Chinese Chemical Letters 37 (2026) 112200

Contents lists available at ScienceDirect

Chinese Chemical Letters

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Electrochemical α-glycosylation of *exo*-glycals

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ARTICLE INFO

Article history:
Received 8 September 2025
Revised 18 November 2025
Accepted 28 November 2025
Available online 1 December 2025

Keywords:
Exo-glycals
1′-*C*-methyl-*O*-glycosides
Stereoselectivity
Electrocatalysis
Glycosylation

ABSTRACT

Electrosynthesis has emerged as a powerful and sustainable strategy in organic chemistry, with growing applications in carbohydrate synthesis. Herein, we report an electrochemical method for the regio- and stereo-selective *O*-glycosylation of *exo*-glycals with a wide range of substrates under mild conditions. This approach employs simple setup in an undivided cell with KOTf as electrolyte, facilitating the efficient synthesis of bioactive *O*-glycosides in good yields and with exclusive α-selectivity. The reaction exhibits broad substrate compatibility, accommodating both sugar- and non-sugar-based acceptors, including complex natural products and pharmaceuticals. Furthermore, gram-scale reactions proceed without loss of efficiency or stereocontrol, underscoring the practicality of this method. Mechanistic studies suggest a radical cation-based pathway. This strategy provides a robust and scalable platform for *O*-glycosides synthesis.

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Nucleic acids, proteins, and polysaccharides constitute the three fundamental classes of biomolecules essential for life. Among these, polysaccharides play critical roles in numerous biological processes, including bacterial and viral infections, cell recognition, immune regulation, inflammatory responses, and cancer metabolism [1–3]. Advances in understanding carbohydrate-receptor interactions have spurred significant progress in the synthesis of carbohydrate analogs and mimetics [4–7]. Nonetheless, significant challenges remain in glycoscience, particularly in achieving the efficient and stereoselective synthesis of bioactive oligosaccharides and glycoconjugates. Addressing these challenges requires the development of innovative synthetic platforms capable of generating diverse carbohydrate-based structures, thereby bridging the gap between fundamental glycoscience and its medical applications.

In recent years, electrochemical organic synthesis has emerged as a powerful tool for green synthesis, offering advantages such as high atom economy, mild reaction conditions, and scalability [8–17]. These attributes make electrochemical methods particularly attractive for glycosylation reactions [18–28].

Glycals represent an important class of polyhydroxylated chiral synthons that serve as versatile synthetic intermediates in the synthesis of oligosaccharides, glycoconjugates, and bioactive natural products [29–34]. They are broadly classified into two categories: *Endo*-glycals, which contain an endocyclic carbon-carbon double bond, and *exo*-glycals (or 1-methylene sugars), featuring an exocyclic double bond (Fig. 1a) [35]. *Endo*-glycals have been extensively explored and widely employed in *O*- and *C*-glycosylation reactions [20,21,26,34,36–52]. In medicinal chemistry, *exo*-glycals are valued as glycosidase inhibitors [53–55] and have been used in the synthesis of *C*-glycoside analogs *via* various strategies, including hydroboration/Suzuki cross-coupling [56], Stille cross-coupling [57], Heck reactions [58], radical addition [59], epoxidation [60], and others methods [56–66].

In contrast, *O*-glycosylation reactions employing *exo*-glycals as glycosyl donors remain underdeveloped [67–73]. Existing approaches are largely limited to two conventional pathways: (a) Lewis acid-catalyzed direct *O*-glycosylation [72,73] and (b) Lewis acid-promoted rearrangement reactions [69,70], both of which have been applied to synthesize biologically relevant 1′-*C*-methyl-*O*-glycosides. These methods, however, suffer from several drawbacks, including narrow substrate scope, limited stereocontrol, and harsh reaction conditions. Thus, the development of efficient and stereoselective strategies for synthesizing 1′-*C*-methyl-*O*-glycoside analogs is highly desirable.

Our group recently reported an electrochemical method for glycosylation using *endo*-glycal, which afforded products in high yields with excellent selectivity, offering an efficient and novel strategy for *O*-glycosylation (Fig. 1d) [20]. Inspired by the structural similarities between the double bonds in *exo*- and *endo*-glycals, and encouraged by the efficiency and sustainability of electrochemical synthesis, we sought to extend this electro-promoted approach to achieve efficient and stereoselective *O*-glycosylation of

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https://doi.org/10.1016/j.cclet.2025.112200
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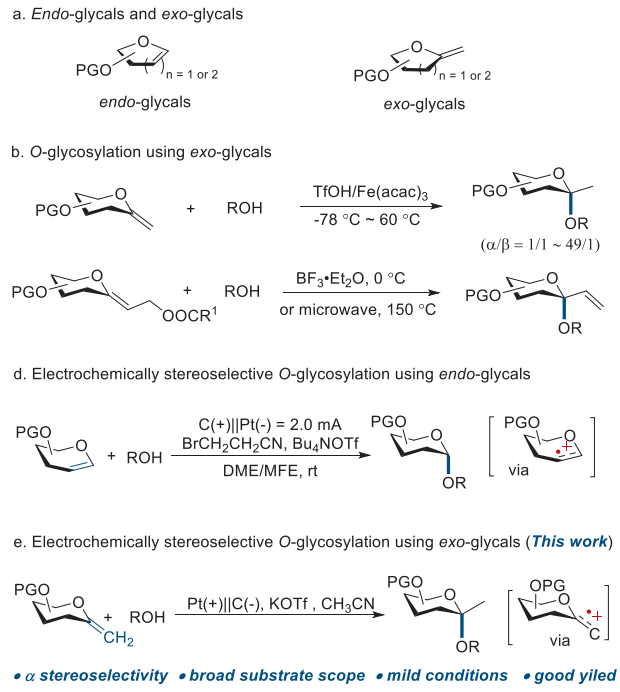
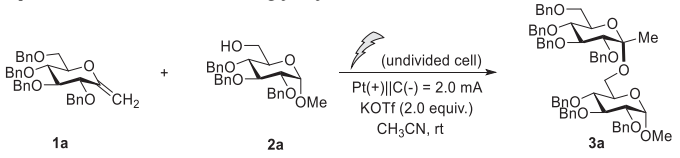


Fig. 1. Structural comparison of *endo*- and *exo*-glycals and their applications in *O*-glycosylation.

exo-glycals (1-methylene sugars) to form 1′-*C*-methyl-*O*-glycosides (Fig. 1e). Herein, we present an electrochemical method for the efficient synthesis of *O*-glycoside analogs from *exo*-glycals and nucleophilic acceptors. The electrochemical method exhibits a combination of broad substrate scope, outstanding functional group tolerance (especially for alkenes), excellent stereocontrol, and enhanced reaction efficiency. In addition, this strategy not only expands the synthetic utility of *exo*-glycals but also aligns with green chemistry principles by reducing environmental impact.

We initiated our investigation by examining the electrochemical glycosylation of benzyl protected-1-methylenesugars **1a** and **2a** in an undivided electrochemical cell (Table 1). Under initial conditions using Bu_4NOTf as the electrolyte, a graphite anode, a platinum plate cathode, CH_3CN as the solvent, room temperature, and a constant current of 2 mA, the target product **3a** was achieved with 44 % yield and exclusive α-selectivity (entry 1). Solvent screening revealed a significant influence on the reaction outcome (entries 2–4). Low or negligible yields were observed with 1,2-dimethoxyethane (DME), diethyl ether, or 1,4-dioxane. Subsequent evaluation of electrolytes, including Bu_4NBF_4, Bu_4NOAc, Bu_4NBr, KOTf and NaOTf, identified KOTf and NaOTf as superior, affording **3a** in 51 % and 50 % yields, respectively (entries 5–9). Based on these results, KOTf (2.0 equiv.) was selected for further optimization. We then evaluated alternative electrode configurations. Employing graphite for both electrodes resulted in a diminished yield of 31 % (entry 10), while a Pt/Pt setup completely inhibited the reaction (entry 11). Notably, reversing the electrodes to a Pt anode and graphite cathode significantly improved the yield to 87 % (entry 12). Varying the current intensity did not lead to further enhancement (entries 13 and 14). Control experiments confirmed that both electricity and electrolyte are essential, as no reaction occurred in their absence (entries 15 and 16).

Table 1
Optimization of electrochemical glycosylation reaction conditions.[a]



Entry	Conditions	Yield (%)[a]
1	C(+)||Pt(-) = 2.0 mA, Bu_4NOTf (2.0 equiv.), CH_3CN	44
2	C(+)||Pt(-) = 2.0 mA, Bu_4NOTf (2.0 equiv.), DME	0
3	C(+)||Pt(-) = 2.0 mA, Bu_4NOTf (2.0 equiv.), Et_2O	0
4	C(+)||Pt(-) = 2.0 mA, Bu_4NOTf (2.0 equiv.), 1,4-dioxane	<10
5	C(+)||Pt(-) = 2.0 mA, Bu_4NBF_4 (2.0 equiv.), CH_3CN	<5
6	C(+)||Pt(-) = 2.0 mA, Bu_4NOAc (2.0 equiv.), CH_3CN	<5
7	C(+)||Pt(-) = 2.0 mA, Bu_4NBr (2.0 equiv.), CH_3CN	<5
8	C(+)||Pt(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN	51
9	C(+)||Pt(-) = 2.0 mA, NaOTf (2.0 equiv.), CH_3CN	50
10	C(+)||C(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN	31
11	Pt(+)||Pt(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN	0
12	Pt(+)||C(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN	87
13	Pt(+)||C(-) = 1.0 mA, KOTf (2.0 equiv.), CH_3CN	85
14	Pt(+)||C(-) = 0.5 mA, KOTf (2.0 equiv.), CH_3CN	86
15	Pt(+)||C(-) = 2.0 mA, no electrolyte, CH_3CN	0
16	No current, KOTf (2.0 equiv.), CH_3CN	0

[a] Reaction conditions: undivided cell, **1a** (0.1 mmol, 2.0 equiv.), **2a** (0.05 mmol, 1.0 equiv.), solvent (3.0 mL), 0.5 h, r.t.

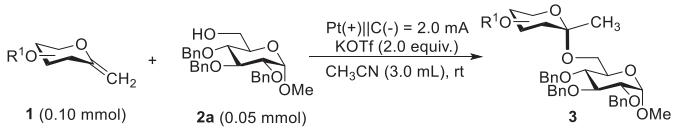
Having established the optimal electrochemical conditions, we next evaluated the scope of glycosyl donors (Table 2). Firstly, we synthesized a series of *exo*-glycals, including *gluco*– (**1a** and **1g**), *galacto*- (**1b**), *manno*- (**1c**), *rhamno*-(**1d**), *fuco*- (**1e**) and *lacto*- (**1f**), following established literature procedures [35,63,74]. Under the standard conditions, the reaction between 1-methylidene-2,3,4,5-tetra-*O*-benzyl-D-glucopyranoside (**1a**) and acceptor **2a** afforded disaccharide **3a** in 87 % yield (entry 1). Notably, a variety of per-benzylated 1-methylene sugars (**1b**-**1e**) acted as competent donors, yielding the corresponding disaccharides **3b**-**3e** in excellent yields ranging from 90 % to 97 % (entries 2–5). Trisaccharide **3f** was also obtained in good yield when *lacto*-derived donor **1f** was employed (entry 6). Furthermore, the 1-ethylsugar was found to be compatible with the system, furnishing glycoside **3g** in 75 % yield (entry 7).

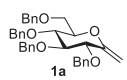
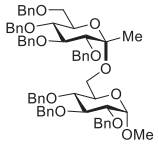
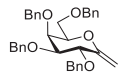
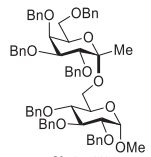
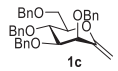
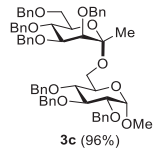
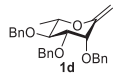
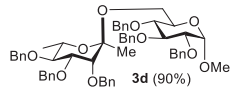
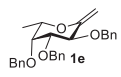
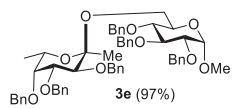
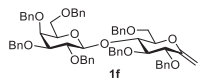
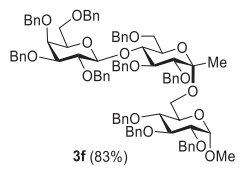
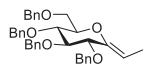
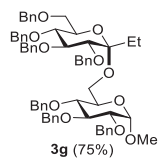
We then explored the acceptor scope of the transformation (Table 3). Both primary and secondary non-sugar alcohols were suitable reaction partners under the standard conditions, delivering glycosides **4a**-**4d** in 71 %–95 % yields. Among these, product **4d** was obtained with an α/β selectivity of 22/1 (entry 4), while the others were exclusively α-configuration. Notably, the reaction could be scaled up to furnish **4b** in 86 % yield (739 mg, entry 2). Reactions of cyclododecanol with either *exo*-glucal or *exo*-galcal donors provided **4c** and **4d** in 71 % and 73 % yields, respectively (entries 3 and 4). Acceptors containing a free C-6 hydroxyl group underwent smooth glycosylation to afford disaccharides **4e**-**4f** in 83 %–91 % yields (entries 5 and 6). Reactions with acceptors containing free C-3 or C-2 hydroxyl groups were less effective at room temperature; however, reducing the temperature to −30 °C improved the yields of products **4g** and **4h** to moderate to good levels (entries 7 and 8). Di-glycosylation of 1,1- bis(hydroxymethyl)cyclopropane and 1,12-dodecanediol with *exo*-glucal afforded **4i** and **4j** in 87 % and 77 % yields, respectively (entries 9 and 10).

To further demonstrate the practical utility of this method, we applied the electro-glycosylation reaction to the late-stage glycosylation of natural products (Table 4). Epiandrosterone was successfully coupled with *exo*-glucal donor under standard reaction conditions to afford **5a** in 61 % yiled. Similarly, podophyllotoxin was glycosylated to give **5b** in 60 % yield. Beta-sitosterol was also modified using different glycosyl donors (**1a** and **1b**), yielding products **5c** and **5d** in 84 % and 85 % yields, respectively. Cholesterol proved to be a suitable substrate as well, providing glycosides **5e** and **5f** in good yields.

To gain further insight into the reaction mechanism, a series of mechanistic experiments were conducted (Scheme 1). First, control experiments confirmed the essential role of electrical current and

Table 2
Scope of *exo*-glycal donors in the *O*-glycosylation reaction.[a]



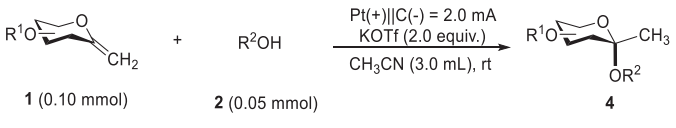
Entry	Donor	Acceptor	Product (yield)
1	 **1a**	**2a**	 **3a** (87%)
2	 **1b**	**2a**	 **3b** (97%)
3	 **1c**	**2a**	 **3c** (96%)
4	 **1d**	**2a**	 **3d** (90%)
5	 **1e**	**2a**	 **3e** (97%)
6	 **1f**	**2a**	 **3f** (83%)
7	 **1g**	**2a**	 **3g** (75%)

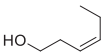
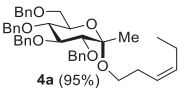
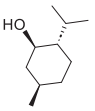
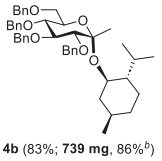
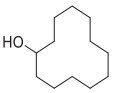
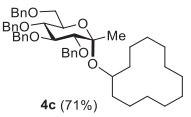
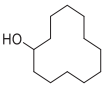
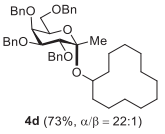
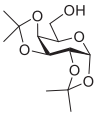
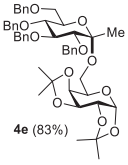
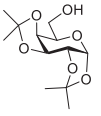
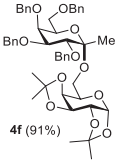
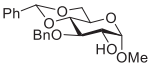
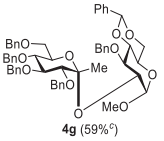
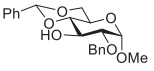
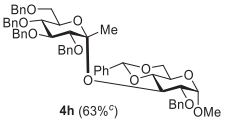
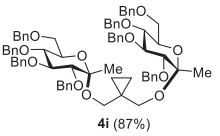
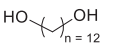
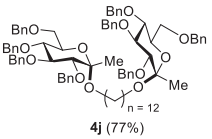
[a] Reaction conditions: Pt(+)||C(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN (3.0 mL), undivided cell, 0.5 h, r.t.

electrolyte, as no glycosylation occurred in their absence (Schemes 1a and b). Subsequently, the introduction of the radical scavenger TEMPO under standard conditions completely suppressed the formation of product **3a**. Notably, intermediates **6** and **7** were detected *via* ESI-MS, suggesting the involvement of radical species and a radical-inhibited pathway (Scheme 1c).

Deuterium labeling experiments provided additional insight into the source of the methyl hydrogen in the product. When the reaction between **1a** and **2a** was performed in CD_3CN, no deuterium incorporation was observed in the product, ruling out the solvent as the hydrogen source (Scheme 1d). In a complementary experiment, reaction of **1a** with CD_3OD in CH_3CN afforded deuterated product **8**, confirming that the methyl proton originates from the nucleophilic acceptor (Scheme 1e).

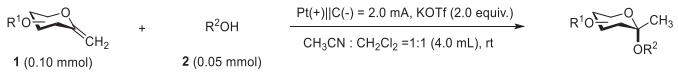
Table 3
Scope of acceptors in the electrochemical *O*-glycosylation.[a]

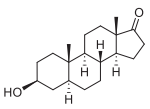
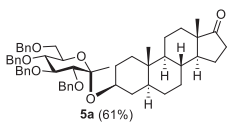
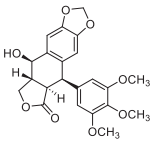
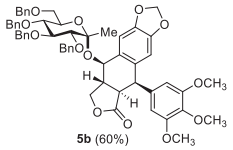
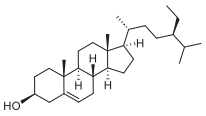
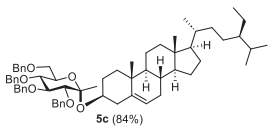
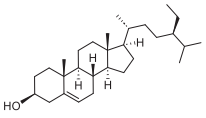
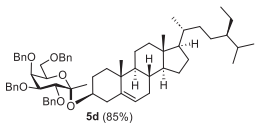
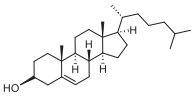
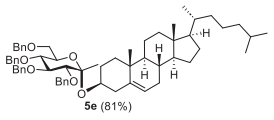
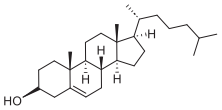
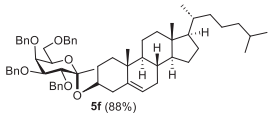


Entry	Donor	Acceptor	Product (yield)
1	**1a**		 **4a** (95%)
2	**1a**		 **4b** (83%; **739 mg**, 86%[b])
3	**1a**		 **4c** (71%)
4	**1b**		 **4d** (73%, α/β = 22:1)
5	**1a**		 **4e** (83%)
6	**1b**		 **4f** (91%)
7	**1a**		 **4g** (59%[c])
8	**1a**		 **4h** (63%[c])
9	**1a**		 **4i** (87%)
10	**1a**		 **4j** (77%)

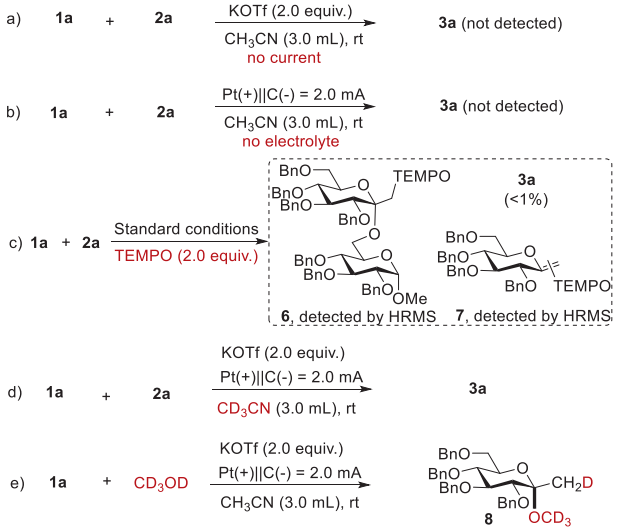
[a] Reaction conditions: Pt(+)||C(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN (3.0 mL), undivided cell, 0.5–1 h, r.t.
[b] **1a** (1.85 mmol, 1.5 equiv.), **1b** (1.24 mmol, 1.0 equiv.), 1 h.
[c] -30 °C, 2 h.

Table 4
Late-stage glycosylation of natural products and pharmaceuticals.[a]



Entry	Donor	Acceptor	Product (yield)
1	**1a**		 **5a** (61%)
2	**1a**		 **5b** (60%)
3	**1a**		 **5c** (84%)
4	**1b**		 **5d** (85%)
5	**1a**		 **5e** (81%)
6	**1b**		 **5f** (88%)

[a] Pt(+)||C(-) = 2.0 mA, KOTf (2.0 equiv.), CH_3CN:CH_2Cl_2 = 1:1 (4.0 mL), undivided cell, 1 h, r.t.



Scheme 1. Control experiments and isotopic labeling.

Based on these experimental results, along with supporting evidence from cyclic voltammetry (Figs. S7-S9 in Supporting information) and previous literature reports [73,75–80], a plausible reaction mechanism is proposed (Fig. 2). Initially, the glycosyl donor **1** is oxidized at the anode to form a radical cation intermediate **I**. Concurrently, the alcohol nucleophile is reduced at the cathode,

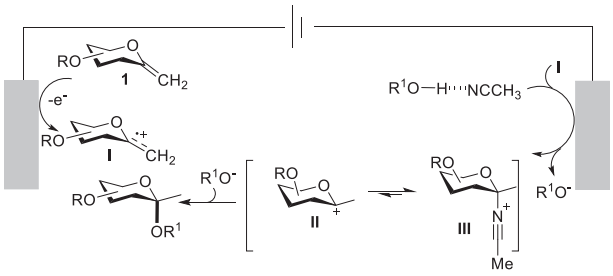


Fig. 2. Proposed mechanism for the electrochemical *O*-glycosylation.

generating a hydrogen radical and an alkoxide ion. The hydrogen radical combines with intermediate **I** to form carbocation intermediate **II**. The approach of acetonitrile is directed by the anomeric effect preferentially from the α-face, leading to the selective formation of intermediate **III**. In acetonitrile solvent, **III** predominates due to effective stabilization. Significant steric hindrance from the C1-methyl group prevents nucleophilic approach from the β-face, while favoring minor dissociation back to intermediate **II**. Ultimately, the alkoxide attacks intermediate **III** from the sterically accessible α-face, resulting in the thermodynamically favored α-glycoside product.

In concluding, we have developed an electrochemical glycosylation method for the stereoselective synthesis of *O*-glycosides using *exo*-glucals and various nucleophilic acceptors. This approach operates under simple conditions in an undivided cell, employing KOTf as the electrolyte to afford the corresponding glycosylation products in high yields with excellent selectivity. The reaction accommodates a broad range of glycosyl and non-glycosyl acceptors, consistently yielding products with exclusive α-selectivity. Scalability was demonstrated through gram-scale synthesis without compromising yield or stereoselectivity. Furthermore, the method proved effective for the late-stage glycosylation of natural products and pharmaceuticals, highlighting its potential for the discovery of carbohydrate-based drugs. This electrochemical strategy thus represents an efficient and attractive platform for synthesizing *O*-glycosides.

CRediT authorship contribution statement

Jing Zhang: Writing – original draft, Investigation. **Chen-Fei Gao:** Investigation. **Xin-Shan Ye:** Resources. **De-Cai Xiong:** Writing – review & editing, Writing – original draft, Supervision, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work was supported by grants from the National Natural Science Foundation of China (Nos. 22177003, 22377003) and the Fundamental Research Funds for Central Universities.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.112200.

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