

## Sustainable Chemistry

## Electrochemical Synthesis of Hydroxylamine

Minghao Guo, Yuhan Zhang, Chengying Guo,\* and Yifu Yu\*

**Abstract:** Hydroxylamine (NH<sub>2</sub>OH) serves as an important industrial feedstock. The conventional production and transportation of NH<sub>2</sub>OH requires harsh conditions. Recently, electrochemical hydrogenation of nitrogen-containing oxidative species, such as nitrate, nitrite, and nitric oxide (NO<sub>3</sub><sup>−</sup>/NO<sub>2</sub><sup>−</sup>/NO), into NH<sub>2</sub>OH has been developed as a sustainable strategy. However, the over-hydrogenation product of ammonia is preferentially obtained under electroreduction condition. Thus, the rational design of catalysts structure is crucial to selective electrosynthesis of NH<sub>2</sub>OH. In this minireview, we summarize recent advances in electrochemical synthesis of NH<sub>2</sub>OH with emphasis on the design of catalysts structure. Electrochemical synthesis strategy we discussed is categorized by the existence form of product, including indirect electrosynthesis and direct electrosynthesis. Finally, the techno-economic analysis (TEA) of electrochemical NH<sub>2</sub>OH production and outlook of performance improvement strategies are carried out to further guide the optimization of reaction system.

## 1. Introduction

Hydroxylamine (NH<sub>2</sub>OH), as an important industrial feedstock, has been widely used in nylon, medicine, fuel, and agrochemical<sup>[1,2]</sup> The conventional craft for NH<sub>2</sub>OH production rely on complex thermocatalytic reactions, which require harsh conditions (Scheme 1a).<sup>[3–7]</sup> Meanwhile, the toxic and explosive risks of NH<sub>2</sub>OH cause difficulties during transportation and storage. Thus, it is of great significance to develop a sustainable technique to in-place produce NH<sub>2</sub>OH for downstream application. Interestingly, numerous works have reported the electroreduction of nitrogen-containing

oxidative species including nitrate, nitrite, and nitric oxide (NO<sub>3</sub><sup>−</sup>/NO<sub>2</sub><sup>−</sup>/NO) to ammonia (NH<sub>3</sub>), in which NH<sub>2</sub>OH was usually found as an intermediate.<sup>[8–13]</sup> Compared with NH<sub>3</sub>, NH<sub>2</sub>OH has much higher value (1600 \$·ton<sub>NH<sub>2</sub>OH</sub><sup>−1</sup> versus 600 \$·ton<sub>NH<sub>3</sub></sub><sup>−1</sup>). Thus, electroreduction of NO<sub>3</sub><sup>−</sup>/NO<sub>2</sub><sup>−</sup>/NO provides a sustainable strategy for in-place production of NH<sub>2</sub>OH. However, the theoretical reduction potential of NH<sub>2</sub>OH to NH<sub>3</sub> is +1.35 V (versus NHE).<sup>[14]</sup> The very positive potential indicates that NH<sub>2</sub>OH is unstable and easily over-reduced to NH<sub>3</sub> during electroreduction process. Therefore, the rational design of catalytic system is crucial to selective electrosynthesis of NH<sub>2</sub>OH.

To date, electrochemical synthesis of NH<sub>2</sub>OH from NO<sub>3</sub><sup>−</sup>/NO<sub>2</sub><sup>−</sup>/NO follows two categories (Scheme 1b): 1) Indirect electrosynthesis: in situ capture of NH<sub>2</sub>OH intermediate (\*NH<sub>2</sub>OH) by spontaneous oximation reaction with aldehyde/ketone and then NH<sub>2</sub>OH is obtained after oximation hydrolyze. 2) Direct electrosynthesis: selective hydrogenation of NO<sub>3</sub><sup>−</sup>/NO<sub>2</sub><sup>−</sup>/NO to directly generate NH<sub>2</sub>OH. In this minireview, we summarize recent advances in the electrochemical synthesis of NH<sub>2</sub>OH with emphasis on the aforementioned two strategies. To further guide the application of reaction system in the future, we carry out techno-economic analysis (TEA) with faradaic efficiency (FE) and current density (*j*) of NH<sub>2</sub>OH as the two key parameters. Finally, we outlook catalyst design and reactor engineering to improve the reactive activity and energy efficiency purposefully.

2. Indirect Electrosynthesis of NH<sub>2</sub>OH

Industrially, NH<sub>2</sub>OH is produced by oxime hydrolysis and oxime is usually synthesized by spontaneous nucleophilic addition–elimination reaction between unstable NH<sub>2</sub>OH intermediate and trapping agent of aldehyde/ketone (RCOH/RCOR). Thus, oxime can also be formed by in situ capturing \*NH<sub>2</sub>OH intermediate with RCOH/RCOR during electroreduction. As a result, NH<sub>2</sub>OH is indirectly electrochemical synthesis. Recently, dozens of oximes have been obtained by in situ capturing \*NH<sub>2</sub>OH through adsorbed or free RCOH/RCOR (1. \*NH<sub>2</sub>OH + RCOH/RCOR → oxime, 2. \*NH<sub>2</sub>OH + \*RCOH/\*RCOR → oxime, Figure 1).<sup>[15–18]</sup> Zhang's group proposed the mechanism of oxime generation during NO<sub>3</sub><sup>−</sup>-RR, in which the over-reduction of \*NH<sub>2</sub>OH to NH<sub>3</sub> competes with the oximation reaction.<sup>[19]</sup> So, the stabilization and enrichment of \*NH<sub>2</sub>OH is a possible direction for catalyst design. It was reported that the desorption of \*NH<sub>2</sub>OH was promoted thermodynamically via alloying Zn and Cu, and thus NH<sub>2</sub>OH could be stabilized.<sup>[20]</sup> Moreover, \*NH<sub>2</sub>OH

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

could be enriched over Fe nanoparticles via increasing the activation barrier for further hydrogenation of  $^*\text{NH}_2\text{OH}$  ( $^*\text{NH}_2\text{OH} \rightarrow ^*\text{NH}_2$ ).<sup>[21]</sup> The indirect synthesis performances are summarized in Table S1, in which the C-selectivity has been achieved almost 100%, but the N-selectivity is still low. Higher N-selectivity could be achieved by increasing the concentration of capture agent, but the C-selectivity is bound to decrease. Thus, the development of novel strategies to simultaneously improve the C-selectivity and N-selectivity is highly requirable.

### 3. Direct Electrosynthesis of $\text{NH}_2\text{OH}$

Comparing  $\text{NO}_3^-/\text{NO}_2^-$  reduction and NO reduction,  $^*\text{NO}$  is formed by  $\text{NO}_3^-/\text{NO}_2^-$  reduction and NO adsorption, respectively. Thus, the mechanistic difference between NO reduction and  $\text{NO}_3^-/\text{NO}_2^-$  reduction lies in the formation process of  $^*\text{NO}$ . For  $\text{NO}_3^-/\text{NO}_2^-$  reduction, NO is the possible by-product, thus high hydrogenation ability is needed to reduce  $^*\text{NO}$ , but it enhances the competition between  $\text{NH}_3$  and  $\text{NH}_2\text{OH}$  in turn. For NO electroreduction, the reactant NO exists in the form of gas molecules, so the hydrogenation of  $^*\text{NO}$  must take into account of NO enrichment and adsorption. For the hydrogenation of  $^*\text{NO}$ , it is shared in  $\text{NO}_3^-/\text{NO}_2^-/\text{NO}$  electroreduction and determines the selectivity for  $\text{NH}_3$  or  $\text{NH}_2\text{OH}$ .

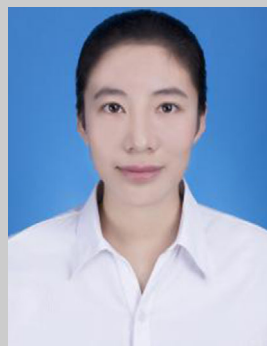
$\text{NH}_2\text{OH}$  has been identified as an unstable intermediate during  $\text{NO}_3^-/\text{NO}_2^-/\text{NO}$  electroreduction, with a propensity to undergo over-hydrogenation to  $\text{NH}_3$ . Single-atom catalysts (SACs) have been widely used in direct electrosynthesis of

$\text{NH}_2\text{OH}$  owing to two advantages as follows (Figure 2a). 1) SACs modulated the adsorption configuration of reactants to selectively generate  $^*\text{NH}_2\text{OH}$ . Zhou et al. simultaneously synthesized Co-based catalysts with single atom and nanoparticles (NPs) structure to explore the relationship between  $\text{NH}_2\text{OH}$  selectivity and adsorption configuration, which is related to catalyst structure.<sup>[22]</sup> In situ attenuated total reflection fourier-transform infrared (ATR-FTIR) spectra proved the linear and bridge adsorption of  $^*\text{NO}$  on Co SACs and Co NPs, respectively. The linear adsorption of  $^*\text{NO}$  brings the maintenance of N–O bond during electroreduction, resulting in the selective generation of  $\text{NH}_2\text{OH}$  ( $\text{FE}_{\text{NH}_2\text{OH}} = 81.3\%$ ). On the contrary, the bridge adsorption of  $^*\text{NO}$  induces the breaking of N–O bond during electroreduction, resulting in the selective generation of ammonia ( $\text{FE}_{\text{NH}_3} = 92.3\%$ ). Furthermore, Wu et al. confirmed the maintenance of N–O bond on SACs during reduction reaction by calculations.<sup>[23]</sup> 2) SACs possessed the weak absorption ability for  $^*\text{NH}_2\text{OH}$  and exhibited spontaneous thermodynamic desorption properties to selectively generate  $\text{NH}_2\text{OH}$ . Generally, the isolated active site of SACs anchored by N/C atoms brings partial positive charge, which weakens the adsorption strength of  $^*\text{NH}_2\text{OH}$  and exhibits high selectivity for  $\text{NH}_2\text{OH}$ .<sup>[24,25]</sup> It has been reported that the real active species is positive charged  $\text{Fe}^{2+}$  in Fe–SACs during NO electroreduction through in situ X-ray absorption near edge structure (XANES) spectra, which directly proved the positive charge of SACs for  $\text{NH}_2\text{OH}$  selective synthesis ( $\text{FE}_{\text{NH}_2\text{OH}} \approx 60\%$ ).<sup>[26]</sup>

To achieve both high activity and selectivity, Yu's group proposed to utilize lattice hydrogen ( $\text{H}_{\text{lat}}$ ) with weaker reduc-



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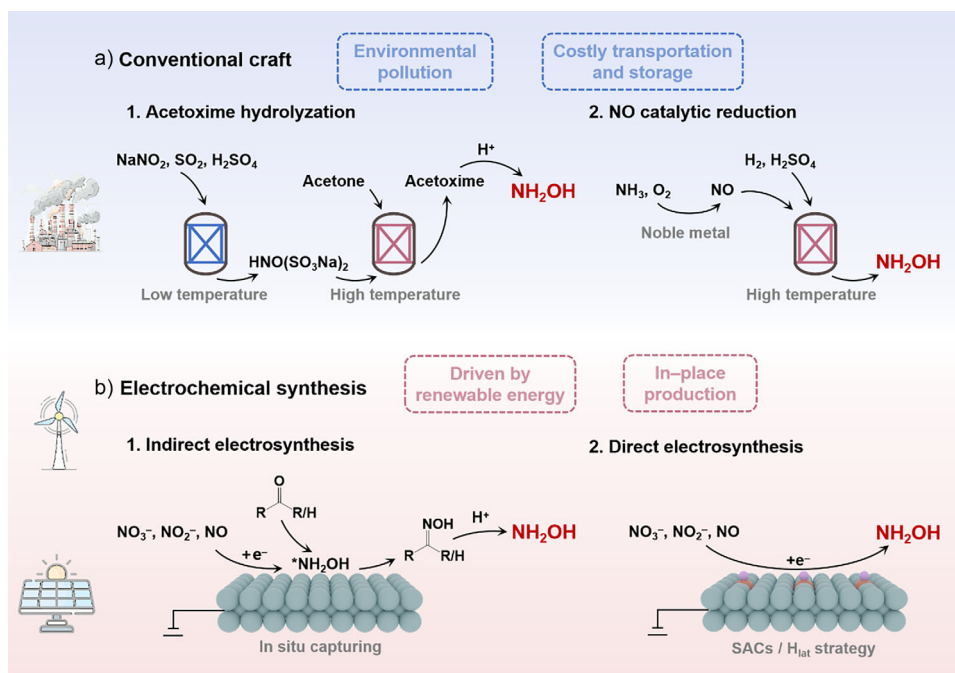
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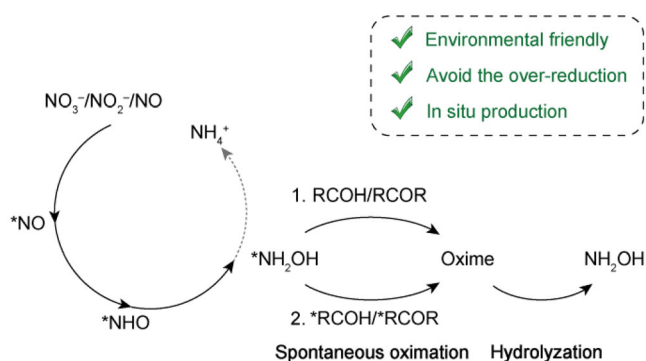
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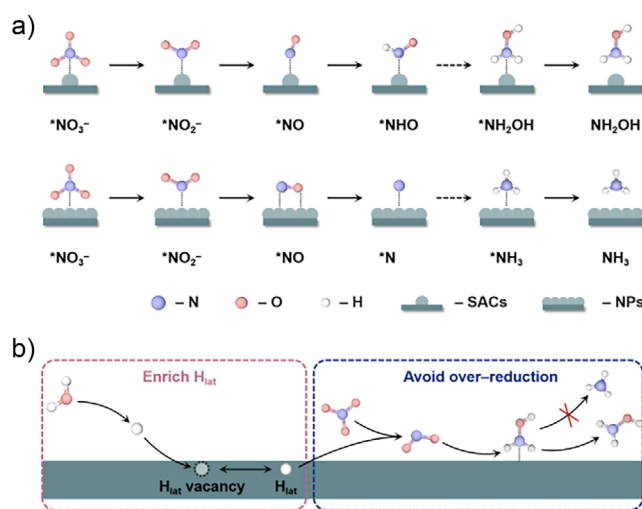


**Scheme 1.** a) Conventional craft of  $\text{NH}_2\text{OH}$  production. b) Electrochemical synthesis of  $\text{NH}_2\text{OH}$  via indirect and direct strategy.



**Figure 1.** Schematic illustration for indirect  $\text{NH}_2\text{OH}$  generation.

tion ability than active hydrogen ( $^*\text{H}$ ) for  $\text{NH}_2\text{OH}$  selective electrosynthesis (Figure 2b).<sup>[27]</sup> The proton is inserted into the lattice of  $\text{MnO}_2$  catalysts to generate  $\text{H}_{\text{lat}}$ , which directly participates in  $\text{NO}_3^-$  hydrogenation and then produces  $\text{H}_{\text{lat}}$  vacancy. Subsequently, the hydrogen tends to replenish the  $\text{H}_{\text{lat}}$  vacancy rather than to form  $^*\text{H}$ . Thus, a dynamic balance between consumption and replenishment of  $\text{H}_{\text{lat}}$  is achieved. The as-produced  $^*\text{NH}_2\text{OH}$  tends to desorb from the surface of catalyst rather than be over-reduced to  $\text{NH}_3$  when using  $\text{H}_{\text{lat}}$  as H-source owing to its weak reduction ability, confirming the buffer effect of  $\text{H}_{\text{lat}}$ . Moreover, the doping of Cu can induce Cu-triggered Jahn–Teller distortion of  $[\text{MnO}_6]$  octahedron, which significantly increases the number of  $\text{H}_{\text{lat}}$ . As a result, the faradaic efficiency and yield of  $\text{NH}_2\text{OH}$  achieve 91.1% and  $396.6 \text{ mmol g}_{\text{cat}}^{-1} \text{ h}^{-1}$ , respectively. Compared to  $^*\text{H}$  from water dissociation,  $\text{H}_{\text{lat}}$  plays the dual-function of enrichment and buffer, which can provide abundant  $\text{H}_{\text{lat}}$  for  $\text{NO}_3^-$  selective reduction to



**Figure 2.** Schematic illustration for the direct electrosynthesis of  $\text{NH}_2\text{OH}$  a) over single-atom catalysts and nanoparticles, b) using  $\text{H}_{\text{lat}}$  as H-source.

$\text{NH}_2\text{OH}$  without over-hydrogenation. This work paved a new path for the design of efficient electrocatalysts for direct electrosynthesis of  $\text{NH}_2\text{OH}$ .

#### 4. $\text{NH}_2\text{OH}$ Detection

In this section, we summarized two-type methods for detection and quantification of  $\text{NH}_2\text{OH}$  based on the above-mentioned indirect electrosynthesis and direct electrosynthesis. For the indirect electrosynthesis, the  $^*\text{NH}_2\text{OH}$  is captured

by RCOH/RCOR to form oxime via a spontaneous oximation reaction. The formed oxime has  $^1\text{H}$  nuclear magnetic resonance ( $^1\text{H}$  NMR) characteristic peaks, which can be precisely detected and quantified by  $^1\text{H}$  NMR method with suitable internal standard (such as dimethyl sulfoxide, maleic acid). In this method, the high stability of RCOH/RCOR and the high oximation rate during electrosynthesis process are important for the accuracy of the data. For the direct electrosynthesis, the free  $\text{NH}_2\text{OH}$  is obtained in electrolyte. The  $^1\text{H}$  NMR method can be also used to detect  $\text{NH}_2\text{OH}$  after adding RCOH/RCOR to as-produced electrolyte. In addition, the colorimetric method is also widely used to directly quantify  $\text{NH}_2\text{OH}$  in aqueous solution.<sup>[22,28]</sup> Notably, the  $^1\text{H}$  NMR and colorimetric methods can be carried out simultaneously to verify the accuracy of the data.

## 5. Techno-Economic Analysis (TEA)

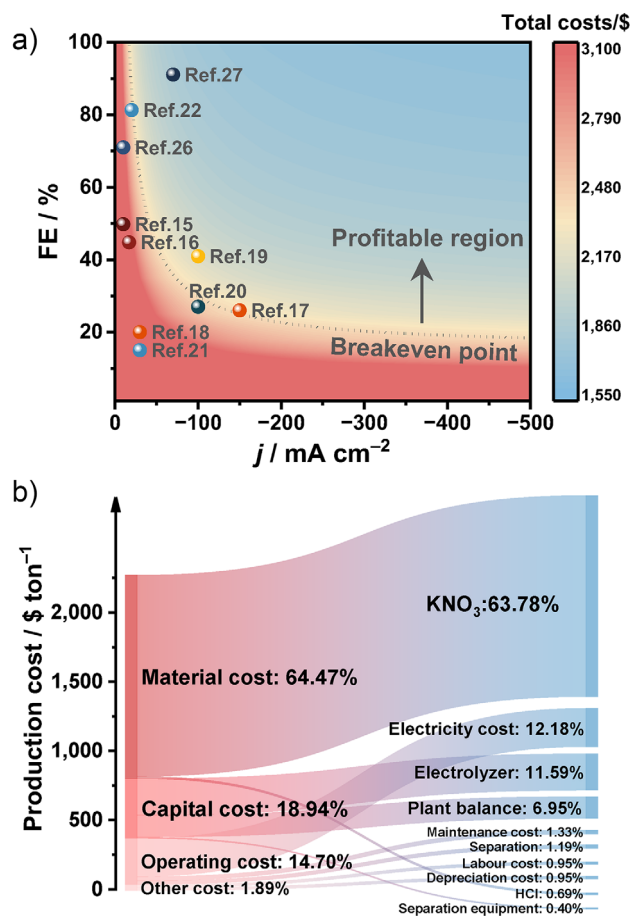
To assess the economic potential of in-place utilization of  $\text{NH}_2\text{OH}$  driven by renewable electricity, a modified TEA model was employed with  $\text{FE}_{\text{NH}_2\text{OH}}$  and current density ( $j$ ) as two key parameters (details in Supporting Information). The performances of recently reported works were added in this TEA picture, in which the improvement of  $\text{FE}_{\text{NH}_2\text{OH}}$  or  $j$  is expected to achieve economic profitability (Figure 3a). In previous works (Table S1), the FE and  $j$  were around 50% and  $-50 \text{ mA cm}^{-2}$  in  $\text{NH}_2\text{OH}$  generation, so the specific costs were directly quantified and provided as Figure 3b. It can be seen that the materials cost was dominant for  $\text{NH}_2\text{OH}$  generation. When using N-containing wastewater as materials, the  $\text{NH}_2\text{OH}$  generation has much higher economic profit and developing advanced catalysts to enrich low concentration nitrate in wastewater also exhibits high research prospect.

## 6. Summary and Outlook

Over past years, electroreduction of  $\text{NO}_3^-/\text{NO}_2^-/\text{NO}$  provides a sustainable strategy for in-place production of  $\text{NH}_2\text{OH}$ . Remarkable advancements have been made in indirect and direct electrosynthesis of  $\text{NH}_2\text{OH}$ . However, several challenges remain: 1) The low efficiency of indirect synthesis strategy and the incompatibility of activity and selectivity. 2) The limitation of traditional batch reactor on performance. In this section, we propose a few possible future directions for addressing the challenges of current researches.

### 6.1. Catalyst Design

To improve the efficiency of indirect synthesis strategy, the spontaneous nucleophilic addition-elimination reaction efficiency between  $^*\text{NH}_2\text{OH}$  and RCOH/RCOR needs to be further improved. As known, the nucleophilicity of  $^*\text{NH}_2\text{OH}$  can be enhanced after the electron transfer from active site to



**Figure 3.** a) TEA result for in-place production of  $\text{NH}_2\text{OH}$  with FE and  $j$  as variates. b) Subdivided cost of the entire process for in-place production of  $\text{NH}_2\text{OH}$  by using nitrate as raw materials.

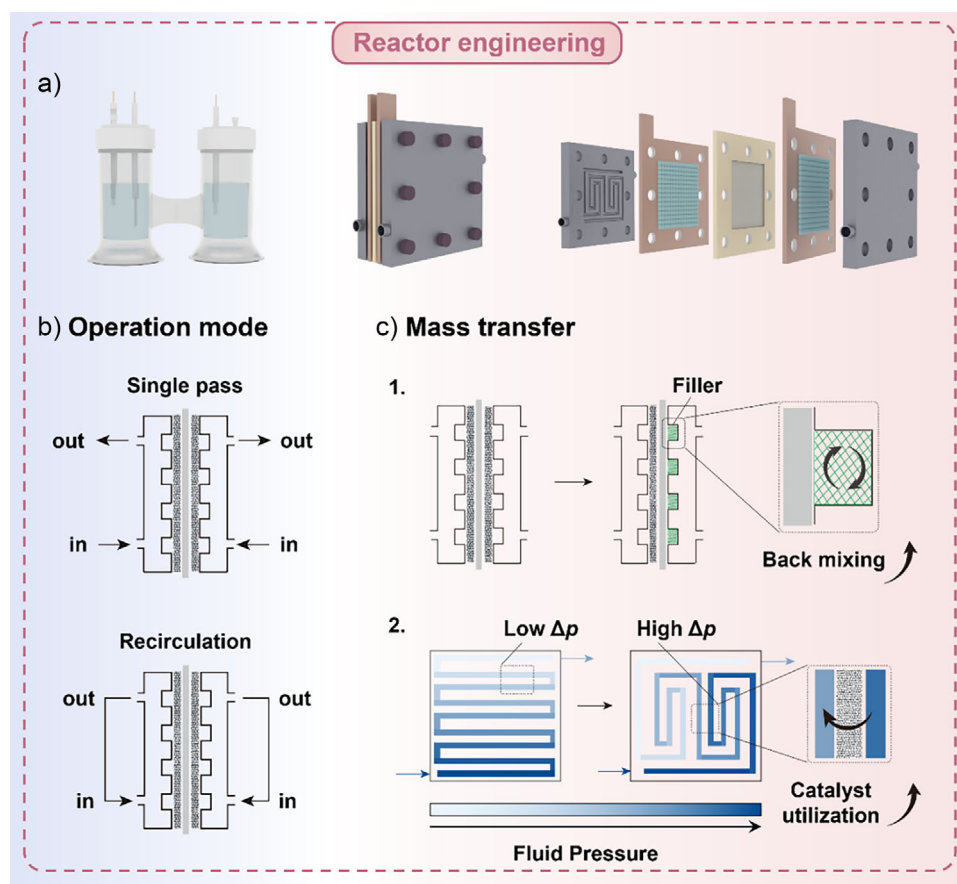
$^*\text{NH}_2\text{OH}$ . Thus, the construction of electron-rich active site is expected to improve the efficiency of nucleophilic attack.

To simultaneously improve the activity and selectivity of direct strategy, the adoption of  $\text{H}_{\text{lat}}$  is promising. As mentioned in section 3,  $\text{Cu-MnO}_2\text{H}_x$  can synthesize  $\text{NH}_2\text{OH}$  with high activity and selectivity via  $\text{H}_{\text{lat}}$ . But,  $\text{MnO}_2$  is not intrinsically active and has a large overpotential for  $\text{H}_{\text{lat}}$  generation. Thus, the design and construction of catalysts with thermodynamical advantage for the formation of  $\text{H}_{\text{lat}}$  is expected to reduce the overpotential for direct electrosynthesis of  $\text{NH}_2\text{OH}$ .

### 6.2. Reactor Engineering

In addition to catalysts, the design of reactor to match reaction conditions is also a pathway to enhance performance of unstable products.  $\text{NO}_3^-/\text{NO}_2^-/\text{NO}$  electroreduction is usually done in batch H-type reactor. But, the ratio of electrode area to electrolyte volume and the mass transfer of batch reactor is lower than continuous flow cell, which inevitably increases the over-reduction of  $\text{NH}_2\text{OH}$  to  $\text{NH}_3$  (Figure 4a). Thus, we proposed two directions in reactor engineering.





**Figure 4.** a) Scheme of batch H-type reactor and continuous flow cell. b) Scheme of different operation modes. c) Scheme of different mass transfer effect.

(a) Operation mode: For indirect synthesis, high N-selectivity could be achieved using high concentration of capture agent, but C-selectivity of capturing agent is low. The recirculation operation can reuse the residual capture agent to improve utilization, achieving high C-selectivity and high N-selectivity simultaneously (Figure 4b down). For direct synthesis strategy, the obtained  $\text{NH}_2\text{OH}$  is unstable and easily over-reduced to  $\text{NH}_3$  during electroreduction process. The single-pass operation is an important operation mode to reduce the duration time of unstable  $\text{NH}_2\text{OH}$  (Figure 4b up).

(b) Mass transfer: Mass transfer plays a central role in continuous flow cell owing to the enhancement in reaction efficiency (Figure 4c). On one hand, filling the flow channels and loading catalyst on the filler can reduce concentration polarization and enhance radial mass transfer between electrode and electrolyte. For indirect electrosynthesis, the enhancement of mass transfer improves the capture efficiency. For direct electrosynthesis, it decreases the duration time of unstable  $\text{NH}_2\text{OH}$  and thus improve the  $\text{NH}_2\text{OH}$  activity and selectivity. On the other hand, there is a pressure difference ( $\Delta p$ ) between the flow channels due to the fluid drag. The high  $\Delta p$  can improve mass transfer in catalysts surface between the neighboring flow channels, resulting in the improvement of catalyst utilization. Therefore, rational

design of the flow channels sequence to increase  $\Delta p$  can improve the reactive activity and efficiency available.

### Acknowledgements

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### Conflict of Interests

The authors declare no conflict of interest.

### Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

**Keywords:** Direct strategy • Hydroxylamine electrosynthesis • Indirect strategy • Reactor engineering

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## Minireview

## Sustainable Chemistry

M. Guo, Y. Zhang, C. Guo\*,  
Y. Yu\* **e202509053**Electrochemical Synthesis of  
Hydroxylamine

This minireview focuses on the indirect and direct electrosynthesis of  $\text{NH}_2\text{OH}$ . The outlook of performance improvement strategies including catalysts and reactor design is also provided.

