

SUPPORTING INFORMATION

Rigorous Evaluation of Liquid Products in High-rate CO₂/CO Electrolysis

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Materials

Anion exchange membrane (AEM, Fumasep FAA-3-PK-75), cation exchange membrane (Nafion™ 212), bipolar membrane (BPM, Fumasep FBM) and gas-diffusion electrode (GDE, Sigracet 39 BB) purchased from Fuel Cell Store were employed. Iridium dioxide (IrO₂) coated on GDE was used as an anode in flow electrolyzers of high-rate electrolysis. For exploring the crossover of hydrated ionic species with different ion-selective membranes, potassium bicarbonate (KHCO₃, ≥99.99% metals basis), potassium carbonate (K₂CO₃, GR ≥99.5%), potassium hydroxide (KOH, ≥95%), lithium sulfate (Li₂SO₄, ≥99.98%) and caesium carbonate (Cs₂CO₃, 99.9%) were utilized without further purification.

By direct current magnetron sputtering, Cu catalyst layers were prepared on top of microporous layer of gas-diffusion electrodes. The thickness of the Cu layers can be controlled via the deposition time of magnetron sputtering. In addition, a fixed active geometric surface area (2 cm²) of Cu layer was used for all the tests in this study.

Water crossover measurement via an AEM

For studying the water crossover accompanied with different anion species through the AEM, the CO₂ electrolysis was carried out in a three-chamber flow electrolyzer made from Teflon at ambient temperature and pressure. A constant current density of 200 mA/cm² was applied for all the tests (the active geometric surface area was 2 cm²).

It should be noted that water in the catholyte should also be consumed via involving in the CO₂ reduction and H₂ evolution. In addition, the gas flow in the gas chamber may enhance the evaporation of water at the GDE/catholyte interface. Thus, the utilization of the variation in the catholyte volume could lead a slight overestimation of water crossover (Figure S6).

The cathodic reactions consume water, and this water consumption rate depends on catalytic selectivity. Although the anodic reactions also consume or generate water, it is much easier to calculate the related water variation of the anodic reactions compared to that of the cathodic reactions (more details in the following section of consideration of anodic reactions). Thereby, the anolyte volume change was measured for the water crossover volume.

However, for the transportation of CO₃²⁻ as domination anion via membranes, if 0.5 M K₂CO₃ is also used as the anolyte, the transformation of 0.5 M K₂CO₃ to 1 M KHCO₃ in the anolyte could occur, which can add extra volume for the anolyte during CO₂ electrolysis. Thus 1 M KHCO₃ anolyte was used for accurately measuring water crossover when transferring CO₃²⁻, as shown in Figure S2.

To ensure the transportation of HCO₃⁻ as domination anion via the AEM, both catholyte and anolyte reservoirs were filled with 50 ml 1 M KHCO₃, and the 1 M KHCO₃ catholyte was purged with CO₂ over the duration of electrolysis, as shown in Figure S1. In addition, CO₂ was purged into gas compartment at a constant flowrate of 45 ml/min. Before starting electrolysis, 1 M KHCO₃ catholyte was bubbled with CO₂ for 30 min. The catholyte pH was detected as a function of electrolysis time, showing that while there was a slight increase in catholyte pH, catholyte pH was maintained at no more than 8.8 over the course of the electrolysis, as shown in Figure S4.

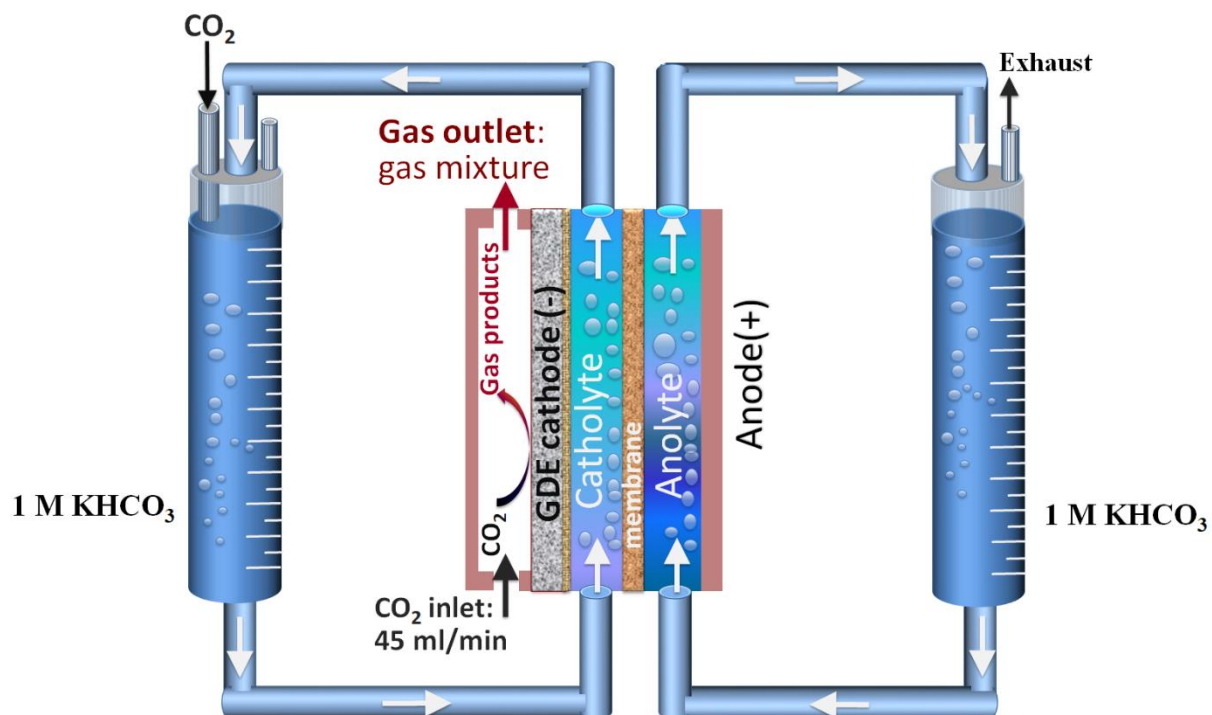


Figure S1. Schematic illustration of flow cell setup for electrocatalytic CO₂ reduction with CO₂-bubbled 1 M KHCO₃ catholyte reservoir (*i.e.* measuring cylinder).

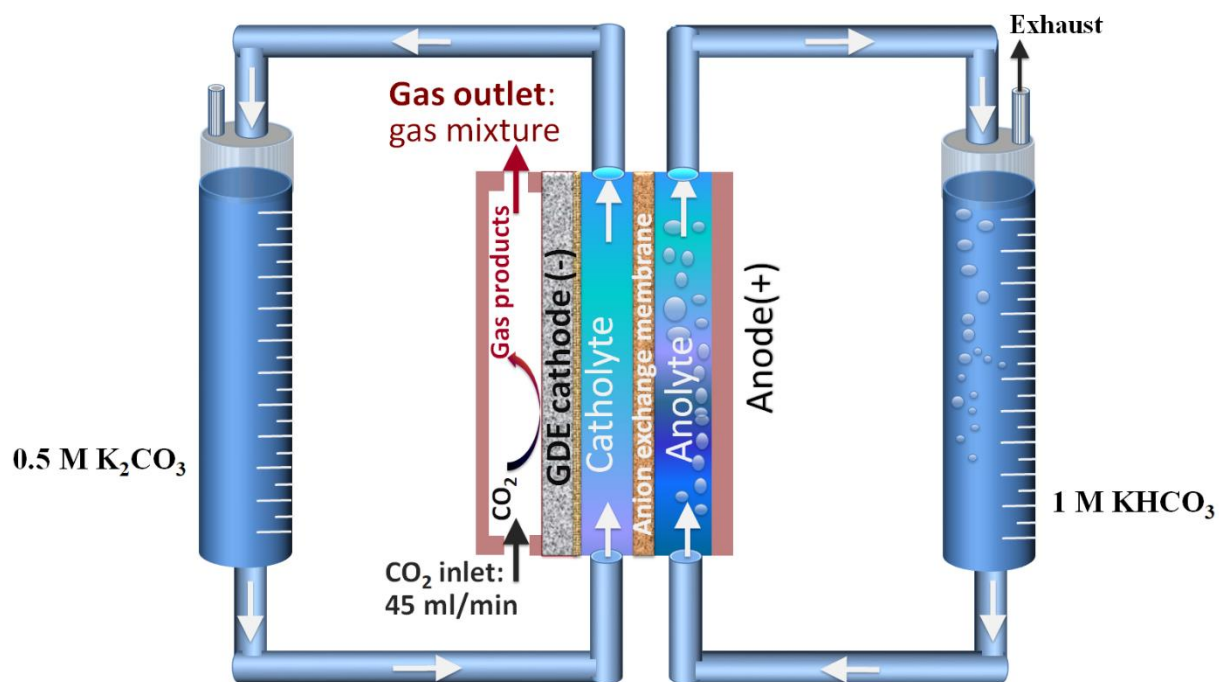


Figure S2. Schematic illustration of flow electrolyzer for electrocatalytic CO₂ reduction in 0.5 M K₂CO₃ (*i.e.* measuring cylinder). 1 M KHCO₃ was used as the anolyte.

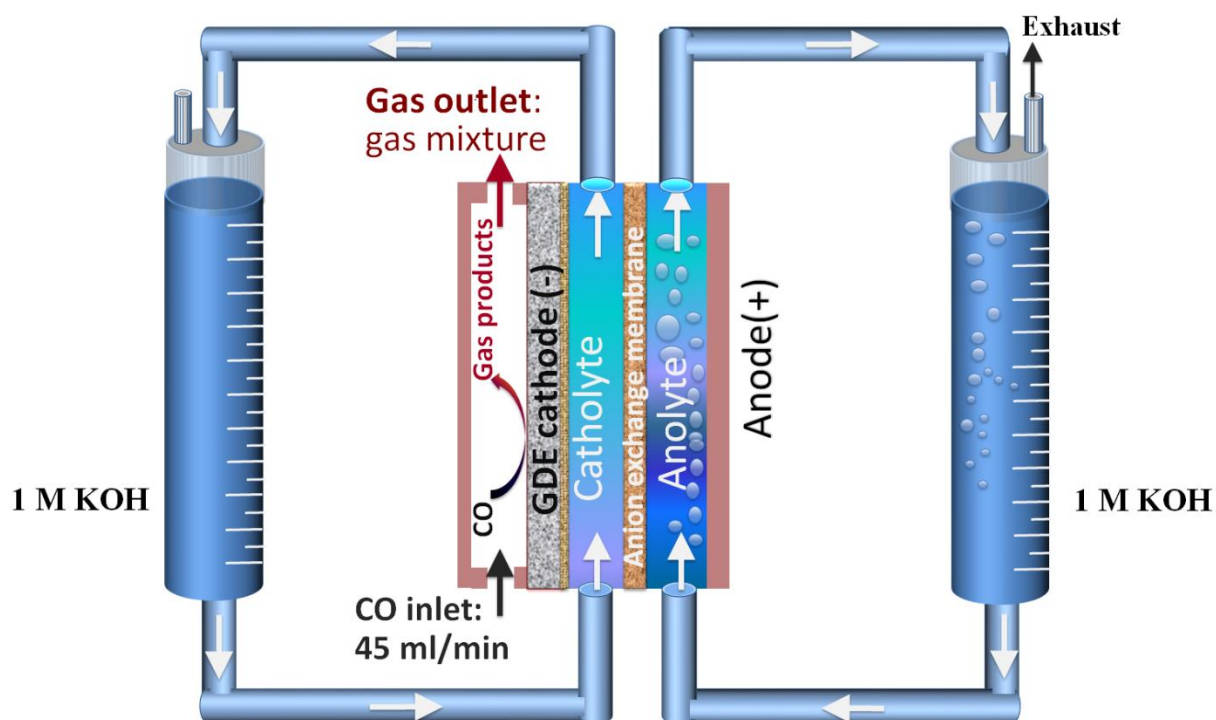


Figure S3. Schematic illustration of flow electrolyzer for electrochemical reduction of CO in 1 M KOH (*i.e.* measuring cylinder). For simplification, Ar purged into the gas chamber instead of CO here, which can avoid the effect of water crossover via the transportation of anionic products through AEMs in CO reduction (due to significant amounts of anionic products could be formed during CO reduction in 1 M KOH¹).

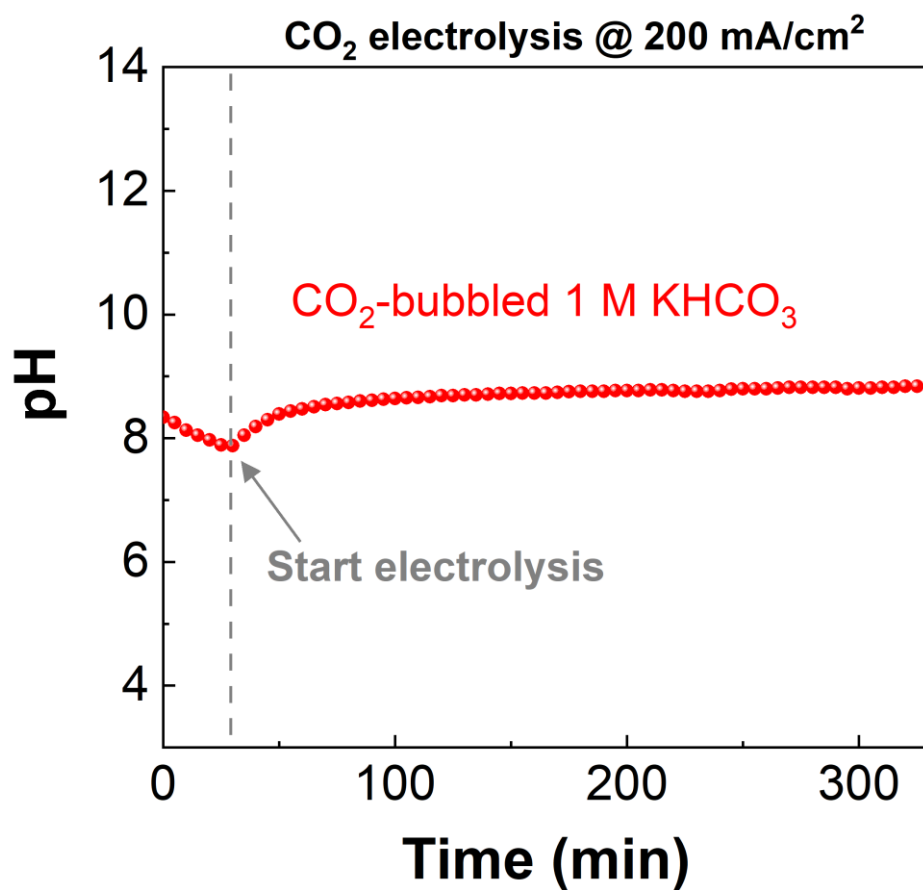


Figure S4. Catholyte pH over the course of CO₂ reduction electrolysis. 1 M KHCO₃ was used as initial catholyte (50 ml), and catholyte reservoir was always purged with CO₂ (the catholyte was bubbled with CO₂ in the initial 30 min before the electrolysis).

Calculation of the carbonate/bicarbonate ratio

The correlation between pH of a buffer solution and the carbonate/bicarbonate ratio can be expressed by the Henderson–Hasselbalch equation:

$$pH = pK_a + \log_{10}\left(\frac{[CO_3^{2-}]}{[HCO_3^-]}\right) \quad (S1)$$

where pKa is the acid dissociation constant, and $[CO_3^{2-}]/[HCO_3^-]$ is the concentration ratio of CO_3^{2-} to HCO_3^- . Here, the pKa is 10.3 at 25 °C based on the below equation:



* This is at a CO_2 partial pressure of 1 bar in 1 M HCO_3^- .

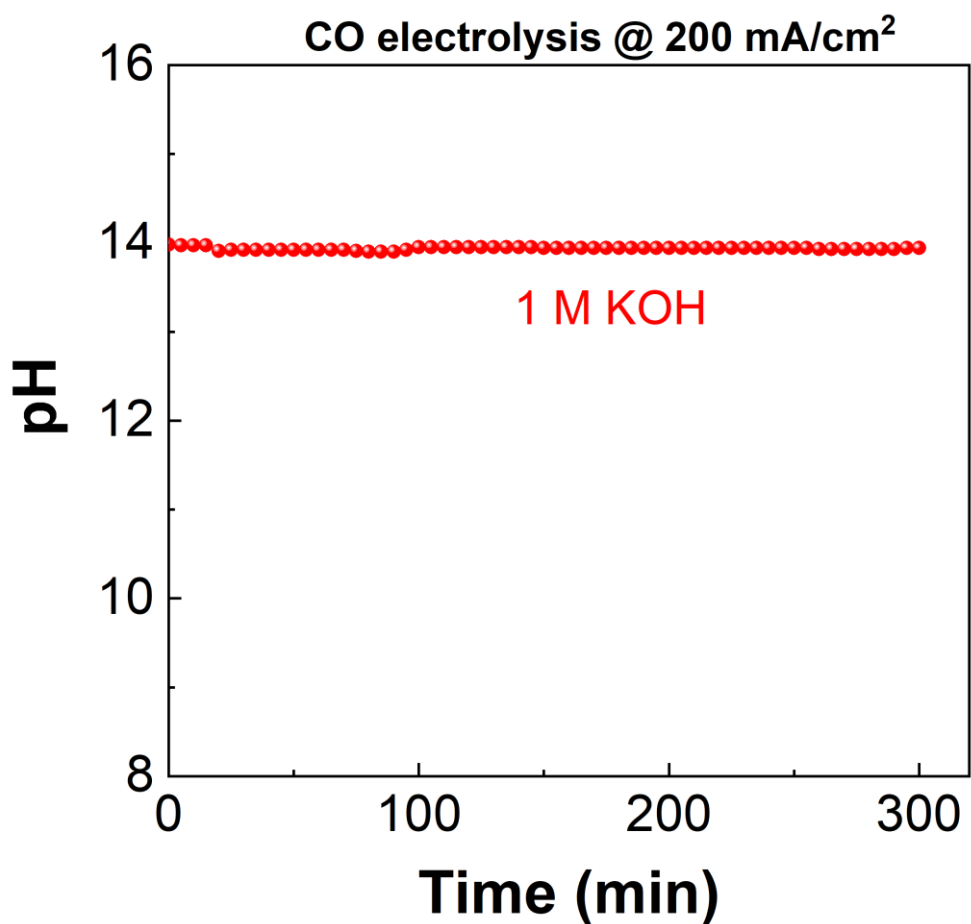


Figure S5. Catholyte pH over the course of electrolysis when feeding Ar/CO into the gas chamber of flow-electrolyzers in 1 M KOH (Figure S3 shows the schematic illustration of flow electrolyzer for CO electrolysis).

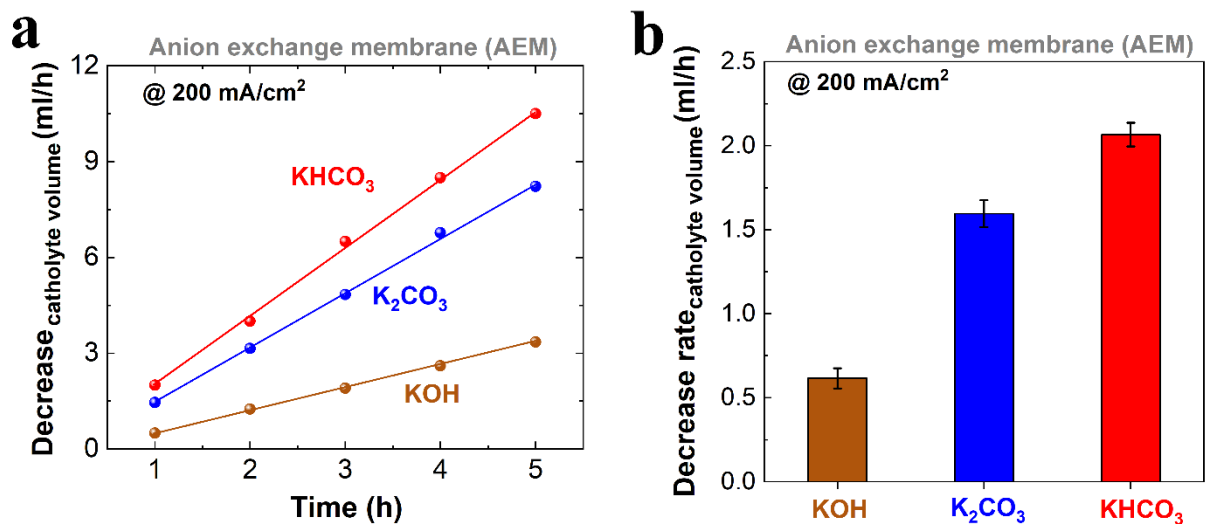
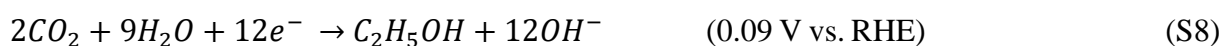
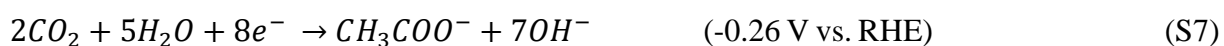


Figure S6 Comparison of the variation in catholyte volume when using different anions with an AEM. (a) Catholyte volume change over electrolysis. (b) Estimated decrease rate of catholyte volume.

The cathodic reactions

In the process of electrochemical conversion of CO₂, CO₂ and H₂O can be electrochemically reduced to a variety of gaseous and liquid products based on the below reactions³⁻⁶:



The competing H₂ evolution is always accompanied with CO₂ reduction:³



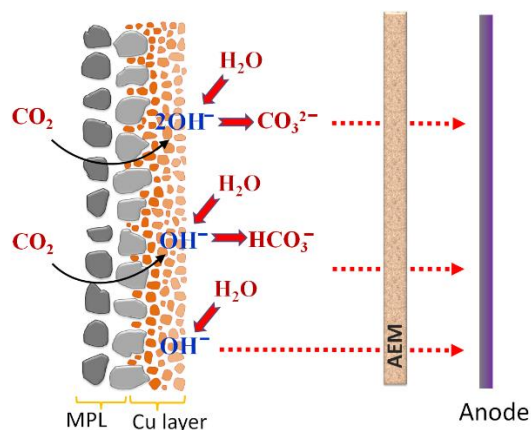
Due to the above cathodic reactions (Equation S(3-9)), different products formation consume distinct amounts of water molecules when transferring one electron. In other words, the water consumption via the cathodic reactions at a constant current is dependent on catalytic selectivity.

The water consumption rate (ml/h) for a certain product formation can be expressed as:

$$\phi_{water\ to\ x} = \left(n_{water} \times \frac{Q_{tot} \times FE_x}{F} \right) \times V_m \quad (S10)$$

where n_{water} is the number of water molecules consumed for transferring per electron during a certain product formation in the cathodic reactions. Q_{tot} and FE_x are total charge passed through the cathode per hour (i.e 1440 C) in the cathodic reactions and Faradaic efficiency (FE) for one certain product, respectively. F is Faradaic constant, and V_m is the molar volume of water (18 ml/mol). Based on the equation S10, we can get the total water consumption rate (0.174 ml/h) in the cathodic reactions in CO₂ reduction, as shown in Table S1.

It should be noted that most of the above water consumption creates OH⁻, which directly transfer via an AEM in the form of OH⁻ or reacts with CO₂ to form (bi)carbonate and then transfer via an AEM in the form of (bi)carbonate, as shown in Scheme S1.



Scheme S1. Water consumption in the cathodic reactions, forming OH^- and related crossover via an AEM in the form of OH^- or (bi)carbonate (OH^- reacts with CO_2 to form (bi)carbonate).

Table S1. Faradaic efficiencies for major products at 200 mA/cm^2 .⁷

	C_2H_4	Ethanol	CO	CH_4	n-Propanol	Acetate	Formate	H_2	Total
Faradaic efficiency (%)	43.3	18.4	15.1	3.0	5.0	3.1	3.0	5.4	96.3
n_{water}	8/12	9/12	1/2	6/8	13/18	5/8	1/2	2/2	0.65
$\Phi_{\text{water to } x}$ (ml/h)	0.0776	0.037	0.0203	0.006	0.0098	0.005	0.004	0.0145	0.174

(The catalytic selectivity over Cu sputtered on GDEs were already tested systematically in our previous work under identical conditions^{2,7}, and the catalytic selectivity in CO_2 reduction is nearly independent of the bulk pH of electrolytes², thus the major products selectivity was adapted based on ref. 7).

Theoretical estimation of crossed molecular ratio of H₂O/ion species

The molecular ratio of H₂O/ion, crossed over via membranes, can be written as:

$$\text{Crossover ratio}(\text{H}_2\text{O}/\text{ion}) = \frac{(\phi_{\text{H}_2\text{O}} \times \rho)/M_{\text{H}_2\text{O}}}{\frac{Q}{F \times n_c}} \quad (\text{S11})$$

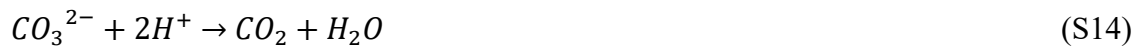
where $\phi_{\text{H}_2\text{O}}$ and ρ are the water crossover rate (ml/h) via the membrane, the density of water (g/ml) at room temperature and atmospheric pressure, respectively. $M_{\text{H}_2\text{O}}$ is the water molecular weight, Q is total charge passed through the membrane per hour, F is Faraday constant, and n_c is the charge number for each anion/cation.

Consideration of anodic reactions

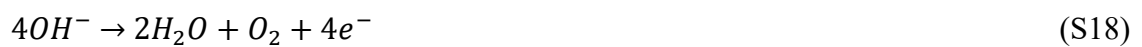
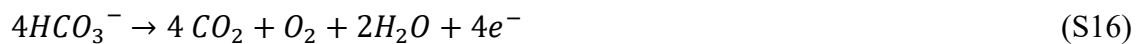
It should be noted that the anodic reactions also influence the variation in the anolyte volume by consuming water or generating water. The water oxidation reaction consumes water via the following equation:



H⁺ generation via this water oxidation (Equation S12) leads to a low pH in the vicinity of the anode. Thus, HCO₃⁻, CO₃²⁻ or OH⁻ coming from catholyte via AEMs will neutralize H⁺ near the anode, generating water via the below reactions⁸:



Here, Equation S12 can be combined with the neutralization reactions (Equation S(13-15)) to get straightforward equations² as follows:



From the Equation S16 and S18, it can be seen that the 0.5 water molecule will be added into the anolyte via the anodic reactions when transferring each HCO₃⁻ or OH⁻ via an AEM,

respectively. Notably, there will be no variation in water volume (the water consumption equals the water generation) if CO_3^{2-} transfers to the anolyte via the AEM (as shown in Equation S17). These findings indicate that the discrepancy for the crossed molecular ratio of $\text{H}_2\text{O}/\text{anion}$ (i.e. the crossed water/charge ratio) should be 0.5 with and without the consideration of the above anodic reactions when transferring HCO_3^- or OH^- . Thus, with considering the anodic reactions, the corrected average number of water molecule should be ~ 1.5 and ~ 6.2 when transferring each HCO_3^- and OH^- , respectively (Figure S7a), which is slightly smaller than those without considering anodic reactions (Figure S7b).

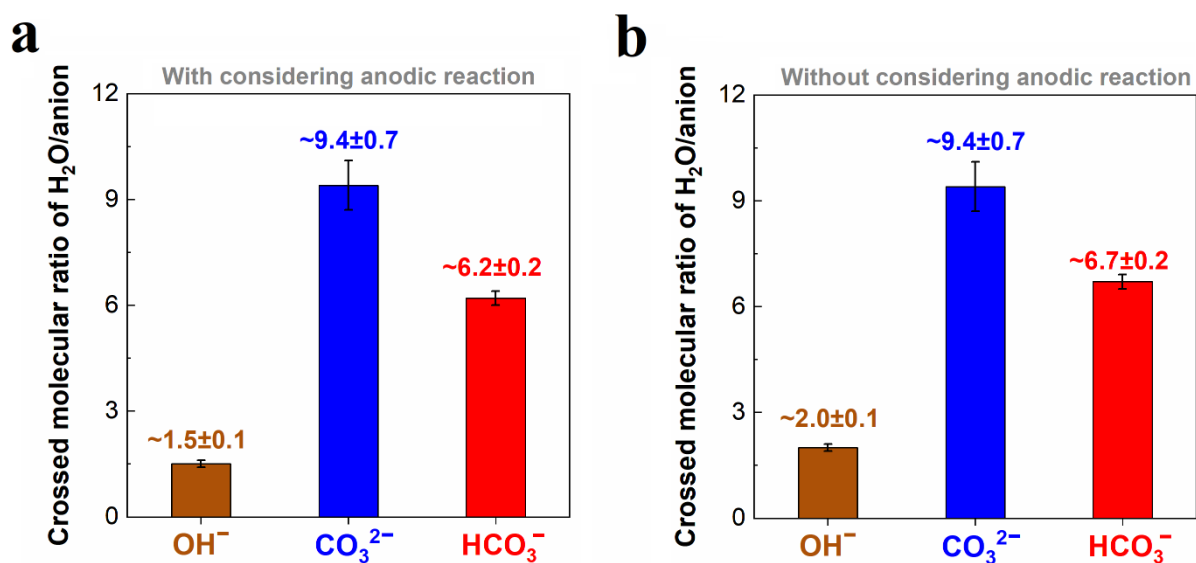


Figure S7. Molecular ratio of water/anion crossed through the AEM with (a) and without (b) considering anodic reactions.

With the use of a CEM, K_2CO_3 and Cs_2CO_3 solutions were utilized for exploring water crossover issue when transferring K^+ and Cs^+ , respectively. Thus, according to the Equation S17, the anodic reactions should not contribute to the variation in anolyte volume when using carbonate solutions. For studying the water crossover issue with Li^+ , Li_2SO_4 electrolyte was used owing to that Li_2CO_3 has a very low solubility. Based on equation S12, 0.5 water molecule will be consumed in the anolyte when transferring each Li^+ (i.e. each charge) via an CEM. Thereby, the corrected molecular ratio of $\text{H}_2\text{O}/\text{Li}^+$ crossed through the CEM should be ~ 5.6 , as shown in Figure S8a.

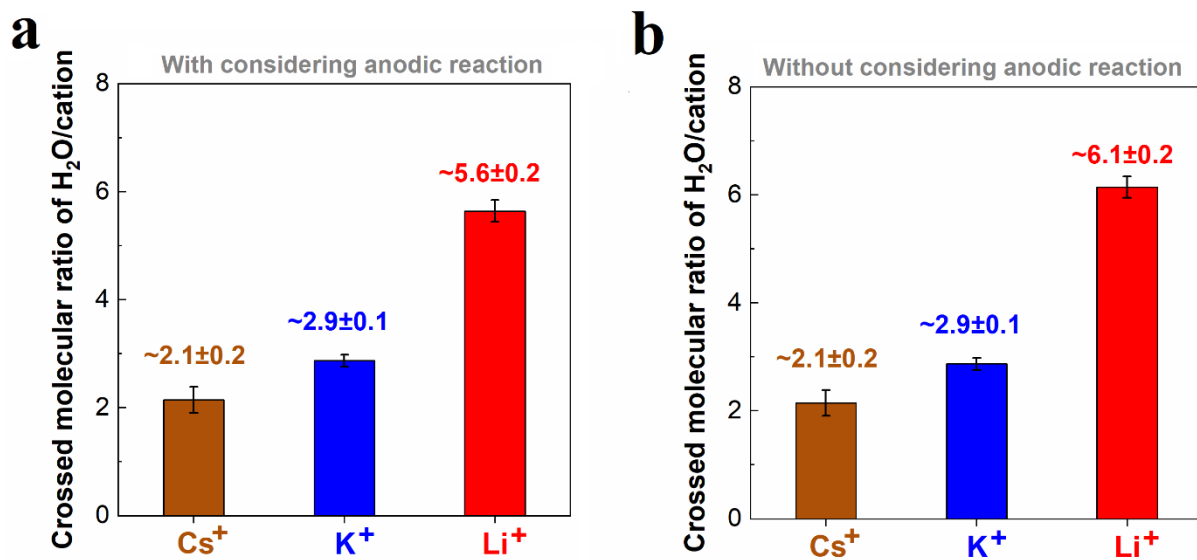


Figure S8. Molecular ratio of water/cation crossed through the CEM with (a) and without (b) considering anodic reactions.

Water crossover as a function of current density

The water crossover volume was measured at various current densities in the typical KHCO_3 electrolyte with an AEM. We found a linear relationship between the water crossover volume and current densities at a fixed electrolysis time, as shown in Figure S9. This finding is attributed to that the amount of water crossover is correlated with the total charge passed through the membranes if the charge-carrying species via the AEM is fixed (i.e. hydration number is fixed).

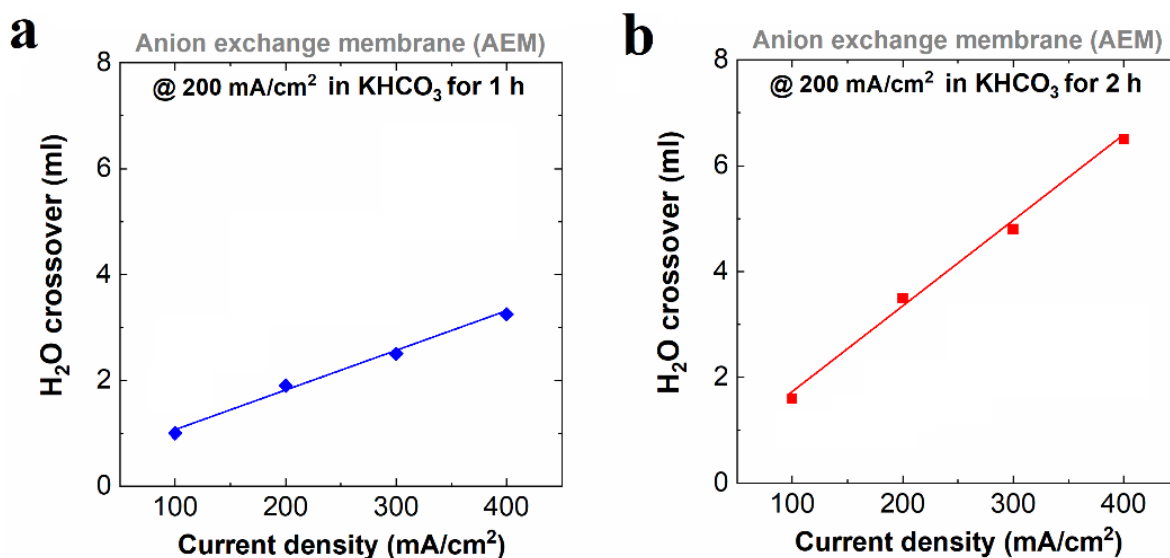


Figure S9. Water crossover volume as a function of current density when transferring HCO_3^- via the AEM after 1 h (a) and 2 h (b), respectively.

Theoretical estimation of H⁺ concentration in the anolyte

The pH decreases locally at the anode/anolyte interface over electrolysis, because of the H⁺ produced by the anodic oxygen evolution reaction (Equation S12) shown as below:



Without using a buffering electrolyte, the substantial amount of H⁺ generated via oxygen evolution reaction at high-rate electrolysis may result in an acidification of anolyte. Here, by assuming that the produced H⁺ is collected in the anolyte (50 ml) during CO₂ reduction in 2 M Li₂SO₄, the H⁺ concentration in the anolyte over electrolysis can be expressed as:

$$C_{H^+} = \frac{Q_{tot}}{FV_A} = \frac{It}{FV_A} \quad (S19)$$

where Q_{tot} , F and V_A are total charge passed through the anode, Faraday constant and anolyte volume (50 ml), respectively. I is the current and t is the electrolysis time. All our tests were performed at 200 mA/cm², and the cathode with 2 cm² geometric active area was used. According to Equation S19, we can easily get H⁺ concentration in the anolyte as a function of electrolysis time in 2 M Li₂SO₄, as shown in Table S2.

Table S2. The concentration of H⁺ in the anolyte at 200 mA/cm² in 2 M Li₂SO₄ over CO₂ reduction electrolysis (assuming that no H⁺ transports to catholyte via the CEM and ignoring the anolyte volume variation). The cathode with 2 cm² geometric active area was used.

Electrolysis time (h)	Total current (mA)	Concentration of H ⁺ (M)
1	400 mA	0.298
2	400 mA	0.597
3	400 mA	0.895
4	400 mA	1.194
5	400 mA	1.492

Water crossover via a CEM when using Na⁺

Considering the fact that the electrolyte containing Na⁺ is frequently utilized in CO₂ electrolysis, we also studied the water crossover issue via a CEM at flowing-catholyte electrolyzers using Na⁺. The initial test with 2 M Na₂CO₃ electrolyte failed, due to a part of Na₂CO₃ transformed to NaHCO₃ in the anolyte via CO₂ generation, creating precipitation (relatively low solubility of NaHCO₃). Thus, a mixture of 1.3 M Na₂SO₄ and 0.5 M Na₂CO₃ was used as the anolyte. In addition, this anolyte mixture containing 0.5 M CO₃²⁻ can avoid the variation in anolyte volume derived from the anodic reactions (Equation S17) for slightly more than 3 h.

Figure S10 shows water crossover from anolyte to catholyte via a CEM when transporting Na⁺. Based on Na⁺ data in the initial 3 h electrolysis, the molecular ratio of H₂O/Na⁺ crossed through the CEM was found to be ~4.7, which is in line with the hydration number of Na⁺ (4±1).⁹ The corresponding water crossover rate was ~0.89 μl/C when transferring Na⁺ via the CEM.

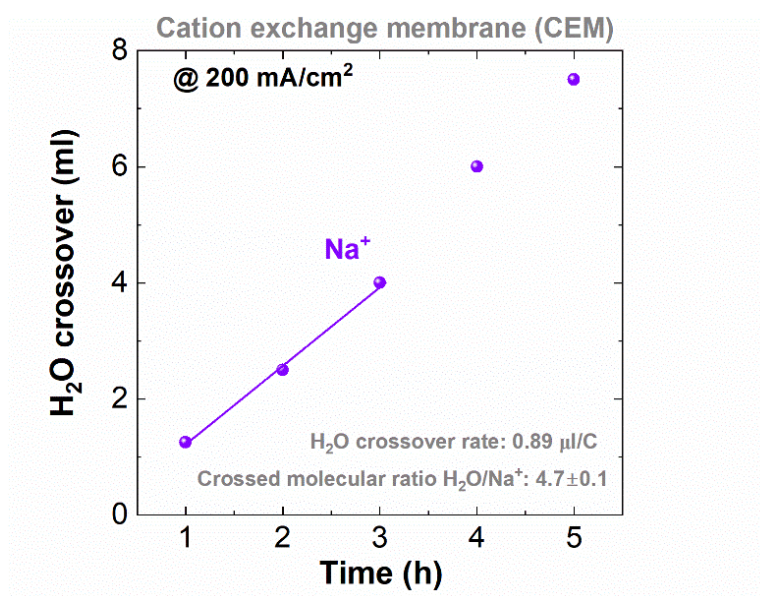


Figure S10. Water crossover from anolyte to catholyte via a CEM when transporting Na⁺ over electrolysis (variation in anolyte volume was utilized here).

Typical cell potentials of the electrolyzers

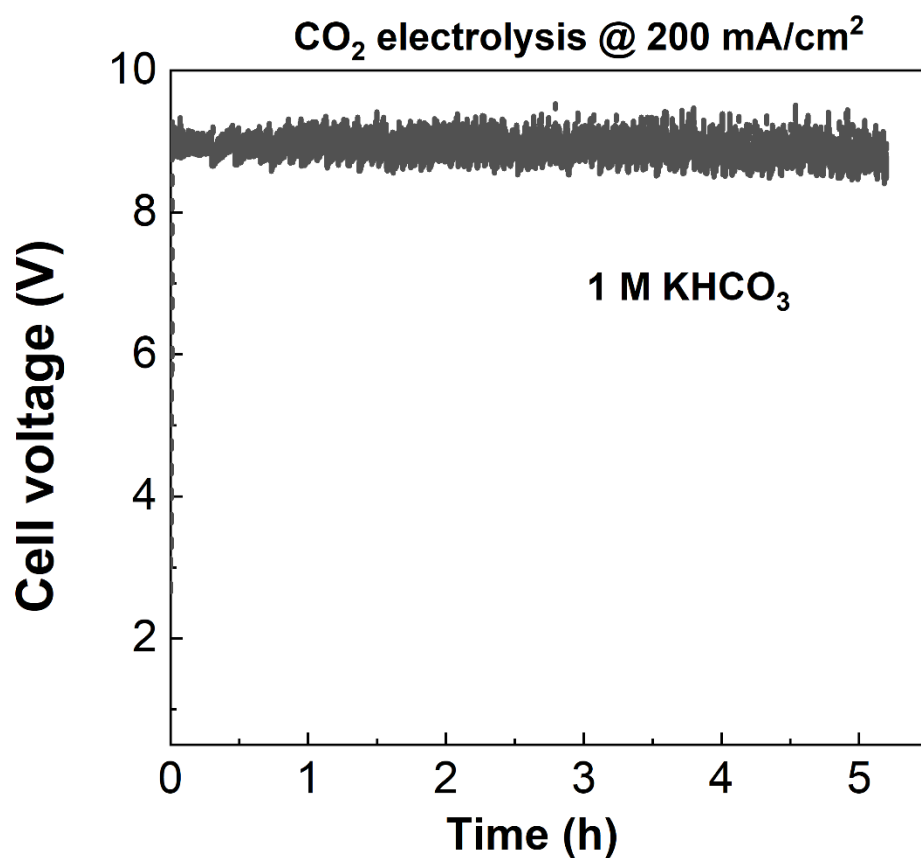


Figure S11. Cell voltage as a function of time when using the AEM over the course of CO₂ reduction electrolysis. 1 M KHCO₃ was used as the initial catholyte and anolyte. The distance between the Cu cathode and the membrane was ~15 mm (the distance between the membrane and the anode was ~3 mm).

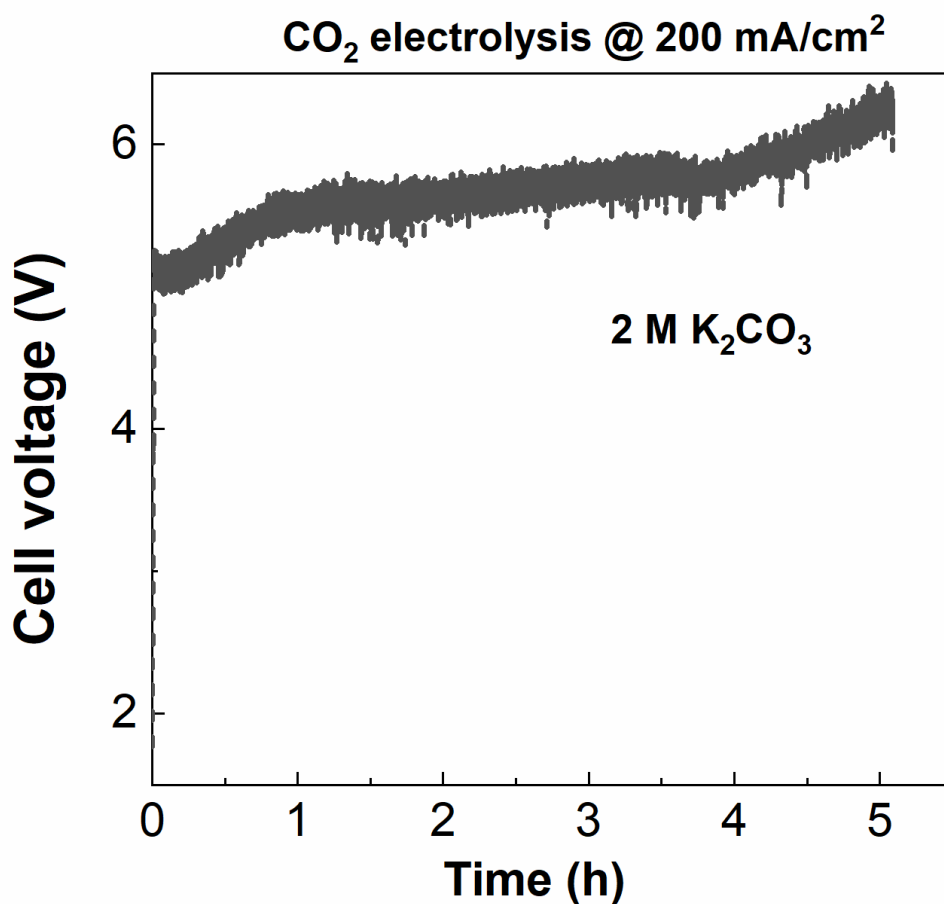


Figure S12. Cell voltage as a function of time when using the CEM over the course of CO₂ reduction electrolysis. 2 M K₂CO₃ was used as the initial catholyte and anolyte. The distance between the Cu cathode and the membrane was ~15 mm (the distance between the membrane and the anode was ~3 mm).

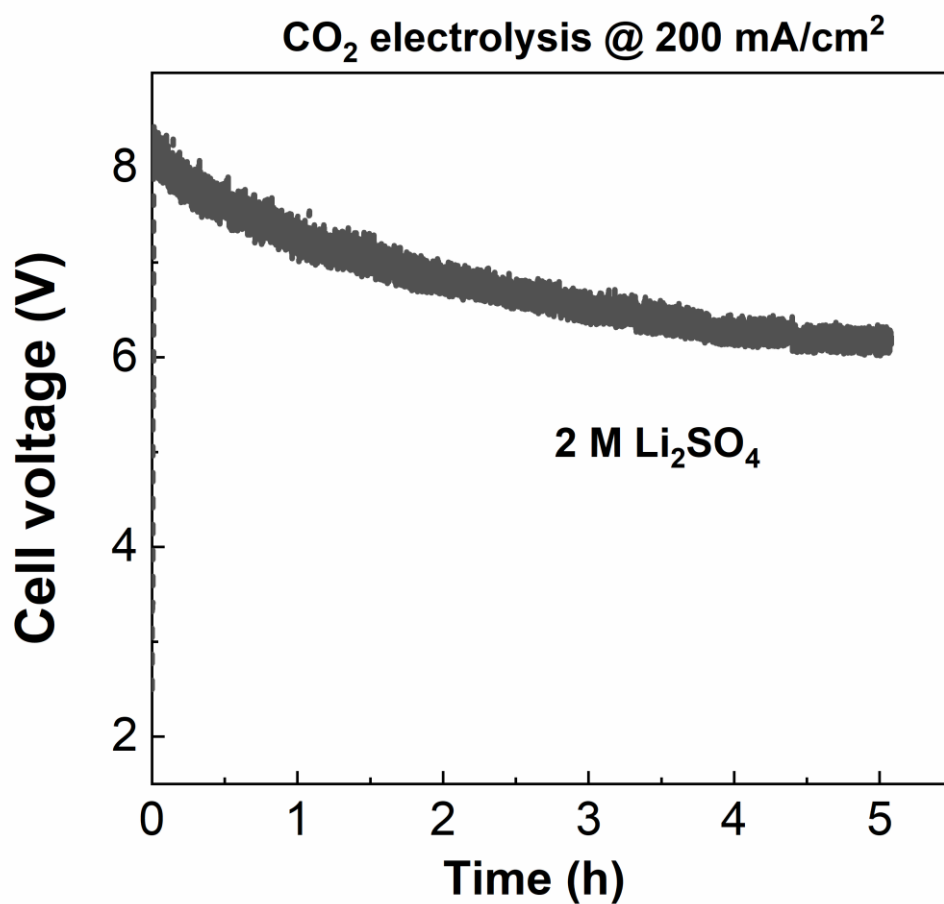


Figure S13. Cell voltage as a function of time when using the CEM over the course of CO₂ reduction electrolysis. 2 M Li₂SO₄ was used as the initial catholte and anolyte. The distance between the Cu cathode and the membrane was ~15 mm (the distance between the membrane and the anode was ~3 mm).

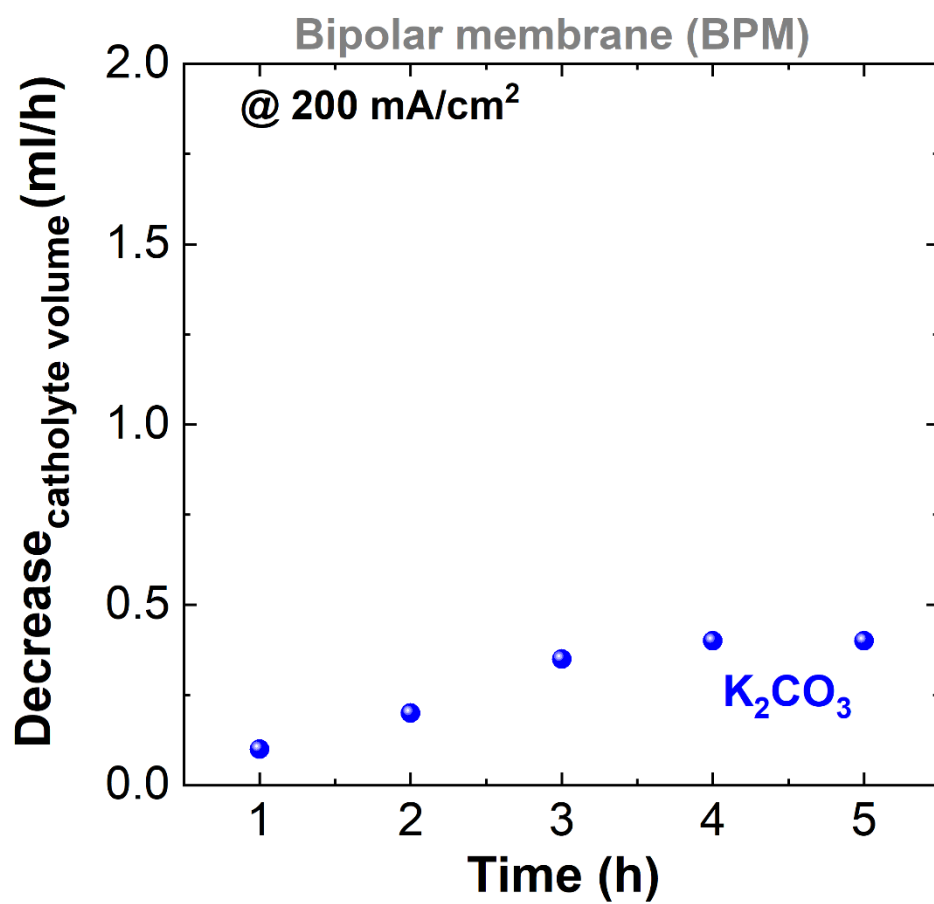


Figure S14. Decrease in catholyte volume when using a BPM.

Effect of water crossover on evaluation of catalytic selectivity

Without considering water crossover from catholyte to anolyte, the Faradaic efficiencies of liquid products could be overestimated when using AEMs in flowing-catholyte electrolyzers. We have previously demonstrated that there should be almost negligible crossover of neutral liquid products from catholyte to anolyte.⁷ Here, by ignoring the crossover of liquid products for AEMs, we can define the overestimation ratio of catalytic selectivity for liquid products in the catholyte without consideration of water crossover by the following equation:

$$\text{Overestimation ratio (\%)} = \frac{FE_{\text{initial volume}} - FE_{\text{final volume}}}{FE_{\text{final volume}}} \times 100\% \quad (\text{S20})$$

where $FE_{\text{final volume}}$ and $FE_{\text{initial volume}}$ are the Faradaic efficiency of liquid products dissolved in electrolyte (*i.e.* catholyte) with and without the consideration of water crossover over electrolysis, respectively. Based on the Faradaic efficiency calculation of liquid products (Equation 1), the Equation S20 can be rewritten as:

$$\text{Overestimation ratio (\%)} = \frac{V_{H_2O \text{ Cross}}}{V_C - V_{H_2O \text{ Cross}}} \times 100\% \quad (\text{S21})$$

where V_C and $V_{H_2O \text{ Cross}}$ are the initial catholyte volume before electrolysis and the volume of water crossed via membranes during the entire electrolysis experiment, respectively. After considering the calculation equation of water crossover volume (Equation 2), the Equation S21 can be rewritten as:

$$\text{Overestimation ratio (\%)} = \frac{N_h \frac{Q_{\text{tot}}}{Fn_c} \cdot \frac{M_{H_2O}}{\rho}}{V_C - N_h \frac{Q_{\text{tot}}}{Fn_c} \cdot \frac{M_{H_2O}}{\rho}} \times 100\% \quad (\text{S22})$$

where N_h and Q_{tot} are the hydration numbers of ions in water and the total charge passed through the membrane, respectively. F is the Faradaic constant, n_c is the number of charges for each anion, M_{H_2O} is the water molecular weight, and ρ is the density of water at ambient temperature and pressure. If CO_2/CO reduction is carried out at 200 mA/cm^2 (50 ml catholyte before electrolysis, active area of cathodic GDE is 2 cm^2), the overestimation ratio for Faradaic efficiency of liquid products as a function of electrolysis time can be roughly calculated using Equation S22 for different anion species when using AEMs. For simplification, the

overestimation ratio of Faradaic efficiency for liquid products only represents the liquid products in the catholyte.

In addition, with the consideration of the water loss in the catholyte caused by both water crossover and the cathodic reactions (Table S1), the overestimation ratios for FE of liquid products can be expressed as:

$$\text{Overestimation ratio (\%)} = \frac{(N_H) \frac{Q_{tot}}{Fn_c} \cdot \frac{M_{H_2O}}{\rho}}{V_C - (N_H) \frac{Q_{tot}}{Fn_c} \cdot \frac{M_{H_2O}}{\rho}} \times 100\% \quad (S23)$$

where N_H is the corrected hydration number of anions with adding water consumption induced by the cathodic reactions:

$$N_H = N_h + n_{average\ water} \cdot n_c \quad (S24)$$

$n_{average\ water}$ is average number of water molecules consumed for transferring per electron for products formation in cathodic reactions. In this work, $n_{average\ water}$ is 0.65 (Table S1). Thus, based on the Equation S23, we can get the overestimation ratios for FE of liquid products with considering the water loss in the catholyte caused by both water crossover and the cathodic reactions, as shown in Figure S15.

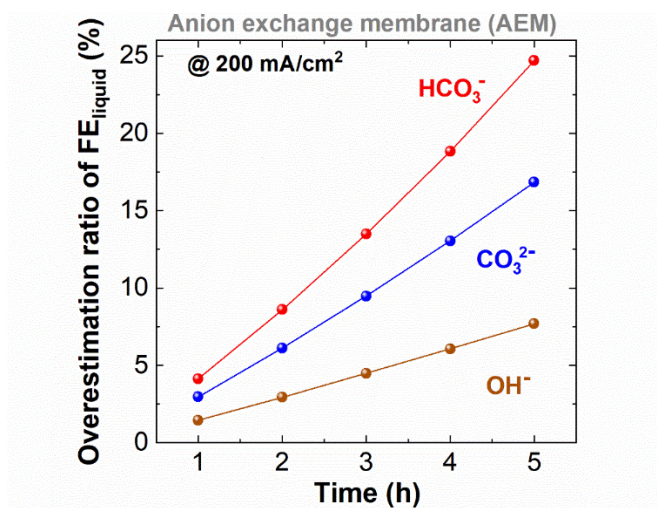


Figure S15. Overestimation ratio for Faradaic efficiency of liquid products as a function of electrolysis time for different anion species (50 ml initial catholyte before electrolysis, active area of cathodic GDE is 2 cm²). The water loss in the catholyte caused by both water crossover and the cathodic reactions were considered.

Exploration of cation crossover via AEMs

In order to explore the possible transport of K^+ from the anolyte to the catholyte when using the AEM in $KHCO_3$ electrolyte, the concentration of K^+ was quantified in both catholyte and anolyte over the course of electrolysis using high pressure ion chromatography (HPIC, Thermo Scientific Dionex Integriion). As shown in Table S3, while the concentration of K^+ increased in the catholyte and decreased in the anolyte, the total amount of K^+ retained at a relatively constant value in both catholyte and anolyte after considering electrolyte volume variation. Thus, we did not observe obvious transport of K^+ from the anolyte to the catholyte using the AEM, which means that the cation should have a minimal effect on water crossover in this work when studying the anion transport via the AEM. It should be noted that different AEMs may show distinct cation crossover properties via membranes.

Table S3. Concentration of K^+ and total amount of K^+ in catholyte and anolyte when using the AEM over the course of electrolysis at 200 mA/cm².

Electrolysis time (h)	Catholyte ($KHCO_3$)		Anolyte ($KHCO_3$)	
	K^+ concentration (M)	Total amount of K^+ (mol)	K^+ concentration (M)	Total amount of K^+ (mol)
0	1.02722	0.03082	1.02722	0.03082
3	1.12187	0.03141	0.94843	0.03035
5	1.17814	0.03051	0.96683	0.03181

(Here, volume of catholyte and anolyte was measured at electrolysis time of 0 h, 3 h and 5 h for calculating the total amount of K^+ in catholyte and anolyte, respectively. In this cation concentration measurement, 1 M $KHCO_3$ was used as the initial catholyte (30 ml) and anolyte (30 ml).)

The water crossover rate with unit of $\mu\text{l}/\text{C}$

Table S4. Water crossover rate for typical anions when using the AEM (anodic reactions were considered).

Typical anion species	Water crossover rate ($\mu\text{l}/\text{C}$)
OH^-	~0.28
CO_3^{2-}	~0.88
HCO_3^-	~1.16

Table S5. Water crossover rate for typical cations when using the CEM (anodic reactions were considered).

Typical cation species	Water crossover rate ($\mu\text{l}/\text{C}$)
Cs^+	~0.40
K^+	~0.54
Li^+	~1.05
Na^+	~0.89

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