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Copper-catalyzed aerobic oxyalkylation of alkenes with cyclopropanols: A straightforward access to 1,5-diketones

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ABSTRACT

We report here an aerobic oxyalkylation reaction of alkenes with cyclopropanols that enables the facile synthesis of 1,5-diketones. Key to success of this reaction is utilization of a $CuCl_2$·Phen catalyst, with the ambient air serving as both the oxidant and oxygen source. The reaction proceeds *via* a domino process involving a free-radical ring-opening addition of cyclopropanols to alkenes and radical capture by O_2. Both intra- and intermolecular variants are feasible, providing stream-lined access to a broad range of 1,5-diketones possessing fused, spiro, bridged, and linear frameworks. The synthetic utility of this method is showcased by late-stage functionalization of bioactive molecules and synthesis of natural products (analogues).

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The oxidation and coupling of readily accessible feedstock chemicals constitute a powerful strategy for synthesis of structurally intricate molecules in modern organic chemistry. Within this paradigm, the catalytic oxyalkylation of alkenes has become a cornerstone methodology, enabling simultaneous elevation of oxidation states and molecular complexity from simple precursors [1–6]. While significant progress has been achieved in this area, the pursuit of practical and sustainable protocols characterized by the use of earth-abundant metals, cost-effective ligands, and green oxidants remains a pivotal frontier.

One noteworthy application of alkene oxyalkylation is the synthesis of 1,4-diketones *via* a sequence involving the generation of α-keto radicals from ketones, addition to alkenes, and oxidation of the resulting γ-keto radicals (Fig. 1A) [7–13]. In 2014, Klussmann's group developed a *para*-toluenesulfonic acid-catalyzed alkylation-peroxidation reaction of styrene with ketones to afford γ-peroxyketones, which was then converted to the 1,4-diketones [7]. The groups of Huang [8], Wan [9], Xing [10], and Thakur [11] independently developed direct oxidative couplings of terminal vinylarenes with ketones to furnish 1,4-diketones directly, employing Cu/Mn co-catalysis or cobalt salts in conjunction with TBHP. Maity [12] and Senadi [13] reported an alternative strategy involving photoredox-catalyzed 1,2-oxoalkylation of vinyl arenes with 1,3-diketones, employing air as the terminal oxidant, sequentially. Inspired by these advances on 1,4-diketone synthesis through α-keto radicals, we envisioned that β-keto radicals generated by oxidation of cyclopropanols, the most prominent type of the homo-enol equivalents [14–21], might undergo an addition reaction with olefins to form δ-keto radicals, which could then be oxidized by molecular oxygen to afford 1,5-diketones straightforwardly [22–24].

The challenge of achieving this aerobic radical transformation lies in the intrinsic instability of β-keto radicals, which are less stable than α-keto counterparts and readily quenched by molecular oxygen [25–27]. Overcoming this limitation requires a catalytic system capable of efficiently generating β-keto radicals and directing their regioselective addition to alkenes. Recently, our group developed an intramolecular homo-Mannich reaction of cyclopropanols to construct azabicyclo[3.3.1]nonane skeletons [28–31]. In this reaction, β-keto radicals were generated *via* oxidative ring-opening of cyclopropanols and then efficiently trapped by the C=N double bonds of imines with $CuCl_2$ as the catalyst. Inspired by the advances in cyclopropanols chemistry made by our group [28–31] and others [32–47], we surmised that a copper-based catalytic system could be adapted to oxyalkylation of the C=C double bonds of alkenes with cyclopropanols, to furnish diverse 1,5-diketones (Fig. 1B). Herein, we report a copper-catalyzed oxyalkylation of alkenes with cyclopropanols under an atmo-

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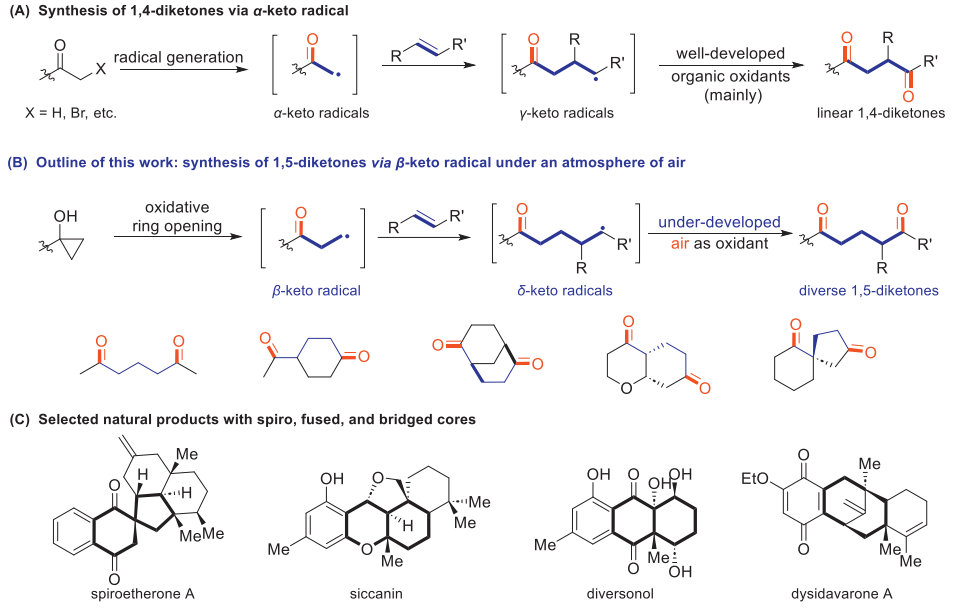
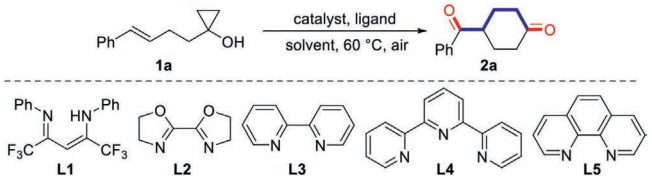


Fig. 1. Background and study synopsis. (A) Synthesis of 1,4-diketones *via* α-keto radical. (B) Outline of this work: Synthesis of 1,5-diketones *via* β-keto radical under an atmosphere of air. (C) Selected natural products with spiro, fused, and bridged cores.

Table 1
Optimization of the oxyalkylation of alkenes with cyclopropanols.[a]



Entry	Catalyst	Ligand	Solvent	Yield (%)[b]
1	$CuCl_2$	—	THF	26
2	CuCl	—	THF	<5
3	$CuCl_2$	—	THF	32
4	$Cu(OTf)_2$	—	THF	0
5	$CuCl_2$	**L1**	THF	37
6	$CuCl_2$	**L2**	THF	0
7	$CuCl_2$	**L3**	THF	30
8	$CuCl_2$	**L4**	THF	21
9	$CuCl_2$	**L5**	THF	69
10	$CuCl_2$	**L5**	1,4-Dioxane	63
11	$CuCl_2$	**L5**	MeCN	61
12[c]	$CuCl_2$	**L5**	THF	69
13[d]	$CuCl_2$·**L5**[f]	—	THF	75
14[e]	$CuCl_2$·**L5**[f]	—	THF	79

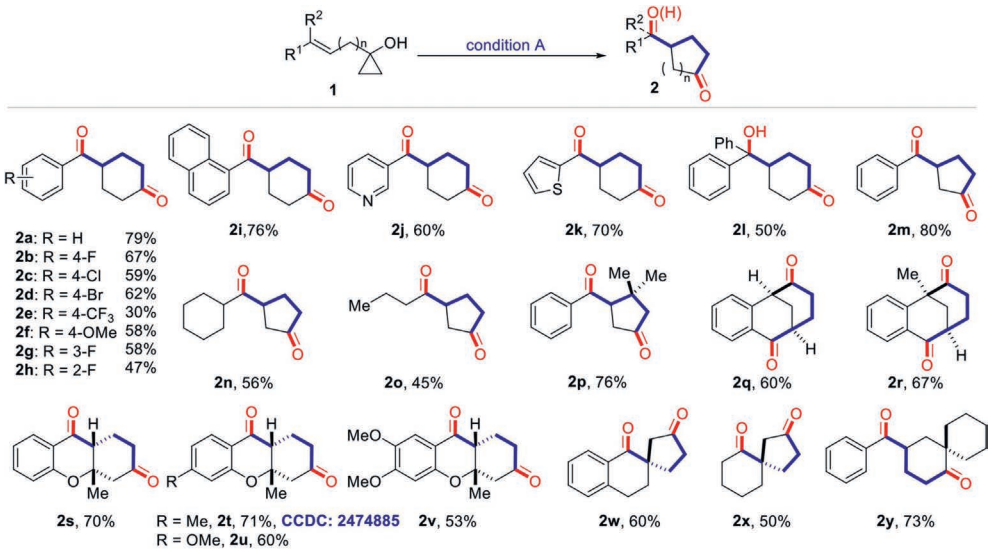
[a] Typical reaction conditions unless otherwise noted: **1a** (0.1 mmol), $CuCl_2$ (0.02 mmol), THF (4.0 mL), 12 h, 60 °C, open to air.
[b] isolated yield.
[c] 2 h of reaction time.
[d] 0.02 mmol $CuCl_2$·**L5** as the catalyst.
[e] 0.01 mmol $CuCl_2$·**L5** as the catalyst.
[f] $CuCl_2$·**L5** is pre-prepared.

sphere of air. This operationally simple protocol accommodates both inter- and intramolecular variants [48–55], providing rapid access to 1,5-diketones bearing linear, fused, spirocyclic, or bridged scaffolds-motifs commonly found in natural products such as spiroetherone A, siccanin, and dysidavarone A (Fig. 1C) [56–63].

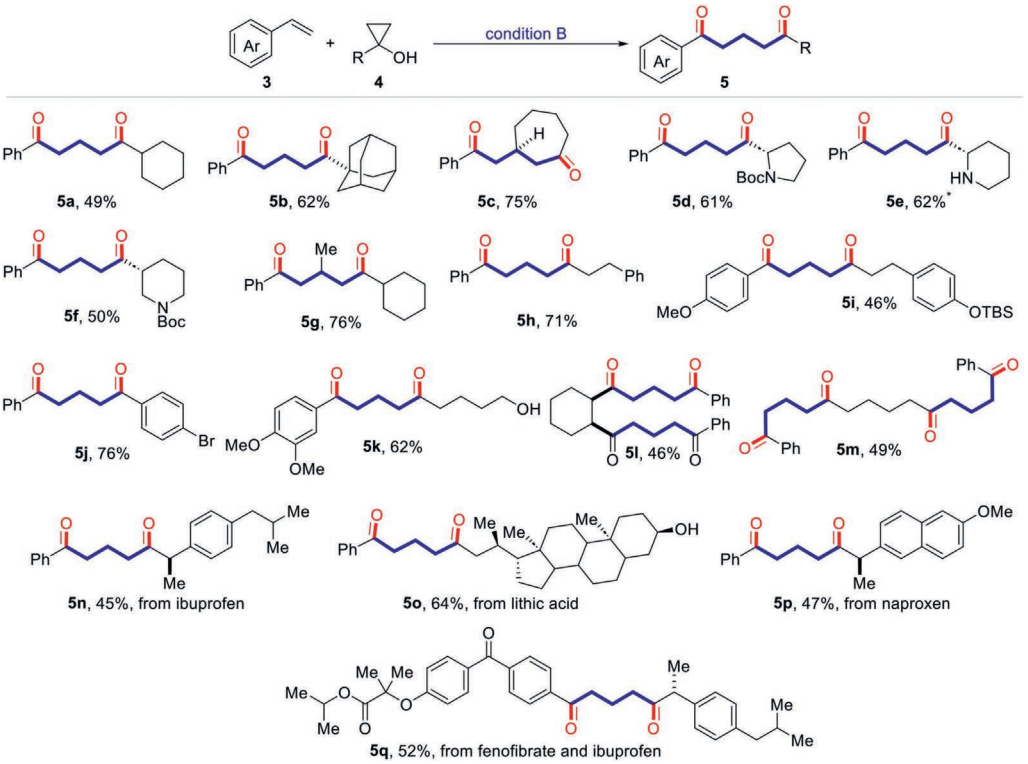
As shown in Table 1, we first commenced our study by screening various metal salts using cyclopropanol **1a** as the model substrate (entries 1–4), and $CuCl_2$ was identified as the optimal copper salt, yielding product **2a** in 32% yield (entry 3). Investigation of ligands (entries 5–9) indicated that 1,10-phenanthroline (Phen, **L5**) was the ligand of choice, increasing the yield to 69% (entry 9). Examination of solvents indicated that substitution of THF with 1,4-dioxane or MeCN was detrimental to the product yield (entries 10 and 11). When the reaction time was shortened to 2 h, the yield remained at 69%, underscoring the efficiency of this reaction protocol (entry 12). Further improvement was achieved using the preformed $CuCl_2$·Phen complex as the catalyst, which delivered **2a** in 79% yield at a 10 mol% catalyst loading (entries 13 and 14).

With the optimized reaction conditions established, we first explored the intramolecular oxyalkylation of alkenes with cyclopropanols (Scheme 1). Styryl-substituted substrates, bearing electron-donating or -withdrawing groups on the aryl ring, underwent a smooth transformation to afford the corresponding 4-benzoylcyclohexanone derivatives **2a**–**2h** in moderate to good yields. Other olefins with aryl groups including 1-naphthyl, 3-pyridyl and 2-thienyl were well tolerated, delivering products **2i**–**2k** in good yields. Importantly, the 1,1-diphenylethene-derived substrate **1l** also furnished the hydroxylated cyclohexanone **2l**. Aliphatic olefins proved to be compatible as well, affording **2n** and **2o** in good yields. Significantly, the methodology enabled the synthesis of a diverse array of bridged, spiro, and fused polycyclic products **2q**–**2y**. For instance, cyclopropanols **1q** and **1r** produced functionalized bicyclo[3.3.1]nonanes **2q** and **2r**, structural analogues of Dysidavarone A [53]. Likewise, fused products **2s**–**2v** were successfully generated, which are the core scaffold of the succinate dehydrogenase inhibitor Siccanin [54]. Importantly, the product **2u** could be obtained in 48% yield when the reaction was carried out on a 5 mmol scale, and the structure of **2t** was confirmed by X-ray crystallography (see details in Supporting information). Furthermore, cyclopropanols with dihydronaphthalene and cyclohexene substituents yielded spirocyclic products **2w**–**2y**. Among them, **2w** represents the spirocyclic core structure found in the natural product Spiroetherane [55].

Next, this method was adapted to the intermolecular oxyalkylation under slightly modified reaction conditions (Scheme 2). A broad range of cyclopropanols were compatible, affording the corresponding linear 1,5-dicarbonyl compounds **5a**–**5m** in moderate to good yields. Notably, cyclopropanol **4k** with a free hydroxyl group delivered product **5k** in 62% yield. Substrates bearing two cyclopropanol units (**4l** and **4m**) also participated in the transformation, providing products **5l** and **5m** with two 1,5-dicarbonyl



Scheme 1. The intramolecular oxyalkylation of olefins with cyclopropanols. Reaction condition A: substrate **1a–1y** (0.20 mmol), $CuCl_2$·Phen ($CuCl_2$·**L5**, 10 mol%), THF (4.0 mL), 60 °C, open to air, isolated yield.



Scheme 2. The intermolecular oxyalkylation of styrenes with cyclopropanols. Reaction condition B: substrate **3a**–**3c** (0.40-0.80 mmol), **4a**–**4m** (0.20 mmol), $CuCl_2$·Phen (10 mol%), CH_3CN (4.0 mL), 80 °C, open to air, isolated yield. * The yield of product was achieved *via* a two-step procedure after deprotection.

fragments. Furthermore, the utility of this protocol for drug derivatization was demonstrated. Cyclopropanols derived from ibuprofen, naproxen, and lithocholic acid reacted well with styrene to furnish products **5n**–**5q** in 45%–64% yields. Remarkably, coupling of an ibuprofen-derived cyclopropanol with a styrene derived from fenofibrate generated a conjugate of two drugs (**5q**), which may be of pharmacological interest in drug discovery.

Additionally, this versatile synthetic strategy was further applied to the synthesis of natural products or their analogues (Fig. 2) [56–63]. First, the natural product analogue 4-dehydroxydiversonol (**7**) [56,57] with multiple oxygen functionalities was synthesized from **2t** by taking advantage of the two ketone groups generated by our new method. The linear 1,5-dicarbonyl compounds can be employed in generating diverse range of heterocycles (**9** and **10**). For instance, a lasubine II analogue **9** was successfully prepared from oxyalkylation product **5k** through a sequence of reductive amination and intramolecular alkylation [60,61]. (±)-Centrolobine (**10**), which was isolated from the heartwood of *Centrolobium robustum* and exhibits biological activity against *Leishmania amazonensis* promastigotes, was synthesized in two steps from **5i** [62,63].

Control experiments were conducted to study the reaction mechanism (Fig. 3A). No product formation was observed in the

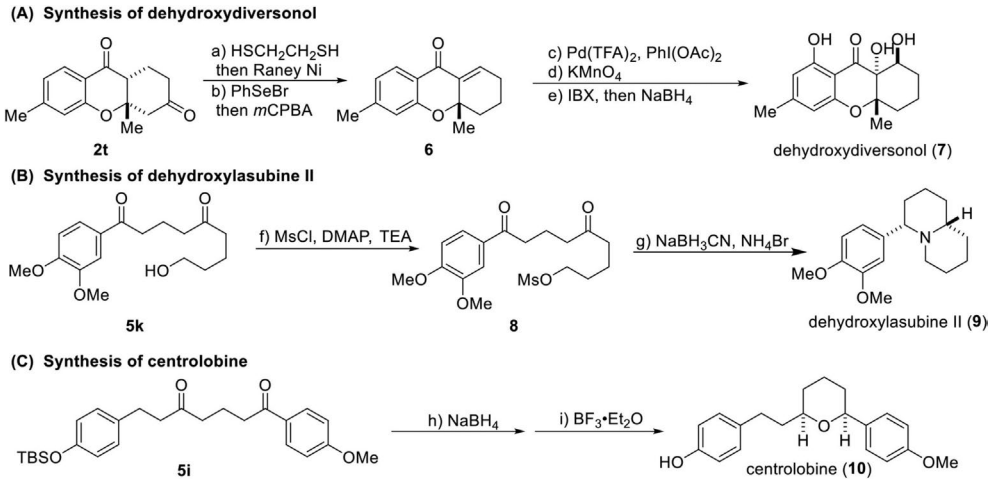


Fig. 2. The synthesis of natural products or their analogues. (A) Synthesis of dehydroxydiversonol. (B) Synthesis of dehydroxylasubine II. (C) Synthesis of centrolobine. Reagents and conditions: (a) 1,2-ethanedithiol (1.2 equiv.), $BF_3{\cdot}Et_2O$ (1.3 equiv.), $CHCl_3$, 0 °C; then Raney Ni (1.0 g), EtOH (10.0 mL), 50 °C, 68 % for 2 steps; (b) KHMDS (1.2 equiv.), PhSeBr (1.2 equiv.), THF, −78 °C; then *m*CPBA (1.0 equiv.), CH_2Cl_2, 64 % for 2 steps; (c) $Pd(TFA)_2$ (0.1 equiv.), $PhI(OAc)_2$ (2.0 equiv.), DCE, 100 °C, 86 %; (d) $KMnO_4$ (1.0 equiv.), EtOH, H_2O, 0 °C, 38 %; (e) IBX (1.0 equiv.), DMSO, 70 °C; then $NaBH_4$ (0.5 equiv.), MeOH/CH_2Cl_2, −78 °C, 48 % for 2 steps; (f) MsCl (1.0 equiv.), Et_3N (2.0 equiv.), DMAP (0.1 equiv.), CH_2Cl_2, 88 %; (g) $NaBH_3CN$ (1.0 equiv.), NH_4Br (2.0 equiv.), MeOH, then 2 mol/L HCl, 66 %; (h) $NaBH_4$ (2.0 equiv.), MeOH, −60 °C, 85 %; (i) $BF_3{\cdot}Et_2O$ (2.0 equiv.), CH_3CN, −40 °C, 93 %.

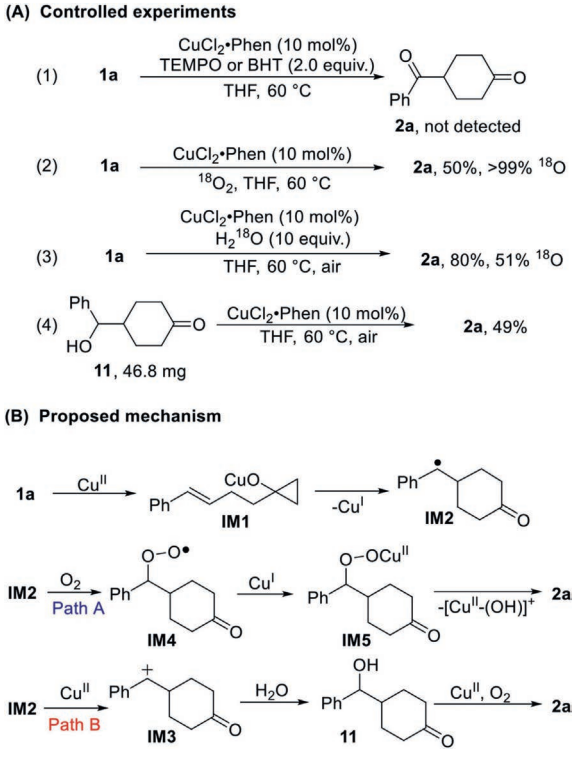


Fig. 3. Mechanistic studies and proposed mechanism. (A) Controlled experiments. (B) Proposed mechanism.

presence of radical scavengers TEMPO or BHT, the corresponding adducts of the β-carbonyl radical with TEMPO or BHT were detected by HRMS, indicating the involvement of a radical process. To probe the source of the incorporated oxygen atom, isotopic labeling studies were performed. When $^{18}O_2$ was employed as the oxidant in place of air, the resulting **2a** was obtained in 50 % yield with complete ^{18}O incorporation; while 10 equivalents of $H_2{}^{18}O$ were added to the standard reaction conditions, the product **2a** displayed 51 % ^{18}O incorporation as determined by high-resolution mass spectrometry. To further examine the possibility of water as the oxygen source, compound **11** was prepared and subjected to the standard conditions, furnishing product **2a** in 49 % yield. These findings suggest that both molecular oxygen and water may contribute as sources of the incorporated oxygen atom.

Based on the results of control experiments and previous reports [28–31,64–70], a plausible reaction mechanism is proposed (Fig. 3B). Initially, association of Cu^{II}-**L5** with cyclopropanol **1a** yields intermediate **IM1**, which undergoes ring opening and radical-type 6-*exo-trig* cyclization onto the alkene to afford δ-ketone radical **IM2** [68]. Subsequently, two possible pathways may account for the formation of the final product **2a**. The dominant route is Path A, in which radical **IM2** is intercepted by molecular oxygen to generate peroxyl radical **IM4**, which undergoes reduction by [$\mathbf{Cu^{I}}$] to afford **IM5**. Then, **IM5** go through a elimination reaction to ultimately afford product **2a** and release [$\mathbf{Cu^{II}}$-(OH)]$^+$ species [66,67]. In this pathway, molecular oxygen serves a dual role as both the oxidant and oxygen source. A plausible alternative mechanism (Path B) involves the further oxidation of radical **IM2** to form carbocation **IM3**, which is subsequently attacked by water to yield hydroxylated intermediate **11**, which is then followed by oxidation to deliver the final diketone **2a** [69,70].

In summary, we have developed an efficient method for the synthesis of 1,5-diketones *via* Cu-catalyzed oxyalkylation of alkenes with cyclopropanols under ambient air. The use of $CuCl_2{\cdot}$Phen complex as the catalyst proved critical to the success of this transformation. This protocol offers notable synthetic advantages, including operational simplicity, mild conditions, and broad substrate scope, enabling the conversion of unactivated alkenes into structurally diverse 1,5-diketones. Importantly, the intramolecular variant of this reaction grants access to valuable fused, spiro, bridged polycyclic scaffolds.

CRediT authorship contribution statement

Song Xi: Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **La Yin:** Writing – review & editing, Methodology, Investigation. **Changhong Xie:** Writing – review & editing, Software, Methodology. **Quanying Long:** Writing – review & editing, Methodology, Investigation. **Yulian Zhang:** Writing – review & editing, Software, Methodology, Investigation. **Ling He:** Writing – review & editing, Methodology, Funding acquisition. **Chenyang Wang:** Writing – review & editing, Writing – original

draft, Supervision, Methodology, Investigation, Conceptualization, Funding acquisition. **Min Zhang:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Methodology, Funding acquisition, 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.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2026.112433.

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