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Research targets for upcycling of CO₂ to formate and carbon monoxide with paired electrolysis



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Electrochemical CO₂ reduction to formate and CO holds potential of upcycling waste into useful chemicals and has industrial viability. Goal of this review is to help align research targets for fundamental research with requirements for industrial viability to accelerate adoption by the industry. We identify which Key Performance Indicators (KPI's) are influential on manufacturing costs for CO and formate and we propose research targets for these KPI's and compare these research targets with the state of the art for CO production and formate production. Some influential KPI's, such as current density, are well addressed in the state of the art. Other KPI's, such as product purity, are commonly insufficiently addressed. To further improve process economics, the oxidation of water at the anode can be replaced by a more valuable reaction. We explain which innovations in anodes material and in bipolar membranes (BPM's) can enable paired electrolysis with CO2 reduction.

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Introduction

To fulfill the demand for carbon materials in sustainable ways, we must move away from fossil-based feedstocks. Recycled carbon, upcycled CO₂, and bio-based feedstocks are the sustainable sources for carbon materials in the future [1]. For upcycling CO₂, electrochemical CO₂ reduction has the advantage that, unlike thermocatalytic CO₂ conversion, it directly uses electricity to drive the reaction and that it does not require hydrogen input. Conventionally, CO₂ reduction is performed with water oxidation at the anode. Replacing this oxidation reaction with a more valuable reaction, a process called paired

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CO₂ to carbon monoxide (CO) and formate and the possibilities for paired electrolysis. To accelerate adoption of these innovative technologies by the industry, the research targets for early-stage research (technology readiness level [TRL] 3 or lower [2]) must be aligned with requirements to achieve TRL 6 and beyond. However, in practice, it is difficult to early on identify the key performance indicators (KPI's) that are most influential for economic viability, which can result in a discrepancy between early-stage and applied research. To solve this gap, we reviewed publications from the last three years on techno-economic analyses (TEA's) to assess which KPI's are most influential on the manufacturing costs of CO and formate by electrochemical CO₂ reduction, and we propose research targets for early-stage technology development. We compare these research targets with the state of the art for CO and formate production. For CO production, we consider solid oxide electrolysis (SOE), because this is commercially available on the laboratory scale and is the most mature technology. SOE generally operates at temperatures above 600 °C. The state of the art for formate production is low-temperature electrolysis. We conclude that purity of the CO and formate product streams is commonly insufficiently addressed in earlystage research, although this KPI has a big influence on the manufacturing costs. In addition, we highlight successful examples of paired electrolysis process with CO₂ reduction to formate and CO. We discuss how innovations in bipolar membranes (BPM's) and anode materials capable of operating in the acidic electrolyte can enable paired electrolysis processes.

electrolysis, can benefit process economics. This review

deals with technologies for electrochemical upcycling of

Important key performance indicators for the reduction of CO₂ to formate and CO

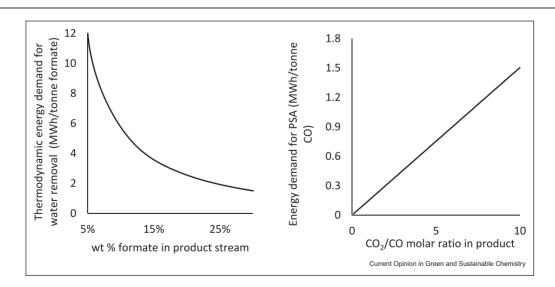
TEA's are tools to define research targets for technology development and to identify the KPI's that are most influential on manufacturing costs. Various TEA's specific on CO₂ reduction to C₁ products are reported [3– 7]. Although these studies vary in approach and assumptions, a general consensus is that under the applied assumptions for process performances, electrocatalytic CO₂ reduction can be cost-competitive for formate and CO production. Electricity cost (between 0.01 and 0.06 US\$/kWh) and cell stacks costs are highlighted as highly influential factors on the manufacturing costs [4,5]. The large differences in assumed cell stack costs (920– 15,000 US\$/m² [4-6]) suggests uncertainty about the realistic costs. Current density (CD) is also commonly mentioned as highly influential. Increasing the partial CD decreases the overall cell stack costs because less cell area is required for a specific production target. However, Sargent et al. report a nonlinear response of the manufacturing cost on the CD for CO production. Increasing the CD from 100 to 300 mA/cm² results in a significant cost reduction, further increasing the CD to 1000 mA/cm² only slightly decreases the manufacturing costs [5]. With increasing CD, an increased cell potential translates into high electricity consumption owing to increased overpotentials. This is an important observation, because it shows that there is an upper limit (we suggest a value of 500 mA/cm² for CO production) at which the catalytic rate can be considered satisfactory. For formate production, lower CD already results in positive TEA [6] or shows less sensitivity in CD than for CO production [4]. The cell potential determines the energy consumption of electrocatalytic conversions. However, variations in the cell potential between 1.6 and 2.6 V seem to have a small influence on CO and formate manufacturing costs [4,5]. KPI's that influence the efforts for downstream processing (DSP) generally have a large influence on the manufacturing costs. Jiao et al. [6] report that the CO_2 conversion per pass strongly influences the manufacturing cost, especially for the production of CO, and specifies a desired conversion per pass of >30%. At low conversions per pass, the CO product stream contains large amount of CO₂. Removal of CO₂, often by pressure swing adsorption

Figure 1

(PSA) or membrane separation [8], is needed to achieve significant CO purity. In the case of formate production, the CO₂ stream that leaves the cell contains products formed owing to cathodic inefficiencies (mostly H_2). Removal of these products is crucial for recycling the CO_2 back into the process. At low CO_2 conversion per pass removal of H_2 is complicated, because the H_2 will be more dilute. For DSP of formate, it is crucial to produce concentrated product streams ($\sim 20 \text{ wt.\%}$) [9]. Formate has a high solubility even at low temperatures, and thus crystallization cannot be used for formate recovery. The energy demand for DSP methods (distillation of water for formate isolation and PSA for CO purification) as function of product purity is shown in Figure 1 to highlight the importance of product purity. For comparison, the energy demand for electrochemical CO production is ~ 5.5 mWh/tonne and for formate production \sim 3.4 MWh/tonne (assuming low-temperature electrolysis with 90% Faraday yield (FY) and $E_{cell} = 2.6 \text{ V}$).

The stability of the electrodes and membranes required for industrial viability varies from ~ 2.3 to 5 years [4,6]. However, stability measurements are time-consuming, and accelerated stability tests reduce reliability of the outcome. A more realistic research target is set by Jiao et al. [6], who define 100-day stability of the first target for an electrolyzer prototype.

In Table 1 we propose research targets for the KPIs for electrochemical production of formate and CO from



DSP energy demands for varying product purities. Thermodynamic energy demand for water removal as function of formate concentration in product stream (left graph) and estimated energy input for PSA as function of the CO₂/CO molar ratio in product. The formulas for calculating the energy demands are given in the section at the end of this review. Assumptions for PSA energy consumption: we assumed the energy consumption for state-of-the-art technology for precombustion CO₂ capture (95.7 kWh/ton CO2) [10]. Assumptions for water removal: thermodynamic energy demand for water removal (0.72 kWh/kg water) is built up from the energy required for heating water up from 21 to 100 °C and the heat of evaporation at 100 °C. PSA, pressure swing adsorption.

Table 1

Proposed research targets for electrochemical CO₂ reduction to formate and carbon monoxide. KPI Research targets Remarks CO₂ to formate CO₂ to CO Current density (mA/cm²) 200-400 300-500 For CO production: influential for CD < 300 mA/cm², less influential for $CD > 300 \text{ mA/cm}^2$ [5]. Lower CD is assumed for formate production [6]. CO₂ conversion per pass (%) >75 >30 The conversion per pass determines the purity of the gas that leaves the cell and required DSP to purify this. Product concentration in product stream mole fraction >0.3 >20 wt% The product purity affects the energy requirement for DSP Demonstrated system stability (days) 100 For industrial viability, 2.3-5 years is desired. For prototype demonstration, 100 days is specified as target [6] Cell potential (V) 2 - 2.6CO production by SOE has lower cell potential owing to thermal contribution to total energy demand.

CD, current density; CO, carbon monoxide; DSP, downstream processing; SOE, solid oxide electrolysis.

 CO_2 . These values are based on the assumed values that result in positive base cases in the highlighted TEA studies [3–7].

State of the art for CO₂ reduction to carbon monoxide and formate

In this paragraph, we review the performances in the state of the art for electrocatalytic CO and formate production and compare these with the research targets as we defined in Table 1. For the state of the art, we considered articles from the last two years, including low-temperature electrolysis and SOE. For low-temperature electrolysis, gas diffusion electrode (GDE) technology is the dominant method for promoting CO_2 transport to the cathode [11]. Several sophisticated catalyst designs are recently reported for formate production at very high CD with high FYs using GDE technology. A single atom Pb-alloyed Cu catalyst operates at 1 A/cm² with 92% FY [12]; a subnanostructured Bi catalyst operates at 500 mA/cm² at 92% FY [13], and a catalyst based on InP quantum dots operates at 930 mA/ cm² at 93% FY [14]. An Sn-Bi/SnO₂ catalyst showed stable catalytic operation over 2400 h at 100 mA/cm² at 95% FY. The same catalyst in a more relevant reactor produced a concentrated formate product stream (3.4 M, 15 wt%) with 100 h stable operation [15]. When operating at high CD, an alkaline environment is formed at the cathode. In this environment, CO₂ that transports through the porous layer of the GDE forms bicarbonate. Bicarbonate can accumulate, precipitate, and mechanically degrade and block the GDE. This 'salting out' is a common degradation pathway for GDE's [16]. When short durability measurements (>100 h) are performed, such slow degradation pathways can be overlooked, whereas a good understanding is required to achieve industrially relevant system lifetimes. Performing relevant durability measurements requires investing in automated equipment, including inline analysis and safeguarding controls, in which an industrial process with product streams suited for DSP is mimicked on the laboratory scale. Strikingly, besides the publication of Zhong et al. [15], we did not find reports that perform long-term stability measurements at high formate concentration. In most reports, the formate concentration and conversion per pass are not addressed at all, although these are crucial KPIs for industrial implementation. This is in sharp contrast with the very high CDs that are reported, which generally are higher than the suggested research target.

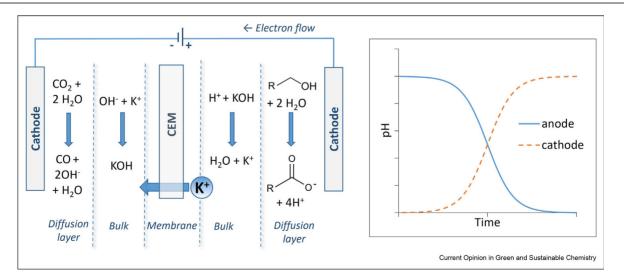
For the conversion of CO_2 to CO, high-temperature solid oxide electrolysis cells (generally operating between 600° and 850 °C) are the most mature technology [17]. Stable operation under industrially relevant conditions is demonstrated for almost 8000 h [18]. In solid oxide electrolysis cells, a large portion of the total energy demand for CO₂ conversion is supplied as heat, and the electrical energy demand is reduced. Partial CD above 1 A/cm^2 is achieved at cell potentials of 1.5 V or lower [18,19]. For comparison, state-of-the-art low-temperature CO production with GDE technology operates at 3.4 V with a stable CO partial CD around 700 mA/cm² [20]. Compared with other technologies discussed in this review, SOE for CO production appears most likely to be most competitive with fossil-based production technologies in the near future.

Paired electrolysis

The economics of electrochemical reduction processes can significantly improve when the oxidation of water at the anode is replaced with a valuable oxidation reaction (paired electrolysis) [3]. Paired electrolysis increases the total production capacity of the electrolyzer and appendages and reduces energy requirement compared with 'unpaired' redox reactions. In this paragraph, we discuss some requirements for successfully pairing redox reactions. We only consider low-temperature electrolysis because paired electrolysis for the SOE would be a completely new concept. In our opinion, to make an industrially feasible paired electrolysis process, it is advantageous to have synergy between the anodic and cathodic reactions. Examples are CO production coupled with chlorine evolution to produce feedstocks for phosgene [21] and the reduction of CO_2 to formate paired with biomass oxidation to formate [6,22]. Another notable example of synergy between anodic and cathodic reactions is reported by Whang et al. where formate production is combined with oxidation of hydrogen to form protons. The protons and formate combine in a porous membrane between anodic and cathodic compartments to form a pure stream of formic acid [14]. When pairing redox processes, compatibilities of pH and CD are important. The CD of both reactions must be in the same order, meaning a $CD \ge 200 \text{ mA/cm}^2$ is needed to match CO_2 reduction, whereas $>1000 \text{ mA/cm}^2$ can be coupled with proton exchange membrane (PEM) hydrogen evolution. Selective oxidation reactions at such high CD are rare. In a review by Janaky et al. on CO₂ reduction paired with valuable anodic reactions, the highlighted examples operate at lower CD than the state-of-the-art CO_2 reduction processes [23] and seem limited in CDof the anodic process. To pair redox reactions, pH compatibility of both reactions is needed for efficient ion transport. When the anodic process operates at high pH, no protons are available for transport to the

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Figure	2
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cathode, and other cations, such as potassium, can cross through a PEM. This is generally undesired, because this will increase the pH at the cathode and decrease that at the anode (Figure 2). Acid and base inputs for active pH control are very costly at industrial scale. Anion exchange membranes are a good alternative when sufficient OH⁻ is available at the cathode, but generally, this is not the case for CO₂ reduction reactions, because bicarbonates will form rapidly [24]. Bipolar membranes (BPMs) can operate at high anodic pH and cathode pH < 7 while maintaining the pH stable, but the long-term stability of BPMs at relevant catalytic conditions ($>>100 \text{ mA/cm}^2$) is not demonstrated, and this class of membranes is less mature than PEM technology. However, BPMs capable of operating at high CD are reported [25], and some benefits of BPMs in CO₂ reduction processes are reported, such as reduced crossover of molecules through the membrane [5,26]. Innovating BPMs to have stable operation at high CD ($>>100 \text{ mA/cm}^2$) can enable paired electrolysis processes with CO₂ reduction reactions. Alternatively, to ensure pH compatibility, it is preferred to operate the anodic reaction at low pH, so sufficient protons are available for transport to the cathode. However, developing oxidation reactions in the acidic electrolyte is challenging; generally, reactions suffer from lower selectivity, whereas anode materials are prone to corrode at low pH. Often noble metals (e.g. Ru, Ir) are used because of their stability, which is a huge cost driver for cell stacks. Alternative, cheap anode materials capable of operating in acidic electrolyte are generally oxygenates of for instance post-



Ion transport in an electrochemical cell. When applying a CEM with a high pH at the anode, alkali-metal cations cross the membrane (left image). This results in an increase in cathode pH and decrease in anode pH. CEM, cation exchange membrane.

transition metals, such as lead and tin. Such anodes are already applied for energy storage [26] and wastewater treatment [27]. Exploring their reactivity in electrosynthesis can be an interesting research line to enable paired electrolysis processes.

Conclusion

In summary, based on recently reported TEAs, we propose research targets for CD, CO₂ conversion per pass, product purity, cell potential and demonstrated stability for electrochemical CO production and formate production. We highlight the idea that product purity and demonstrated lifetime at relevant conditions should more commonly be addressed in low TRL research. Few mature technologies are available for pairing CO₂ reduction with valuable oxidation reactions. Generally, synthetic oxidation reaction operates at lower CD and thereby limits the activity of the total system. In addition, oxidation reactions generally operate at high pH. This causes an incompatibility in the pH between redox reactions; there are no protons available for transport from the anode to the cathode. To facilitate paired electrolysis with oxidation reactions operating at high pH and CO₂ reduction reactions, BPMs capable of stable operation at high CD must be developed. If valuable oxidation reactions in acidic electrolyte are developed, these can be paired with CO₂ reduction with well-developed PEM's.

Calculations for process energy consumption

Thermodynamic energy demand for water removal: The thermodynamic energy demand for water removal is calculated with the following formula:

Therm. energy demand =
$$\frac{d\left(\frac{kg}{m3}\right)*(100 - wt\% \text{ formate})}{d\left(\frac{kg}{m3}\right)*wt\% \text{ formate}}$$

where *d* is the density which was determined experimentally. The energy for evaporation of water is 0.72 kWh/kg water, which is the sum of the energy required to heat water from 22 to 100 °C (specific heat of water = 4.186 J/ kg/°C) and the energy to evaporate water at 100 °C (heat of evaporation is 40.65 kJ/mol). Conversion of J to Wh: 1 J = 0.000278 Wh. Energy demand for PSA energy consumption: We assumed the energy consumption of state-ofthe-art technology for precombustion CO₂ capture (95.7 kWh/ton CO₂). Conversion gives $4.21*10^{-6}$ MWh/ mol CO₂. The energy requirement is calculated with the following formula:

$$PSA \ energy\left(\frac{MWh}{ton\ CO}\right) = 4.21 \ * \ 10^{-6} \ \frac{MWh}{mol\ CO_2}$$
$$* \ \frac{mol\ CO_2}{mol\ CO} \ * \ 35071 \ \frac{mol\ CO}{ton\ CO}$$

where 35,071 is the number of moles CO_2 that goes into 1 tonne CO_2 .

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 article.

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