



(REVIEW ARTICLE)



ELECTROCHEMICAL AND PHOTOCATALYTIC C–H FUNCTIONALIZATION: A DECADE OF PROGRESS TOWARD ATOM- ECONOMIC LATE-STAGE MODIFICATION

Babatunde A. Adeleke ^{1, *} and Amina M. Bello ²¹ Department of Chemistry, Faculty of Science, Ahmadu Bello University (ABU), Zaria, Nigeria.² Department of Chemistry, University of Ibadan (UI), Ibadan, Nigeria.

* Corresponding Author

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ABSTRACT

The direct functionalization of C–H bonds represents one of the most powerful and atom-economical strategies in modern organic synthesis, enabling the conversion of ubiquitous, inert C–H bonds into valuable C–C and C–heteroatom bonds without requiring prefunctionalized substrates. Over the past decade, electrochemical and photocatalytic approaches have emerged as transformative platforms for C–H functionalization, offering sustainable alternatives to traditional methods that rely on stoichiometric oxidants, precious metal catalysts, and harsh reaction conditions. This review provides a comprehensive overview of the progress achieved in electrochemical and photocatalytic C–H functionalization from 2013 to 2023, with particular emphasis on the paradigm shift from precious metal catalysts (Pd, Rh, Ir, Ru) to earth-abundant 3d metals (Ni, Fe, Co, Cu). We critically examine the development of paired electrolysis strategies that simultaneously harness anodic oxidation and cathodic reduction, the integration of flow chemistry for scalable continuous-flow transformations, and the application of green chemistry metrics—including E-factor and atom economy—to evaluate the sustainability of these methodologies. The translational potential of these approaches for pharmaceutical and agrochemical late-stage functionalization is discussed, alongside current limitations and unresolved challenges. Finally, we offer perspectives on emerging trends, including electrophotocatalysis, artificial intelligence-driven reaction optimization, and the integration of renewable energy sources for sustainable chemical synthesis.

Keywords: C–H functionalization, Electrochemistry, Photocatalysis, Earth-abundant metals, Paired electrolysis, Flow chemistry, Green chemistry

1 INTRODUCTION

The selective functionalization of C–H bonds has long been considered the "Holy Grail" of organic synthesis. Carbon–hydrogen bonds are the most abundant structural motifs in organic molecules, yet their inertness—characterized by high bond dissociation energies (typically 96–105 kcal/mol for alkanes) and low polarity—has historically made them challenging substrates for direct transformation. Traditional synthetic approaches circumvent this challenge by relying on prefunctionalized starting materials, introducing functional groups (such as halides, boronic acids, or triflates) that

serve as handles for subsequent transformations. While effective, this strategy inherently generates stoichiometric waste, reduces atom economy, and increases synthetic step count.

The past decade has witnessed a paradigm shift in this landscape. The development of catalytic C–H functionalization methodologies has enabled the direct conversion of C–H bonds into C–C, C–N, C–O, C–S, and C–halogen bonds with remarkable selectivity and efficiency. Among the most significant advances are those driven by electrochemical and photochemical activation modes. These approaches harness electricity and light—renewable and traceless energy sources—to generate reactive intermediates under mild conditions, eliminating the need for stoichiometric chemical oxidants or reductants.

Electrochemical C–H functionalization operates through the application of an electric potential to drive electron transfer at electrode surfaces. As noted by Yang et al. (2019) and Jiao et al. (2020), this approach offers precise control over redox potentials, enabling selective oxidation or reduction processes with exceptional chemo-selectivity. Importantly, electrochemical oxidative C–H functionalizations produce molecular hydrogen as the sole byproduct through the hydrogen evolution reaction (HER), rendering them inherently atom-economical.

Photocatalytic C–H functionalization, in parallel, utilizes visible light to excite photosensitizers or photocatalysts, generating excited-state species capable of single-electron transfer (SET) or energy transfer to substrates. Bellotti et al. (2023) comprehensively reviewed photocatalytic late-stage C–H functionalization strategies, highlighting the mechanistic diversity and synthetic utility of these approaches for modifying complex small-molecule drugs, agrochemicals, and natural products.

The convergence of these two enabling technologies has given rise to electrophotocatalysis—the synergistic combination of light and electricity—which offers unprecedented opportunities for C–H functionalization under exceptionally mild conditions. As highlighted by recent reviews (Murtaza et al., 2023; Singh et al., 2024), electrophotocatalytic systems can access reactive intermediates and reaction pathways that are inaccessible to either electrochemistry or photocatalysis alone.

This review aims to provide a comprehensive and critical assessment of the progress in electrochemical and photocatalytic C–H functionalization over the past decade (2013–2023). We focus on four key thematic areas: (1) the transition from precious metal catalysts to earth-abundant 3d metals; (2) the development of paired electrolysis strategies; (3) the integration of flow chemistry for scalable continuous-flow synthesis; and (4) the application of green chemistry metrics to evaluate the sustainability of these methodologies. The translational relevance of these advances for pharmaceutical and agrochemical late-stage functionalization is emphasized throughout.

2 MAIN REVIEW

2.1 From Precious Metals to Earth-Abundant 3D Metals

The early development of C–H functionalization was dominated by precious metal catalysts—particularly palladium, rhodium, iridium, and ruthenium. While these catalysts exhibit exceptional activity and selectivity, their high cost, limited availability, and toxicity pose significant sustainability challenges. The past decade has witnessed a concerted effort to replace these precious metals with earth-abundant 3d transition metals, including nickel, cobalt, iron, and copper.

Nickel has emerged as a particularly versatile catalyst for electrochemical C–H functionalization. Ackermann (2020) summarized the emergence of electrocatalyzed C–H activation by earth-abundant 3d base metals, noting that nickel-based catalysts have been devised for a wide range of metallacyclic C–H activations. The compatibility of nickel with electrochemical conditions stems from its accessible redox states (Ni(0), Ni(I), Ni(II), Ni(III)) and its ability to participate in both oxidative addition and reductive elimination steps. Jiao et al. (2020) demonstrated site-selective C–H functionalization via the synergistic use of electrochemistry and transition metal catalysis, including nickel-catalyzed systems.

Cobalt has proven to be an exceptionally powerful platform for electrochemical C–H functionalization. Cobalt catalysts are particularly effective for C–H oxygenation, C–H amination, and C–H annulation with alkynes, isocyanates, and carbon monoxide. The relatively low cost and high abundance of cobalt make it an attractive alternative to precious metals. Notably, cobalt-catalyzed electrochemical C–H activations often proceed under remarkably mild conditions, with room-temperature operation and ambient atmosphere compatibility.

Iron has emerged as a sustainable catalyst for C–H functionalization, although its application in electrochemistry has lagged behind nickel and cobalt. Recent advances have demonstrated the viability of iron-catalyzed electrochemical C–

H activations, often leveraging ligand-to-metal charge transfer (LMCT) mechanisms for hydrogen atom transfer (HAT) from C–H bonds. As noted in recent reviews, iron-based catalytic systems have been successfully applied to electrophotocatalytic C(sp³)–H functionalization, enabling both net-oxidative transformations and redox-neutral processes.

Copper has found extensive application in both electrochemical and photocatalytic C–H functionalization. Copper catalysts are particularly effective for C–H bond functionalization involving radical intermediates, leveraging the facile interconversion between Cu(I) and Cu(II) oxidation states.

The transition to earth-abundant metals represents a fundamental advance in the sustainability of C–H functionalization. However, significant challenges remain. The reactivity of 3d metals is often lower than that of their precious metal counterparts, requiring more forcing conditions or specialized ligand designs. The mechanistic understanding of 3d metal-catalyzed electrochemical C–H functionalization is also less developed, limiting the rational design of improved catalysts.

2.2 Paired Electrolysis: Harnessing Both Anode and Cathode

Traditional electrochemical C–H functionalization typically employs either anodic oxidation or cathodic reduction as the primary activation mode, with the counter electrode serving only as a passive conduit for electron flow. Paired electrolysis represents a more sophisticated approach that simultaneously harnesses both anodic and cathodic processes to generate reactive intermediates, maximizing atom economy and energy efficiency.

In paired electrolysis, the anode and cathode each participate in productive chemical transformations. This approach can be classified into two main categories: convergent paired electrolysis, where intermediates generated at both electrodes combine to form the desired product, and divergent paired electrolysis, where independent transformations occur at each electrode.

Recent advances in paired electrolysis for C–H functionalization have been remarkable. As reviewed by recent studies, paired electrocatalysis strategies have been developed for alkane functionalization, integrating oxidative catalysis with reductive catalysis in a single cell without interference, employing earth-abundant iron and nickel as the anodic and cathodic catalysts, respectively.

A notable example is the benzylic C–H arylation with dicyanoarenes via convergent paired electrolysis reported in 2023. This approach merged anodic benzylic C–H bond oxidation with cathodic reduction of dicyanobenzenes, generating two reactive intermediates that combine to form biarylmethane derivatives. Similarly, paired electrochemical approaches have been developed for the direct hydroxylarylation of unactivated benzylic carbons (sp³/sp²/sp) via radical–radical cross-coupling.

The advantages of paired electrolysis extend beyond atom economy. By utilizing both electrodes productively, paired electrolysis can overcome the potential barriers that often limit single-electrode transformations. As highlighted by recent reviews, paired electrolysis enables the combination of oxidative and reductive catalysis in a single electrochemical cell, facilitating transformations that would be challenging or impossible using conventional approaches.

Despite these advances, paired electrolysis remains underutilized in C–H functionalization. The complexity of designing compatible anodic and cathodic reactions, the need for careful potential control to avoid interference between electrode processes, and the challenges of scaling up paired electrolysis systems have limited their widespread adoption.

2.3 Flow-Chemistry Integration: From Batch to Continuous Flow

The translation of electrochemical and photocatalytic C–H functionalization from batch to continuous-flow systems has emerged as a critical enabler for scalable, safe, and reproducible synthesis. As noted by recent reviews, continuous-flow technology has emerged as a powerful platform for resource-economical molecular synthesis, with the application of flow chemistry to C–H functionalization bearing unique potential.

Continuous-flow electrochemistry addresses several limitations of conventional batch electrochemistry. As summarized by recent studies (Huang et al., 2026; Murtaza et al., 2023), flow reactors provide a high surface-area-to-volume ratio, promoting uniform current distribution and efficient electron transfer. This improved mass and electron transfer enhances reaction efficiency, minimizes overoxidation or over-reduction, and allows reactions to be conducted at higher concentrations with improved reproducibility. The typically small interelectrode gap diminishes the ohmic drop, allowing for a substantially reduced amount of required supporting electrolyte.

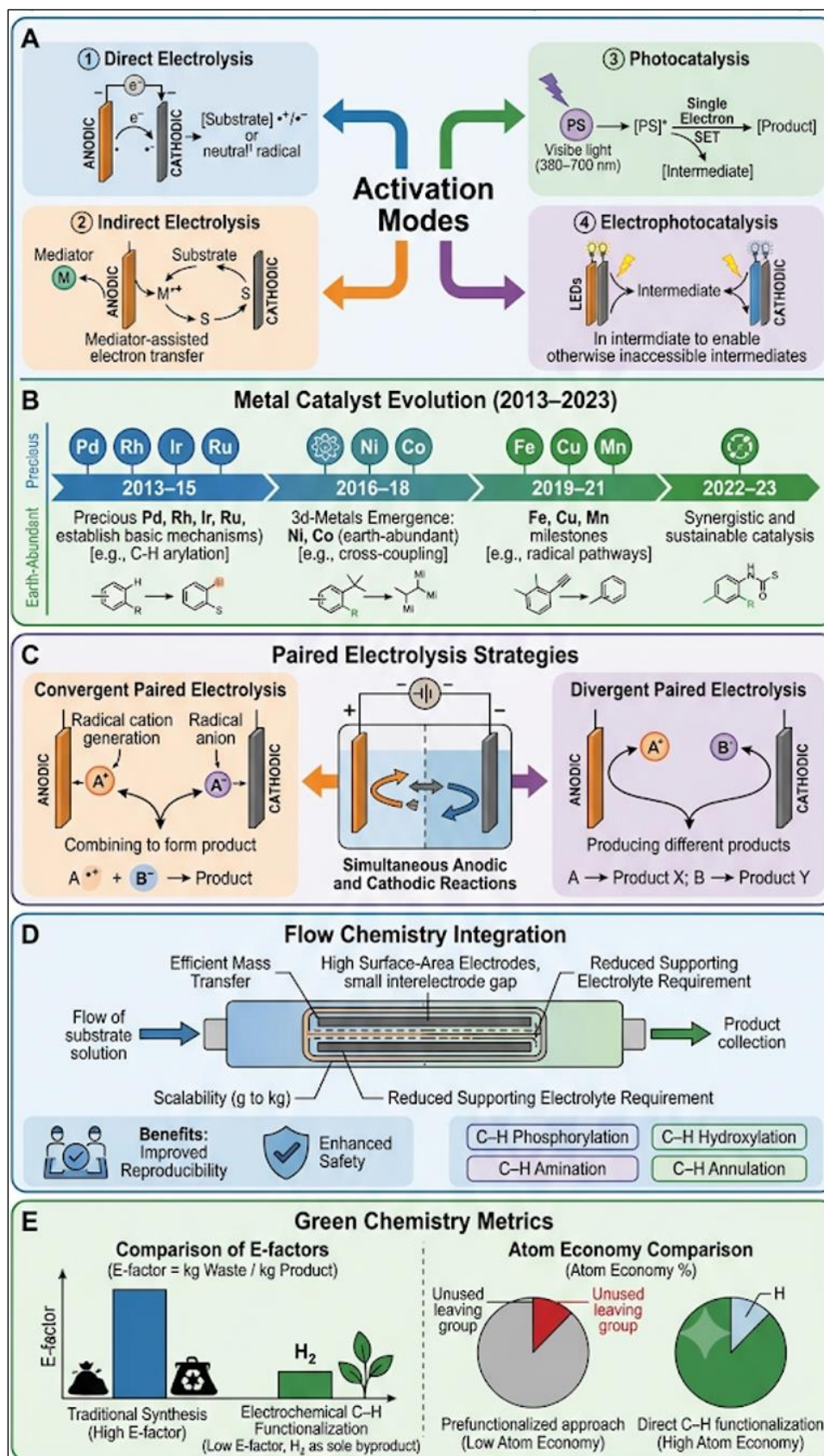


Figure 1: Schematic Overview of Electrochemical and Photocatalytic C–H Functionalization Strategies

Continuous-flow setups also facilitate scalability and improved safety, making them particularly attractive for industrial applications. Recent studies have showcased the potential of electrocatalysis under continuous-flow conditions for C–H functionalization via SET-enabled radical pathways. Notable examples include the synthesis of benzothiazoles via dehydrogenative intramolecular C–S bond formation, where successful omission of supporting electrolyte and gram-scale-up were achieved. Subsequent work demonstrated electrochemical manifolds for direct C–H phosphorylation,

hydroxylation, oxidation, and amination, with exceptional residence times as low as 22 seconds and broad scale-ups up to 204 grams.

Continuous-flow photocatalysis offers complementary advantages. The precise control over irradiation geometry, efficient light penetration, and the ability to conduct photochemical reactions at high concentrations make flow systems particularly attractive for photocatalytic C–H functionalization. The combination of photochemistry and electrochemistry in continuous-flow systems offers a powerful strategy for modern organic synthesis, enabling multistep transformations with enhanced performance, scalability, and mechanistic insight.

The integration of flow chemistry with electrochemical and photocatalytic C–H functionalization represents a significant advance toward industrial applicability. However, challenges remain, including the development of robust electrode materials for flow systems, the management of gas evolution (particularly hydrogen) in flow reactors, and the optimization of residence times for complex, multistep transformations.

2.4 Green Chemistry Metrics: Evaluating Sustainability

The assessment of sustainability is essential for evaluating the true environmental benefit of electrochemical and photocatalytic C–H functionalization. Two fundamental green chemistry metrics are particularly relevant: atom economy and the E-factor (environmental factor).

Atom economy, as defined by Trost (1991), measures the proportion of reactant atoms incorporated into the final product. C–H functionalization reactions are inherently atom-economical because they directly functionalize C–H bonds without requiring prefunctionalization. This contrasts with traditional approaches that introduce functional groups (such as halides or boronic acids) that ultimately become waste. The directness of C–H functionalization minimizes atom loss and reduces the generation of byproducts.

The E-factor, defined as the mass of waste per mass of product, provides a quantitative measure of waste generation. Recent studies have compared the sustainability of C–H functionalization strategies with classical approaches in the synthesis of active pharmaceutical ingredients (APIs). As reviewed by the Green Chemistry journal (2023), exemplary case studies analyzed holistically on the basis of waste production (E-factor) and environmental and safety hazard scores demonstrate that direct C–H functionalization technologies are not always the most effective strategy compared to classic approaches, highlighting the need for case-by-case sustainability assessment.

Electrochemical C–H functionalization offers particularly favorable E-factors because the electron serves as a traceless reagent, and the sole byproduct of oxidative transformations is molecular hydrogen. As noted by recent reviews, electrochemical synthesis is typically carried out under environmentally benign conditions and is increasingly used in organic reactions. The elimination of stoichiometric oxidants or reductants substantially reduces waste generation.

Photocatalytic approaches similarly benefit from the use of light as a traceless energy source. However, the need for photocatalysts—often based on precious metals such as ruthenium or iridium—can compromise sustainability. The shift toward earth-abundant metal photocatalysts (e.g., iron, copper, manganese) addresses this concern, though the performance of these catalysts often lags behind their precious metal counterparts.

A comprehensive sustainability assessment must consider additional factors beyond E-factor and atom economy, including solvent choice, energy consumption, catalyst toxicity, and the environmental impact of reagent production. As highlighted by recent studies, the integration of green chemistry metrics with environmental and safety hazard scores provides a more holistic evaluation of synthetic methodologies.

Table 1: Comparison of Catalytic Systems for Electrochemical and Photocatalytic C–H Functionalization

Catalyst Class	Representative Metals	Activation Mode	Key Advantages	Key Limitations	Typical Applications	Sustainability Rating
Precious Metals	Pd, Rh, Ir, Ru	Electrochemical or Photocatalytic	High activity; broad substrate scope; well-established mechanisms	High cost; limited availability; toxicity	Late-stage functionalization; C–C and C–N bond formation	Low
Nickel (Ni)	Ni(0), Ni(II)	Electrochemical (anodic/cathodic)	Earth-abundant; versatile	Air sensitivity; requires	C–C cross-coupling; C–heteroatom	High

			redox states; compatible with paired electrolysis	specialized ligands	bond formation	
Cobalt (Co)	Co(II), Co(III)	Electrochemical (anodic)	Powerful for C–H oxygenation and amination; mild conditions	Limited substrate scope for some transformations	C–H oxygenation; C–H amination; C–H annulation	High
Iron (Fe)	Fe(II), Fe(III)	Electrophotocatalytic (LMCT-HAT)	Most abundant; low cost; environmentally benign	Lower reactivity; limited mechanistic understanding	C(sp ³)–H functionalization; alkane activation	Very High
Copper (Cu)	Cu(I), Cu(II)	Photocatalytic or Electrochemical	Effective for radical pathways; inexpensive	Limited to specific substrate classes	C–H borylation; C–H arylation; C–H amination	High
Manganese (Mn)	Mn(I), Mn(II), Mn(III)	Photocatalytic	Emerging earth-abundant photocatalyst	Limited examples; early stage of development	C–H functionalization; alkene modification	High
Organocatalysts	None (organic dyes, etc.)	Photocatalytic (visible light)	Metal-free; tunable; low cost	Lower efficiency; limited substrate scope	C–H functionalization; C–C bond formation	Very High

Sustainability rating based on relative abundance, cost, toxicity, and waste generation, evaluated qualitatively.

3 FUTURE PERSPECTIVES

Several emerging trends and unresolved challenges promise to shape the future of electrochemical and photocatalytic C–H functionalization over the next decade.

Electrophotocatalysis represents one of the most exciting frontiers. The synergistic combination of light and electricity enables access to reactive intermediates and reaction pathways that are inaccessible to either technology alone. Recent advances in electrophotocatalytic C(sp³)–H functionalization enabled by decatungstate, cerium-based, and iron-based catalytic systems have demonstrated the power of this approach. Future developments will likely focus on expanding substrate scope, improving enantioselectivity, and developing robust, scalable electrophotocatalytic systems.

Artificial intelligence and machine learning are poised to accelerate reaction discovery and optimization. Predictive models that correlate catalyst structure, reaction conditions, and substrate properties with C–H functionalization outcomes could dramatically reduce the time and cost of method development. Machine learning approaches may also enable the identification of optimal conditions for challenging substrates and the prediction of site-selectivity in complex molecules.

Sustainable energy integration represents a long-term goal for electrochemical C–H functionalization. The direct coupling of electrochemical synthesis with renewable energy sources—such as solar or wind power—would further enhance the sustainability of these methodologies. Photoelectrochemical systems that directly harness solar energy for C–H functionalization are an emerging area of research.

Expansion of substrate scope remains a critical challenge. While significant progress has been made in functionalizing activated C–H bonds (benzylic, allylic, and α -heteroatom positions), the selective functionalization of unactivated aliphatic C–H bonds—particularly in complex molecules—remains difficult. The development of catalysts and

conditions that enable selective C–H functionalization at remote, unactivated positions would represent a transformative advance.

Enantioselective C–H functionalization via electrochemical and photocatalytic approaches is an emerging area with enormous potential. The combination of chiral catalysts with electrochemical or photochemical activation offers the possibility of asymmetric C–H functionalization under mild conditions. Recent advances in asymmetric electrochemical C–H cross-coupling and photoelectrochemical asymmetric catalysis suggest that this area will see significant growth.

Standardization and reproducibility remain challenges for the field. The lack of standardized protocols for electrochemical and photochemical reactions—particularly regarding electrode materials, cell design, and light sources—hampers reproducibility and cross-study comparison. The development of community-accepted standards and best practices would accelerate progress.

Scale-up and industrial application represent the ultimate translational goal. While significant progress has been made in scaling electrochemical and photocatalytic C–H functionalization to gram and kilogram scales, further development is needed to enable industrial-scale production. The integration of flow chemistry, advanced reactor design, and process optimization will be essential for translating these methodologies from academic discovery to industrial practice.

4 CONCLUSION

The past decade has witnessed remarkable progress in electrochemical and photocatalytic C–H functionalization, transforming these approaches from academic curiosities into powerful synthetic tools with genuine translational potential. The paradigm shift from precious metal catalysts to earth-abundant 3d metals—nickel, cobalt, iron, and copper—has substantially improved the sustainability of C–H functionalization while maintaining or even enhancing synthetic utility. The development of paired electrolysis strategies has enabled the simultaneous harnessing of anodic and cathodic processes, maximizing atom economy and energy efficiency. The integration of flow chemistry has addressed the scalability and reproducibility challenges that have historically limited the industrial application of electrochemical and photochemical methods.

The application of green chemistry metrics—particularly E-factor and atom economy—has provided quantitative evidence for the sustainability benefits of electrochemical and photocatalytic C–H functionalization. However, as highlighted by recent sustainability comparisons, direct C–H functionalization is not always the most effective strategy, and case-by-case assessment remains essential.

Despite the substantial progress achieved, significant challenges remain. The selective functionalization of unactivated aliphatic C–H bonds, particularly in complex molecules, remains a formidable challenge. The mechanistic understanding of 3d metal-catalyzed electrochemical C–H functionalization lags behind that of precious metal systems, limiting the rational design of improved catalysts. The scale-up of electrochemical and photocatalytic processes to industrial scale requires further development of reactor design, electrode materials, and process optimization.

Looking forward, the convergence of electrochemistry, photocatalysis, flow chemistry, and artificial intelligence offers unprecedented opportunities for the development of sustainable, selective, and scalable C–H functionalization methodologies. The integration of renewable energy sources and the development of electrophotocatalytic systems promise to further enhance the sustainability of these approaches. As the field continues to mature, electrochemical and photocatalytic C–H functionalization are poised to become indispensable tools for pharmaceutical, agrochemical, and fine chemical synthesis—enabling the direct, atom-economical, and sustainable construction of molecular complexity from the simplest of starting materials.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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