Trends and Challenges in Electrifying Technical Organic Synthesis

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DOI: 10.1002/cite.202400155

Organic electrosynthesis is a potential enabler for the energy and resource transition in the chemical industry as it offers a sustainable alternative to homogeneous or heterogeneous processes for producing fine and commodity chemicals. It utilizes electricity instead of hazardous reagents and, thus, also allows reducing the product's carbon footprint and waste production. It enables dynamic operation and safe operation due to galvanostatic process control. Electro-organic processes are not yet widespread. Engineering tools have not yet been tailored to electro-organic processes, and more quantitative and model-based insight is needed. Extensive adoption in the industry requires also efforts regarding electrode material performance and stability, scalable reactor design, and process digitization. Addressing these issues requires interdisciplinary collaboration, particularly between chemists and engineers, to accelerate process implementation with high efficiency and economic feasibility.

Keywords: Electrochemical engineering, Electrochemical synthesis processes, Electrode and cell concepts, Modeling and simulation

Received: November 26, 2024; *revised:* January 31, 2025; *accepted:* February 12, 2025

1 Introduction

With the climate crisis, humanity is facing one of the most significant threats in millennia. The primary challenge in the upcoming decades will be to limit the anthropogenic increase in global temperatures and mitigate the resulting natural disasters. One key contributor to the rising global temperature is the excessive reliance on finite fossil fuels. In addition to energy production, the chemical industry utilizes fossil fuels in the production of commodity and fine chemicals. The challenge in our generation is to replace fossil fuels with sustainable energy sources and transform the chemical industry for a more sustainable future. Electrifying energy-intensive industries and leveraging the Power-to-X principle to distribute or store intermittent energy from renewable sources is one promising approach [1, 2].

A notable guideline towards greener chemistry is given by Anastas and Warner [3]. Their "Twelve Principles of Green Chemistry" can be applied in many contexts in electrochemistry by substituting toxic and hazardous chemicals with inexpensive and, when generated from renewable sources, sustainable electricity. Replacing stoichiometric quantities of redox reagents reduces waste and increases atom efficiency of chemical reactions. Solvents and supporting electrolytes are the only source of waste originating from many actual electrochemical transformations, but even these components can be efficiently recycled, making these processes more cost effective [4, 5]. Precise control over a reaction is achieved by simply controlling the supply of electric current, which makes electrosynthesis an inherently safe production technique – a feature of particular interest on the technical scales. This approach generates the reactive intermediates in situ and avoids runaway reactions. In addition, electrochemistry uniquely enables certain reactivities

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that offer synthetic shortcuts and thus facilitate resource efficiency [6–8].

1.1 Why Is Organic Electrosynthesis a Key Technology for the Energy and Resource Transition?

In the chemical industry, most syntheses of commodity and fine chemicals are typically conducted under thermochemical conditions or with the aid of catalysts, either heterogeneous or homogeneous ones, to surmount high activation barriers. These conventional methods are often energy intensive and require hazardous reagents, posing significant environmental and safety concerns.

Organic electrosynthesis presents a promising alternative by leveraging electricity as the primary redox agent. This approach aligns with the growing demand for greener and more sustainable production pathways in chemical synthesis. Electricity, the most versatile and cost-effective redox agent available, can replace conventional, toxic, and environmentally harmful reducing and oxidizing reagents such as osmium(VIII) oxide, lead(IV) acetate, sodium hydride, and other metal redox agents. By controlling the applied current density or voltage, many electro-organic reactions can proceed at room temperature, further lowering their energy demands. Another significant advantage of organic electrosynthesis is its ability to enable carbon-neutral chemical synthesis when coupled with renewable energy sources like solar or wind power. This attribute makes it a crucial technology for the transition towards more sustainable industrial processes. Its importance becomes even greater as it potentially allows dynamic operation, i.e. using the intermittent electricity from renewables [9]. However, achieving this goal requires robust electro-transformations capable of operating across a broad range of current densities [10, 11].

Despite its many advantages, organic electrosynthesis has not yet achieved widespread use among synthetic chemists and chemical engineers. As a result, only a limited number of organic electrosynthesis processes have so far been implemented at the technical or industrial pilot-plant scale. Nonetheless, the integration of green processes via electrosynthesis presents a challenging, yet significant pathway to more sustainable and efficient methods. However, broader implementation faces significant challenges. Among these are: enabling electrodes with high stability, selectivity, and resistance to fouling or corrosion; the optimization of cell designs and operating procedures to achieve high productivity, scalability, and energy efficiency; and the setup, validation, and usage of computational tools and digitization for process design, control, and optimization.

Similar challenges were encountered by the classical chemical research and industry in the last century. The successful transfer of fundamental chemical research into applied engineering disciplines was key to realizing many processes at scale. A similar transition is now expected to take place in organic electrosynthesis. Initiatives such as the BMBF-funded Cluster4Future "Electrifying Technical Organic Electrosynthesis (ETOS)" [12] serve as an innovation network between chemistry and engineering by fostering interdisciplinary collaborations, addressing technical challenges, and laying the groundwork for scalable and sustainable electro-organic processes in the chemical industry.

The scope of this review focuses on the topics and technologies essential for advancing organic electrosynthesis beyond the reactions themselves and toward technical implementation. Emphasis is placed on the electrosynthesis of commodity and fine chemicals, which involve more complex transformations compared to the production of base chemicals like ethanol or ethylene via CO_2 reduction. It highlights the critical aspects necessary to transition electrosynthesis from laboratory research to technical implementation, as well as the challenges encountered during this process. This includes the selection of electrode materials, the design of electrolyzers, and the use of model-based methods for process analysis and optimization. While this review focuses on the fundamental aspects and challenges of organic electrosynthesis, it is important to note that key system parameters shift depending on the scale. Laboratory-scale studies primarily emphasize reaction selectivity and mechanistic insights whereas industrial applications require additional optimizations for energy efficiency, process stability, and scalability.

2 Electrolytes in Organic Electrosynthesis

An essential component of electrochemical cells is the electrolyte, which consists of a solvent or solvent mixture and a chemically inert salt – the supporting electrolyte – ensuring ionic conductivity in the reaction medium [13]. Although organic electrosynthesis is widely considered a sustainable method, its environmental impact strongly depends on the choice of the solvents and supporting electrolytes. Many electrosynthetic processes rely on protic or aprotic polar solvents such as acetonitrile, methanol, tetrahydrofuran, or *N*,*N*-dimethylformamide, often combined with excessive amounts of salt. This contradicts the principles of green chemistry and undermines the concept of "supporting electrolyte-free systems". Typical supporting electrolytes include alkylated ammonium salts and weakly coordinating anions.

Aqueous electrolytes offer a more sustainable alternative due to their high specific conductivity, which enables lower cell voltages and reduces energy losses [14]. They are typically hydroxides, strong acids, or halides [15]. However, their use in organic electrosynthesis remains limited. Organic solvents generally provide better solubility for nonpolar compounds; thus, they are more frequently used in organic electrosynthesis compared to aqueous electrolytes.

Furthermore, conventional organic supporting electrolytes are often toxic, volatile, and flammable, emphasizing the urgent need for safer and more environmentally friendly alternatives [16–18]. Moreover, organic supporting electrolytes are expensive and often difficult to recycle, which limits their economic viability in large-scale applications. However, few examples on strategies for improved separation and reuse exist, such as chemical modifications that facilitate the precipitation of conductive salts or the immobilization of these salts on polymer beads, allowing for efficient recovery via size-exclusion filtration [19]. Nowadays, there are commercially available alternatives like 2-methyltetrahydrofuran (2-MeTHF), propylene carbonate, and dihydrolevoglucosenone, which is a substitute for dimethylformamide [20–22].

For a long time, supporting electrolytes were considered merely ionic conductors, providing stable electrochemical conditions. However, recent studies have shown that electrolyte ions can actively influence the reaction rates and selectivity [23, 24]. For instance, Blanco et al. [25] demonstrated that the size and concentrations of alkali cations significantly impact the overpotential and selectivity in adipodinitrile synthesis. Smaller cations, such as Na^+ and K^+, exhibit weaker water solvation, leading to enhanced surface charge density and lower reaction overpotentials [25]. Similar effects can be found by quaternary ammonium salts. Beyond conductivity, supporting electrolytes can also act as reaction participants. Both solvents and conductive salts may undergo oxidation or reduction, functioning as electrophiles or nucleophiles. In some cases, conductive salt ions mediate electron transfer between the electrode and the substrate, effectively enhancing the reaction kinetics.

Ionic liquids have gained attention as potential replacements for conventional organic solvents. Their advantages include high ionic conductivity, nonvolatility, thermal stability, and recyclability [26–28]. These liquids typically consist of bulky organic cations paired with weakly coordinating anions, providing a broad electrochemical window and tunable solvent properties. A major advantage in organic electrosynthesis is their ability to dissolve nonpolar substrates without requiring additional supporting electrolytes. However, high viscosity, environmental concerns, and high production costs limit their broader adoption [29, 30].

Solid polymer electrolyte membranes offer a promising alternative to traditional liquid electrolytes by addressing some of the aforementioned drawbacks [31]. These ion-exchange membranes ensure high ionic conductivity while minimizing electrode distance and ohmic resistance, improving energy efficiency. Furthermore, they enable the use of gaseous and nonpolar solvents that are otherwise non-conductive, significantly expanding the range of applicable reactants [32]. However, membrane contamination and electrode durability remain major challenges, restricting their widespread use in organic electrosynthesis [33–36]. A commonly used polymer electrolyte membrane is Nafion® [37], a proton-conducting material widely applied in hydrogen fuel cells and water electrolyzers due to its high stability. Nafion® is compatible with mildly to strongly acidic conditions, making it well suited for various electrosynthetic applications. In organic electrosynthesis, polymer electrolyte membranes offer the advantage of simplified purification, as they eliminate the need for supporting electrolyte removal and recycling. For example, Nafion® has been successfully used in the electrochemical reduction of cyclic ketones to alcohols [36]. In contrast, alkaline exchange membranes (AEM) have seen limited application in organic electrosynthesis, primarily due to slow improvements in performance and durability [38, 39]. However, AEM electrolysis has recently gained attention, particularly for biomass-derived molecules that require alkaline conditions for conversion into value-added products [40]. Additionally, alkaline environments enable the use of base-metal catalysts, promoting cost-effective and sustainable electrosynthetic processes.

3 Electrode Materials and Concepts

Electron transfer occurs at the electrode surface, and it is directly linked with the transport of reactant molecules from the bulk electrolyte through the diffusion layer to the electrode surface, or to product transport from the electrode into the bulk. These internal cell processes are essential for direct electrolysis, where the reactant undergoes electron transfer at the electrode, and for mediated electrolysis, where a redox-active species does the electron transfer at the electrode and subsequently acts as a homogeneous redox agent with the reactant in solution. Electrical conductivity is an important requirement for an electrode material, but also the material strongly influences the electrochemical reaction due to differences in stability, mechanical durability, and reactivity. The electrode can either serve as an inert conductor, solely facilitating electron transfer, or it functions as an electrocatalyst, making it a critical parameter in process optimization. Noteworthy, the nature of the supporting electrolyte can substantially alter the characteristics of the electrodes and directly affect the electrolysis outcome [41–43]. Consequently, electrodes and electrolytes have to be considered as a functional couple [44].

Platinum anodes offer desirable physical and electrochemical properties; materials such as non-precious metals and carbon-based materials such as graphite and glassy carbon are an attractive and frequently applied low-cost alternative for technical applications [45]. Carbon electrodes consist of a wide variety of sp^2 and sp^3 carbon allotropes and their mixtures, resulting in significant performance variations in productivity, selectivity, and durability of electrochemical processes [46]. Therefore, it is important to consider different carbon materials during the optimization of organic electrosynthesis if the required productivity or selectivity is not achieved. Besides platinum- and carbon-based materials, metals such as silver, nickel, mercury, copper, lead, or leaded bronzes are commonly used [47].

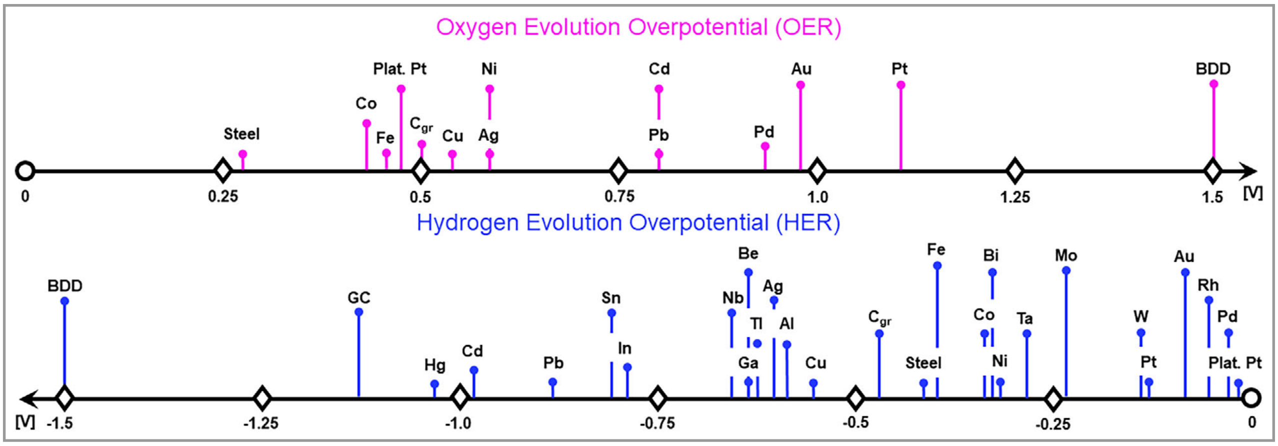

Figure 1. Electrode materials categorized by their potentials for the HER and the OER. HER overpotentials are measured at 1 mA cm^{-2} in 0.5–1 M H_2SO_4 at 25 °C, whereas OER overpotentials are measured at 1 mA cm^{-2} in 1 M KOH at 25 °C. Adapted from [45].

Some of the electrode materials are also used in the form of foams to achieve higher electrochemical active surface areas and thus conversion rates, e.g., nickel foam, leaded bronzes, graphite felt, or reticulated vitreous carbon (RVC) [48].

The downside of carbon as electrode material is that a passivation layer may form on it after a certain time of operation, also known as electrode fouling. Fouling may alter the electrochemical properties of the electrode and reduce its productivity and selectivity [49]. Mechanical cleaning can be performed on flat, non-porous electrodes to restore their original properties. Conversely, metal electrodes can be subject to corrosion. This latter issue is particularly severe in reductive electro-organic syntheses [50] and poses significant challenges for sensitive synthesis processes with demand for highly pure or metal-free products, such as the synthesis of pharmaceutically relevant compounds [51]. Anodic corrosion can alter the electrode surface through the formation of porous metal salt films or oxide layers [52]. Even noble metal electrodes like Pt are susceptible to dissolution. Ranninger et al. [53] reported Pt ion dissolution during direct Kolbe electrolysis and its dependence on water impurities and the addition of a base. In direct electrolysis, substrate conversion is often accompanied by additional challenges, such as over-oxidation of the substance, passivation through film deposition, and unwanted competing reactions like the hydrogen or oxygen evolution reaction (HER/OER), which depend on the electrode material and the solvent used. For a wide range of electrode materials, Fig. 1 gives an overview of the potentials below which the HER and above which the OER may occur.

A commercially available high-performance material that suppresses the HER and OER due to very high overpotentials is boron-doped diamond (BDD). It is a p-type semiconductor. Boron atoms can induce splitting in the diamond energy band and create impurity states near the Fermi level, thereby enhancing electron conductivity [54]. With its high stability, BDD can be used in a broad electrochemical window for various solvents. Carbon-based materials can be used as anode or cathode material [55].

When no suitable counter-reaction is available, sacrificial electrodes – often anode materials, such as magnesium or nickel – can be employed [44]. These electrodes dissolve through oxidation, releasing metal ions into the electrolyte. An even electric field is essential, as surface heterogeneities lead to nucleation of metal salts that passivate the electrode [56]. However, the dissolved ions can then stabilize intermediates or products, thereby promoting the electrosynthesis process. Since sacrificial electrodes are consumed during operation and large amounts of electricity are required for their production, sustainability is not given with most of these electrodes. Modern developments in electrodes for electro-organic synthesis use alloying concepts [57] and foam-type electrodes, which turned out to be stable under cathodic polarization [58, 59].

Electro-organic reductions are much less widespread than electro-oxidations, also due to competitive hydrogen evolution; consequently, there is a clear need for cathode materials that can operate in protic media at strong negative potentials without the predominant evolution of hydrogen. Furthermore, in non-aqueous electrosynthesis, there are so far no gas diffusion electrodes (GDE) that can deliver gas directly to the electrode to overcome the limitations of mass transport due to low gas solubility. The challenge in GDE for organic electrolytes is that electrolyte, electrode, and gas are hydrophobic; the resulting capillary pressure is so small that the electrodes are easily wetted by the electrolyte and no gas-liquid interface can be established in the pores. Lazouski et al. [60] reported on GDE with lithium-coated stainless-steel cloths that can realize a stable liquid-gas interface in tetrahydrofuran for ammonia synthesis; however, no report in organic electrosynthesis exists so far.

4 Electrochemical Cells and Electrolyzers

The selection of a suitable cell design for a given electrosynthesis reaction is a critical factor, as the design influences the productivity, energy efficiency, and scalability of the entire electrochemical process. Presently, the majority of new organic electrosynthesis processes are conducted within batch cells [61–63]. So far, relatively few employ flow reactors for continuous flow synthesis on a gram-to-kilogram scale [31, 36, 64, 65]. They are typically operated in single-pass mode, i.e., without recirculation of the exit stream to the inlet stream; recirculation is used for incremental conversions per pass, as it allows for a quite homogeneous concentration distribution in the cell per pass. In scaled-up processes, single-pass continuous flow is rarely used; instead, continuous-flow electrolyzers in recirculation mode are more common. While several electrochemical processes have reached pilot or commercial scale, detailed documentation on these systems and chemical processes remains limited [66–70].

Cell designs should enable easy handling and support the electrochemical reaction process as well as post-reaction maintenance, including easy cleaning and minimal reliance on consumable parts [70–72]. High mass transfer to the electrodes, supported by efficient mixing and effective removal of produced reaction gases, is essential to prevent side reactions caused by reactant depletion and local fluctuations in potential. Additionally, cell materials must be designed to withstand the temperatures specific to the targeted electrosynthesis process while also minimizing leakage risks and ensuring material compatibility to prevent corrosion. Although many processes operate near room temperature, larger electrolyzers can experience temperature increases due to ohmic resistance.

Identifying the optimal cell design for scaling up organic electrosyntheses remains challenging, as achieving high productivity and selectivity requires a thorough understanding and optimization of the process conditions. To date, the transition from batch cells in laboratories to other technical cell formats is often carried out intuitively, frequently without the application of physical-chemical principles and methods. As a result, process conditions are often hard to reproduce and need to be re-optimized from scratch to achieve technically relevant efficiencies. There is a lack of empirical concepts as well as physically grounded methods and models to describe processes and conditions in electrolyzers.

Electro-organic reactions often require careful selection of the electrode and cell design and the cell operating conditions. Due to the interfacial nature of electrochemical reactions and the high sensitivity of the reaction to local concentrations, mass transport to and from the electrode as well as the hydrodynamic properties can heavily influence the reaction rates and outcomes, with each reactor type offering distinct advantages and limitations. It is important to keep in mind that only for some electro-organic reactions, the complete reaction occurs at the electrode; frequently, a reactive intermediate is generated by the electron transfer and follow-up reaction steps take place in the electrolyte. In mediated processes, only a mediator is oxidized or reduced, which subsequently reacts in the electrolyte with the substrate [73]. Cell-specific properties include: mixing efficiency, suitability for handling various reaction mixtures – whether homogeneous, partially heterogeneous, or biphasic – and a uniform electric field distribution; undivided or divided operation capabilities in flow cells; use of sacrificial anodes; and temperature control options.

Several cell designs have demonstrated versatility for a wide range of applications in organic electrosynthesis [67]. These designs include: (1) beaker-type cells, using plate-in-tank or rod-in-tank setups, (2) parallel-plate reactors, in single-cell or stack configurations, (3) capillary gap in stacked-disk flow setups, and (4) continuous stirred-tank reactors (CSTR), such as rotating-cylinder electrode cells.

Fig. 2 illustrates a beaker cell-type setup used in batch electrolysis mode, which replicates smaller batch electrolysis cells [74, 75]. Scaling these reactors typically involves increasing the total electrode surface area by expanding the size of the cylindrical vessel, either with large rectangular electrodes or with multiple cylindrical rods in the reservoir [76–79].

While this design offers simplicity and ease of use, it also presents certain limitations, including non-optimal mixing, inhomogeneous electric fields with cylindrical-rod electrodes, and an unfavorable electrode surface area-to-volume ratio. For instance, as the cylindrical cell volume increases, the electrode surface area-to-volume ratio declines, limiting scalability. Nevertheless, its straightforward design, assembly, and operation make it easy to use for batch reactions on decagram-to-kilogram scales.

Parallel-plate flow reactors offer high electrode area-to-electrolyte volume ratios. They are used for various reactions and facilitate scale-up as the area-to-volume ratio and conditions are very similar across scales [66, 71, 72]. Their design resembles electrolyzers used in alkaline and proton-exchange membrane (PEM) water electrolysis. These reactors operate in single-pass or recirculating-flow modes. In single-pass mode, a long channel design and slow flow rates are employed to extend the reactant's residence time within the cell, thereby optimizing conversion; this holds benefits for unstable electro-organic products that need immediate isolation post electrolysis [80].

In contrast, enhanced mixing compared to batch and single-pass systems can be achieved by using parallel-plate flow reactors with high flow rates combined with operation in recirculating-flow mode [81, 82]. In cases where gas evolution occurs, such as hydrogen evolution from cathodic proton reduction, low flow rates in single-pass mode may lead to a fluctuating residence time distribution and gas buildup in the electrolysis cell, reducing the active electrode

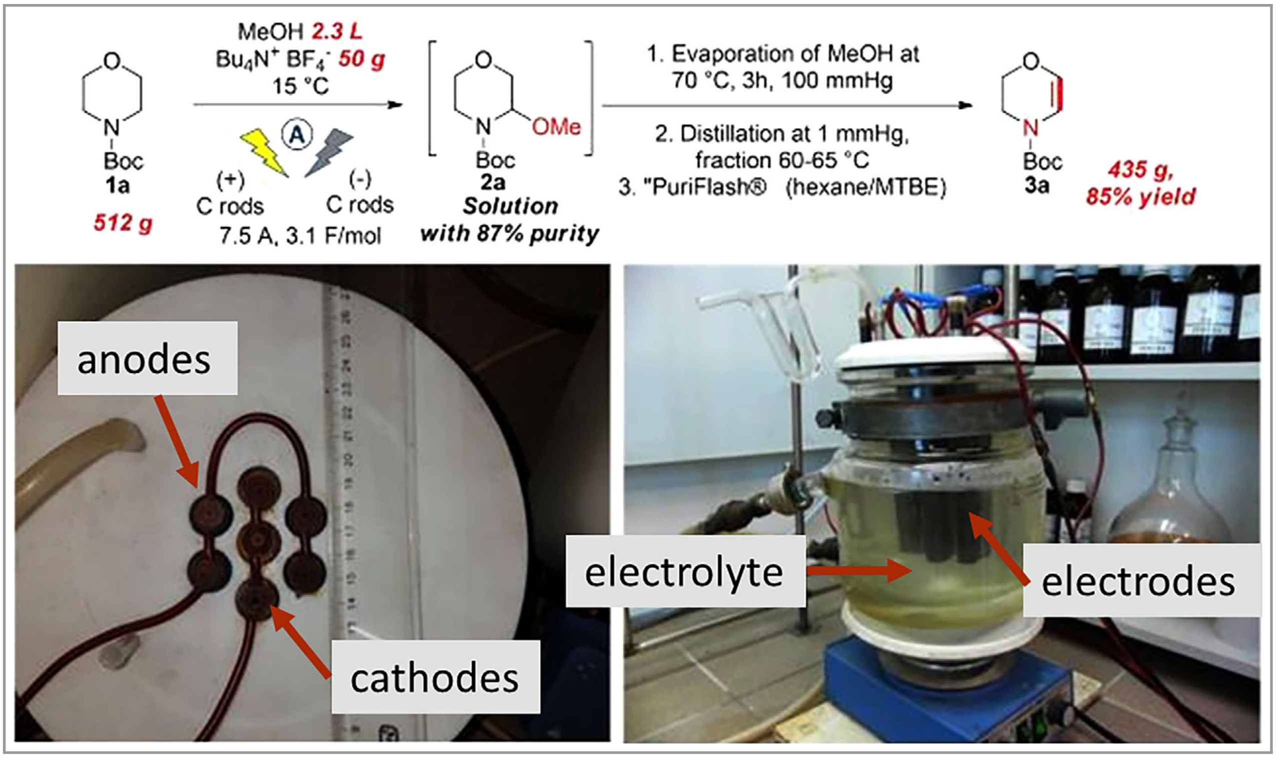

Figure 2. Beaker cell-type reactor setup used in batch electrolysis mode for the synthesis of cyclic enecarbamates [74] (reprinted with permission from Wiley-VCH).

surface area and increasing cell resistance. To counter these effects, higher flow velocities are used, albeit with lower single-pass conversion rates, making the recirculating mode necessary to achieve complete conversion. Fig. 3 depicts a parallel-plate electrolysis cell operating in recirculating-flow mode in divided mode; this cell can also be used without separating membrane, i.e., as undivided cell [82].

Capillary gap or stacked-disk electrolysis cells employ disk-shaped electrodes with narrow interelectrode spacing (2–3 mm), which feature reduced cell resistance and enable the use of lower electrolyte concentrations [83]. These undivided electrolysis cells feature bipolar electrodes, where only the end electrodes of the stack are directly connected to the power source. As the same electrode material is used for both the anode and cathode, the cells can be operated with alternating polarity. Capillary gap reactors have been employed in large-scale processes, such as the oxidation of toluene derivatives, with BASF demonstrating their industrial utility [84, 85].

CSTR have been extensively studied in organic electrosynthesis, including rod-in-tank and rotating-cylinder electrode configurations. These reactors can operate in single-pass, cascade, or recirculating-flow modes to achieve full conversion [78]. While CSTR resemble batch reactors, their ability to operate under steady-state conditions offers significant advantages. However, it is important to note that electrochemical CSTR do not inherently provide sufficient mixing to completely eliminate mass transport limitations and gas evolution issues. However, since mass transport often determines the limiting current in organic electrochemical processes, high-mixing reactors can operate at higher current densities, thereby increasing the productivity [86]. Similar to other stirred-tank reactors, electrochemical CSTR face challenges such as an unfavorable electrode surface area-to-volume ratio, uneven potential distribution across the electrode surface, and the formation of local hot spots. The presence of electrode structures within the reactor further complicates homogeneous mixing. Nevertheless, their major advantage over CSTR lies in their continuous operation and the ability to optimize the residence time distribution, which is crucial for maintaining reaction stability and improving process efficiency.

Scaling up CSTR equipped with electrodes presents a hands-on approach, allowing for the transfer of expertise from traditional chemical process development to electrosynthesis. Typically, a central high-speed mixer is surrounded by electrode rods [78, 87]. Fig. 4 illustrates a CSTR cascade used for the methoxylation of cyclic carbamate **1**. The residence time in each CSTR was set to 22 min, resulting in a total flow-through time of 88 min across all four reactors. The first two CSTR operated at 6 V, while CSTR 3 and CSTR 4 were set to 4 and 3.5 V, respectively. This voltage adjustment was done to minimize overoxidation of product **2** to the dimethoxylated side product **3**, which occurs

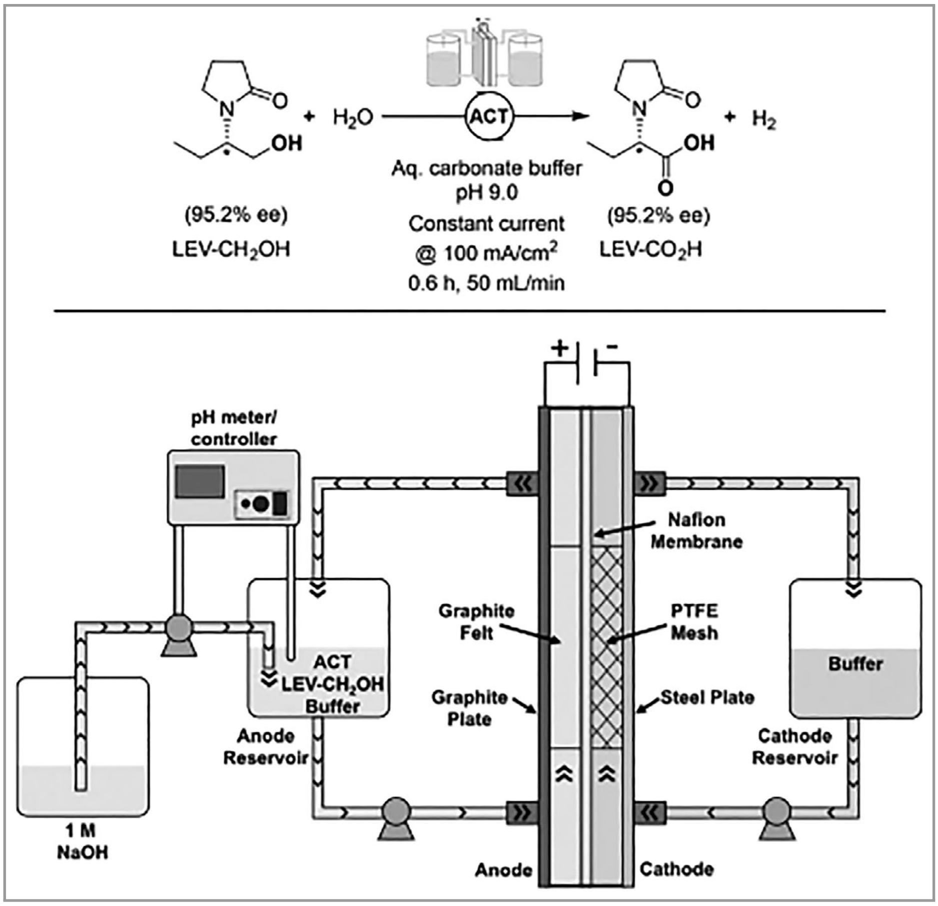

Figure 3. Schematic illustration of the divided flow cell with graphite felt anode, stainless-steel plate cathode, and Nafion® N324 membrane for the synthesis of levetiracetam. A pH meter/controller maintains the pH of the anode reservoir solution [82] (reprinted with permission from the American Chemical Society).

at these high potentials when mass transport limitations restrict the availability of the starting material (**1**) (Fig. 4a). Fig. 4b presents the conversion of starting material **1** as a function of the flow-through time of the cascade. Under steady-state conditions, conversion is expected to remain linear over time and depends on the applied potential. The use of multiple CSTR instead of a single reactor with an extended residence time was chosen to allow for better control over intermediate formation and to sequentially adjust the reaction conditions, particularly the applied voltage, to optimize selectivity.

Unlike in chemical reactors, mixing is not ideal due to the electrodes acting as barriers. Therefore, homogeneous process conditions are difficult to realize in the cell vessel. CSTR for electrochemical applications are not widely available commercially across scales, from laboratory to manufacturing.

Overall, while significant advancements have been made in the development and application of electrochemical cells for organic electrosynthesis, several challenges persist. The standardization of reactor setups remains a key issue, as does the reproducibility, transferability, and comparability of results across different scales and systems. Furthermore, the development of robust methods for identifying optimal process conditions within cells and the establishment of comprehensive cell models at multiple levels are crucial for enhancing process understanding. Addressing these open questions will not only improve the scalability and efficiency of electrochemical processes but will also accelerate the integration of academic innovations into technical applications.

5 Digitization in Organic Electrosynthesis Processes

5.1 DoE-Based Screening and Optimization of Electrochemical Synthesis

Significant efforts have been made to establish design of experiments (DoE) approaches in organic electrosynthesis as a toolkit usable for every chemist. The scope of practicable transformations in electrosynthesis rapidly progresses [7, 88]. The goal is to streamline the experimental protocols to improve reproducibility, enabling other researchers to easily adopt them, as several parameters are often not well reported [46, 89]. However, optimization remains challenging as attention has to be paid to many parameters of the system studied. The already large parameter space of conventional organic synthesis is further expanded by

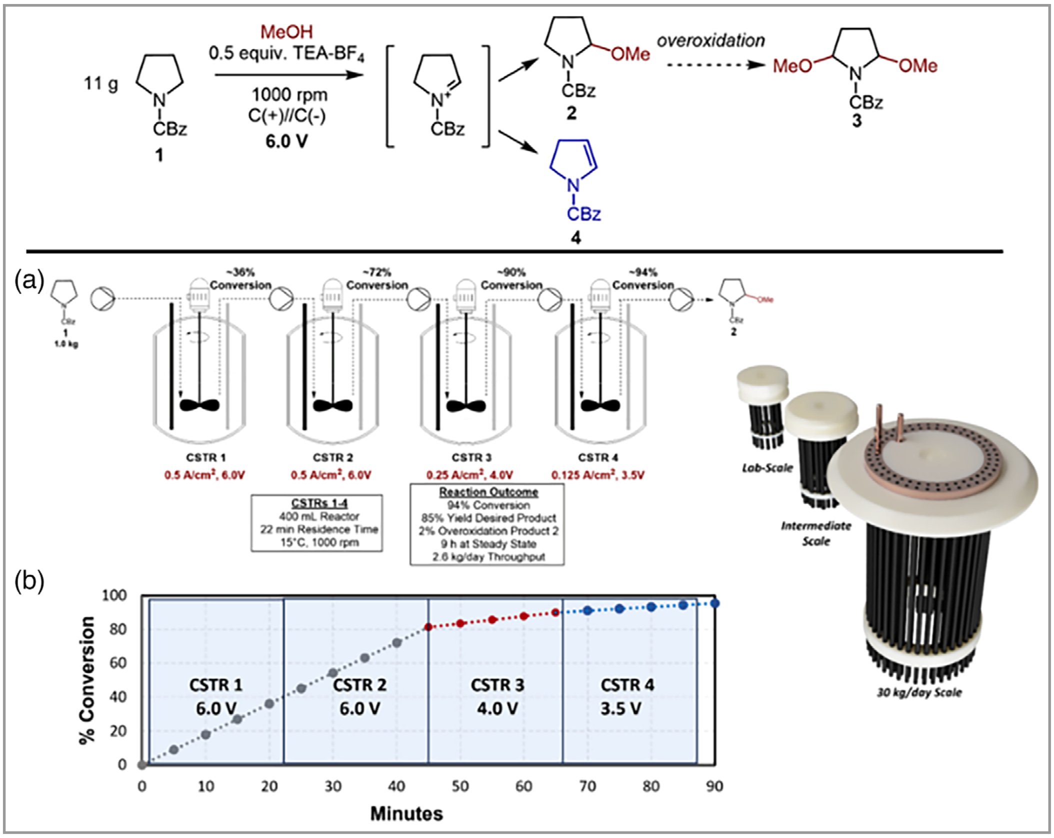

Figure 4. (a) Design of the four-stage CSTR cascade. The cell potential was varied across stages to balance throughput and selectivity for the electrolysis. (b) Batch data from different cell potentials supported the different applied cell potentials [78].

adding specific electrolysis parameters, such as the applied charge or current density. Additionally, factors typically considered minor – such as mixing rate and interelectrode distance – can have unexpectedly significant impacts in electrosynthesis due to its interfacial nature [90–92].

This complexity necessitates systematic optimization strategies capable of handling these mostly nonlinear systems. When the one-variable-at-a-time approach is insufficient, a viable step forward in process optimization is using DoE techniques that incorporate regression modeling, thus providing detailed information about the studied system [93–98]. When considering multiple substrate combinations, incorporating experimental and theoretical molecular parameters can be highly effective. However, this approach necessitates the use of advanced techniques such as predictive modeling methods, including multivariate linear regression, principal component analysis, and machine learning [99–102]. These methods balance experimental data to produce robust models; so, researchers must value negative results equally to obtain high-quality data for both positive and negative experiments [103, 104]. Examples for DoE-based process optimization are electrochemical lignin degradation [105, 106], iodine-mediated processes for heterocycle synthesis [107, 108], the ex-cell generation of a platform oxidizer [109, 110], and the subsequent lignin conversion to save energy and reagents [111, 112].

Automated setups push the boundaries of rapid data generation, favoring flow over batch operation mode in this context. This requires the integration of lab appliances and has generated quicker results [113]. By such achievements, the operation of a divided cell can be switched to an undivided setup, since the contact time to the electrodes was minimized [114, 115]. It is anticipated that automated screening for operational electrolysis conditions and self-optimization of flow electrolyzers can be realized within the next couple of years and may evolve to a standard tool in electro-organic synthesis. This will require almost full automation of a robotic flow synthesis platform for organic compounds, as accomplished by Coley et al. [116]. Such an approach includes several artificial neural networks and a Monte Carlo tree search algorithm for synthesis planning [116].

5.2 Model-Based Analysis of Organic Electrosynthesis Processes

Models give highly valuable insights into processes, local compositions and states, and into limitations that occur during operation at the electrode, in the electrolyte and at the cell or system level.

Molecular simulations focusing on the electrosynthesis of large organic molecules are rare. The number of molecular dynamics simulations published so far is in the single-digit range. Most papers on the molecular dynamics of organic molecules in electrochemistry focus on the synthesis of smaller molecules ($\leq C_2$) [117], the stability of organic solvents in batteries [118, 119], or on organic metal corrosion inhibitors [120, 121]. The existing publications on electro-organic synthesis focus on the interaction of the electrolyte with the reactant and electrode. Hollóczki et al. [122] showed that the solvent composition can strongly influence the reactant adsorption at the electrode surface. Blanco et al. [25] and Hymel et al. [123] found that the accumulation of ions in the electrical double layer helps to stabilize radical ion intermediates, thereby promoting the target reaction. The smaller the alkali cations, the more the radical anions were stabilized; this effect was shown to increase aqueous adiponitrile production while simultaneously increasing the unwanted HER side reaction [25]. The group of Koper [124] came to similar conclusions for CO_2 reduction, using a combination of simulation and experiment: The size and coordination of the supporting anions and cations strongly influenced the CO_2 reduction reaction efficiencies and selectivities [25, 124]. Molecular modeling may in future contribute to advancing organic electrosynthesis also by improving the understanding of electrode fouling, steric effects, or the influence of impurities, and of how to mitigate associated negative effects.

Compared to molecular dynamics, the density functional theory (DFT) is more frequently used in electro-organic systems – so far, though, with a focus on interpreting the light absorbance of chromophores [125, 126] and organic framework catalysis [127, 128]. When specifically investigating organic electrosyntheses, DFT methods are mostly used to calculate the Gibbs free energies of reactants and intermediate species, to explain either reaction paths or reaction selectivities. Wong et al. [129] predicted the catalyzed and uncatalyzed mechanism for the electroreduction of NH_2-phenyl and found the overpotential-determining reaction step [129]. Richers et al. [130], Xiao et al. [131], Osˇeka et al. [132], and Novaes et al. [133] used experiments and DFT simulations to develop new synthesis routes for benzoxazines, cyclic carbonates, aziridines, and methylated amines, respectively. DFT was used in these cases to explain the underlying reaction mechanisms. By studying the electrostatic interaction and adsorption, Haya et al. [134] and Liu et al. [135] were able to gain further insights on their reaction systems. Haya et al. [134] showed that the cause for the regioselectivity of carbamate methoxylation is steric hindrance due to dipole orientation close to the electrode surface. Liu et al. [135] found an explanation for the transition from oxygen evolution towards Kolbe electrolysis on platinum anodes with increasing electrode potential. Blanco et al. [25] and Hymel et al. [123] integrated DFT calculations into their molecular dynamics routine in order to account for electrostatic interactions, solvent effects, and adsorption. It is notable that adsorption and electrode effects are so far often neglected for mechanistic investigations; electrode-substrate interactions are ignored although such interactions can have a significant influence on the dynamic behavior of the electrodes [136]. This opens the opportunity for further research.

Molecular simulations, machine learning, and artificial intelligence have only been rather recently employed to better (understand) organic electrosynthesis processes. In contrast, models on the continuum and system scale have been around for over 40 years [137]. Yet, also continuum modeling of electro-organic processes is still a niche, as evidenced by the rather low number of publications (two-digit range), the high diversity of models including the lack of a "standard" model, and the low diversity of the studied processes. The high diversity in the way how processes are modeled, and to which degree, is illustrated in Tab. 1. Here, we categorize continuum-scale models into kinetic models [137, 138], which focus on reaction and local transport effects, fluid dynamic models [139, 140], which target the convective flow and concentration distribution in cells, and process models [141, 142], which search to analyze and improve the full process including balance-of-plant components. Although the majority of electro-organic syntheses were conducted on the laboratory scale, the majority of models focus on undivided flow cells rather than the lab-typical H-cells. The modeled systems use two of the operating modi introduced above: Kinetic investigations mostly consider a recycle loop with differential conversion during every loop, while fluid dynamic and system models consider single-pass operation.

The processes at the electrode surface are typically modeled with lumped kinetic models with only few physical or chemical details. Only a few models consider adsorption in their models. Yu et al. [143] use adsorption only for the reaction kinetics of H_2 evolution as a side reaction. No single model assumes adsorbed organic species, so far. On the same note, none of the reviewed models was applied to model a detailed reaction mechanism with intermediate species. This is in stark contrast to electrolysis or fuel cell models, which frequently assume intermediates and adsorbates [144–146].

The reason for the so far only lumped models was the focus on a different aim compared to fuel cells and electrolysis: In fuel cells and electrolysis, high electrochemical performance with low overpotentials is in the focus, whereas in organic electrosynthesis, it is reactant conversion and evolution of the product concentration, and especially reproduction of concentration measurements from high-performance liquid chromatography (HPLC), gas chromatography (GC), or UV spectrometry. Thus, different lumped kinetic descriptions were applied. The most prominent approaches for the reaction rates are Butler-Volmer kinetics [137, 143, 147–151], which contains concentration

Table 1. Overview on continuum models for organic electrosynthesis: The models differ in their degree of details regarding description of the cell configuration, mass transfer, and reaction kinetics.

		Cell configuration					Mass transfer				Reaction kinetics				
Model type	Authors	Half cell	Full cell	Flow cell	Single-pass operation	Divided cell	Convection	Mass transfer coefficient	Detailed diffusion	Multiphase	Constant rate	Mass transfer-limited rate	Mass action law	Butler-Volmer	Other
Kinetic models	Alkire et al. [137]	X		X			X	X						X	
	Yu et al. [147]		X	X			X	X						X	
	Yu et al. [143]		X	X			X	X						X	
	Scott et al. [152]		X	X				X					X		
	Wendt et al. [148]	X		X				X						X	
	Ferrigno et al. [156]		X	X	X		X		X		X				
	Michel et al. [157]	X		X		X	X		X						X
	Attour et al. [158]	X						X							X
	Kristal et al. [149]		X	X										X	
	Wei et al. [154]	X		X			X					X			
	Suwanvaipattana et al. [153]		X					X		X			X		
	Morrison et al. [150]		X						X					X	
	Weng et al. [151]		X						X					X	
	Kunkel et al. [155]	X		X				X				X			
	Delparish et al. [138]	X		X	X			X		X					X
	Hodgson et al. [159]	X							X					X	
Fluid dynamic models	Santos et al. [139]		X	X	X		X		X			X			
	Folgueiras-Amador et al. [140]	X		X	X		X			X	X				
	Cao et al. [164]		X	X	X		X		X					X	
System models	Boor et al. [142]				X						X				
	Shin et al. [141]				X				X					X	
	Na et al. [165]				X						X				

and potential dependency, the classical chemical rate law, which contains only concentration dependency [152, 153], mass transfer-limited reaction rates [139, 154, 155], and simple current-dependent rates, i.e. Faraday's law [140, 156]. Not all models therefore require a coupling of the reaction rate with the current or potential. For this reason, the calculation of rates for side reaction is likely to mismatch the experimental system in most cases; hence, no Faraday (or Coulombic) efficiencies are calculated. The considered side reactions are mostly the H_2 or O_2 evolution.

Mass transfer processes and their impact on organic electrosynthesis are another central aspect of continuum models. Similar to the reaction kinetics, there are various implementations of mass transfer (Tab. 1). Likely for the sake of computation time and reduced complexity, most models are discretized in only one spatial dimension. Since mostly flow cells are considered, convection is implemented in many models, and flow is discretized in the direction of flow [137, 139, 140, 143, 147, 154, 156, 157]. In these cases, the diffusion of reactants from the convection-driven flow to the electrode and back was implemented using analytical expressions of local reactions, which contain mass transfer coefficients [137, 143, 147, 153]. Models without any spatial discretization of the cells also used integral mass transfer expressions [138, 152, 155, 158]. These simple zero-dimensional models can already provide

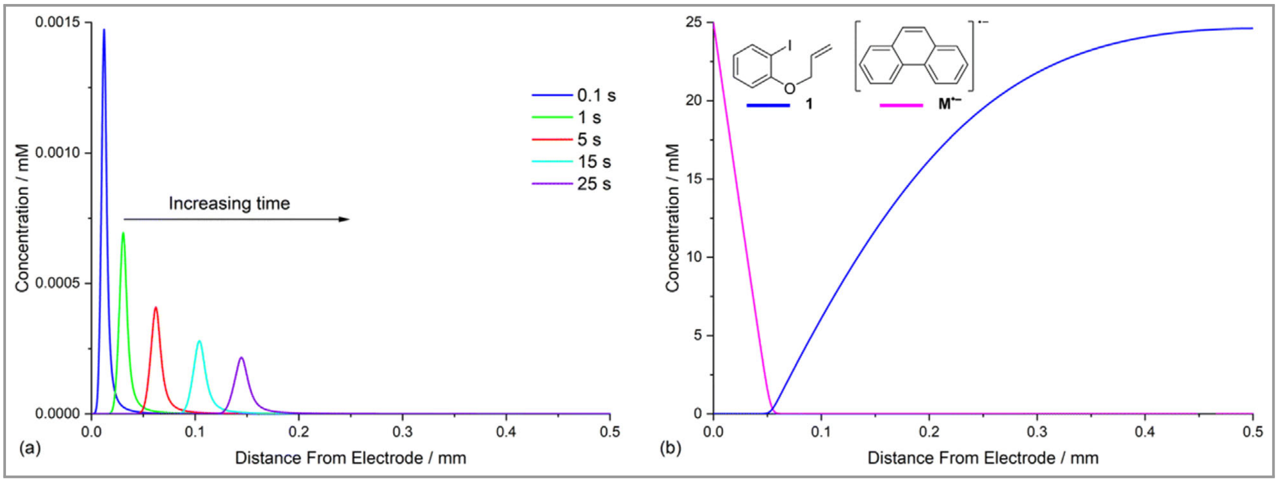

Figure 5. Demonstration of the reaction layer detachment by showing (a) the concentration profile of a radical intermediate formed by a mediated reduction and cyclization for different electrolysis times and (b) the reactant and reduced mediator concentrations after 5 s [159] (reprinted with permission from the Royal Society of Chemistry).

significant insights into the modeled system. Kunkel et al. [155] developed a model for the electroreduction of vanillin. This model was then applied to improve the operation conditions, yielding a two-step procedure that reduced the energy consumption by ~26 % [155]. If dispersion effects or bulk reactions play a major role in the modeled system, diffusion is explicitly included in the model [156, 157]. Hodgson et al. [159] used a combined approach of fluid simulation and one-dimensional modeling to investigate the unintuitive selectivity of mediators with reduction potentials below the reactant reduction potential. They found that a complex interplay of mass transfer and reaction kinetics yields a detachment of the mediation reaction layer from the electrode, causing the mediator transport to dictate the selectivity (Fig. 5) [159].

While many processes involve gas production, e.g., H_2 evolution, only few models so far contain gas and liquid phases and thus are multiphase models. Suwanvaipattana et al. [153] modeled a liquid-liquid reaction system for adiponitrile production by considering mass transfer between a dispersed drop phase and an aqueous phase. Multiphase flow is more prominent in actual computational fluid dynamic simulations; they target to understand the influence of the second liquid or the gas phase on mass transfer and mixing effects. Folgueiras-Amador et al. [140], for example, used such simulations to show the positive effect of non-ionic surfactants on bubble formation; the surfactant reduced the gas-blocked surface area and increased mixing from bubbles, and thus the current. Mass transfer by migration was not explicitly implemented in any of the identified models, so far. It is typically assumed that, due to high concentrations of supporting electrolyte, migration effects are negligible.

It can be concluded that continuum-scale models of organic electrosynthesis are present, but with a limited depth regarding electrochemical performance and Faradaic/Coulombic efficiency. As both aspects will become more relevant in the future, further investigations are required to gain a more detailed understanding of the kinetics, adsorption, multiphase systems, and migration involved. The coupling of molecular simulations with continuum-scale simulations can help to gain a deeper understanding of these processes, both phenomenologically as well as descriptively [160].

Along with model-based approaches, experimental electroanalytical methods are important tools for investigating reaction mechanisms or operating conditions. While a detailed discussion of electroanalytic methods would exceed the scope of this review, it needs to be mentioned that experimental methods are of high importance, as simulations require experimental data for parameterization and validation [161–163].

6 Conclusion and Perspective

Organic electrosynthesis is presently experiencing a renaissance and is evolving as a powerful tool for electrifying organic synthesis processes and making them more sustainable. The research efforts in this field have led to a few examples of technical-scale electrosynthesis of highly value-added products in the range of 1–20 tons per year [51, 109, 166–169], but the real potential for the chemical industry is far from being realized. There are many reactions that can be electrified, but so far there have been few transfers to applications. Most of these processes lead to fine chemicals for the agrochemical or pharmaceutical sectors. This aspect is very important as these industries have the financial resources to quickly implement new technologies such as electrosynthesis. Development and scale-up of the usually rather small processes was done within companies, frequently without significant involvement of reaction or process engineering divisions or engineers from academia. In contrast to organic chemists, chemical engineers in academia are just starting to work in this field, developing the required engineering and model tools and training engineers on electrosynthesis processes.

With the engineering and modeling tools currently being developed, processes can be scaled up faster and can be optimized towards higher energy efficiency, productivity, and selectivity, thus making them more cost-competitive compared to classical organic synthesis routes. The tools and established knowhow and lessons-learned will also benefit other fields as, e.g., the electrosynthesis of products with a lower added value, such as commodities or base chemicals. The usage of electrosynthesis in the high-price segments is expected to increase strongly in the next few years; it has the potential to become a standard production method, whereas it will gain ground more slowly in base and commodity chemicals due to the larger-scale production and thus higher investment costs with lower product price. However, electrosynthesis will advance the field due to lower electricity costs and a better CO_2 balance in the long term.

Whereas past reports about electrochemical setups in academic literature often lack reproducibility due to complex and custom-made equipment, the growing availability of standardized cells and electrolyzers accelerate the development of new concepts and synthetic methods. Since organic electrosynthesis is a small but rapidly growing field with ongoing research efforts, the increasing availability of commercial cells, electrodes, electrolyzers, and synthetic as well as analytical methods will continuously evolve. In this regard, the implementation of high-throughput experimentation or the development of automated syntheses in continuous flow systems are worthwhile research areas, which are promising to impact modern organic synthesis.

Further, modeling tools, which are already standard for chemical processes, are expected to play a transformative role in the knowledge-driven design and optimization of electrochemical processes. From molecular processes at electrode surfaces to complete systems, these models will aid in risk assessment, cost-benefit analysis, and the realization of economically viable and sustainable chemical processes.

An important topic not covered in detail in this review but critical for advancing research in the field is the education of skilled professionals. Currently, many undergraduate theory and practical courses in both chemistry and engineering lack organic electrosynthesis topics, providing students with limited exposure to electrochemical methodologies. This lack of familiarity reduces awareness of the field among future chemists and engineers. To attract the next generation of researchers, it is vital to highlight the benefits of electrochemical processes, setups, and principles for process development and operation.

Despite these challenges, the remarkable versatility of electro-organic processes in generating diverse high-value products highlights their importance in advancing sustainability within the chemical industry. With continued interdisciplinary collaboration between chemists and engineers, innovations in organic electrosynthesis will not only extend its influence across key sectors but it will also establish itself as a cornerstone of the global transition toward greener, more sustainable industrial processes.

Acknowledgements

The authors acknowledge the financial support by the BMBF – Federal Ministry for Education and Research of Germany as part of the Cluster4Future ETOS (Electrifying Technical Organic Electrosynthesis) and the projects DigiEtos (03ZU1205NA), Outreach4ETOS (03ZU1205TB), Training4ETOS (03ZU1205QC), E-Scope 2.0 (03ZU1205LD), and EAGLE (03ZU1205JA).

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Abbreviations

CSTR	continuous stirred-tank reactor
DoE	design of experiments
DFT	density functional theory
GDE	gas diffusion electrode
HER	hydrogen evolution reaction
OER	oxygen evolution reaction

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