How to Minimize Faradaic Efficiency Error in Electrochemical CO_2 Reduction for Gas Products

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Abstract

Faradaic efficiency (FE) is an important metric for evaluating electrochemical processes, such as carbon dioxide reduction, that is often used for performance comparisons. Although the equation to calculate FE is well-known, details needed to yield accurate values are often overlooked, potentially leading to errors exceeding 100%. Avoiding any errors is crucial for drawing correct conclusions, especially during a catalysis optimization process. Factors with high potential for FE errors include incorrect mass flow rates and temperature values, imprecise gas chromatography calibrations, and uncorrected gas viscosities, while factors with less potential are also identified. This manuscript presents FE calculation guidelines intended for both newcomers and experts in the field of electrochemical fuel production.

Keywords

Carbon Dioxide, Electrolysis, Water Splitting, Gas Chromatograph Calibration, Gas Mixture Viscosity

Introduction

Renewable and decentralized production of fuels and chemicals via electrolysis has received a lot of attention recently. One of the main criteria to assess these electrochemical processes is the faradaic efficiency (FE), which describes how much of the charge passed by an electrical current goes into each product. While electrochemical water splitting is expected to yield only one cathodic product (hydrogen), other electrolysis processes such as CO_2 reduction (CO_2R) can yield a variety of products,[1] making the determination of the FE more complex. Although there have been some publications describing the FE calculation for gas products from electrochemical CO_2R,[2,3] it is clear that some details still get overlooked frequently and equations are sometimes used incorrectly. As shown in this article, it is crucial to pay attention to these details to prevent potential analytical errors in FE determination, which are shown can exceed 100%. This is especially important when catalysts are tuned carefully to gain incremental increases in FE for certain products.[4] These gains could easily be a result of imprecise FE calculations or conversely, potential improvements may go unnoticed. Therefore, it is necessary to elaborate on the determination of this very important value often used for performance comparisons across different studies.

Discussion

Generally, Equation (1) can be used to calculate the FE of a CO_2R product *i*:

$$FE_i = \frac{N_i \, n_e \, F}{Q} \tag{1}$$

with N_i as the number of moles of product i generated, n_e, the number of electrons necessary to form one product molecule from the reactant, F is the Faraday constant, and Q is the total charge passed during CO_2R testing. For the quantification of liquid products, nuclear magnetic resonance

(NMR) spectroscopy is often chosen,[2,5,6] while a gas chromatograph (GC) is typically used for gas products.

With continuous flow from the CO_2R cell to a GC (see Figure S1 for a simplified experimental setup), the calculation of the FE for a gas product *i* can be done with Equation (2):

$$FE_i = \frac{\dot{m}_{corr}\, p_{STP}\, x_i\, n_e\, F}{I\, R\, T_{STP}} \tag{2}$$

with $\dot{m}_{corr}$ as the corrected mass flow rate at the cathode outlet (measured in sccm), p_{STP} and T_{STP}, the pressure and temperature at standard conditions, x_i, the mole fraction of component *i* in the gas mixture, *I* gives the total current, and *R* is the gas constant. While Equation (2) is generally straightforward, several factors need to be considered to minimize errors. In the following paragraphs, the relative error (RE) of each variable in the numerator of Equation (2) to the FE was calculated according to Equation (3):

$$RE = \frac{Used\ value}{Actual\ value} - 1 \tag{3}$$

The individual error of each term in the denominator of Equation (2) can also be determined with Equation (3). However, to account for how error of a variable in the denominator contributes to error propagation in the FE equation, Equation (4) was used:

$$RE = \frac{Actual\ value}{Used\ value} - 1 \tag{4}$$

Positive error values calculated with Equations (3) and (4) indicate an FE overestimation, while negative error values indicate an FE underestimation.

1. Mass Flow Rate

As mentioned by Dutta et al.,[2] mass flow meters (MFMs) should be utilized to measure the cathode outlet flow rate and calculate the FE. Placing an MFM behind the cathode outlet has the additional advantage of being able to detect potential leaks, which would affect the interpretation

of CO_2R results, especially if hydrogen (H_2) leaks more than the other gases. Ratios between different product gases are often used to analyze CO_2R performance, and would be skewed if some gases leak disproportionally. Using the cathode inlet flow rate instead of the outlet flow rate can lead to rather large FE inaccuracies. As shown in **Figure 1**, the flow rate error can easily exceed 100% under certain conditions and lead to FE over- and underestimation.

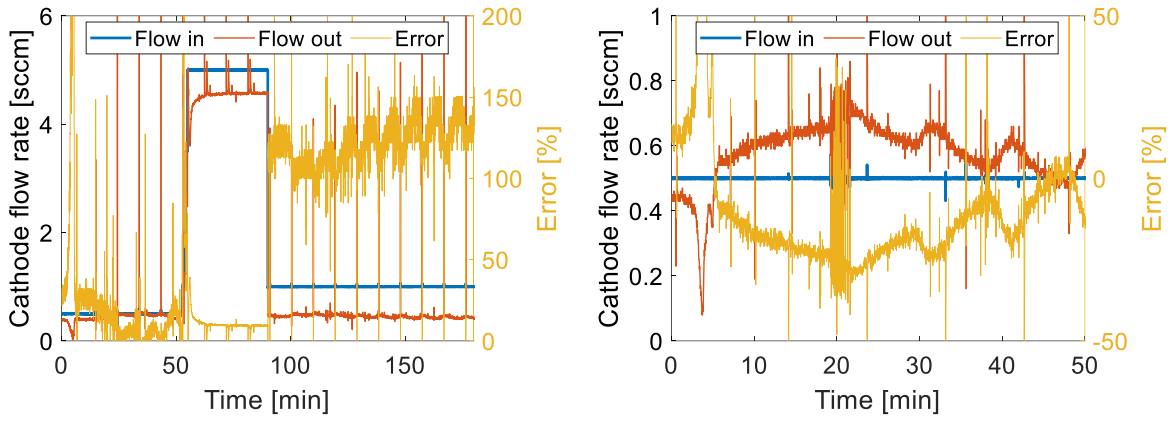

Figure 1 Measured cathode flow rates during CO_2R experiments with operating currents of 80 mA. Measured gas product concentrations are shown in Figure S2. (a) Product streams with comparatively low H_2 concentrations may lead to lower outlet flow rates due to reaction and crossover of CO_2. If the inlet flow rate is used for FE calculations, errors may exceed 100% in that case. (b) At high H_2 evolution rates, the outlet can exceed the inlet flow rate, leading to negative error values if the inlet flow rate is used to determine the FE. Such high H_2 evolution rates can, for example, be the result of cathode flooding.

Such mismatches between cathode inlet and outlet flow rates can be due to several reasons. On the one hand, when operating in alkaline conditions, CO_2 can react with hydroxide ions to form carbonate and bicarbonate species which can easily cross anion exchange membranes that are typically used under these conditions. Any CO_2 crossing the membrane as carbonate or bicarbonate

would result in a reduced cathode outlet flow rate. Additionally, formation of liquid and C_{2+} gas products is expected to lower the cathode outlet flow rate compared to the inlet, because the number of gas molecules is reduced at the outlet.[7] On the other hand, cathode outlet flow rates can exceed inlet flow rates during conditions of excessive H_2 evolution, because H_2 is not a direct product of the CO_2 feed gas, increasing the number of gas molecules leaving the cathode.

1.1 Temperature and Pressure

With commonly used MFMs that rely on differential pressure measurements it is important to note that they typically do not display units of mass, but rather standardized volumetric flow rates. To convert these volumetric flow rates to true mass or molar flow rates using the ideal gas law, the relevant standard conditions for temperature and pressure (p_{STP}, T_{STP}), based on the (adjustable) settings of the MFM, must be used.[8] The correct standard conditions need to be picked carefully and these values should not be replaced by the reaction or GC sample loop conditions (instead the GC calibration should be done at the relevant conditions). For example, MFMs from Alicat use default STP conditions of 25 °C and 1 atm.[8] If alternative STP conditions of 0 °C and 1 atm or reaction conditions of 80 °C and 1 atm are instead used for the FE calculation, errors of 9% or -16%, respectively, will be introduced. Similarly, if the electrolyzer is pressurized to 2 atm and this value is used for the FE calculation, it will result in a much larger error of 100%.

1.2 Gas Mix Viscosity

Additionally, it is important to note that differential pressure MFM measurements rely on the correct selection of the gas and their respective viscosity values.[9] While for small gas product concentrations it may be acceptable to select the reactant (CO_2) as the gas, errors of several percent can be reached when gas product concentrations exceed 10% in the effluent flow, especially when the viscosity values of the gases differ significantly, as in the case for ethylene (C_2H_4) and CO_2.

However, even if the wrong gas (mixture) is selected, a correction to the mass flow rate can easily be made using Equation (5):[9]

$$\dot{m}_{corr} = \dot{m}_{MFM} \frac{\mu_{sel}}{\mu_{mix}} \tag{5}$$

with $\dot{m}_{MFM}$ as the mass flow rate indicated by the MFM, μ_{sel} the viscosity of the selected gas and μ_{mix} the viscosity of the real gas (mixture). The μ_{mix} values are not listed for most gas mixes, but can be calculated from the viscosities of the individual gases as described by Davidson.[10] The method involves calculating the momentum fractions of a gas mixture and its corresponding fluidity (f_{mix}), which is correlated to the viscosity as shown in Equation (6):

$$\mu_{mix} = \frac{1}{f_{mix}} \tag{6}$$

According to Davidson,[10] f_{mix} can be calculated by Equation (7):

$$f_{mix} = \sum_{i,j} \left(\frac{y_i y_j}{\sqrt{\mu_i}\sqrt{\mu_j}} E_{i,j}{}^{A} \right) \tag{7}$$

where y_i and y_j describe the momentum fractions of gas components i and j, μ_i and μ_j the viscosities of components i and j, and $E_{i,j}$ the weighted mean efficiency of momentum transfer between two bodies, adjusted by an empirical exponent A which was found to be 0.375 by Davidson.[10] The efficiency $E_{i,j}$ can be determined using the masses of the participating molecules (m_i and m_j):

$$E_{i,j} = \frac{2\sqrt{m_i}\sqrt{m_j}}{m_i + m_j} \tag{8}$$

which are determined using the molar mass of the gas (M_i) and the Avogadro constant (N_A):

$$m_i = \frac{M_i}{N_A} \tag{9}$$

Finally, the momentum fraction of a gas component is calculated according to Equation (10):

$$y_i = \frac{x_i\sqrt{M_i}}{\sum_i\left(x_i\sqrt{M_i}\right)} \tag{10}$$

Using the equations above, mixture viscosities for some gas compositions that could be expected from CO_2R electrolyzers with elevated CO_2 conversion were calculated (see Python code in Supporting Information) and are displayed in **Table 1**.

Table 1 Viscosity values of gas mixes according to Davidson[10] that could exit a CO_2R electrolyzer and corresponding $\dot{m}_{corr}$ based on $\dot{m}_{MFM}$ = 10 sccm. Davidson mentioned a root-mean-square deviation of 1.99% for a range of binary mixtures.[10] However, it would be good to confirm viscosities experimentally for some of the gas mixes listed below. The following values were used as the viscosities of pure gases (with units of µPa s): H_2 (8.915355), carbon monoxide (CO, 17.649330), methane (CH_4, 11.075950), CO_2 (14.931840), C_2H_4 (10.318390).[11] While high CO concentrations in the gas stream lead to overestimations, high H_2, CH_4 and C_2H_4 concentrations yield underestimated mass flow rates without viscosity correction.

Gas mixture	**Calculated viscosity [µPa s]**	$\dot{m}_{corr}$ **[sccm]**	**Mass flow error to FE [%]**
70% CO, 30% CO_2	16.694355	8.94	11.9
40% H_2, 29% CO, 11% CH_4, 20% CO_2	15.302921	9.76	2.5
20% H_2, 20% CO, 10% C_2H_4, 50% CO_2	14.905622	10.02	-0.2
10% C_2H_4, 90% CO_2	14.473647	10.32	-3.1
70% H_2, 4% CO, 1% CH_4, 25% CO_2	14.328471	10.42	-4.0
70% H_2, 10% CO, 10% C_2H_4, 10% CO_2	13.295019	11.23	-11.0
40% C_2H_4, 60% CO_2	13.088345	11.41	-12.4
70% CH_4, 30% CO_2	12.740169	11.72	-14.7

2. Electrical Current

Furthermore, the total current *I* (and <u>not</u> the current density, which would give the FE units of area) should be averaged over a relevant timeframe (ensuring that the average current is not taken from a current spike only) before the injection of gas into the GC. Since electrodes with

areas significantly different from 1 cm^2 (or 1 m^2) are frequently assembled, errors above 100% could easily be added to the FE calculation if the current density is used. However, because the error here can be so significant, it is less likely to be unnoticed. Based on the author's experience, the current density is usually just displayed incorrectly in the FE equation but for actual calculations the correct current is used.[2] However, with electrode areas close to 1 cm^2 (or 1 m^2) the incorrect use of current density could lead to an unnoticed error. Additionally, for pulsed experiments only the faradaic (as opposed to capacitive) current should be considered.[12]

3. Mole Fractions from Gas Chromatography

To determine the mole fractions x_i accurately, it is highly encouraged to use automated sampling valves rather than manual syringe injections to increase reproducibility.[13] Furthermore, it is crucial to generate high-quality GC detector calibration curves, ideally using at least five points for each compound. Any GC peak area used for a calibration curve should be an average value from at least 3 injections with a low standard deviation. This requires waiting until the GC peak areas stabilize, which typically takes at least 30 minutes at flow rates below 10 sccm and with commonly used tubing between the mass flow controller and GC sample loop. Importantly, the GC needs to be calibrated in a relevant concentration range covering all the cathode outlet concentrations. As shown in **Figure 2**a-b, calibrating for low concentration ranges and extrapolating the calibration curves to larger concentrations can lead to significant deviations between calculated and actual concentration values.

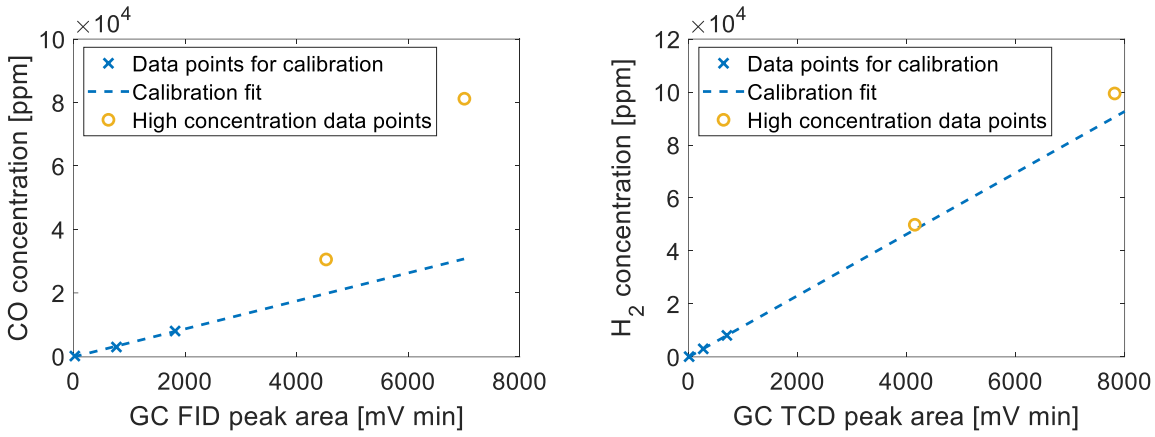

Figure 2 GC calibration curves for concentrations ranging from 100 ppm to 25% using a 1 ml sample loop. (a) CO concentration as a function of flame ionization detector (FID) peak area. At a peak area of ~7000 mV min, the calibration curve generated from low concentration data points would yield a CO concentration of ~3%, while the actual concentration is ~8% (roughly -63% error to the FE). (b) H_2 concentration as a function of thermal conductivity detector (TCD) peak area. The response of the TCD is closer to linear behavior than the FID at concentrations above 1%. (c) Different methods to fit the CH_4 concentration data points as a function of the GC FID area. Weighted fits were generated

with the weighted least squares method using a weighting factor of $\frac{1}{x}$, where x is the GC peak area.[14] (d) Low concentration range of (c), indicating significant deviation (up to -5000%) between data points and unweighted fits despite an R-squared value of 0.99 (**Table 2**).

The R-squared value is often used to confirm good representation of the data points by the calibration curve, but it can be extremely misleading. When back-calculated concentrations using the equations from the curve fits are compared against the known concentrations of the calibration standards, the relative error can be calculated using Equation (3). As shown in **Table 2** and **Figure 2**c-d, even with an R-squared value of 0.99 the average RE can still exceed 1000%. Therefore, it is recommended to prioritize the RE over the R-squared value when making an assessment about the calibration curve quality.[15]

Table 2 Relative error of GC calibration with 1 ml sample loop using different curve fitting methods. All percentage values listed here are calculated by taking the average of absolute values of the REs for each individual data point. The R-squared (R^2) values are shown for the (unweighted) linear fit. Weighted fits use a weighting factor of $\frac{1}{x}$, where x is the GC peak area. The last column displays the orders of magnitude covered by the fits ($log_{10}(max.concentration) - log_{10}(min.concentration)$), for example the CH_4 calibration data points range from 99.44 ppm to 25.01%.

	Gas	Linear fit	R^2 value	Weighted linear fit	Quadratic fit	Weighted quadratic fit	Orders of magnitude
TCD	H_2	369.76%	1.00	7.60%	52.67%	1.45%	3.29
FID	CO	39.98%	1.00	2.27%	3.73%	0.44%	2.49
	CH_4	1081.32%	0.99	11.47%	56.15%	2.20%	3.40
	C_2H_4	297.13%	1.00	5.29%	15.10%	1.03%	3.30
	Ethane	57.99%	1.00	1.96%	6.06%	0.52%	3.00

When looking at the average values of the REs in **Table 2**, it becomes clear that quadratic fits are generally superior over linear fits for the large concentration ranges covered by these fits since the FID shows nonlinear behavior at concentrations exceeding 1%.[16] However, a linear fit is generally considered more robust in the concentration range where linear behavior is expected and when a small amount of data points is available for the calibration curve. As shown in Figure S3, a quadratic fit should not be considered reliable with only three data points. It is possible to get the FID response closer to linear behavior (and reduce the RE of linear fits) by reducing the GC sample loop size (Table S1). Furthermore, the cathode outlet may be diluted with an inert gas before injection into the GC (Figure S1b),[17] or the compounds which are typically quantified with the FID could alternatively be analyzed with the TCD, which may be more suitable in the concentration range above 1% (Table S2).

Additionally, ordinary least squares curve fitting typically leads to biasing the fit in favor of the high concentration calibration standards, which can lead to large REs in the low concentration range, especially if the fit spans several orders of magnitude (**Figure 2**c-d).[14] As shown in **Figure 2**d, the RE can exceed -1000% for the lowest concentration data points. To reduce the RE across the entire range of relevant concentrations, weighted least squares fits with a weighting factor of $\frac{1}{x}$, where x is the GC peak area, can be used.[14] As shown in **Table 2**, the average REs are significantly lower using the weighted fits, making them highly suitable when calibration curves spanning several orders of magnitude are required. An Excel Spreadsheet detailing the steps for creating weighted fits can be found in the Supporting Material.

Aside from low REs across the calibration range, the effective carbon number can be used to ensure good FID calibration curves have been created (or to estimate calibration curves for compounds without calibration standards). For example, CH_4 should yield peak areas half that of

C_2H_4 or ethane (C_2H_6), when the concentrations are kept equal.[18] Additionally, CO and CO_2 should yield the same response as CH_4 since these are typically methanized before reaching the FID. Significant differences between the CO, CO_2 and CH_4 FID calibration curves indicates incomplete conversion of CO or CO_2 to CH_4, especially when it occurs at high concentrations. In that case, it is recommended to reduce the GC sample loop volume to reduce the amount of CO or CO_2 that needs to be methanized, and compare the peak areas again.

4. Number of Electrons

Finally, the number of electrons n_e needs to selected correctly. While these numbers can easily be calculated from the differences in oxidation states between reactant and product, it is worth noting that n_e is reduced when the same reduction product is formed from CO compared to CO_2. Additionally, when calculating n_e from the oxidation state difference between reactant and product, this difference needs to be multiplied by the number of reactant molecules needed to form one product molecule to get the correct n_e. For example, multi-carbon products such as C_2H_4 or propylene (C_3H_6) require two or three reactant molecules of CO/CO_2, respectively. Ignoring these principles could lead to FE errors exceeding -50%. **Table 3** lists n_e for more and less common gas products from CO_2R.

Table 3 Number of electrons (n_e) for gas products seen during CO_2R experiments.

Gas product	n_e
H_2	2
CO	2
CH_4	8
C_2H_4	12
C_2H_6	14
C_3H_6	18
Propane	20

5. Summary

As shown in the above paragraphs, there are multiple factors which can significantly skew FE values. A summary of these factors is shown in **Table 4**. Since the product concentrations determined from GC injections also affect the gas mixture viscosity correction, it becomes even more important to use high-quality GC calibration curves.

Table 4 Summary of possible error sources for FE calculation. It should be noted that the potential errors are shown in absolute values and could always be larger than indicated in this table, but the values displayed here are based on calculations and observations by the author.

Factor	|Potential error|	Risk of being unnoticed
Inlet instead of outlet flow rate	> 100%	High
Wrong temperature value	> 15%	High
Wrong pressure value	> 100%	Low
Gas mixture viscosity uncorrected	> 10%	Very high
Current density instead of current	> 100%	Low
Nonlinear GC detector behavior	> 50%	High
Improper GC calibration fit	> 100%	High
Wrong number of electrons	> 50%	Low

Conclusions

Determining precise FE values is crucial to compare performance across different studies. This manuscript outlines some of the more and less common FE calculation errors and explains how to avoid them. Some errors should become obvious to anyone looking at their data in sufficient detail, while other error sources can be difficult to spot. For example, not measuring the cathode outlet flowrate or GC peak areas at high analyte concentrations, would make it difficult to judge the difference between assumed and actual values. However, resulting errors can surpass 100% and could easily exceed any measured performance gains, especially at conditions of elevated reactant utilization with high product concentrations and differences between inlet and

outlet flow rates. Therefore, to ensure comparability between future reports, it is highly recommended to check for the error sources mentioned in this manuscript.

Supporting Information

Illustration of an experimental setup for quantification of gas products, measured gas product concentrations for results shown in Figure 1, additional GC calibration graph, and relative error values for GC calibration curves (PDF). Spreadsheet for creating weighted GC calibration curve fits (XLSX). Python code for calculating gas mix viscosities (TXT).

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