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Electrochemical Dehydration in Flow: Fatty Acid Nitriles From Their Carboxamides

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ABSTRACT

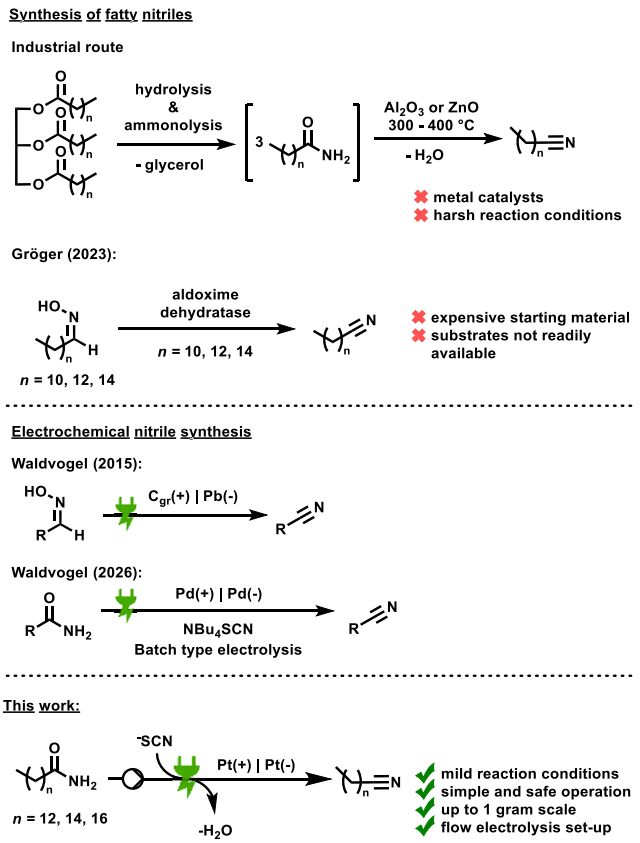
We established a sustainable and scalable electrochemical dehydration of highly lipophilic amides to their nitriles in recirculating flow electrolysis conditions, expanding on our previously published work on the electrochemical dehydration of carboxamides in batch-type cells. With our electrochemical flow setup, long-chain aliphatic carboxamides (C14–C18) and related substrates were converted efficiently, affording lipophilic nitriles in yields up to 88% (2.5 mmol) and 86% (4.4 mmol). The method proved robust across different flow reactor designs, including small cells for rapid screening and optimization, larger systems for scale-up, and adapted setups capable of handling suspensions, further highlighting the versatility of the flow approach. Tetrabutylammonium thiocyanate functions as both redox mediator and supporting electrolyte, while hexafluoroisopropanol is crucial to suppress side reactions. The reaction proceeds under mild conditions, avoiding harsh catalysts and temperatures. Importantly, solvent recovery and reuse under recirculating flow operation maintained comparable yields, reinforcing the sustainability and practicality of this flow-based electrochemical process for producing valuable fatty nitriles.

1 | Introduction

Fatty nitriles are considered valuable platform molecules and intermediates for the pharmaceutical, polymer, and surfactant industries [1]. Moreover, the reduction of fatty nitriles is the most common route for the production of lipophilic amines, which are widely used in a range of household products and form the basis for the industrial manufacture of, e.g., lubricants, surfactants, and corrosion inhibitors [2, 3]. Industrially, fatty nitriles are produced from biomass feedstocks, such as vegetable oils (triglycerides) or fats, making them attractive in the frame of renewable resources [4]. Their conventional synthesis involves the conversion of fat or oil to fatty acids by hydrolysis, followed by an ammonolysis-dehydration strategy [5]. In this sequence, the fatty acid first reacts with ammonia to form the easily accessible fatty amide (ammonolysis), followed by dehydration of this intermediate to yield the fatty nitrile (Scheme 1) [6]. However, this process has several major drawbacks. The overall reaction is typically carried out at elevated temperatures (300–400 °C), and the dehydration reaction requires the addition of a metal oxide catalyst, such as alumina or zinc oxide. Moreover, the process calls for the continuous removal of water from the reaction to minimize undesirable side reactions leading to impurities in the final lipophilic nitrile, providing another obstacle in reaction engineering [7, 8].

To circumvent the harsh reaction conditions needed for this dehydration reaction, alternative routes to fatty nitriles have been previously investigated. A known process to obtain nitriles is the dehydration of oximes, which can be conducted either *via* enzymatic [9–11] or electrochemical [12–17] methods (Scheme 1). Beyond dehydration strategies, electrochemical approaches have also enabled the synthesis of nitriles from alternative and readily

Enrico Lunghi and Annemijn M. van Koten contributed equally to this study.



SCHEME 1 | Conventional synthesis approaches of fatty nitriles and the electrochemical synthesis of nitriles.

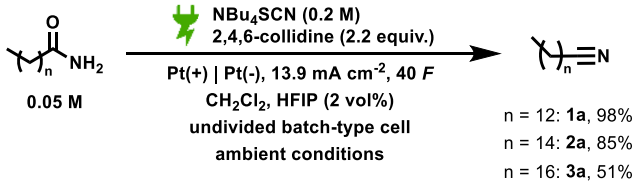
available precursors, like primary alcohols. In particular, amines, aldehydes, and alcohols have been successfully converted into the corresponding nitriles through oxidative electrochemical transformations [13–17]. For example, Gröger et al. [2] developed the biocatalyzed dehydration of long-chain fatty aldoximes to their fatty nitriles with high conversion at high substrate concentrations. Although operating under ambient conditions, these methods rely on hydroxylamine and the corresponding long-chain fatty aldehydes as precursors, which are less readily accessible than the fatty acids and fatty amides conventionally employed as intermediates in fatty nitrile production. In contrast, carboxamides, which are also used as intermediates in the industrial synthesis of fatty nitriles, are readily accessible and bench-stable substrates. A direct synthesis of fatty nitriles from their carboxamides at ambient temperature and pressure, without the need for metal catalysts, therefore represents an attractive sustainable alternative [18]. In this context, organic electrosynthesis has experienced a renaissance in recent years due to its ability to provide sustainable, energy-efficient, and reagent-saving processes at ambient temperature and pressure [19–25].

Electrochemical methods allow reactions to proceed under relatively mild and safe reaction conditions [26]. Electrical current, preferably from renewable sources [27, 28], can be employed as a controllable and inherently clean redox reagent [29, 30], thereby substantially reducing the generation of hazardous waste compared to stoichiometric amounts of classical oxidizers and reducing agents. These characteristics have made electrosynthesis a powerful and attractive alternative in the toolbox of modern organic chemistry [31, 32]. Following these principles, our group recently published the electrochemical dehydration of aliphatic and aromatic carboxamides to their corresponding nitriles, providing an operationally simple, safe route to nitrile products under environmentally benign conditions in good yields (Scheme 1) [33]. While this study established the general feasibility of electrochemical amide dehydration, its scope was primarily limited to aromatic and short-chain aliphatic substrates, and scale-up was demonstrated only in batch-type electrolysis cells. The translation of this methodology to long-chain fatty carboxamides is not trivial because of their distinct physicochemical properties, particularly their poor solubility and the challenges associated with handling highly lipophilic substrates during electrolysis. Furthermore, implementation under continuous flow conditions has not previously been demonstrated. More generally, flow electrolysis has emerged as an effective strategy for the scale-up and process intensification of electro-organic transformations, supported by recent developments in scalable and modular electrochemical flow-cell technologies [34–37].

In this work, this methodology is extended to the dehydration of long-chain fatty carboxamides in a circular flow electrolysis setup. The reaction proceeds under ambient temperature (20 to 25 °C) and pressure, affording fatty nitriles in good yields on a gram scale. Beyond simple scale-up, the electrochemical approach offers remarkable flexibility in flow-cell reactor design, enabling the use of a variety of flow-cell configurations, including small-scale cells for rapid screening, larger flow cells for increased throughput, and specially adapted systems capable of handling slurry-based conditions. This versatility is particularly valuable when considering translation to industrial processes, where reactor design and material handling are critical parameters [38]. Importantly, this operationally straightforward electrochemical method eliminates the need for metal oxide catalysts and high operating temperatures, thereby circumventing the hazardous dehydration conditions typical of conventional processes. As a result, it provides a safe, scalable, and adaptable alternative for the synthesis of fatty nitriles under mild conditions.

2 | Results and Discussion

The method previously established in our group for the dehydration of carboxamides to their nitriles was used as a starting point for the current reaction optimization and scale-up [33]. These reaction conditions are summarized in Scheme 2. First, under these reaction conditions, the dehydration of linear C14–C18 fatty amides was tested in a batch-type cell. Even-numbered long-chain fatty amides were chosen for this process



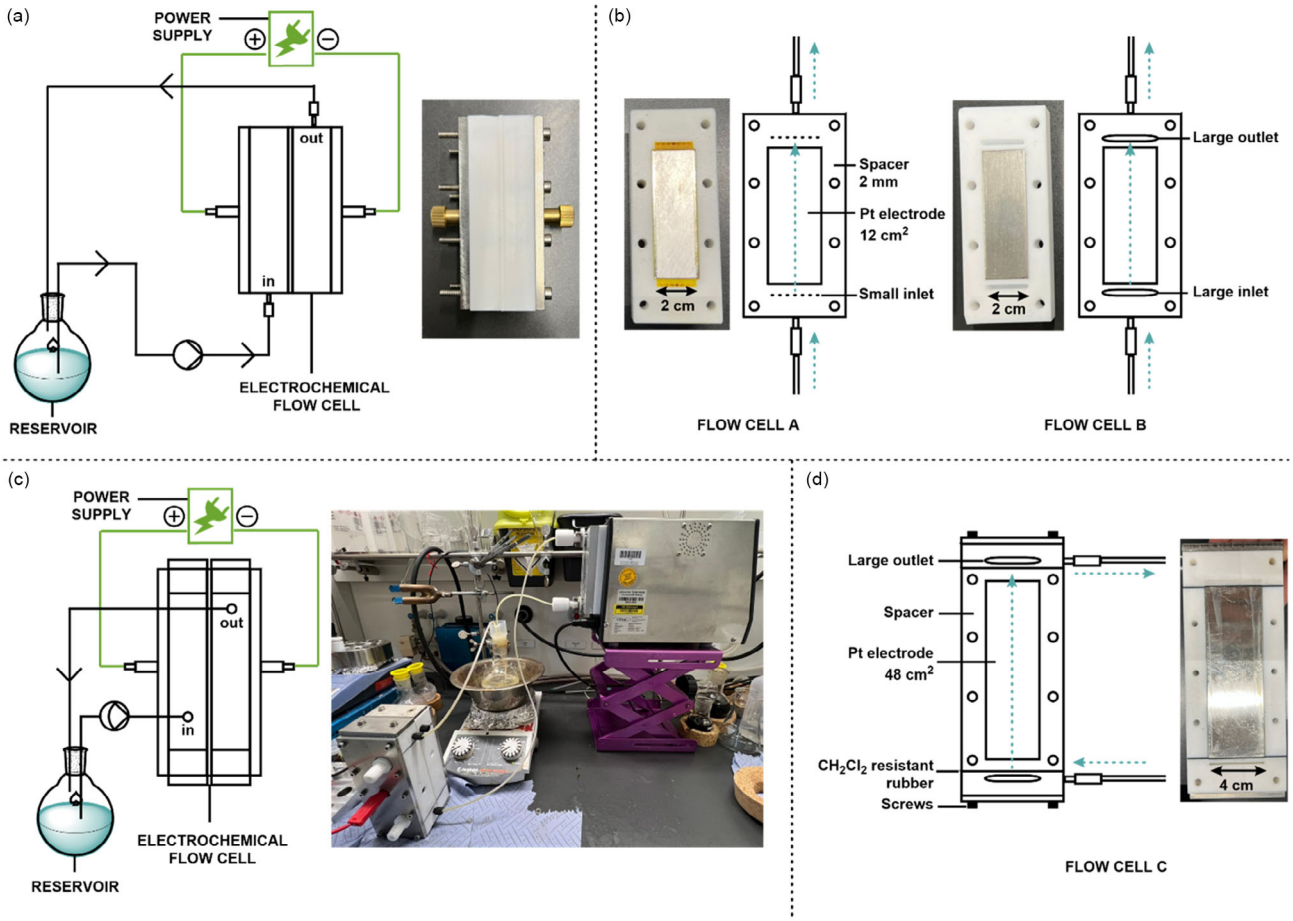
SCHEME 2 | Initial reaction conditions in the batch-type cell. Isolated yields are given.

as these can be produced from the most abundant bioderived fatty acids [7].

During the batch cell reactions, we experienced solubility issues of the starting material as the long-chain aliphatic carboxamides were not or barely soluble in dichloromethane and tended to float on the surface of the reaction mixture during electrolysis. Despite this fact, with these conditions, stearonitrile **3a** was isolated in a 51% yield starting from the C18-amide stearamide. To enhance the solubility of the starting material, the electrochemical dehydration of stearamide was repeated at 35 °C, and after product isolation, a yield of 54% was obtained. However, due to significant solvent evaporation during electrolysis at higher temperatures and only a slight increase in yield, this was not deemed a viable solution. We therefore moved on to testing different solvents for this reaction. A range of solvents was tested at higher temperatures (see Table S2). Unfortunately, although the solubility of the starting material was increased in some cases, the elevated temperatures likely enhanced the formation of parathiocyanogen, a polymerized byproduct formed during electrolysis of the thiocyanate anion [39], and as a result, the yield of the reaction did not improve. Therefore, the use of dichloromethane as a solvent remained crucial to achieve the desired high yields, and further electrolysis experiments were performed at room temperature. The electrochemical dehydration of tetradecanamide gave the corresponding tetradecanitrile (**1a**) in an isolated yield of 98% after purification. Finally, using palmitamide, an isolated yield of 89% of **2a** was obtained.

To test the scalability of the electrochemical dehydration of fatty amides, the reaction was first scaled-up in batch cell conditions. Tetradecanamide was subjected to electrolysis on a 5-fold preparative scale to generate tetradecanenitrile under the standard reaction conditions shown in Scheme 2. After filtration over a silica pad, the product was isolated in a yield of 85%, indicating that the reaction can be reliably scaled up without a large loss of yield. Additional experimental details and operational considerations for the scale-up procedure in batch and for the following recirculation flow electrolyzer setup are provided in the Supporting Information.

After establishing the scalability of the reaction in batch, we moved on to performing the reaction in a recirculation setup with a flow-type electrolysis cell, as shown in Scheme 3a. To better illustrate the successful translation of the methodology from batch to continuous-flow electrolysis, the key features of both approaches are compared in Table 1.



SCHEME 3 | (a) Schematic of the recirculation flow electrolysis setup, including a flow electrochemical cell used for initial reaction optimization in flow. (b) Schematics and pictures of the internal cell design of flow cells A and B, including the direction of the flow. The size of the Pt electrodes is 2 × 6 cm. (c) (Left) Schematic of the recirculation flow electrolysis setup used for reaction scale-up with flow cell C. (Right) Picture of the recirculation flow electrolysis setup used for reaction scale-up with flow cell C. (d) Schematic (left) and picture (right) of the internal cell design of flow cell C, including the direction of the flow. Size of the Pt electrode is 4 × 12 cm.

TABLE 1 | Comparison between the previously reported batch methodology and the continuous-flow electrolysis developed in this work.

Feature	Batch	Flow
Scale-up	✓	✓✓
Cell design	1	3
Slurry	✗	✓
Solvent reuse	✗	✓
Lipophilic substrates	✓	✓

While tetradecanamide and palmitamide afforded good yields under the applied batch-type conditions, stearamide resulted in significantly lower yields. Therefore, a targeted optimization study was conducted for this substrate (Table 2). The optimization was initialized by directly transferring the batch conditions to the flow electrolysis setup using flow-cell design A (Scheme 3b), with a flow rate of 10 mL/min. This gave a low yield of 13% stearonitrile due to the voltage of the reaction spiking after the administration of 20 *F* (Table 2, entry 1). The observed voltage increase may be attributed to a progressive increase in cell resistance during electrolysis, potentially arising from changes in the electrolyte composition and/or partial electrode fouling by poorly soluble or thiocyanate-derived species. Under galvanostatic conditions, such an increase in resistance requires a higher cell voltage to maintain the imposed current. Screening of the current density revealed a lower current density of 8 mA cm^{-2} was optimal. The improved yield at lower current density may result from a better balance between the electrochemical generation of the active thiocyanate species and its subsequent reaction in solution. At higher current densities, increased electrode polarization and mass-transport limitations may favor nonproductive oxidation processes, thereby decreasing the selectivity toward the desired nitrile. Furthermore, due to the low solubility of the starting material and the small inlet and outlet of the flow cell, we encountered frequent clogging of the cell when pumping around the suspension. To remedy this, the reaction mixture in the reservoir was not stirred while pumping, thereby avoiding the formation of a suspension and allowing the undissolved substrate to remain floating on the solvent surface in the reservoir. As a result, only the dissolved substrate was pumped through the electrolysis cell, effectively preventing clogging, while the undissolved substrate in the reservoir could gradually dissolve and react over time. Consequently, an increase in yield to 33% was obtained (Table 2, entry 2).

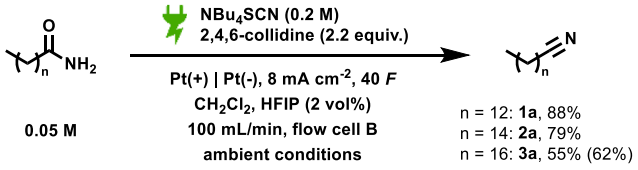
To further investigate the influence of the electrochemical parameters on the reaction, several parameters were systematically varied. Lowering the amount of applied charge to 30 *F* strongly diminished the yield to 7% (Table 2, entry 3), while performing the reaction at 0 °C led to a decrease in yield as well (Table 2, entry 4). When no HFIP was added to the reaction, a yield of 20% was obtained (Table 2, entry 5), and significant solid deposition was observed on the anode, necessitating manual opening and cleaning of the cell after electrolysis. This suggests that HFIP is crucial for achieving selective conversion and suppressing thiocyanate-derived side reactions. It is well known in the literature that HFIP can establish a solvent cage, preventing undesired side reactions or facilitating desired reactions [40, 41]. Importantly, only a low amount of HFIP (2 vol%) is necessary, and it is possible to recover the HFIP after electrolysis, reducing concerns regarding sustainability and process viability. Finally, different flow rates were tested, and a high flow rate of 100 mL/min proved to be ideal, giving a yield of 37% (Table 2, entry 6). When then stirring the reaction mixture in the reservoir to create a fine suspension of the starting material and pumping the slurry through the cell, we obtained a GC yield of 47% (Table 2, entry 7), although the higher flow rates could not reliably prevent clogging of the cell.

Therefore, to reliably pump slurries into the flow cell, we utilized an adapted cell design (flow cell B, Scheme 3b). Here, the small channels through which the reaction mixture is pumped into and out of the cell were adapted to form a big channel instead to prevent clogging of the cell in the small inlet and outlet holes. Using this new design, the optimized conditions were achieved, giving a GC yield of 62% (Table 2, entry 8) and an isolated yield of 55%. The final optimized reaction conditions are shown in Scheme 4.

TABLE 2 | Optimization of the dehydration of stearamide in an undivided electrochemical flow cell.

Entry	Flow rate, mL/min	Amount of applied charge, *F*	Current density, mA cm^{-2}	Temperature, °C	Additive	Flow cell design	Yield of 3a[a]
1	10	20[b]	14	20	HFIP (2 vol%)	A	13%[d]
2	10	40	8	20	HFIP (2 vol%)	A	33%
3	10	30	8	20	HFIP (2 vol%)	A	7%
4	10	40	8	0	HFIP (2 vol%)	A	29%
5	10	40	8	20	—	A	20%
6[c]	100	40	8	20	HFIP (2 vol%)	A	37%
7[c]	100	40	8	20	HFIP (2 vol%)	A	47%[d]
8[c]	100	40	8	20	HFIP (2 vol%)	B	**62%**[d]

Note: Bold elements signify the element in best yield.
Note: Conditions: stearamide (0.5 mmol, 140 mg, 1 equiv.), NBu_4SCN (2 mmol, 600 mg, 4 equiv.), 2,4,6-collidine (0.55 mmol, 150 µL, 2.2 equiv.), CH_2Cl_2 (10 mL, 0.05 M), HFIP (200 µL, 2 vol%), Pt (+) | Pt (−), constant current, and undivided flow cell.
[a]Yields are reported as GC yields, using hexadecane as an internal standard.
[b]Voltage of the reaction spiked after 20 *F*.
[c]Reaction performed on a 2.5 mmol scale.
[d]Reaction mixture in reservoir was stirred at 500 rpm.

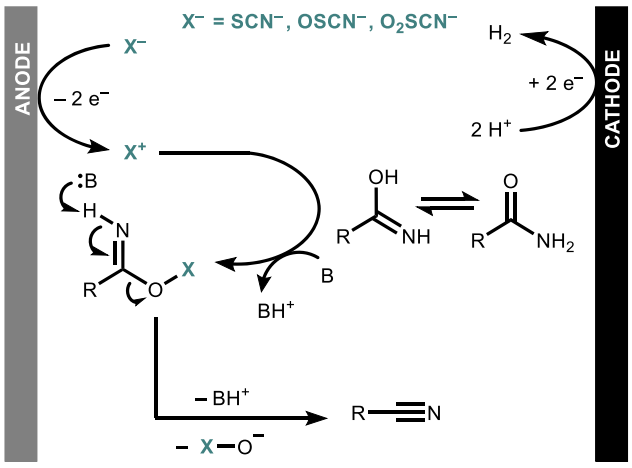


SCHEME 4 | Final optimized conditions in an undivided recirculating flow cell setup. GC yield is given in parentheses.

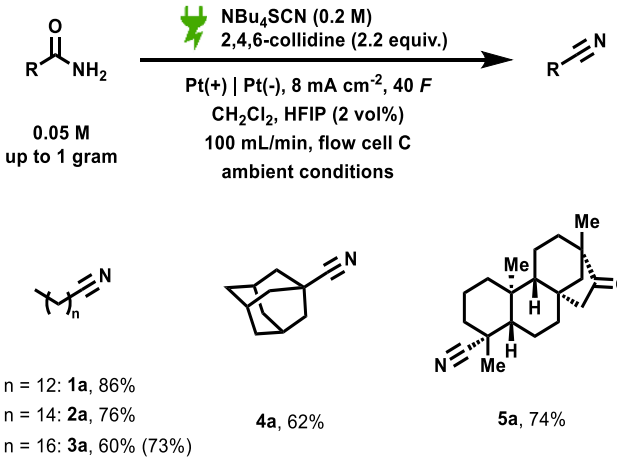
The cell-voltage evolution under the optimized electrolysis conditions is provided in Figure S8. The comparatively modest Faradaic efficiency (Table S5) reflects the high charge requirement of the current process and identifies charge efficiency as a target for further optimization. With these optimized conditions, several control experiments were done. In the absence of NBu_4SCN as electrolyte, no product yield was detected (Table S5, entry 2), highlighting the necessity of the thiocyanate supporting electrolyte. Based on previous mechanistic investigations conducted by our group, the oxidation of the thiocyanate anion of the supporting electrolyte acts as a mediator for the electrochemical dehydration reaction (Scheme 5) [33, 42–44]. When no current was applied, the reaction likewise did not yield the desired nitrile product (Table S5, entry 1), confirming that electrolysis plays a key role in promoting this reaction.

Using the optimized conditions in flow, the electrochemical dehydration of tetradecanamide and palmitamide was also attempted in flow cell B. On a 2.5 mmol scale, the dehydration of tetradecanamide using the recirculation flow setup gave **1a** in an isolated yield of 88%. Using palmitamide, an isolated yield of 79% of **2a** was obtained. Interestingly, we were able to isolate a minor side product when performing the reaction at a larger scale, which was identified using NMR methods as the fatty HFIP ester (see Figure S12). For the dehydration of tetradecanamide, a yield of 13% of the HFIP ester was obtained, accounting for the remainder of the carbon balance and explaining the non-quantitative yield of the reaction.

We then moved on to realize the scale-up of the dehydration of long-chain aliphatic carboxamides on a one-gram scale. For this, a flow-type cell with an anodic area of 48 cm^2 was used (flow cell



SCHEME 5 | Proposed reaction mechanism [33].



SCHEME 6 | Scale-up of the reaction in an undivided recirculating flow cell setup. Given yields are isolated yields. GC yield is given in parentheses.

C), with a large inlet and outlet to efficiently handle slurry-based reaction mixtures and prevent clogging of the cell during electrolysis (Scheme 3c,d). Under the optimized conditions, good yields were obtained for the fatty nitrile products (Scheme 6). Starting from 1 g of tetradecanamide, **1a** was isolated in an 86% yield. Dehydration of palmitamide gave **2a** in a 76% yield. Finally, **3a** was obtained in a 60% isolated yield. The minimal reduction in yield when scaling up underlines the robustness of the reaction in flow. To validate the transfer from batch to flow, previously established substrates, such as isosteviol and adamantane carboxamides, were re-examined under flow conditions (**4a** and **5a**). Flow electrolysis of these highly lipophilic carboxamides afforded isolated yields of 62% and 74% for **4a** and **5a**, respectively, compared with 71% and 84% in batch-type cells, respectively [33]. Their successful conversion with only a slight decrease in yield demonstrates that the method retains its efficiency for structurally demanding substrates beyond linear fatty amides. At the present stage, the process has been demonstrated on a gram scale using an electrode area of 48 cm^2 and a current density of 8 mA cm^{-2}, corresponding to a total current of approximately 0.38 A. For the gram-scale synthesis of tetradecanenitrile, the applied charge of 40 F corresponds to an electrolysis time of approximately 12 h and an isolated production rate of approximately 0.07 g h^{-1}. These values demonstrate successful translation to a larger flow electrolysis configuration while also highlighting that further process intensification will be required for industrial implementation. In particular, increasing the productive current density and reducing the required charge and electrolyte loading represent key targets for future optimization. While gram-scale synthesis was successfully achieved, further scale-up is currently limited by long electrolysis times and the high loading of tetrabutylammonium thiocyanate. Addressing these aspects will be key to enhancing the overall efficiency and sustainability of the process in future developments.

To further highlight the sustainability of the reaction, we investigated possibilities for solvent recycling after electrolysis. Simple solvent recovery using a rotary evaporator directly after electrolysis and additional rinsing of the cell yielded a solvent mixture of dichloromethane and HFIP. As the additional rinsing diluted the

solvent mixture, the concentration of HFIP was checked and adjusted before the subsequent reaction. Using ^{1}H NMR (Figure S21), the concentration of HFIP in the recovered solvent mixture was determined to be 0.8 vol%. For a repeat reaction, additional HFIP was added to reach the necessary 2 vol% HFIP (see Supporting Information, S9). Using this recycled mixture, the electrochemical dehydration of tetradecanamide was repeated on a small scale, giving an isolated yield of 83% of **1a**, thereby showing that solvent recycling is an effective way to generate less reaction waste and contribute to an overall more sustainable process while still maintaining the desired high reaction yields.

3 | Conclusion

This work demonstrates that flow electrolysis enables the efficient and scalable dehydration of highly lipophilic carboxamides to nitriles, achieving high yields of up to 88% on a 2.5 mmol scale and up to 86% on the gram scale. The successful translation from batch to flow conditions highlights the robustness of the method and the advantages of flow processing for controlled, reproducible electrochemical transformations. Crucially, the use of tailored flow cell designs allows reliable handling of poorly soluble, long-chain aliphatic substrates and slurry systems, addressing key challenges such as mass transport limitations and clogging that often hinder electrochemical scale-up. Beyond these results, this study demonstrates how the combination of electrochemistry and continuous-flow processing can contribute to more sustainable synthetic methodologies. By replacing conventional high-temperature dehydration processes with an electrochemical protocol operating under ambient conditions, eliminating the need for metal oxide catalysts, and enabling solvent recovery and reuse without significant loss of reaction efficiency, the present approach provides a practical alternative for the synthesis of fatty nitriles. Considering that fatty nitriles are key intermediates derived from renewable lipid feedstocks, these advances further support the development of more sustainable manufacturing processes for bio-based chemicals. More generally, this work illustrates how continuous-flow electrolysis can facilitate the translation of sustainable electro-organic transformations from laboratory-scale methodology toward practical and industrially relevant manufacturing.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.