

Scaling-up electro-organic synthesis: challenges and approaches

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The electrosynthetic preparation of organic compounds experiences a strongly increasing attention and evolves into a future methodology. The scalability of these synthetic approaches generated several electrolyzer concepts, including monopolar, bipolar, and rotating electrode setups. With such strategies, scaling into the hectogram and kilogram range is readily viable.

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Current Opinion in Chemical Engineering 2026, 51:101238

This review comes from a themed issue on **Towards Industrial Application of Electrochemical Processes**

Edited by **Elias Klemm, Tom Breugelmans** and **Luis F. Leon-Fernandez**

Available online xxxx

<https://doi.org/10.1016/j.coche.2026.101238>

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Introduction

The chemical industry is faced with increased scrutiny concerning the environmental impact and sustainability of both established and newly developed processes. In response to this, the replacement of energy-intensive processes by new technologies has become a focus over the past decade. Simultaneously, the electrical energy for these new processes should also originate from

renewable and sustainable sources. Gratifyingly, the costs for electricity generated by wind power and photovoltaics have massively decreased over the past decade as well, making their use ever more prevalent. Consequently, electrifying chemical processes appears both economically and ecologically desirable [1,2].

This revolution in the chemical industry will significantly rely on the development of chemical processes using electricity. Electro-organic synthesis employs electrical current to perform oxidations and reductions in a reagent waste-free, safe, and sustainable fashion [3]. While researchers have come to increasingly embrace this technology, most studies are performed on the scale of a few mmol. These approaches are perfectly suited for fast and efficient screening and are also facilitated by multiple commercially available setups. However, with increased industrial interest in these technologies, as evidenced by both industry surveys [4–6] and the formation of research consortia on the topic [7], larger-scale applications of electro-organic synthesis are of huge interest, albeit these studies are still rare enough that they can be reviewed comprehensively [8,9]. However, scaling electro-organic reactions comes with a unique combination of challenges, which go beyond those addressed when screening for reaction conditions at a small scale. Specifically, the complex interplay of mass transfer, charge transfer, and chemical reactivity needs to be balanced in order to achieve a successful electrochemical process. One matter of paramount importance is operational simplicity; hence, they are mostly performed using a galvanostatic (constant current) setup, but with this simple electrical periphery, a variety of electrolyzer geometries are in use. Most of the setups used at scale are undivided cells, since the membranes used in divided cells introduce operational complexity, are expensive, and increase the resistance of the cell [10,11]. Furthermore, a simple and elegant downstream processing is also a prerequisite for the translation onto a larger scale [12].

Batch-type reactors

The majority of electrosynthetic investigations is carried out in simple batch-type cells with a single anode and cathode, such as the Electrasyn 2.0, IKA screening system, or most beaker cells [13]. A reaction in such a setup can be scaled up by increasing the size of the

vessel and the electrodes. An operationally simple example of this is the scale-up of the electrochemical C-H chlorination developed by Liu [14]. In this reaction, the chlorination of secondary C-H moieties is performed using a *N*-hydroxymaleimide mediator, and graphite felt as electrodes (Figure 1a). The reaction was initially optimized on a 1 mmol scale in a beaker cell, resulting in yields up to 71%. Subsequently, the process was scaled to the hectogram range, whereby 64% of the yield was observed, and the kilogram scale, where 51% of the product could be isolated. These experiments were conducted using larger electrodes in a larger beaker cell, with their decreasing yields owing to the fact that no further optimization took place for the larger setups.

Larger commercial cells are also available, such as the SynLectro series by Merck, which has been used by Waldvogel for the electrochemical decagram production of, for example, phosphinimines [15] (Figure 1a). In this reaction, a phosphine and a sulfonamide are coupled electrochemically in an iodide-mediated process. The reaction was initially optimized in a 25 mL batch-type cell with two 12 cm² electrodes, reaching a yield of up to 90% with a current density of 30 mA/cm². These conditions were subsequently transferred to a 200 mL batch-type cell with two 48 cm² electrodes. Using the previously optimized conditions in the larger cell resulted in 79% of the isolated product. However, this approach is quickly limited by the available size of the electrodes employed and will generally display low ratios of electrode area to electrolyte volume.

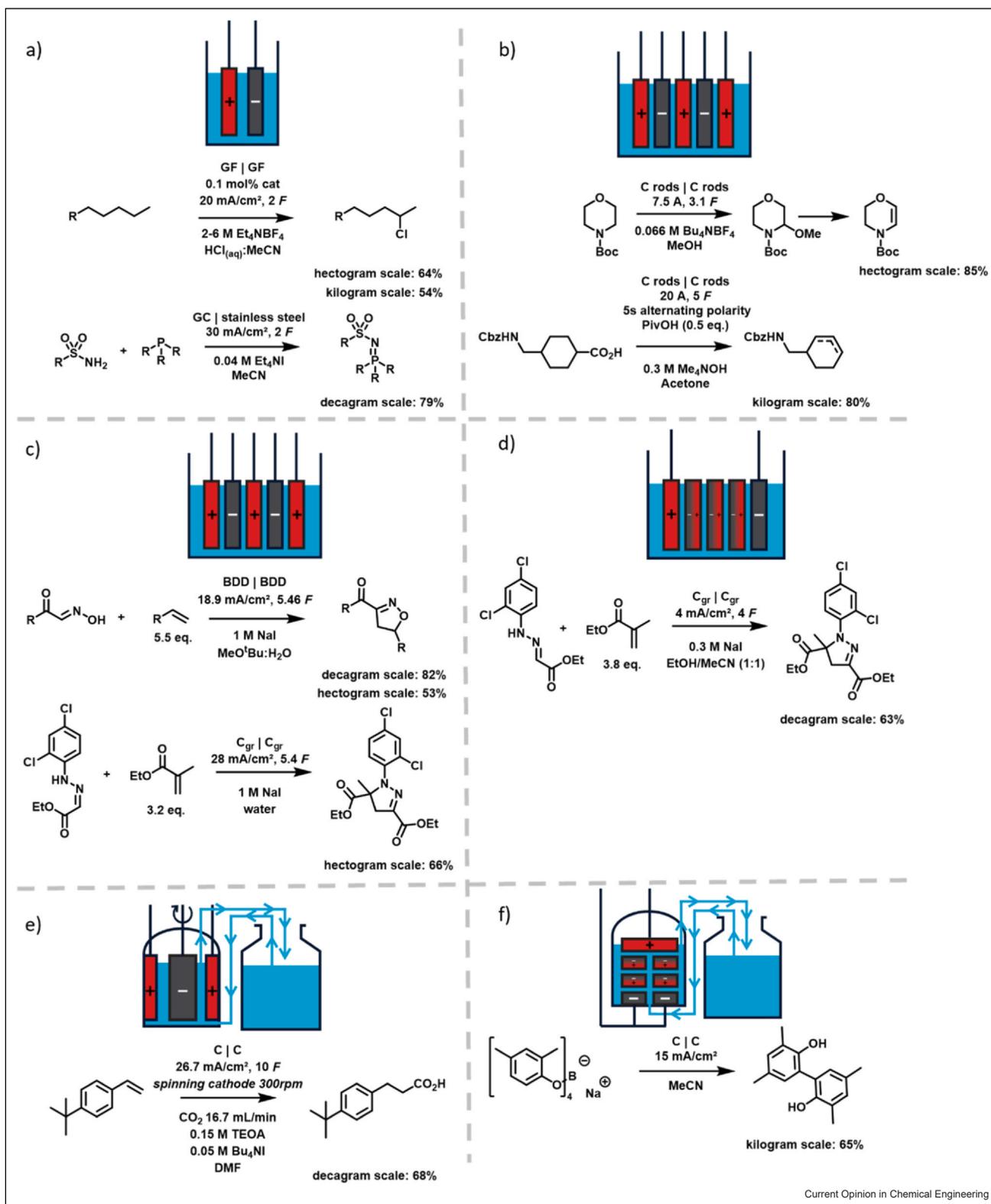
Consequently, more electrodes in the shape of either rods or plates are employed, leading to setups with a higher electrode surface to electrolyte volume ratio. Electrode arrays comprised of both rods and plates are employed. However, such arrays often provide a less defined electrical field. The electrode arrays can be electrically connected in two general ways: either by contacting each of the electrodes individually (monopolar, Figure 1b, c) so that it serves as either an anode or a cathode or using them in a bipolar arrangement (Figure 1d), in which all electrodes, except those at the very ends of the arrays, serve as anode and cathode. A monopolar arrangement of rods (Figure 1b) has been employed by the researchers at Enamine [16] in the Shono-type oxidation of saturated nitrogen heterocycles, which was followed by an elimination reaction to result in enecarbamates. Their cell consisted of a beaker cell with an array of 7 graphite rods, and their procedure was shown to be applicable to a variety of substrates with yields of 71–85% on a hectogram scale, with the best example even being converted on 500 g scale and retaining 85% yield. Larger rod arrays have also been used, such as in the work of Baran: [17] Here, an electrochemical decarboxylative olefination was performed on a kilogram scale using an 8 L batch-type reactor

equipped with 96 graphite rods connected in a monopolar fashion. At this large scale, the reaction yielded 80% of product, which is comparable to the results on smaller scales, despite the high electrode surface necessary for lowering the current density due to instrumental limitations.

However, an inherent limitation in the use of rod arrays is the inhomogeneous electrical field due to the diverse interelectrode gaps in the system. This is less pronounced in plate arrays (Figure 1c), which the group of Waldvogel has used in the electrosynthesis of Isoxazolines [18]. The condition screening was initially performed in 5 mL beaker cells with 100 mg of starting material. When the reaction was scaled to 25 g scale, a significant decrease in yield (from 82% to 40%) was initially observed, prompting further optimization during which both mono- and bipolar arrangements were investigated, resulting in a protocol with identical yields to those achieved in the screening cell. Upon further scaling up the reaction to the hectogram scale, a decrease in yield to 53% was reported, which is attributed to an inhomogeneous substrate concentration in the largest reactor.

The same group also showed the electrosynthesis of the herbicide safener mefenpyr-diethyl from hydrazone species [19], with methods for the conversion of both the *E* and *Z* isomers being developed and scaled up. For the *Z* isomer, a solvent-free method was developed that allowed for the gram-scale conversion of the hydrazone in a 5 mL screening cell with 93% yield. These conditions were subsequently scaled up to result in the hectogram scale generation of mefenpyr diethyl using a 1.5 L beaker cell equipped with a monopolar stack of 6 graphite electrodes, resulting in 66% yield. The pathway involving the *E* hydrazone was also investigated, with the method optimized on a small scale, reaching 73% yield, which was subsequently scaled to decagram scale in a 1.5 L beaker cell with a stack of six graphite electrodes connected in a bipolar fashion (Figure 1d). This method resulted in a 63% yield of the herbicide safener. In most of these cases, an increase in scale goes along with a decrease in yield, which is often attributed to mass transport limitations [20]. One approach to mitigating this drawback is the use of cells with rotating electrodes (Figure 1e). These setups have been used by Kappe and Cantillo for a number of studies [21–24], including the hydrocarboxylation of activated alkenes [25]. In the former case, an optimization in a 3 mL batch-type reactor allowed for the generation of hydrocinnamic acid from 4-*tert*-butyl styrene in 70% yield. The scale-up reaction was then performed in a 50 mL cell with one static electrode along the outer wall of the vessel and a spinning, tubular electrode in the center. By using the spinning electrode as the cathode, a method could be developed that gave the desired acid in 77% isolated

Figure 1



Batch-type reactors used in electro-organic scale-up studies and example reactions. **(a)** plate-in-tank reactor, **(b)** rod array reactor, **(c)** monopolar plate array reactor, **(d)** bipolar plate array reactor, **(e)** rotating electrode reactor with reservoir, **(f)** capillary gap reactor with reservoir.

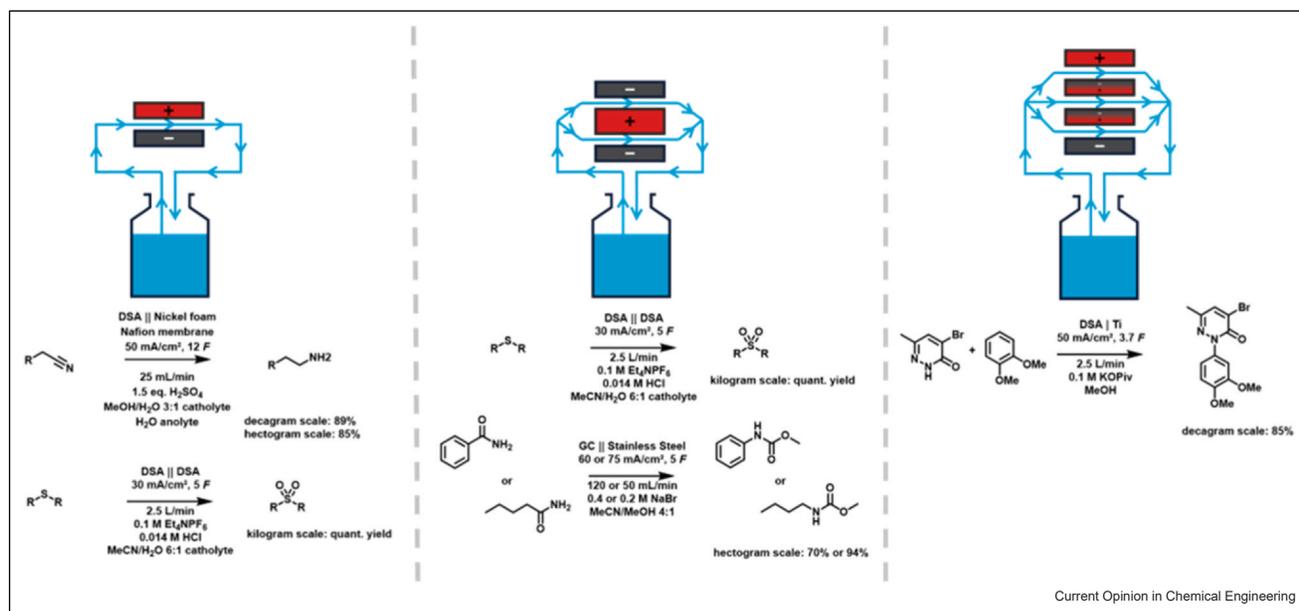
yield. However, the increased mass transport in this setup also has the drawback of increased electron shuttling between the anode and cathode, which necessitated an increase in the applied charge from $3.5 F$ in the traditional batch-type cell to $10 F$ in the cell with the rotating electrode. Due to these cells being more challenging to fabricate, the further scale-up was performed in a semi-batch fashion, with a 0.5 L reservoir connected to the 50 mL cell. This setup allowed for the generation of hydrocinnamic acid on 20 g scale with 68% yield. This semi-batch approach has also been used with simpler batch-type reactors [26,27]. An alternative way to overcome mass transport limitations is the use of capillary gap electrolyzers (Figure 1f). In these setups, the solution is pressed through narrow gaps in a bipolar electrode stack. This cell type has been used, for example, in the kilogram scale electro-synthesis of biphenols by Waldvogel and coworkers in 65% yield [28].

Flow cells

Performing electrochemical reactions in flow tends to significantly increase the electrode surface to electrolyte volume ratio. However, it further increases the parameter space, since the influence of, for example, flowrate and residence time also need to be considered. The most used cell type in such studies is the plate and frame reactor, which allows for a high modularity and thus a facile exchange of electrode materials (Figure 2a). A number of such systems have been commercialized [29–31]. While a single pass operation is feasible for these electrolyzers [32], it also leads to a convolution of

residence time, current density, and starting material conversion. In order to avoid this, most systems are used in a recirculatory mode. An example of how simple, yet powerful, these reactors can be is the electrochemical hydrogenation of nitrile compounds, developed by the Waldvogel group [33,34]. In this study, the reaction was first investigated in a divided batch-type cell, where 90% yield was achieved, before being transferred first to a flow cell with 12 cm^2 of active cathode surface (86% yield), after which it was scaled to a larger flow cell with 48 cm^2 of active electrode surface. In this latest cell, the reaction could be performed at a decagram scale with 89% yield at a high current density of 50 mA/cm^2 and concentration of 0.7 M. In a follow-up study, the reaction was even performed in decagram scale for the anti-oxidant spermine with 85% yield. This same pathway from optimization to scale-up is also outlined in a study on the electrochemical generation of sulfones from thioethers by Bottecchia and coworkers [35]: After finding initial, successful reaction conditions, they optimized their reaction in batch-type screening cells, before moving to flow cells. They first performed studies in a cell with 10 cm^2 cathode surface, followed by a setup with 100 cm^2 cathode surface and finally one with 400 cm^2 of cathode surface. However, even the largest cell could not match the desired productivity target. As such, the authors used an array of three cathodes and two anodes to reach a total of 1600 cm^2 of cathode surface, allowing for the electrochemical generation of the desired sulfones on a kilogram scale. Such a sandwich-type flow cell (Figure 2b) has also been employed by the

Figure 2



Flow reactors used in electro-organic scale-up studies and example reactions. (a) Plate and frame reactor, (b) monopolar sandwich reactor, (c) bipolar sandwich reactor.

Waldvogel group in scaling the electrochemical Hofmann rearrangement to hectogram scale [36], albeit only using two cathodes and one anode. In this study, the optimization was performed in one reaction compartment of the final electrolysis cell with 81 cm² of cathode surface for both benzamide and valeramide.

However, bipolar electrode arrays can also be employed in flow cells (Figure 2c), as shown in the industrial production of aromatic acetals by BASF [1,37] and also illustrated in the investigation into the electrochemical synthesis of an *N*-arylpiperidazinone by Scarborough and coworkers [38]. While Scarborough's study also followed the optimization workflow established previously, they also facilitate scalability by already ensuring a turbulent reagent flow at small scale [39]. Furthermore, the largest cell in this study employs an electrode array consisting of 5 bipolar graphite plates, with a total electrode surface of 1296 cm², allowing for the decagram-scale synthesis of the desired product in up to 85% yield. Finally, it should be highlighted that the employed setup was investigated in long-term experiments, which also revealed a, albeit slow, corrosion of the graphite electrodes over time.

Downstream processing

Besides the electrolysis itself, efficient workup procedures are also of paramount importance and should directly be considered when scaling up electro-organic reactions [12]. While on smaller scales, the purification via chromatography is often used to isolate the product, this cannot be applied to large-scale operations due to the tremendous costs. As outlined before, a less soluble product in the electrolyte mixture has many benefits, and the precipitated product can be removed by filtration. Ideally, this enables the direct reuse of the remaining electrolyte [15]. Although this down-streaming concept is commonly employed for electrolysis of inorganics [40], it is not so often used in electro-organic synthesis [41]. In most cases, a combination of extraction, filtration, crystallization, or distillation is still employed. The isolation of the product should also require as few steps as possible, with direct crystallizations from the reaction medium, as demonstrated for iminophosphoranes [24,42], by Lehnher and coworkers. Aside from the isolation of the product, the fate of the supporting electrolyte should be considered. While it is treated as problematic waste in many studies [43–45], a simple aqueous extraction often allows for the recovery and subsequent reuse of these species [15,19,36], thus further increasing the atom economy of the underlying processes.

Conclusion and perspective

Electro-organic synthesis is recognized as a common synthetic method, and more operations on a larger scale will happen within the next years. Several approaches

for electrolyzers on a larger scale are established, and some are commercially available. The path is paved well. Albeit the reporting of additional metrics, such as the specific energy consumption of the developed systems is often lacking and should be included in future studies. In past decades, most attention was given to electrode development [46–48], whereas in the future, the engineering of the electrolyte systems is required: being greener, enabling phase separation with the product, or being completely evaporated in downstream processing [49,50]. Combining the electrochemical conversion with novel concepts such as solvation science will propel electrosynthesis far beyond current capability. This is considered to be a fruitful scientific playground for academic research and industrial development.

Data Availability

No data were used for the research described in the article.

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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