Significant efforts have been dedicated to developing Ti$_3$C$_2$T$_z$-based MXene aqueous supercapacitors (SCs) with improved energy and power densities. Notably less research has been devoted to an equally important characteristic of aqueous SCs, viz. self-discharge (SD). The SD rates are rarely reported despite their crucial importance from a practical point of view. Herein, the SD rates in four different aqueous electrolytes: H$_2$SO$_4$, KOH, LiCl, and LiBr in Ti$_3$C$_2$T$_z$-based aqueous SCs are compared. For the latter two, the SD rates vary as a function of salt concentration in the electrolytes with higher LiCl or LiBr concentrations having the lowest SD rates, viz. 78.3% and 81.5% in 14M LiCl and LiBr, respectively, after 10 h. Further, the influence of dissolved oxygen and the purities of the starting powders are examined, and it is concluded that parasitic reactions, including oxygen, are responsible for the SD.

1. Introduction

Supercapacitors (SCs) are recognized as promising next-generation energy storage devices due to their fast charge/discharge rates, high power densities, excellent cycling stabilities, and good safety record, compared with much the more widely used lithium-ion batteries.[1] In general, and depending on the electrolytes, SCs are either aqueous or organic-based. Benefiting from the low cost, environment benignity, and fast charging capabilities, great efforts have been dedicated to developing aqueous SCs.

Due to their quite high electronic conductivities, excellent hydrophilicities, and pseudocapacitive response, MXenes have shown great potential as electrodes in aqueous SCs with high volumetric capacitances ($C_v$) and volumetric energy densities ($E_v$).[2] MXenes, a family of early 2D transition metal carbides/nitrides, have a general formula of M$_{n+1}$X$_n$T$_z$ or M$_1.33$XT$_z$, where M is transition metal, X is C, and/or N, T$_z$ represents surface terminations such as $-$F, $-$OH, $-$O, $-$Cl, etc., and n is 1–4.[2,3] They are derived by etching the A layers—mostly Al—from their layered parent materials, the MAX phases.[4]

The preponderance of research on aqueous SCs however, has focused on maximizing and energy and power densities of various systems.[5] For example, in 2018, Yang et al. reported a $C_v$ for Ti$_3$C$_2$T$_z$ of $\approx2800$ F cm$^{-2}$,[6] which exceeded that of another much more expensive pseudocapacitive electrode, viz. RuO$_2$. This is typically accomplished using aqueous electrolytes and in many cases quite low loadings. In this work, the low loadings are not addressed. What we focus on is one that is as important, viz. self-discharge, SD. From a practical point of view, an energy storing device that cannot store charges for long times will have limited applicability. It is thus noteworthy that this aspect has, for the most part, been almost ignored in the aqueous SC literature in general, and in the MXene literature in particular. For example, in the MXene literature, sulfuric acid (H$_2$SO$_4$) is quite popular because of the very fast redox reactions between hydronium ions and MXenes,[7] resulting in ultrahigh $C_v$ values combined with good power characteristics.[8] Another widely used electrolyte is potassium hydroxide (KOH), that has a relatively smaller water of hydration (3.31 Å) and relatively higher ion conductivity,[9] allowing rapid K$^+$ (de)intercalation in between the MXene interlayers.[10] As noted above the SD performance in these papers is largely not reported.

In the few reports on SD performance, Li et al. modified the surface functional groups of MXene by heat treatment, and found $-$O-rich Ti$_3$C$_2$T$_z$ could slow the SD rate in KOH electrolytes.[11] However, this surface modification method only slightly slowed the SD rates (voltage retention after 10 h, increased to 30% from 25%). Avireddy et al. tested Ti$_3$C$_2$T$_z$/MnO$_2$ asymmetric SCs in a 21 M potassium acetate-based electrolyte and lowered the SD rate. The open cell voltage dropped from 2.2 to $\approx$1.5 V in 14 h.[12] In our previous work, we showed that a highly concentrated LiCl electrolyte could significantly improve the SD performance, resulting in low SD rates ($\approx66$% of the voltage was retained after 54 h).[13] However, the reasons for this enhancement were not studied.

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1. Introduction

Supercapacitors (SCs) are recognized as promising next-generation energy storage devices due to their fast charge/discharge rates, high power densities, excellent cycling stabilities, and good safety record, compared with much the more widely used lithium-ion batteries.[1] In general, and depending on the electrolytes, SCs are either aqueous or organic-based. Benefiting from the low cost, environment benignity, and fast charging capabilities, great efforts have been dedicated to developing aqueous SCs.

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The purpose of this work is to systematically study the SD performance of Ti3C2T x MXene film electrodes, in four electrolytes: H2SO4, KOH, LiCl, and LiBr with various concentrations. The results clearly show that one straightforward method to mitigate SD is to work with highly concentrated salt electrolytes with high purity and low oxygen contents.

2. Experimental Section

2.1. Materials Preparation

The synthesis process of Ti3C2T x films can be found in our previous work.[14] Briefly, Ti3C2T x nanosheet suspensions were prepared by etching the Al layers from a Ti3AlC2 precursor using LiF and HCl mixtures. The latter was then sonicated in an ultrasonic bath for 1 h under argon, Ar, protection. Films were obtained by vacuum filtration of a known suspension volume. The solid loading in the colloidal suspension was in the 3–5 mg mL−1 range.

2.2. Materials Characterization

XRD was conducted to characterize the phase constitutions of samples on a diffractometer (PANalytical X’Pert company, Netherlands) with Cu Kα (40 kV, 40 mA) in the 3°–70° 2θ range. The step size was 0.02° and the scan rate was 0.5 s/step.

An SEM (LEO 1550 Gemini, Germany) was used to examine the morphologies of the samples.

2.3. Electrochemical Measurements

The symmetric supercapacitors (SSCs) were assembled with two MXene discs in plastic Swageloks. Before measurements, all cells were precycled at 20 mV s−1 for 100 cycles to stabilize the performance on a potentiostat (VSP, Biologic, France). All cells used glassy carbon (CHI Instruments, China) as current collectors and glass microfiber membranes (Whatman, General Electric, USA) as separators.

The MXene films were cut into 4 mm diameter discs and used as working electrodes with areal and volumetric densities of ≈1.1 mg cm−2 and ≈3.0 g cm−3, respectively. To prepare the electrolytes, we started with H2SO4 (Merck, ≥98%, Germany), potassium hydroxide, KOH (Sigma-Aldrich, ≥85%, balance water pellets, Sweden), high-purity lithium chloride (LiCl, Sigma-Aldrich, ≥99.98%, Sweden), lower-purity LiCl (Sigma-Aldrich, ≥99%, Sweden), henceforth referred to as LiCl(Li), and high-purity lithium bromide (LiBr, Sigma-Aldrich, ≥99.99%, Sweden). Characteristics of these reagents can be found in Table S1, in Supporting Information. The MXene films were mixed with deionized water with a resistance of 18.2 MΩ cm to prepare 1 m H2SO4, 1 m KOH, 1 m LiCl, or 1 m LiBr. We also prepared electrolytes with 1, 5, 10, and 14 m LiCl and a 14 m LiBr. Before using the salt electrolytes, nitrogen, N2, gas (99.999%, Nippon Gases, Sweden) was bubbled overnight through them. In two cases, air was bubbled through the 1 m H2SO4 and 14 m LiCl to check the influence of oxygen on the SD performance.

The SD performance was measured by first holding the cell at certain voltage for 0.5 h and then monitoring the cell voltage as a function of time for 10 h. The choice of voltage in the different electrolytes varied due to their different electrochemical stability windows (ESWs). For example, the SSC in 1 m H2SO4 was stable between 0 and 0.6 V. Therefore, the 0.6 V was selected as the cell’s voltage. Similarly, the SSC in 1 m KOH was 1 V. In the LiCl or LiBr electrolytes, the voltage was 1.2 V, regardless of their molarity. In all cases, the ESWs for the salt electrolytes were higher than those in the acid or base electrolytes.

The CV was calculated from CV curves, assuming

$$C_v = \frac{1}{\nu (U_+ - U_-)} \int_{U_-}^{U_+} I(V) dV / \text{vol} (F \text{ cm}^{-3})$$

where $U$ is the potential, $\nu$ (mV s−1) is the scan rate, $I$ (mA) is the current, vol is the volume of the two electrodes, which is 1 m, and $U_+$ and $U_-$ are the positive and negative potentials, respectively.

The volumetric energy density ($E_v$) and power density ($P_v$) were calculated assuming

$$E_v = C_v U^2 / 7.2 \text{ (mWh cm}^{-3})$$

$$P_v = \nu E_v / U \times 3.6 \text{ (mWh cm}^{-3})$$

3. Results and Discussion

3.1. Results

A typical X-ray diffraction (XRD) pattern for a Ti3C2T x film (Figure 1a) shows the flakes to be well aligned along the stacking direction. All peaks belong to the (00l) plane family of Ti3C2T x and are typical of MXenes. Two (002) peaks at 2θ = 7.56° (high intensity) and 4.64° (low intensity) are visible. They correspond to d-values of 11.78 and 18.95 Å, respectively, and have been ascribed to the different number of intercalated water layers between the sheets.[15] For example, the peak at 4.64° is consistent with Li+ in the interlayer space with three H2O layers, whereas the peak at 7.56° only has Li+ and one H2O layer.

Figure 1b shows a digital photograph of a typical MXene film, showing good flexibility. The cross-sectional SEM of the highly stacked film is shown in Figure 1c. This dense stacking leads to a relatively high density (≈3 g cm−3), which in turn results in higher $C_v$ values. Interestingly, the MXene films’ surfaces are not smooth but appear wrinkled (Figure 1d).

Figure 2a shows our SD results, after 10 h, in the various electrolytes after bubbling N2; Figure 2b shows selected results for SCs in which N2 was not bubbled prior to testing the SD. Figure 2c shows the SD as a function of electrolyte concentrations. Figure 2d shows the SD as a function of different starting voltages. From the totality of these results, the following observations are salient: 1) Bubbling of N2 prior to assembling the cell significantly slows down the SD rate. This is most dramatically demonstrated for the H2SO4 electrolyte; after N2 bubbling, the...
cell maintains 19.7% of the initial voltage after 10 h. Without bubbling N₂, the voltage is almost zero after 4 h (Figure 2b). The effect is even more dramatic after 1 h (see Figure S1, Supporting Information), where after N₂ bubbling, 39.3% of the voltage is retained; without, only 1.2%. In addition, after N₂ bubbling through the 14M LiCl cell the voltage retention after 10 h is 78.3%, which is greater than the 69.5% obtained when N₂ is not bubbled (Figure 2b). 2) At a concentration of 1 M, the cells with LiCl and LiBr electrolytes show comparable voltage retentions with the latter being slightly higher than LiCl (30.7% vs 27.8%). As noted earlier, the cells’ voltages in the salt electrolytes (1.2 V) are higher than H₂SO₄ (0.6 V) or KOH (1 V). 3) The results clearly show that when starting with the lower purity LiCl-LiCl (L), 69.5% of the initial voltage is retained. For its high purity counterpart, 78.3% is retained in 10 h (Figure 2a). 4) When air is bubbled in the 14 M LiCl cell, the SD rate (Figure 2b) is higher than the as produced cells for which N₂ is not bubbled (Figure 2b). 5) The SDs in dilute LiCl electrolytes are significantly faster than those in their concentrated counterparts (Figure 2c). 6) The voltage retentions in low concentrated acid, base, and salt electrolytes are quite low, with the acid electrolyte being the worst. 7) Starting with higher voltages result in lower voltage retentions (Figure 2d). The SD voltages however, never cross. Note that, the cyclic voltammetry (CV) shape at 1.4 V have some distortions from rectangular shape (Figure S2, Supporting Information), which could probably be ascribed to side reactions on the electrodes and thereby leads to faster SD rates.

The CV curves of the cells in the different electrolytes are shown in Figure 3. All CV curves exhibit near ideal rectangular shapes, indicating capacitive charge storage mechanism. The galvanostatic charge–discharge (GCD) curves are shown in Figure S3, Supporting Information. In all electrolytes, the charge and discharge times are almost equal indicating good reversibility.

The corresponding specific capacitance versus scan rates are shown in Figure 4a and S4, Supporting Information. Not surprisingly, the cell with the 1 M H₂SO₄ electrolyte has the highest Cₛ at all scan rates. The second highest is 1 M KOH, again not surprisingly. At the same concentration, the Cₛ values in LiBr are higher than those in LiCl.

Interestingly, at low scan rates (2–10 mV s⁻¹), the Cₛ in LiCl and LiBr at 14 M is higher than that in 1 M electrolytes. This may be ascribed to the dense hydrated Li⁺ in the interlayer space of the charged MXene.[16]

From the Ragone plots (Figure 4b and S5, Supporting Information), the LiCl and LiBr cells deliver higher Pₑ values than those in the H₂SO₄ and KOH electrolytes because of the higher voltages and comparable Cₛ. The highest Pₑ is 17.4 mW h cm⁻² at a Pₜ of 104.7 mW cm⁻² in the 14 M LiBr cells. Not surprisingly, the Pₑ values of the concentrated salt electrolytes drop faster than the less concentrated ones.

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**Figure 1.** Phase composition and morphology of Ti₃C₂Tₓ films: a) XRD pattern; b) digital photo of filtered film; and c) cross-sectional and d) top-view SEM images of typical films.
3.2. Discussion

In general, there are three reasons for SD: 1) ohmic leakage, 2) parasitic faradaic reactions, and/or 3) charge redistribution across the electrodes.\[^{17}\]

It is unlikely that ohmic leakage is responsible here as all the cells are of identical build and design.\[^{17,18}\] Charge redistribution can be an issue in highly porous carbon electrodes. As the thin-film electrodes tested herein are readily fully charged by holding the cells at a given voltage for 0.5 h and given the planar structure of the MXene sheets and the lack of very small pores, it is reasonable to assume that charge redistribution is not the main culprit in the SD.

Then it leaves the parasitic faradaic reactions. All our results are consistent with this notion. For example, there is little doubt that O\(_2\) in the electrolyte results in higher SD rates (Figure 2b). It is established that impurities or O\(_2\) in electrolytes participate in some redox reactions (like water decomposition, current collector corrosion, surface terminations changes, or O\(_2\)→H\(_2\)O\(_2\)/HO\(_2\)\(^+\)) and consume electrons, leading to faster SD rates.\[^{18}\]

Furthermore, starting with a purer LiCl salt results in lower SD rates (Figure 2a), a result consistent with parasitic reactions. Based on the concentration of parasitic reactant species, the SD mechanisms can be further divided in two: 1) activation-controlled mechanism, and 2) diffusion-controlled mechanism.\[^{17}\] For the former, the measured potential scales with ln\(t\); For the latter, the measured potential scales with \(t^{1/2}\).\[^{19}\]

Figure 5a shows the voltage plotted versus ln\(t\). These results suggest that at short times, the process may be activation controlled. The results shown in Figure 5b further confirm the \(t^{1/2}\) dependence, especially at longer times. In highly concentrated electrolytes, the dense hydrated Li\(^+\) ions in the interlayer space of MXene shield their surfaces from parasitic reactions. Least square fitting of the 14 M LiCl plot, at \(t > 400\) s, yields a \(R^2\) value of 0.9999. In comparison, at lower salt concentrations, the diffusion-controlled process is significantly postponed to \(t > 6400\) s in 10 M LiCl, \(t > 10000\) s in 5 M LiCl, and \(t > 17000\) s in 1 M LiCl. The fact that in the 14 M LiCl electrolyte...
the process is diffusion controlled after only 400 s is an important result that cannot be overemphasized.

We thus conclude that concentrated, high-purity electrolytes are much more suitable for MXene-based SCs. While acknowledging the merits of the high-concentrated electrolytes, it should be noted that their viscosities clearly limit their power densities (Figure 4). Also, the inevitable salt precipitation at lower temperatures may restrict their applicable temperature range. In addition, the weight of these electrolytes reduces their gravimetric energy density.

4. Conclusions

In summary, we compared the SD rates and energy densities in SSCs in four electrolytes: H₂SO₄, KOH, LiCl, and LiBr aqueous electrolytes and concluded that parasitic reactions were responsible for the SD.

It follows that high-purity salts in high concentrations and the exclusion of oxygen improve the SD performance.

From a practical point of view, and in most applications, the SD rates in H₂SO₄ and KOH are unacceptably fast. An energy
storage device cannot be considered as such if it cannot store energy for extended time periods. We, therefore, plea with the MXene community to routinely report the SD rates of their SCs; it is a simple yet crucial experiment.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
Research data are not shared.

Keywords
concentrated electrolytes, MXene, self-discharge, supercapacitors

Figure 5. Plot of V as a function of a) ln t and b) \( t^{1/2} \) in different concentrated LiCl electrolytes. The white straight lines in (a) and (b) are fitting results for each SD curve.

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