%, indicating that these hydrogen atoms were partially derived from water in the reaction system (eqn (c)). When the reaction was carried out in absolute anhydrous DMF, the yield sharply decreased to 25%, which indicated that the reaction was inhibited in the absence of water as a proton source (eqn (d)). This result also implied that some protons were derived from sources other than water and DMF, namely tetrabutylammonium iodide (^{n}Bu$_4$NI). The hydrogenation process was further explored (Fig. 4C). In the presence of 1 equivalent of CH_3I, a benzyl monomethylated product was detected, indicating the formation of a monoanionic intermediate (eqn (e)). Replacement of ^{n}Bu$_4$NI with KI resulted in negligible product formation, demonstrating that the iodide anion (I^-) alone is insufficient to initiate the reaction (eqn (f)). Notably, adding 0.5 equiv. of ^{n}Bu$_3$N to the aforementioned reaction afforded the reductive coupling products in 30% yield, highlighting the significant role of ^{n}Bu$_3$N in facilitating this reaction (eqn (g)). However, the yield obtained in the presence of both KI and ^{n}Bu$_3$N remained significantly lower than that with ^{n}Bu$_4$NI, suggesting that ^{n}Bu$_4$NI or its equilibrium products may play a more active role in promoting this reaction.

To further investigate the mechanism, cyclic voltammetry (CV) testing was carried out (Fig. 5). The CV curve of styrene (blue) showed a slight increase in reduction peak current compared to the blank group (black), suggesting that the reduction reaction of styrene might occur directly on the electrode. The reduction potential of ^{n}Bu$_4$NI (red) was observed at −0.804 V, indicating that ^{n}Bu$_4$NI could be reduced on the electrode. When ^{n}Bu$_4$NI and styrene coexisted (green), the reduction peak appeared at −0.776 V, with its peak position largely consistent with that of styrene alone (blue), but the current intensity significantly increased, indicating that the presence of ^{n}Bu$_4$NI promoted the reaction. Since the reduction peak position remained unchanged, it was more likely that styrene was directly reduced at the cathode.

Based on the control experiments and CV test, a possible reaction mechanism was proposed (Fig. 6). Initially, ^{n}Bu$_4$NI decomposed to ^{n}BuI and Bu_3N. Meanwhile, styrene could be directly reduced at the cathode under an applied potential of 8 V, forming the radical anion **B**. An alternative coexisting pathway that cannot be ruled out is the cathodic reduction of nBuI to form radical anion **A**, followed by a single-electron transfer (SET) process between styrene and intermediate **A**, generating the unstable radical anion **B**. Subsequently, radical anion **B** underwent a radical addition reaction with another styrene molecule, forming coupled radical anion **C**. Radical anion **C** then captured a proton from water to generate radical **D**. Radical **D** was further reduced at the cathode to form anion **E**, which captured another proton from water to yield the target molecule (**T.M.**). An alternative pathway involved a hydrogen atom transfer (HAT) process between radical anion **C** and an amine radical cation, producing anion **E**, which subsequently captured a proton from water to form the **T.M.**

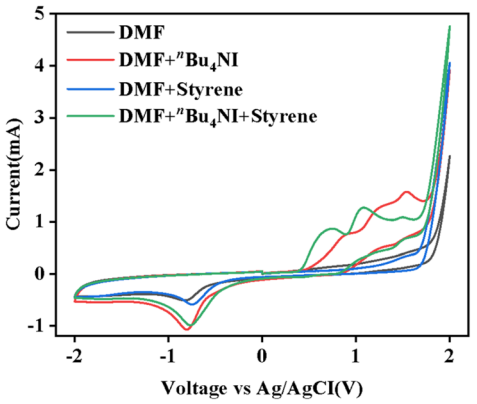


Fig. 5 Cyclic voltammetry experiments. The related compounds were dissolved in 0.1 M $LiClO_4$/DMF using Pt disk as working electrode, and Ag/AgCl (in saturated KCl solution) as counter and reference electrodes at 0.1 V s^{-1} scan rate.

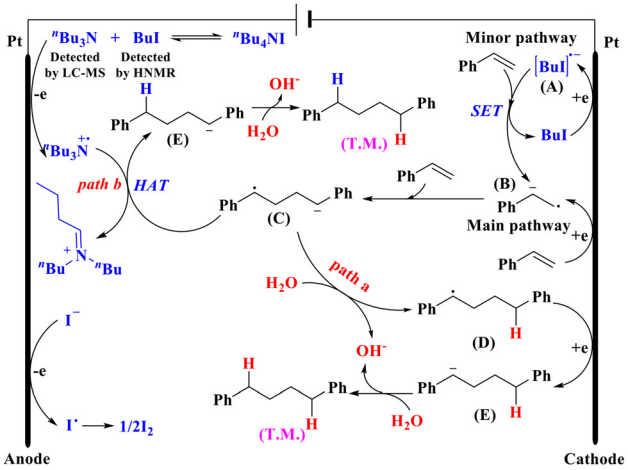


Fig. 6 Proposed mechanism.

Conclusions

In conclusion, an efficient electrocatalytic protocol has been developed for the reductive coupling of simple alkene components in a head-to-head fashion, utilizing a radical anion as the key intermediate. This method enables the one-step construction of 1,4-diphenylbutane moieties, which are biologically active, demonstrating the practical applicability of this method. Given the ubiquity of alkenes in organic molecules, this general electrocatalytic coupling strategy offers a powerful tool for organic chemists. The facile and mild generation of radical anions from alkenes *via* electroredox catalysis, without the need for metals, oxidants, or reductants, paves the way for the broad application of these highly reactive intermediates in the synthesis of various chemicals.

Author contributions

J. X. and F. L. carried out the synthesis and characterization. S. Y., H. L. and C. C. analyzed the results. H. G. conceived and planned the study. All authors contributed to the preparation of manuscript.

Data availability

The data supporting this article have been included as part of the ESI.†

Conflicts of interest

There are no conflicts to declare.

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