Flow batteries are an attractive option for storing electricity at the utility-scale, offering benefits such as the increased uptake of intermittent renewable power sources such as solar and wind. Zinc/bromine redox flow batteries (Zn/Br RFBs) are a viable candidate for such applications due to factors such as a high theoretical specific energy (440 Wh kg\(^{-1}\))\(^9\),10 and relatively low cost of the materials of construction. Consequently, it is of commercial, environmental and social interest to pursue materials research leading to improved performance of such systems.

During the charging phase of the Zn/Br battery, the following reactions occur within the zinc and bromine half-cells, respectively (Eqs. 1–2):

\[
\begin{align*}
2\text{Br}^- (aq) & \rightarrow \text{Br}_2 (aq) + 2e^-, \\
\text{Zn}^{2+} (aq) + 2e^- & \rightarrow \text{Zn} (s). 
\end{align*}
\]

The reverse occurs during the discharge phase, whereby the primary electrolyte (i.e. ZnBr\(_2\)) is regenerated. The Zn/Br electrolyte also contains a bromine sequestration agent which complexes with Br\(_2\) evolved at the positive electrode to form a separate phase which is immiscible with the aqueous electrolyte. Organic quaternary ammonium bromides such as 1-ethyl-1-methylpyrrolidinium bromide are commonly employed as sequestration agents.\(^{11,12}\)

Dendrite formation during the charging phase has long been a major observation and source of concern during the design of RFBs which employ zinc electrodeposition, with various strategies employed to minimize this issue.\(^{13–16}\) Dendrites increase operational risks by causing problems such as short circuiting of cells. It is also beneficial to promote efficient electrodeposition and stripping of zinc during the charge and discharge phases, respectively, in order to maximize the coulombic efficiency of the system. Further improvements can be achieved by minimizing undesirable side-reactions which cause coulombic losses in the system.

An important factor influencing zinc electrodeplating behavior is the electrolyte composition.\(^{17}\) This dependency includes the type and amount of supporting electrolyte used in the Zn/Br battery to maintain conductivity when the primary electrolyte (i.e. ZnBr\(_2\)) is depleted during charging. The acidic nature of Zn/Br electrolytes limits the use of chemicals which would significantly alter the narrow working pH range.\(^{14}\) Chloride-based secondary electrolytes are conventionally used in Zn/Br systems,\(^{9,18–20}\) including bench-scale testing.\(^{21}\) Zinc electrodeposition and behavior in solutions containing chlorides has also been studied for industrial electroplating applications. However, it is possible that chlorides have a negative influence on electrodeposited zinc in the system due to the corrosive action of the former on the latter. This challenge has prompted work to identify suitable alternatives, such as the use of perchlorates.\(^{22}\) The effects of sulfates\(^{22–27}\) on zinc plating have also been studied and could potentially be adapted for use in Zn/Br electrolytes. Consequently, it is of interest to optimize the type and properties of supporting electrolyte employed in the next generation of Zn/Br batteries in order to improve performance and thus the uptake of this system for utility-scale electrical energy storage.

This interest provides motivation for the present work which focuses on improving the electrochemical performance of the zinc half-cell, both in the bulk electrolyte and at the electrode–electrolyte interface. Four Na-based supporting electrolytes (Br\(^-\), SO\(_4^{2-}\), H\(_2\)PO\(_4^-\) and NO\(_3^-\)) are assessed against a chloride-based solution to study the influence of these compounds on the Zn\(^{2+}\)/Zn couple. These electrolytes are selected as part of a targeted approach exploring ions present in conventional solutions (Br\(^-\), Cl\(^-\)) as well as common anions which would not lead to the formation of alkaline Zn/Br electrolytes (SO\(_4^{2-}\), H\(_2\)PO\(_4^-\) and NO\(_3^-\)), i.e. supporting electrolytes which would maintain an acidic working environment. The implications of the findings of this work are potentially relevant to other RFBs containing this redox and similar systems involving metal electrodeposition.

**Experimental**

Electrolyte preparation.— Testing solutions containing 2.25 M zinc bromide (ZnBr\(_2\), Merck) primary electrolyte with 0.5 M of a sodium-based supporting electrolyte were prepared. The supporting electrolytes included the following compounds: sodium bromide (NaBr, Merck, 99%), sodium sulfate (Na\(_2\)SO\(_4\), Mallinkrodt, 100%), sodium dihydrogen phosphate dihydrate (Na\(_2\)H\(_2\)PO\(_4\) \(\cdot\) 2H\(_2\)O, VWR, 99.7%), sodium chloride (NaCl, Ajax, 99.9%) and sodium nitrate (NaNO\(_3\), Ajax, 99.5%).
(NaNO₃, Merck, 99%). 0.8 M N-ethyl-N-methyl-pyrrolidinium bromide was used as the bromine sequestering agent based on the 0.8–1 M concentration range conventionally used in Zn/Br₃ systems. Ionic conductivity was measured using a labCHEM CP (TPS Ltd., Brisbane, Australia) for each testing solution prepared and listed in Table I together with a numerical shorthand used to refer to each supporting electrolyte.

**Table I. Properties and numerical shorthand for supporting electrolytes studied in this work.**

| Supporting electrolyte | Numerical shorthand | Conductivity (mS cm⁻¹) |
|------------------------|---------------------|------------------------|
| Na₂SO₄                 | 1                   | 54.0 ± 0.2             |
| NaBr                   | 2                   | 47.8 ± 0.2             |
| NaCl                   | 3                   | 48.2 ± 0.6             |
| NaH₂PO₄                | 4                   | 43.2 ± 0.9             |
| NaNO₃                  | 5                   | 49.7 ± 0.3             |

Zinc half-cell electrochemical tests.— A three-terminal glass cell was used for electrochemical half-cell studies, with a graphite-coated conductive carbon plastic working electrode (RedFlow Ltd., Brisbane, Australia), vitreous carbon rod counter electrode and silver–silver chloride (Ag/AgCl/3.4 M KCl) reference electrode. Solutions were bubbled with nitrogen gas and maintained under an inert atmosphere to remove dissolved oxygen prior to half-cell testing. A Bio-Logic SP-300 potentiostat was used for all electrochemical tests and the EC-Lab (version 10.32) data acquisition software was used to extract, process and fit the results obtained. Cyclic voltammetry (CV) was carried out between 0 V to −1 V vs Ag/AgCl at 30 mV s⁻¹ for six cycles (scans had stabilized by the end of the final cycle). Tafel analysis was carried out using linear sweep voltammetry (LSV) data obtained between −0.95 V and −0.60 V vs Ag/AgCl at 2 mV s⁻¹. Values of current densities obtained using this setup show good reproducibility within ±2% (standard deviation of ±0.04 mA cm⁻²) for CV and within ±1% (standard deviation of ±0.001 mA cm⁻²) for LSV. The equations used for Tafel fitting on the linear regions of Tafel plots (logarithm of the magnitude of current density against the working electrode potential vs Ag/AgCl) are (Eqs. 3–4):

\[
\log |i| \approx \frac{E - E_0}{\beta_a} + \log (i_0), \quad \text{for } E > E_0
\]

\[
\log |i| \approx \frac{E_0 - E}{\beta_c} + \log (i_0), \quad \text{for } E < E_0
\]

where \(i\) is the current density of the Zn²⁺/Zn reaction, \(i_0\) the exchange current density, \(E\) the working electrode potential vs Ag/AgCl, \(E_0\) the potential vs Ag/AgCl at which \(i_0\) occurs. The Tafel constants \(\beta_a\) and \(\beta_c\) are the anodic and cathodic reaction coefficients, respectively. The polarization resistance \(R_p\) at \(E_0\) is calculated as (Eq. 5):

\[
R_p = \frac{\beta_a \beta_c}{i_0 (\beta_a + \beta_c)} \ln 10
\]

The fitted Tafel parameters presented in this work include \(i_0\), \(E_0\), \(R_p\), \(\beta_a\) and \(\beta_c\). Potentiostatic electrochemical impedance spectroscopy (EIS) scans were carried out by holding the working electrode at −1 V vs Ag/AgCl for 1 minute to generate zinc electrodeposits on the surface, followed by EIS at the open-circuit potential (OCP) with 10 mV perturbation from 4 MHz to 50 mHz. External interferences were minimized by the use of a custom-built Faraday cage and all experiments were carried out at laboratory conditions of 25 ± 1 °C. Differential capacitance tests were also carried out as based on a method described in earlier literature¹⁵ to study relative capacitive behavior arising from ion assembly at the electroplated zinc surface during battery charging (thus forming an interfacial electrical double-layer, EDL). The electroplated zinc surface (after 1 minute of charging) was scanned between −0.2 V and +0.2 V vs OCP at 0.1 V steps, beginning from the OCP in the positive direction, followed by the negative direction. EIS scans were carried out at 20 different frequencies between 200 kHz and 100 Hz to identify a suitable frequency for comparing the capacitive behavior of EDLs formed in each of the solutions studied. Differential capacitance was calculated using the following equation³⁵ (Eq. 6):

\[
C = - (\omega Z_{im})^{-1}
\]

where \(C\) is the differential capacitance calculated for a particular frequency, \(\omega\) the angular frequency of the ac perturbation during the impedance scan, and \(Z_{im}\) the imaginary part of the impedance. Higher frequency leads to lower differential capacitance, hence it is necessary to select a suitable frequency which does not distort the impedance measurements and allows for clear comparison of trends in differential capacitance.⁵ On this basis, 1 kHz was selected as a suitable frequency for such comparisons to be made.

**Zinc electrodeposition preparation and characterization.—** Zinc was electrodeposited for 10 minutes from each electrolyte onto the graphite-coated working electrode using a Neware battery cycler operating at a current density of 20 mA cm⁻². The solution bath was magnetically stirred (300 rpm) to maintain homogeneity of the solution during this process. Electrodeposits were analyzed via scanning electron microscopy (SEM) and X-ray diffraction (XRD). SEM imaging was performed with a JCM-6000 NeoScope Benchtop SEM while XRD scans were carried out between 30°–80° at 5° min⁻¹ and 0.02° step size using a Shimadzu S6000 with a Cu-Kα target operating at 40 kV and 30 mA. Analysis of XRD data was carried out using the PDF4+ software database.

**Results and Discussion**

**CV and Tafel analysis of the zinc redox.—** Voltammograms from zinc half-cell CV tests of solutions containing the various supporting electrolytes are presented in Figure 1. The following electrochemical parameters are calculated for the reduction (red) and oxidation (ox) scans, with the ratios of oxidation to reduction values expressed as half-cell efficiencies (\(\eta\)): highest-magnitude current (\(i\)), maximum power (\(P^*\)), charge (\(Q\)) and energy (\(E^*\)) densities, and nucleation overpotential (\(\eta_{n.o.}\)) for zinc electrodeposition. The magnitude of potentials (\(\eta_{n.o.}\)) at which each electrolyte produced the stripping peak was also recorded. These values are listed in Table II. \(P^*\) and \(E^*\) are calculated with Ag/AgCl as a reference.

![Figure 1. Zinc half-cell CV for solutions containing 0.5 M of the secondary electrolytes studied: 1 Na₂SO₄ (thin solid line), 2 NaBr (thick solid line), 3 NaCl (dotted line), 4 NaH₂PO₄ (thin dashed line) and 5 NaNO₃ (thick dashed line).](image-url)
The conventional chloride option (3) generally performed comparably to, or poorer than, alternative supporting electrolytes employing sulfate (1), bromide (2) or phosphate (4). Electrolytes containing 2 exhibited the best electrochemical performance during both the deposition and stripping scans with the highest magnitude values. The presence of 1 gave the highest energy efficiency while relatively similar current and power efficiencies were obtained for electrolytes utilizing 1–4. The generally comparable (j0, P*) or lower (Qeff, E*) efficiencies calculated for solutions with 3 suggest a possible interference with zinc ions in solution and/or electrodeposited zinc by chlorides compared to other supporting species. Higher bromide concentration led to improved zinc-side performance despite bromide anions being expected to behave as spectators in the zinc half-cell which do not participate in redox reactions there. This observation, combined with the mid-range conductivity of solutions with 2 compared to other electrolytes, suggests that increased bromide concentration could be more beneficial to the zinc electrodeposition process than chlorides, and require closer attention for such systems. It was noted that electrolytes which produced stripping peaks at comparatively lower j0 values to the other electrolytes during charging. This is possibly due to the high charge-transfer resistance. Further study is underway in the zinc electrodeposits section, below.

**Impedance spectroscopy of the zinc half-cell.—** Nyquist plots from potentiostatic EIS of the various electrolytes show single time-constant behavior and are presented in Figure 3, together with an equivalent circuit model (see inset). This simple circuit contains the following elements: R1 represents solution resistance between the reference and working electrodes, R2 represents charge-transfer resistance of the Zn2+/Zn redox reaction, C2 represents capacitance of the electrical double-layer at the electrode surface and W2 represents Warburg diffusion limitations of the redox species. Values for each of the solutions are presented in Table IV.

**Similar solution resistances (R1)** are observed for the electrolytes studied, with a maximum difference of 2 Ω. The lowest R2 value was calculated for those using 2, suggesting improved mobility of ions in the bulk electrolyte for this solution compared to others. Solutions containing 1, 2 and 4 gave better electrochemical performance during CV tests due to lower resistances associated with charge-transfer (R2) and Warburg diffusion limitation (W2), as determined via EIS.

A wide range of capacitance (C2) values were calculated for the electrolytes, with the highest and lowest capacitance recorded for those with 2 and 5, respectively. Such a large range of values (each with a large relative error) indicate significant differences in the organization and behavior of ions forming the EDL, possibly with the densest packing of ions (and thus highest C2) observed from the use of 2. Conversely, a low C2 in the presence of 5 suggests the formation of a comparatively less-compact EDL possibly due to larger ions forming it, notwithstanding any changes in zinc surface morphology. This provides an explanation toward the higher diffusion limitation in electrolytes containing 5 although the reaction is occurring at a slower rate due to the high charge-transfer resistance. Further study is underway

### Table II. Zinc half-cell CV comparing the electrochemical performance of supporting electrolytes.

|        | Na2SO4 | NaBr | NaCl | NaH2PO4 | NaNO3 |
|--------|--------|------|------|---------|-------|
| jred, mA cm⁻² | −6.04 | −6.94 | −6.07 | −6.07 | −5.79 |
| jox, mA cm⁻² | 5.12 | 5.70 | 4.94 | 4.98 | 2.19 |
| j0, mV     | −731 | −728 | −734 | −741 | −758 |
| j0%       | 85%  | 82%  | 81%  | 82%  | 38%  |
| Qred, mC cm⁻² | −25.4 | −30.4 | −26.7 | −25.5 | −27.4 |
| Qox, mC cm⁻² | 20.0 | 23.4 | 18.9 | 20.0 | 6.5 |
| Qeff%     | 79%  | 77%  | 71%  | 79%  | 24%  |
| P*, mW cm⁻² | 6.0  | 6.9  | 6.1  | 6.1  | 5.8  |
| P**, mW cm⁻² | 3.7  | 4.2  | 3.6  | 3.7  | 1.7  |
| P*/P**%   | 62%  | 60%  | 60%  | 61%  | 29%  |
| E°, mV     | 23.8 | 28.6 | 24.9 | 23.9 | 24.9 |
| E°, mJ cm⁻² | 13.3 | 15.0 | 12.1 | 12.8 | 4.9 |
| E°%       | 56%  | 52%  | 49%  | 53%  | 20%  |
| DR, mV    | −926 | −917 | −919 | −924 | −916 |
| COP, mV   | −859 | −855 | −861 | −861 | −838 |
| NOP, mV   | 67   | 62   | 68   | 63   | 78   |

*Standard deviation of ±0.03 mA cm⁻² using this setup.

*Standard deviation of ±0.04 mA cm⁻² using this setup.

| Na2SO4 | NaBr | NaCl | NaH2PO4 | NaNO3 |
|--------|------|------|---------|-------|
| jred, mA cm⁻² | 0.657 | 0.620 | 0.447 | 0.643 | 1.569 | −0.187 |
| jox, mV | −848 | −845 | −845 | −850 | −829 | −781 |
| j0, mV/dec | 84 | 71 | 58 | 73 | inf | inf |
| R0, Ω | 96 | 91 | 106 | 90 | 110 | 110 |
to understand this phenomenon. From comparison of the differential capacitances of the various electrolytes at 1 kHz, test results confirmed that solutions with 2 and 3 had higher differential capacitance than electrolytes with other supporting species while those utilizing 5 had the lowest differential capacitance. However, there was a slight discrepancy in the order of those using 1 and 4 which is attributed to the overlap of \( C_2 \) values within the margin of error and indicating that the ions present in these solutions potentially exhibit similar capacitive behavior.

**SEM and XRD characterization of zinc electrodeposits.**—The zinc electrodeposits produced during the charging phase of the Zn/Br system under a current density of 20 mA cm\(^{-2}\) exhibit significantly different morphology depending on the supporting electrolyte used, and are presented in Figure 4. This observation confirms the significant influence of the anions studied herein on zinc electroplating behavior during battery operation, possibly via complexes formed with Zn\(^{2+}\) in solution. Electrolytes employing 2 and 4 produced bright electrodeposits with highly crystalline regions and crystal sizes generally between 5–10 \( \mu \)m, while solutions with 3 produced comparatively larger crystals in patches which were less bright. Conversely, solutions utilizing 1 and 5 produced highly mossy deposits with needle-like zinc clusters which were dull and had very little apparent ordering during visual observation. This observation suggests a link between high NOP seen during CV and the generation of mossy deposits due to the creation of multiple nucleation points instead of crystal growth. The mossy appearance of deposits in solutions with 1 was unexpected as such deposits are usually associated with poor electrochemical performance,\(^{13,36}\) whereas the opposite was noted during CV tests in this study. Such zinc electrodeposition behavior when utilizing a ZnSO\(_4\) electrolyte has been previously reported\(^{37}\) and it has been postulated the presence of an adsorbate (likely hydrogen) interferes with the normal epitaxial deposition, thereby promoting dendritic growth instead. We believe this model explains the reason behind the mossy deposits obtained from solutions containing 1.

Circular pores with diameters less than 5 \( \mu \)m were found within deposits obtained in the presence of 5. These pores are possibly due to the evolution and escape of hydrogen bubbles on the electrode surface under the electrodeposited zinc. \( \text{H}_2 \) evolution caused by depassivation due to the presence of impurities has been discussed for the case of

![Figure 3](image-url). Zinc half-cell Nyquist curves for solutions containing 0.5 M of the secondary electrolytes studied: (a) 1 Na\(_2\)SO\(_4\) (thin solid line), 2 NaBr (thick solid line), 3 NaCl (dotted line), 4 Na\(_2\)HPO\(_4\) (thin dashed line) and (b) 5 NaNO\(_3\) (thick dashed line). Inset of (b) shows the equivalent circuit model used to simulate each impedance curve.

![Figure 4](image-url). SEM micrographs of zinc electrodeposited on graphite after 10 minutes of charging at 20 mA cm\(^{-2}\) from solutions containing 0.5 M of the secondary electrolytes studied: (a) 1 Na\(_2\)SO\(_4\), (b) 2 NaBr, (c) 3 NaCl, (d) 4 Na\(_2\)HPO\(_4\) and (e) 5 NaNO\(_3\).
zinc electrodeposited from acidic sulfate solutions. In the present work, it is proposed that depassivation at the zinc surface occurs in the presence of electrolytes containing nitrates, thereby promoting H₂ evolution on the electrode surface. The generation of H₂ could explain the significantly poorer electrochemical performance during the oxidation scan compared to the reduction scan observed during CV, as well as presence of double peaks on CV and Tafel plots, for that electrolyte. With the exception of solutions using NaNO₃, those which produced non-mossy zinc deposits were observed to also give relatively good zinc half-cell CV values. A similar observation has been previously reported in the literature. Experimental and molecular modelling work is being undertaken to better understand this phenomenon.

Diffractograms from XRD analysis of zinc electrodeposits obtained from the various electrolytes are presented Figure 5 and are consistent with SEM observations. The Miller indexes of zinc peaks were identified using PDF card #04-014-0230 in the PDF4 database. Solutions containing NaNO₃, Na₂SO₄, NaBr, NaH₂PO₄ and NaNO₃ yielded electrodeposits with a strong (101) preferred orientation and a secondary preference for the (102) configuration. A preference for the (101) orientation has been previously reported for the case of zinc deposited from solutions with 2.5 M ZnBr₂ and relatively high concentration of 3 M NaCl supporting electrolyte, while some preference for the (102) and (103) configurations has also been reported from the use of ZnCl₂ solutions containing HCl. The similarity in XRD peaks obtained for zinc electrodeposited from these solutions is consistent with their generally similar morphology observed during SEM. The mossy deposits produced by electrolytes utilizing NaNO₃ and NaBr exhibiting a preference for the basal (004) orientation.

Selection of suitable alternative supporting electrolytes.— Based on the findings from half-cell CV scans and Tafel analysis of the electrolytes studied, two alternatives to Cl-based supporting electrolytes are suggested. The first suggestion involves the use of higher bromide concentrations (by up to 0.5 M, based on this study). The second alternative is the further development of sulfates- and/or phosphate-based secondary electrolytes. The expected benefits of these two approaches are based on lower Zn⁺/Zn redox charge-transfer resistances and Zn⁺ diffusion limitations, as indicated by impedance spectroscopy. Further investigations regarding the impact of these strategies on electrochemical performances of the bromine half-cell and subsequently a full Zn/Br cell are being undertaken.

It is proposed that the differences in electrochemical performance and behavior of ions in solution are attributable to the different types and degrees of zinc complexes formed in the presence of each supporting electrolyte. Specifically, the complexing of Zn²⁺ by weakly bound ligands of the various anions such as bromides and chlorides. It has been shown that increasing ion size increases the effective EDL thickness and that ion size influences reaction rates at the electrode surface due to the extent of electrode-coverage. Consequently, it is also possible that the formation of large cationic species in high concentrations does not allow for dense packing of ions at the EDL, thereby producing lower EIS capacitance values for some solutions studied in the present work. Consequently, the type and strength of ligands associated with zinc is an important factor to consider, with weaker association possibly giving rise to favorable zinc electrodeposition and thus zinc half-cell electrochemical performance.

Conclusions

Zinc half-cell electrochemical studies (CV, Tafel analysis, EIS) were performed to assess and identify possible alternative supporting electrolytes for Zn/Br flow batteries. Sodium-salts of the following anions were assessed for their viability by comparison against the conventional Cl-based option: Br⁻, SO₄²⁻, H₂PO₄⁻ and NO₃⁻. Electrolytes utilizing NaBr, Na₂SO₄ and NaH₂PO₄ generally exhibited comparable or better performance than those containing NaCl, primarily due to lowering of the charge-transfer resistance and diffusion limitation in the zinc half-cell. SEM imaging and XRD analysis confirmed the strong influence of supporting electrolyte type on the crystal size and morphology of zinc electrodeposits obtained during the charging phase of the battery. Electrolytes which produced smaller crystals also gave good electrochemical performance. Additionally and in contrast with conventional assumption, it was found that the generation of mossy deposits does not translate to poor zinc half-cell performance. The findings of this work are expected to prompt further investigations into, and identification of, beneficial alternative supporting electrolytes which improve Zn/Br performance.

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