

## RESEARCH ARTICLE

View Article Online  
View Journal | View IssueCite this: *Org. Chem. Front.*, 2025,  
12, 5737

## Electrochemical water activation for the oxidative cleavage of alkenes

Ning Zhang,<sup>†</sup> Hui Zhou,<sup>†</sup> Guojuan Liang, Gaochen Liu, Xing Liu, Pengfei Zhou,<sup>†</sup> Dong Zhang<sup>†</sup> and Jing Zhou<sup>†</sup>\*

Oxidative cleavage of alkenes allows rapid access to valuable products, starting from simple petrochemicals. Water is an ideal carrier of oxygen. Here, we report a novel electrochemical water activation for the oxidative cleavage of alkenes. Water was used as an abundant and economical oxygen source, with no additional oxidants or complex workups. A wide range of alkenes such as activated alkenes and unactivated alkenes were tolerated. Additionally, we observed oxidative cleavage of the allylic C–C(vinyl)  $\sigma$ -bond and a unique linear paired electrolysis mode, which are not observed in existing methods for the oxidative cleavage of olefins.

Received 26th June 2025,  
Accepted 4th September 2025

DOI: 10.1039/d5qo00944h

rsc.li/frontiers-organic

## Introduction

Alkenes<sup>1–4</sup> are feedstock materials that are obtained on the ton scale from petroleum and vegetable biomass and are exploited by the bulk chemical industry to access oxygen-enriched synthetic intermediates. Oxidative cleavage of olefinic double bonds to aldehydes or ketones is one of the important reactions, as ketones and aldehydes are one of the few most often used functionalities in organic synthesis. One of the most popular methods for this transformation is ozonolysis,<sup>5</sup> which requires an ozone generator and displays serious safety issues owing to the toxicity and explosiveness of high concentrations of ozone and ozonides (Fig. 1a). Alternative metal or nonmetal oxidants, such as  $\text{KMnO}_4$ ,  $\text{RuO}_4$ ,  $\text{OsO}_4$ ,  $\text{H}_2\text{O}_2$  and  $\text{NaIO}_4$ , are either toxic or strongly oxidizing, and also generate waste<sup>6</sup> (Fig. 1b). Oxidative cleavages using  $\text{O}_2$  and a suitable photocatalyst have also been developed, but they are limited to activated alkenes<sup>7,8</sup> (Fig. 1c). Recently, a purple light-induced oxidative cleavage of alkenes using nitroarenes has been reported, which has greatly expanded the range of olefins. However, this method requires low temperature and complex workups.<sup>9,10</sup> Electrochemical methods for promoting the oxidative cleavage of olefins have also been reported;<sup>11,12</sup> however, their substrate scope is limited to styrene derivatives and requires  $\text{O}_2$  or other specialized redox catalysts<sup>12</sup> (Fig. 1d).

Electrolysis is a powerful and sustainable approach for redox transformations, allows straightforward and mild chemical conversions that are metal- and oxidant-free.<sup>13–16</sup> The

chemical activation of water would allow this earth-abundant resource to be transferred into value-added compounds, and is a topic of keen interest in energy research.<sup>17–19</sup> Water is an ideal carrier of oxygen.<sup>20–22</sup> Developing renewable energy-powered electrochemical technologies to drive water splitting under ambient conditions for organic oxidation reactions is highly desired.<sup>23,24</sup> Here, we demonstrate electrochemical water activation for the oxidative cleavage of all types of alkenes, including both activated and unactivated alkenes, under mild conditions. By avoiding transition metals and chemical oxidizers, this protocol offers an environmentally sustainable method for the oxidative cleavage of alkenes, leading to a valuable contribution to the sustainable conversion of petrochemical feedstocks into synthetically usable fine chemicals and commodities (Fig. 1, bottom).

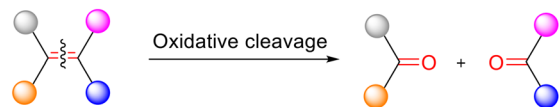
## Results and discussion

Because unactivated aliphatic alkenes have higher oxidation potentials ( $>2.0$  V vs. SCE)<sup>25</sup> and are more difficult to oxidize than activated alkenes, we chose the unactivated alkene 1-allylnaphthalene **1a** as a model substrate for our electrochemical oxidation studies (Table 1). The electrolysis was conducted with an undivided cell (a schlenk tube) with a simple two-electrode configuration. The electrode, serving as a support for a heterogeneous catalyst, is an essential component of an electrochemical reactor and plays a decisive role in the outcome of the transformation.<sup>26</sup> Consequently, we first performed a screening of electrode materials (entries 1–5). Using  $\text{PbO}_2/\text{Ti}$  electrode as the anode, the lead dioxide lattice is partially hydrated, forming gel zones. These zones slow the diffusion of anodically generated molecular oxygen and

Chongqing Research Center for Pharmaceutical Engineering, College of Pharmacy,  
Chongqing Medical University, Chongqing 400016, China.  
E-mail: 102792@cqmu.edu.cn

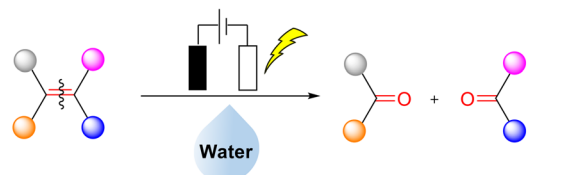
<sup>†</sup> Contributed equally to this work.

## Previous methods:



- Ozonolysis
  - Stoichiometric metal or non-metal oxidants (KMnO<sub>4</sub>, RuO<sub>4</sub>, OsO<sub>4</sub>, H<sub>2</sub>O<sub>2</sub>, NaIO<sub>4</sub> etc.)
  - Photochemical methods with O<sub>2</sub> as oxidant (generally only applicable to styrene derivatives)
  - Electrochemical methods (only applicable to styrene derivatives and require O<sub>2</sub> or other specialized redox catalysts)
- ✗ Safety risk   
 ✗ Multiple steps   
 ✗ Additional reagent waste due to external oxidizer

## Our work:



Electrochemical water activation for the oxidative cleavage of alkenes

- ✓ Mild electrochemical reaction conditions
- ✓ Green oxidant: water, no need for additional oxidants
- ✓ Oxidative cleavage of non-activated and activated alkenes
- ✓ Oxidative cleavage of the allylic C-C(vinyl)  $\sigma$ -bond and a unique linear paired electrolysis mode have been observed in some examples

**Fig. 1** Previous methods and our work. (a) PbO<sub>2</sub>/Ti as anode, Sudan III before electrolysis; (b) PbO<sub>2</sub>/Ti as anode, Sudan III after electrolysis; (c) Pt as anode, Sudan III before electrolysis; (d) Pt as anode, Sudan III after electrolysis.

promote its combination with atomic oxygen to produce ozone.<sup>27</sup> This process potentially enables the electrochemical water activation for alkene cleavage. This electrochemical strategy is advantageous due to the lower stationary concentrations of hazardous ozone and ozonides, resulting from their *in situ* reaction, which reduces safety risks compared to traditional ozonolysis, avoids the problems of over-oxidation and excessive waste production. Furthermore, we observed oxidative cleavage of the allylic C-C(vinyl)  $\sigma$ -bond and a unique linear paired electrolysis mode, which are not observed in existing methods for the oxidative cleavage of olefins. Glassy carbon and boron-doped diamond, both materials possessing high O<sub>2</sub> evolution overpotentials, which are presumed conducive to ozone generation, were therefore also screened as anode materials.<sup>28</sup> The desired product was exclusively obtained utilizing a PbO<sub>2</sub>/Ti electrode (entries 1–3). The cathode electrode screening included nickel foam and graphite felt; however, their performance was inferior to that of the platinum electrode (entries 4 and 5). The optimal conditions for the electrochemical water activation for **1a** cleavage were identified as

**Table 1** Optimization of reaction conditions<sup>a</sup>

Entry	Deviation from standard condition	Yield <sup>b</sup> (%)
1	None	44
2	Glassy carbon as anode	ND
3	Boron-doped diamond as anode	ND
4	Nickel foam as cathode	6
5	Graphite felt as cathode	11
6	70 mA	24
7	20 mA	17
8	60 °C	ND
9	10 °C	13
10	0.15 mmol <b>1a</b>	Trace
11	0.6 mmol <b>1a</b>	9
12	LiBF <sub>4</sub> instead of Et <sub>4</sub> NBF <sub>4</sub> , 20 mA	14
13	Et <sub>4</sub> NCl instead of Et <sub>4</sub> NBF <sub>4</sub> , 20 mA	11
14	Et <sub>4</sub> NBF <sub>4</sub> (0.24 mmol)	40
15	Et <sub>4</sub> NBF <sub>4</sub> (0.96 mmol)	43
16	No EtOAc	ND
17	CH <sub>3</sub> CN instead of EtOAc	Trace

<sup>a</sup> Standard conditions: undivided cell, PbO<sub>2</sub>/Ti anode (1 cm × 2 cm), Pt cathode (1 cm × 1.5 cm), **1a** (0.3 mmol), Et<sub>4</sub>NBF<sub>4</sub> (0.48 mmol), EtOAc (1.8 mL), phosphate buffer (Na<sub>2</sub>HPO<sub>4</sub>: 90 mg mL<sup>-1</sup>, NaH<sub>2</sub>PO<sub>4</sub>: 30 mg mL<sup>-1</sup>, NaF: 0.12 mg mL<sup>-1</sup>, H<sub>2</sub>O: 3 mL), 50 mA, RT, 18 h. Voltage 4.1–4.3 V. <sup>b</sup> Isolated yield. RT, room temperature; ND, not detected.

follows: constant current electrolysis (50 mA) in EtOAc (1.8 mL), phosphate buffer (3 mL, prepared using Na<sub>2</sub>HPO<sub>4</sub>: 90 mg mL<sup>-1</sup>, NaH<sub>2</sub>PO<sub>4</sub>: 30 mg mL<sup>-1</sup>, NaF: 0.12 mg mL<sup>-1</sup> in water) at room temperature for 18 h, PbO<sub>2</sub>/Ti as anode, Pt as cathode, Et<sub>4</sub>NBF<sub>4</sub> as the electrolyte. Under these conditions, the desired alkene oxidative cleavage product **2a** was isolated in 44% yield (entry 1). The role of the phosphate buffer is to protect the lead dioxide electrode from corrosion by the generated H<sup>+</sup>, which facilitates electrode recycling.<sup>29</sup> The function of NaF is to block the O<sub>2</sub> evolution and, thereby, increase the efficiency for O<sub>3</sub> evolution.<sup>30</sup> The phosphate buffer and NaF also serve as supporting electrolytes. Increasing the current to 70 mA or decreasing it to 20 mA both resulted in a reduced yield (entries 6 and 7). Increasing reaction temperature to 60 °C resulted in no product formation (entry 8), while decreasing reaction temperature to 10 °C led to a significant reduction in yield (entry 9). Lowering the concentration of substrate **1a** resulted in trace amounts of product (entry 10), while increasing the concentration of substrate **1a** led to a decreased yield (entry 11). Screening of supporting electrolytes revealed Et<sub>4</sub>NBF<sub>4</sub> as the most efficient (entries 12 and 13). Neither an increase nor a decrease in the amount of Et<sub>4</sub>NBF<sub>4</sub> electrolyte resulted in a higher yield (entries 14 and 15). During solvent screening, no product was observed when ethyl acetate was not used (entry 16). Furthermore, when acetonitrile was used

instead of ethyl acetate, only trace amounts of product were obtained (entry 17).

With the optimized conditions in hand, we investigated the scope of the olefin cleavage (Table 2). First, a series of unactivated aliphatic alkenes, including terminal alkenes and internal di- and trisubstituted alkenes, were examined. In most cases, aldehydes or ketones were obtained in moderate to good yields. To demonstrate the applicability of our method in chiral synthesis, we examined the reactions of the natural products dihydrocarvone and vitamin K1. Gratifyingly, both reacted smoothly, yielding the valuable, synthetically challenging chiral products (2*R*,5*R*)-5-acetyl-2-methylcyclohexan-1-one **6b** and hexahydrofarnesyl acetone **7b**, respectively. Interestingly, unlike the model substrate 1-allylnaphthalene (**1a**), allylic benzene derivatives exhibited different reactivity. The *para*-bromo-substituted allylbenzene (**9a**) and the (*S*)-naproxen-derived analogue (**8a**) produced benzaldehyde derivatives as products (**9b** and **8b**, respectively) *via* allylic C–C (vinyl)  $\sigma$ -bond oxidative cleavage. This oxidative cleavage of the allylic C–C(vinyl)  $\sigma$ -bond<sup>31</sup> is difficult to achieve with existing olefin oxidative cleavage methods. Additionally, the major products from the reaction of long-chain aliphatic terminal alkene 1-heptene **10a**, internal alkene 2-octene **11a**, and a cyclohexyl-substituted terminal alkene **12a** were carboxylic acids. More surprisingly, cyclic alkenes **13a** and **14a** both underwent oxidative double bond cleavage, with the ketone carbonyl group remaining intact and the aldehyde group further reduced to an alcohol. We hypothesize that the generated aldehyde group, upon double bond cleavage of these cyclic alkenes, is further reduced to the alcohol by hydrogen gas produced at the cathode. This linear paired electrolysis mode,<sup>32</sup> involving sequential anodic oxidation and cathodic reduction of the same starting material, has rarely been reported in the literature. Using this linear paired electrolysis approach, we achieved one-step synthesis of the sequential oxidation–reduction products **13b** and **14b**, containing both ketone and hydroxyl groups, with a 40% yield. In contrast, previous literature<sup>33,34</sup> needs a two-step sequence of OsO<sub>4</sub> or O<sub>3</sub> oxidation followed by NaBH<sub>4</sub> reduction, giving only 17% yields. This linear paired electrolysis approach offers a promising platform for exploring further sequential oxidative double bond cleavage and cathodic reduction reactions.

In order to further expand the range of olefins, the oxidative cleavage of mono-, di-, and trisubstituted styrenes derivatives was also examined. It was found that styrenes containing both electron-donating and electron-withdrawing substituents performed well under the reaction conditions, resulting in the corresponding aldehydes or ketone in moderate to good yields. The reaction also demonstrated excellent functional group compatibility, tolerating halogen, nitro, ester, cyano, and methoxy substituents. Heterocycle-substituted alkene, including 2-vinylpyridine **21a**, and electron-deficient alkene, such as (2-nitrovinyl)benzene **25a**, were also tolerated. Diphenylethylene was also tolerated, providing product **23b** in good yield. Cyclic alkene also reacted smoothly to give a product **32b** containing both aldehyde and ketone functional-

Table 2 Scope of the oxidative cleavage of alkenes<sup>a</sup>

unactivated aliphatic alkenes			
Substrate	Product and Yield <sup>b</sup>	Substrate	Product and Yield <sup>b</sup>
<b>1a</b>	<b>1b</b> , 44%	<b>4a</b>	<b>4b</b> , 47%
<b>2a</b>	<b>2b</b> , 50% <sup>[d]</sup>	<b>5a</b>	<b>5b</b> , 61% <sup>[c]</sup>
<b>3a</b>	<b>3b</b> , 66%	<b>6a</b> dihydrocarvone	<b>6b</b> , 51%
<b>7a</b> Vitamin K1	<b>7b</b> , 42% <sup>[d]</sup>		
<b>8a</b> ( <i>S</i> )-naproxen	<b>8b</b> , 44% oxidative cleavage of allylic C–C(vinyl) $\sigma$ -bond		
<b>9a</b>	<b>9b</b> , 54% oxidative cleavage of allylic C–C(vinyl) $\sigma$ -bond	<b>10a</b>	<b>10b</b> , 52%
<b>11a</b>	<b>11b</b> , 59%	<b>12a</b>	<b>12b</b> , 66%
<b>13a</b>	<b>13b</b> , 40% linear paired electrolysis one step		
<b>14a</b>	<b>14b</b> , 40% linear paired electrolysis one step		
			Previous 2 steps ref. 33, 17% yield
			Previous 2 steps ref. 34
mono-, di-, and trisubstituted activated alkenes			
Substrate	Product and Yield <sup>[b]</sup>	Substrate	Product and Yield <sup>[b]</sup>
<b>15a</b> , R <sup>1</sup> = H	<b>15b</b> , R <sup>1</sup> = H, 69%	<b>25a</b>	<b>25b</b> , 53%
<b>16a</b> , R <sup>1</sup> = Me	<b>16b</b> , R <sup>1</sup> = Me, 60%		
<b>17a</b> , R <sup>1</sup> = OCOMe <sup>[d]</sup>	<b>17b</b> , R <sup>1</sup> = OCOMe, 60%		
<b>18a</b> , R <sup>1</sup> = CN	<b>18b</b> , R <sup>1</sup> = CN, 57%		
<b>19a</b> , R <sup>1</sup> = NO <sub>2</sub> <sup>[d]</sup>	<b>19b</b> , R <sup>1</sup> = NO <sub>2</sub> , 46%		
<b>20a</b> , R <sup>1</sup> = CH <sub>2</sub> Cl	<b>20b</b> , R <sup>1</sup> = CH <sub>2</sub> Cl, 75%		
<b>21a</b>	<b>21b</b> , 41%	<b>26a</b> , R <sup>1</sup> = Cl	<b>26b</b> , R <sup>1</sup> = Cl, 75%
<b>22a</b>	<b>22b</b> , 42%	<b>27a</b> , R <sup>1</sup> = Br	<b>27b</b> , R <sup>1</sup> = Br, 71%
		<b>28a</b> , R <sup>1</sup> = CF <sub>3</sub>	<b>28b</b> , R <sup>1</sup> = CF <sub>3</sub> , 43%
		<b>29a</b> , R <sup>1</sup> = OMe	<b>29b</b> , R <sup>1</sup> = OMe, 60% <sup>[c]</sup>
<b>23a</b>	<b>23b</b> , 81% <sup>[c]</sup>	<b>30a</b>	<b>30b</b> , 65%
<b>24a</b>	<b>24b</b> , 60%	<b>31a</b>	<b>31b</b> , 62%
<b>32a</b>	<b>32b</b> , 45%		
<b>33a</b> Gemfibrozil	<b>33b</b> , 54%		

<sup>a</sup> Standard conditions: undivided cell, PbO<sub>2</sub>/Ti anode (1 cm × 2 cm), Pt cathode (1 cm × 1.5 cm), **1a** (0.3 mmol), Et<sub>4</sub>NBF<sub>4</sub> (0.48 mmol), EtOAc (1.8 mL), phosphate buffer (Na<sub>2</sub>HPO<sub>4</sub>: 90 mg mL<sup>-1</sup>, NaH<sub>2</sub>PO<sub>4</sub>: 30 mg mL<sup>-1</sup>, NaF: 0.12 mg mL<sup>-1</sup>, H<sub>2</sub>O: 3 mL), 50 mA, RT, 18 h. Voltage 4.1–4.3 V. <sup>b</sup> Isolated yield. <sup>c</sup> 21 h. <sup>d</sup> 24 h.

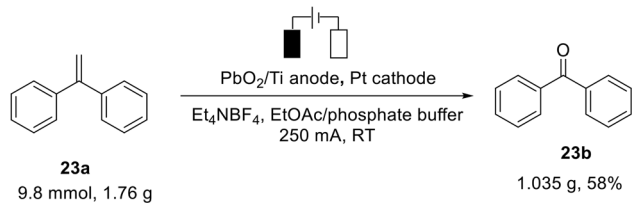
ities. The tetrasubstituted alkene tetraphenylethylene did not yield the target product due to its insolubility in ethyl acetate. A styrene derivative of the drug molecule gemfibrozil also smoothly yielded the aldehyde product **33b**.

As depicted in Fig. 2(A), this system can be scaled up to gram level easily. It indicates that this method might be applied in chemical and pharmaceutical industry. To investigate the oxidation reaction mechanism,  $^{18}\text{O}$ -labeling experiments were conducted to probe the source of oxygen in the product Fig. 2 (B). The successful detection of  $^{18}\text{O}$ -labeled ketone **11b**- $^{18}\text{O}$  demonstrated that the product's oxygen atom was derived from water. This  $^{18}\text{O}$ -labeling experiment also validated the potential of our method to use inexpensive  $\text{H}_2^{18}\text{O}$  for  $^{18}\text{O}$ -labeling of drug molecules. To investigate whether water is

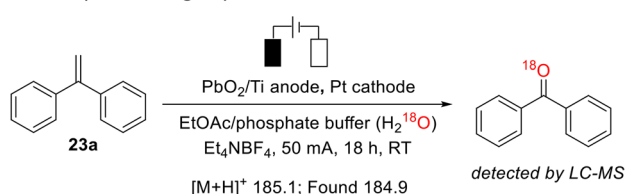
converted to ozone at the  $\text{PbO}_2/\text{Ti}$  anode, we conducted ozone detection experiments Fig. 2(C). Sudan III indicator<sup>5</sup> was added to the reaction system, and before electrolysis, it showed a red color when dissolved in ethyl acetate. After electrolysis, the solution turned yellow, indicating Sudan III oxidation and ozone generation. When the  $\text{PbO}_2/\text{Ti}$  anode was replaced with platinum, the Sudan III remained red before and after electrolysis, indicating no oxidation, thus confirming that the  $\text{PbO}_2/\text{Ti}$  electrode indeed converted water to ozone.

Based on the above mechanism studies and related literature reports,<sup>27</sup> we proposed a possible mechanism for the electrochemical oxidative cleavage of alkenes, as shown in Fig. 3. The  $\text{PbO}_2/\text{Ti}$  anode lattice is partially hydrated, forming gel zones. These zones slow the diffusion of molecular oxygen produced by the anodic electrolysis of water and promote its combination with atomic oxygen, resulting in ozone formation. The ozone then immediately facilitates the oxidative cleavage of alkenes in water phase.<sup>35</sup> Protons, also generated

### A. Gram-scale reaction



### B. Isotope labeling experiment



### C. Investigation on the formation of $\text{O}_3$

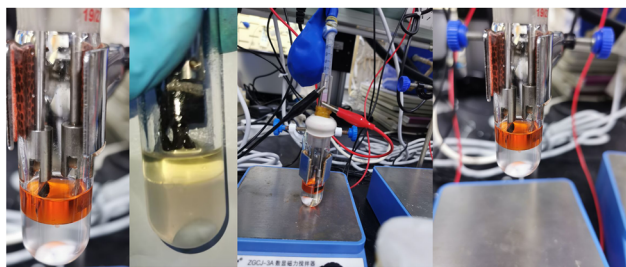


Fig. 2 (A) Gram-scale reaction; (B) isotope labeling experiment; (C) investigation on the formation of  $\text{O}_3$ .

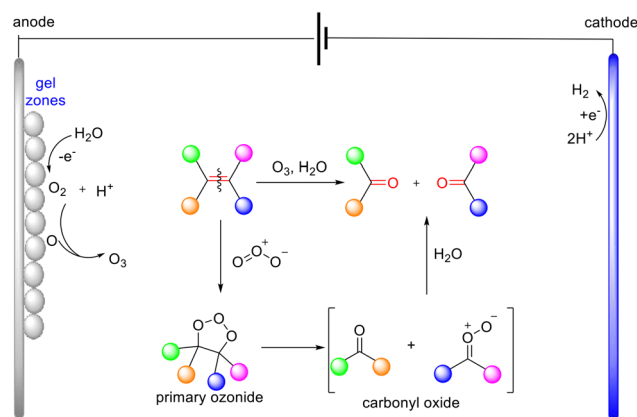


Fig. 3 A plausible mechanism for electrochemical oxidative cleavage of alkenes.

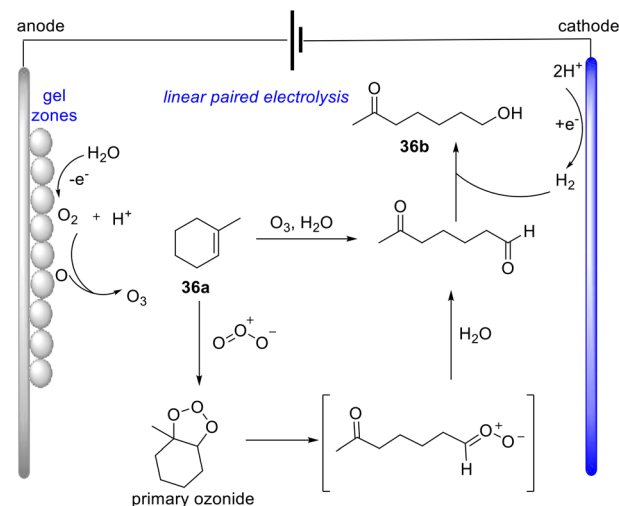


Fig. 4 A plausible mechanism for linear paired electrolysis.

by the anodic electrolysis of water, are reduced at the cathode, producing hydrogen gas. As for linear paired electrolysis, alkenes are first oxidatively cleaved by ozone generated from the anodic electrolysis of water, and then the resulting aldehyde groups are reduced to hydroxyl groups by hydrogen produced at the cathode (Fig. 4).

## Conclusions

In conclusion, we established a novel electrochemical water activation for the oxidative cleavage of alkenes. Water was used as an abundant and economical oxygen source, with no additional oxidants or complex workups. A wide range of alkenes such as activated alkenes and unactivated alkenes were tolerated. Additionally, our method enables the synthesis of challenging chiral products from natural chiral sources. This electrochemical strategy is advantageous due to the lower stationary concentrations of hazardous ozone and ozonides, resulting from their *in situ* reaction, which reduces safety risks compared to traditional ozonolysis, avoids the problems of over-oxidation and excessive waste production. Intriguingly, we observed oxidative cleavage of the allylic C–C (vinyl)  $\sigma$ -bond and a unique linear paired electrolysis mode, which are not observed in existing methods for the oxidative cleavage of olefins.

## Author contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

All data supporting the findings of this study are included within the article and its SI files. Supplementary information is available. See DOI: <https://doi.org/10.1039/d5qo00944h>.

## Acknowledgements

This work is supported by the National Natural Science Foundation of China (21672031, 22301027), Natural Science Foundation of Chongqing (CSTB2024NSCQ-MSX0129, CSTB2024NSCQ-MSX0138), the Science and Technology Research Program of Chongqing Municipal Education Commission (KJQN201900437, KJQN202300427) and CQMU Program for Youth Innovation in Future Medicine (W0152).

## References

- 1 M. S. Faculak, A. M. Veatch and E. J. Alexanian, Cobalt-Catalyzed Synthesis of Amides from Alkenes and Amines Promoted by Light, *Science*, 2024, **383**, 77–81.
- 2 M. L. Li, Y. J. Yuan, W. Harrison, Z. Y. Zhang and H. M. Zhao, Asymmetric Photoenzymatic Incorporation of Fluorinated Motifs into Olefins, *Science*, 2024, **385**, 416–421.
- 3 S. H. M. Kaster, L. Zhu, W. L. Lyon, R. Ma, S. E. Ammann and M. C. White, Palladium-Catalyzed Cross-Coupling of Alcohols with Olefins by Positional Tuning of a Counteranion, *Science*, 2024, **385**, 1067–1076.
- 4 A. Bunescu, Y. Abdelhamid and M. J. Gaunt, Multicomponent Alkene Azidoarylation by Anion-Mediated Dual Catalysis, *Nature*, 2021, **598**, 597–603.
- 5 T. J. Fisher and P. H. Dussault, Alkene Ozonolysis, *Tetrahedron*, 2017, **73**, 4233–4258.
- 6 R. Aashrita, L. Miguel and K. Wolfgang, Oxidative Alkene Cleavage by Chemical and Enzymatic Methods, *Adv. Synth. Catal.*, 2013, **355**, 3321–3335.
- 7 Y. C. Deng, X. J. Wei, H. Wang, Y. H. Sun, T. Noël and X. Wang, Disulfide-Catalyzed Visible-Light-Mediated Oxidative Cleavage of C = C Bonds and Evidence of an Olefin–Disulfide Charge-Transfer Complex, *Angew. Chem., Int. Ed.*, 2017, **56**, 832–836.
- 8 G. Urgoitia, R. SanMartin, M. T. Herrero and E. Domínguez, Aerobic Cleavage of Alkenes and Alkynes into Carbonyl and Carboxyl Compounds, *ACS Catal.*, 2017, **7**, 3050–3060.
- 9 A. Ruffoni, C. Hampton, M. Simonetti and D. Leonori, Photoexcited Nitroarenes for the Oxidative Cleavage of Alkenes, *Nature*, 2022, **610**, 81–86.
- 10 D. E. Wise, E. S. Gogarnoiu, A. D. Duke, J. M. Paolillo, T. L. Vacala, W. A. Hussain and M. Parasram, Photoinduced Oxygen Transfer Using Nitroarenes for the Anaerobic Cleavage of Alkenes, *J. Am. Chem. Soc.*, 2022, **144**, 15437–15442.
- 11 Y. Imada, Y. Okada, K. Noguchi and K. Chiba, Selective Functionalization of Styrenes with Oxygen Using Different Electrode Materials: Olefin Cleavage and Synthesis of Tetrahydrofuran Derivatives, *Angew. Chem., Int. Ed.*, 2019, **58**, 125–129.
- 12 X. Wu, A. P. Davis and A. J. Fry, Electrocatalytic Oxidative Cleavage of Electron-Deficient Substituted Stilbenes in Acetonitrile–Water Employing a New High Oxidation Potential Electrocatalyst. An Electrochemical Equivalent of Ozonolysis, *Org. Lett.*, 2007, **9**, 5633–5636.
- 13 F. X. Bu, Y. Q. Deng, J. Xu, D. L. Yang, Y. Li, W. Li and A. W. Lei, Electrocatalytic Reductive Deuteration of Arenes and Heteroarenes, *Nature*, 2024, **634**, 592–599.
- 14 B. M. Campbell, J. B. Gordon, R. E. Reichert, M. I. Gonzalez, K. G. Reynolds, M. Nava and D. G. Nocera, Electrophotocatalytic Perfluoroalkylation by LMCT Excitation of Ag(II) Perfluoroalkyl Carboxylates, *Science*, 2024, **383**, 279–284.

- 15 I. N. Cloward, T. F. Liu, J. Rose, T. Jurado, A. G. Bonn, M. B. Chambers, C. L. Pitman, M. A. ter Horst and A. J. M. Miller, Catalyst Self-Assembly Accelerates Bimetallic Light-Driven Electrocatalytic H<sub>2</sub> Evolution in Water, *Nat. Chem.*, 2024, **16**, 709–716.
- 16 B. Li, L. I. Oldham, L. Tian, G. Zhou, S. Selim, L. Steier and J. R. Durrant, Electrochemical versus Photoelectrochemical Water Oxidation Kinetics on Bismuth Vanadate (Photo) anodes, *J. Am. Chem. Soc.*, 2024, **146**, 12324–12328.
- 17 J. J. Zhang, C. Mück-Lichtenfeld and A. Studer, Photocatalytic Phosphine-Mediated Water Activation for Radical Hydrogenation, *Nature*, 2023, **619**, 506–513.
- 18 R. Ram, L. Xia, H. Benzidi, A. Guha, V. Golovanova, A. Garzón Manjón, D. Llorens Rauret, P. Sanz Berman, M. Dimitropoulos, B. Mundet, E. Pastor, V. Celorrio, C. A. Mesa, A. M. Das, A. Pinilla-Sánchez, S. Giménez, J. Arbiol, N. López and F. P. García de Arquer, Water-Hydroxide Trapping in Cobalt Tungstate for Proton Exchange Membrane Water Electrolysis, *Science*, 2024, **384**, 1373–1380.
- 19 I. Klose, C. Patel, A. Mondal, A. Schwarz, G. Pupo and V. Gouverneur, Fluorochemicals upon Low-Temperature Activation in Water, *Nature*, 2024, **635**, 359–364.
- 20 J. Y. Zhang, Z. L. Yang, C. L. Liu, H. Wan, Z. Z. Hao, X. R. Ji, P. J. Wang, H. Yi and A. W. Lei, Tailoring Photocatalysts to Modulate Oxidative Potential of Anilides Enhances Para-Selective Electrochemical Hydroxylation, *Nat. Commun.*, 2024, **15**, 6954.
- 21 A. B. Dapkekar, S. K. Nag and G. Satyanarayana, Electrochemically Driven Site-Selective C(sp<sup>2</sup>)-H Bond Hydroxylation of *N*-Substituted Anilines, *Adv. Synth. Catal.*, 2025, **367**, e202401349.
- 22 H. Huang and T. H. Lambert, Electrophotocatalytic C-H Heterofunctionalization of Arenes, *Angew. Chem., Int. Ed.*, 2021, **60**, 11163–11167.
- 23 M. A. Hoque, J. B. Gerken and S. S. Stahl, Synthetic Dioxygenase Reactivity by Pairing Electrochemical Oxygen Reduction and Water Oxidation, *Science*, 2024, **383**, 173–178.
- 24 C. B. Liu, F. P. Chen, B. H. Zhao, Y. M. Wu and B. Zhang, Electrochemical Hydrogenation and Oxidation of Organic Species Involving Water, *Nat. Rev. Chem.*, 2024, **8**, 277–293.
- 25 H. G. Roth, N. A. Romero and D. A. Nicewicz, Experimental and Calculated Electrochemical Potentials of Common Organic Molecules for Applications to Single-Electron Redox Chemistry, *Synlett*, 2016, 714–723.
- 26 D. M. Heard and A. J. J. Lennox, Electrode Materials in Modern Organic Electrochemistry, *Angew. Chem., Int. Ed.*, 2020, **59**, 18866–18884.
- 27 P. C. Foller and C. W. Tobias, Effect of Electrolyte Anion Adsorption on Current Efficiencies for the Evolution of Ozone, *J. Phys. Chem.*, 1981, **85**, 3238–3244.
- 28 S. G. Park, Stable Ozone Generation by Using Boron-Doped Diamond Electrodes, *Russ. J. Electrochem.*, 2003, **39**, 321–322.
- 29 J. C. G. Thanos, H. P. Fritz and D. Wabner, The Influences of the Electrolyte and the Physical Conditions on Ozone Production by the Electrolysis of Water, *J. Appl. Electrochem.*, 1984, **14**, 389–399.
- 30 P. C. Foller and C. W. Tobias, The Anodic Evolution of Ozone, *J. Electrochem. Soc.*, 1982, **129**, 506.
- 31 B. B. Liu, L. Cheng, P. H. Hu, F. N. Xu, D. Li, W. J. Gu and W. Han, Iron-Catalyzed Oxidative C-C(vinyl)  $\sigma$  - Bond Cleavage of Allylarenes to Aryl Aldehydes at Room Temperature with Ambient Air, *Chem. Commun.*, 2019, **55**, 4817–4820.
- 32 L. Nie, J. Y. Yang, Z. Liu, S. B. Zhou, S. M. Chen, X. T. Qi, A. W. Lei and H. Yi, Linear Paired Electrolysis Enables Redox-Neutral (3 + 2) Annulation of Benzofuran with Vinylidazo Compounds, *J. Am. Chem. Soc.*, 2024, **146**, 31330–31338.
- 33 M. Morimoto, W. Cao, R. G. Bergman, K. N. Raymond and F. D. Toste, Chemoselective and Site-Selective Reductions Catalyzed by a Supramolecular Host and a Pyridine-Borane Cofactor, *J. Am. Chem. Soc.*, 2021, **143**, 2108–2114.
- 34 G. Y. Ishmuratov, G. R. Mingaleeva, O. O. Shakhanova, R. R. Muslukhov, M. P. Yakovleva, L. P. Botsman and A. G. Tolstikov, Synthesis from (+)- $\alpha$ -pinene of Optically Active Macrocycles Containing Cyclobutane, Ester, Azine, or Hydrazide Groups, *Chem. Nat. Compd.*, 2011, **47**, 210–214.
- 35 E. S. Charles and H. D. Patrick, Ozonolysis in Solvent/Water Mixtures: Direct Conversion of Alkenes to Aldehydes and Ketones, *J. Org. Chem.*, 2008, **73**, 4688–4690.