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How Water Accelerates Bivalent Ion Diffusion

Fei Wang,[a],[b] Wei Sun, [a] Zulipiya Shadike, [c] Enyuan Hu, [c] Xiao Ji,[a] Tao Gao, [a] Xiao-Qing Yang, [c] Kang Xu* [b] and Chunsheng Wang* [a][d]

Abstract: Multivalent (MV) ion batteries (Mg, Zn, Al, etc) are considered promising alternatives to the Li-ion batteries (LIBs), while their applications suffer from the sluggish MV cation diffusion in electrolytes, electrodes, and the interfaces in between, induced by its strong electrostatic interaction with both solvent molecules and host lattices. Recent reports demonstrated that the presence of H_2O molecules in the electrolyte and/or electrode significantly alleviates these MV cation diffusion barriers, but the rationales varied, and the underneath mechanism remains uncertain. In this work, we systemically investigated the effect of H_2O in electrolyte and in electrode lattice on the thermodynamics and kinetics of reversible MV-ion intercalation chemistry based on a model platform of layered $VOPO_4$. We found that H_2O presence at the electrolyte/electrode interface plays the key role in assisting Zn^{2+} diffusion from electrolyte to the surface, while H_2O in the lattice structure alters the working potential. More importantly, a dynamic equilibrium between bulk electrode and electrolyte is eventually reached for the H_2O transports during the charge/discharge cycles, with the water activity serving as the key parameter determining the direction of water movement and the cycling stability.

Lithium-ion batteries (LIB) satisfy most of our daily digital and mobile energy needs[1], however, their energy limits are set by the intercalation chemistry nature. Aggressive engineering to maximize energy density have brought safety concerns[2]. As alternatives, multivalent (MV) ion batteries (Mg, Zn, Al, etc) have attracted increasing attentions for their comparable or even superior capacities to that of LIB but much lower cost[3]. However, the MV ion batteries typically suffer from the sluggish kinetics, no matter in crystalline solids, in bulk electrolytes or across the interfaces/interphases. The stronger electrostatic interaction between the MV cations and their environment constitutes the following fundamental difficulties: (1) The high energy barriers for the diffusion within the solid electrodes, (2) The much bulkier and clumsier solvation spheres in electrolyte due to the higher solvation numbers (~6) than mono-valent cations, and (3) The especially difficult desolvation at the electrode/electrolyte interfaces. Mitigation or elimination of these barriers thus become the key to unlock these potential battery chemistries.

Recent reports demonstrated that the H_2O molecules in the electrolyte and/or electrode could significantly alleviate these MV cation diffusion barriers [4], but rationales varied. Some believed that H_2O in lattice structure helps open up the diffusion tunnels, or acts as electrostatic shield for MV cations [5] [3b, 4a], while others argued that water molecules move with the MV cations [6]. Overall, the underneath mechanism remains uncertain, mainly because of the limited analytical tools that can precisely identify how water distribute in a MV electrode/electrolyte system [7], as well as the limited host materials that could allow reversible MV-ion insertion. This absence of fundamental understanding prevents us from further exploiting this advantage brought by water and making MV ion batteries successful.

Among the MV ion chemistries, Zn is of particular interest for its high volumetric capacities (5851 mAh cm^{-3}), high abundance, and good compatibility with aqueous electrolytes[8]. Differing from the complications arising from interphase formation on Mg surface at the low potential, Zn resides within the electrochemical stability windows of most non-aqueous (and even aqueous) electrolytes, where facile Zn stripping/plating occurs with high reversibility [6, 9]. Thus, Zn chemistry should serve as an ideal platform to investigate how water influence the diffusion of a MV cation.

Herein, we demonstrate layered $VOPO_4$ and its hydrates as such model platforms, which could be utilized as Zn^{2+} host in both aqueous and non-aqueous electrolytes. Using electroanalytical technologies, X-ray diffraction (XRD), neutron diffraction (ND) and the first-principle calculations, we systemically investigated the effect of H_2O that exists in electrolyte and in lattice on the thermodynamics and kinetics of reversible Zn^{2+}-ion chemistry. We found that the H_2O at the electrolyte/electrode interface accelerates Zn^{2+} diffusion from electrolyte to the bulk surface, while H_2O in the bulk electrode alters the working potential. A dynamic equilibrium between bulk electrode and electrolyte is eventually reached for the H_2O transports during the cycles, with the water activity (α_{H2O}) as the key parameter determining the

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direction of H_2O movement. The mechanistic understanding about how H_2O affects the transport of MV cations from electrode to electrolyte constitutes a solid foundation for the design of future MV ion batteries.

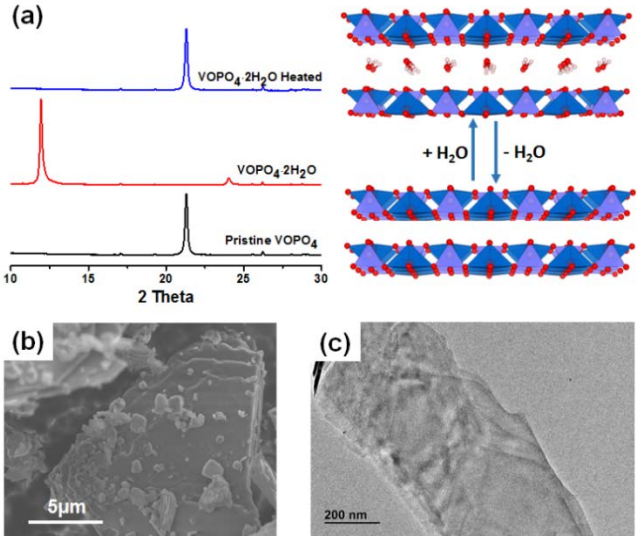

Figure 1. (a) XRD patterns of pristine $VOPO_4$, $VOPO_4{\cdot}2H_2O$ and $VOPO_4{\cdot}2H_2O$ after heat treatment. The schematic illustration shows the transformation between $VOPO_4$ and $VOPO_4{\cdot}2H_2O$. (b) SEM and (c) TEM image of the $VOPO_4$.

Layered $VOPO_4$ has been used as host for monovalent-ions[10], but, to our best knowledge, was never reported for MV cations. $VOPO_4$ has a typical (001) peak at ~21.5°, corresponding to a d value of 4.2 Å [11], while the intercalation of H_2O molecules into the interlayer significantly expanded basal spacing to 7.4Å, thus pushing the peak to ~11.9° for hydrated $VOPO_4{\cdot}2H_2O$[12]. The thermogravimetric analysis (Figure S1) proved that the hydration-dehydration of $VOPO_4$ is reversible, because after being heated to 500 °C, the peak of $VOPO_4{\cdot}2H_2O$ shifted back. $VOPO_4$ displays thin platelets with a lateral size of ~20 μm (Figure 1b), while TEM image reveals $VOPO_4$ as individual and freestanding nanosheets (Figure 1c).

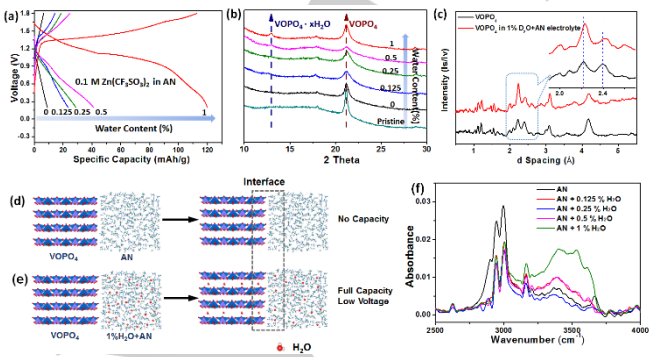

Figure 2. (a) The voltage profile of $VOPO_4$ between 0.2 V and 1.9 V in the 0.1 M $Zn(OTf)_2$-AN with different water contents at 20 mA/g. (b) The XRD patterns of the $VOPO_4$ soaked in the different electrolytes for 24 hours. (c) The neutron diffraction results of $VOPO_4$ soaked in 0.1 M $Zn(OTf)_2$-AN with 1% D_2O. The schematic illustrations of $VOPO_4$ in the 0.1 M $Zn(OTf)_2$-AN (d) without water and (e) with 1% H_2O. (f) The progression of FTIR spectra between 2500 and 4000 cm^{-1} with water contents.

0.1 M $Zn(OTf)_2$ in acetonitrile (AN) was chosen as the base electrolyte, into which different amount of H_2O was added. Only negligible capacity can be accessed from $VOPO_4$ in the water-free electrolyte (Figure 2a), while slight increase in capacity (47 mAh/g) was obtained at 0.5% H_2O, followed by a rapid increase to 122 mAh/g when H_2O increases to 1%. The significant increase is induced by the presence of H_2O diffusion into $VOPO_4$, as evidenced by the shift of XRD patterns (Figure 2b). EDX mapping confirms that the capacity arise from the Zn-ion intercalation (Figure S2). When dry $VOPO_4$ electrode was immersed in 0.1 M $Zn(OTf)_2$-AN electrolytes with H_2O contents below 0.5%, no significant shift in XRD peaks was observed; while a small peak representing hydrate $VOPO_4$ appeared after being exposed to 1% H_2O. This difference strongly suggests that H_2O intercalated into the lattice of $VOPO_4$, and led to the capacity hike. The chemical intercalation of H_2O into $VOPO_4$ can also be confirmed by ND (Figure 2c), where peak shifts to a high *d*-spacing after soaking in electrolyte containing 1% H_2O, reminiscent of solid-solution behavior. The hydration of $VOPO_4$ can also be revealed by the atomic pair distribution function (PDF) technique using neutron scattering (Figure S3). Therefore, as schematically illustrated in Figure 2d and 2e, H_2O migrates from water-containing electrolyte into the $VOPO_4$ lattice, and simultaneously creates a "wet interface" that assists the Zn^{2+} intercalation, whereas a dry interface accepts the Zn^{2+} with significant resistance (see the dotted box).

To understand why H_2O migrates into $VOPO_4$, the chemical environment of H_2O molecules in 0.1 M $Zn(OTf)_2$-AN electrolyte was investigated by FTIR (Figure 2f). When H_2O-content crossed the threshold between 0.5%~1%, a strong absorption abruptly appeared at 3550 cm^{-1} with a shoulder at ~3410 cm^{-1}, which have been indexed to the aggregated H_2O molecules [13]. In other words, at 1% H_2O-content, there are suddenly "free" H_2O-molecules available that neither coordinate with salt cation/anion nor the organic molecule. Upon contact with anhydrate $VOPO_4$, they are thermodynamically driven into the lattice.

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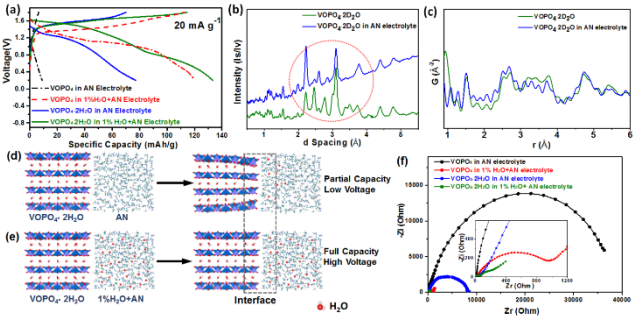

Figure 3. (a) The voltage profile of $VOPO_4{\cdot}2H_2O$ between 0.2 V and 1.9 V in the 0.1M $Zn(OTf)_2$-AN with different water contents at 20 mA/g. (b) The neutron diffraction and (c) the PDF refinement of neutron scattering of the $VOPO_4{\cdot}2H_2O$ equilibrated with anhydrous electrolytes for 24 hours. The scheme of $VOPO_4{\cdot}2H_2O$ in the 0.1 M $Zn(OTf)_2$-AN (d) without water and (e) with 1% H_2O. (f) EIS results of $VOPO_4$ and $VOPO_4{\cdot}2H_2O$ in different electrolytes.

One wonders whether the migration of H_2O molecules could also happen when hydrated $VOPO_4{\cdot}2H_2O$ meets water-free electrolyte, where residual H_2O in $VOPO_4$ lattice serves as the source of hydration for the electrolyte/electrode interface. Indeed, a higher capacity of ~135 mAh/g was delivered by the $VOPO_4{\cdot}2H_2O$ in electrolyte containing 1% H_2O than that (122 mAh/g) of the dry $VOPO_4$ (Figure 3a). Furthermore, crystal H_2O in lattice also shifts Zn^{2+}-insertion potential higher, consistent with previous reports[4e, 5]. In strong contrast, $VOPO_4{\cdot}2H_2O$ in anhydrous electrolyte only delivered ~60% of that in the wet electrolyte. As shown in Figure 3b, after equilibrating $VOPO_4{\cdot}2H_2O$ with anhydrous electrolyte for 24 hours, a new set of diffraction peaks arose, indicating a two-phase transformation, which is also revealed by XRD (Figure S4). PDF analysis revealed a decrease in O-H bond intensity (r=1Å) due to the partial dehydration of $VOPO_4$ [14]. A schematic illustration is thus constructed (Figure 3d and 3e), which visually depicts the water exchange between $VOPO_4{\cdot}2H_2O$ and electrolytes. The extraction of H_2O by the anhydrous electrolyte from the interfacial region of $VOPO_4{\cdot}2H_2O$ leads to a poorly hydrated interface, which disfavors the Zn^{2+} diffusion.

Since full cell kinetics are determined by the cathode (Figure S5), the charge-transfer resistances (R_{CT}) at the cathode–electrolyte interface, as represented in EIS [15] using three-electrode cells, are much higher in the anhydrous electrolytes, while a hydrated electrode $VOPO_4{\cdot}2H_2O$ effectively decreases the resistance (Figure 3f). The minimum in R_{CT} occurs only when H_2O is present in both electrolyte and electrode lattice, confirming that H2O dictates the Zn-ion diffusion. In the optimum scenario, the presence of H_2O in both bulk electrolyte and electrode lattice would create a continuous and fully hydrated ionic passage with little energetic barriers, significantly benefiting the Zn^{2+} desolvation porcess that would be otherwise rather difficult [16].

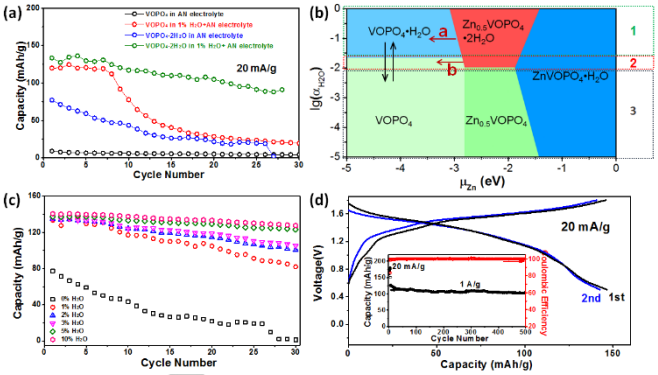

Figure 4. (a) The cycling performance of $VOPO_4$ and $VOPO_4{\cdot}2H_2O$ in different electrolytes. (b) The phase diagram of $Zn_xVOPO_4{\cdot}nH_2O$ as a function of water activity (α_{H2O}) and Zn chemical potential (μ_{Zn}). (c) The cycling performance of VOPO4·2H2O in electrolytes with different water contents at 20 mA/g. (d) The electrochemical performance of $VOPO_4{\cdot}2H_2O$ in 4 m $Zn(OTf)_2$ aqueous electrolyte.

All experimental evidences converge to the conclusion that water-redistribution occurs spontaneously between electrolyte and $VOPO_4$ and does not rely on the electric field. The sheer driving force behind this process should be the water activity (α_{H2O}) or its concentration gradient in two neighboring materials [17]. Given the fact that α_{H2O} in the electrolyte depends on the H_2O concentration that may change with time, and that water uptake for the electrode also depends on α_{H2O}, the water concentration equilibrium between electrolyte and electrode should be a key parameter for cycling stability. Figure 4a shows the cycling performance of $VOPO_4$ and $VOPO_4{\cdot}2H_2O$ in anhydrous and moist electrolytes, respectively. The dehydrated $VOPO_4$ extracts H_2O from the moist electrolyte and became electrochemical active, but as expected, the capacity decayed rapidly after only several cycles, because H_2O diffusion into $VOPO_4$ would dehydrate the interface and reduces α_{H2O} in the electrolyte. Equally, $VOPO_4{\cdot}2H_2O$ showed a persistent capacity fading in the dry electrolyte because the lattice H_2O kept migrating into the electrolyte, leaving a insufficiently hydrated interface and lattice. In comparison, the optimum capacity retention is demonstrated for $VOPO_4{\cdot}2H_2O$ in the moist electrolyte (Figure S6).

To reveal the influence of α_{H2O} during the zincation process of $VOPO_4$, the phase diagram of $Zn_xVOPO_4{\bullet}nH_2O$ as a function of α_{H2O} and Zn chemical potential (μ_{Zn}) are calculated using DFT simulations (Figure 4b). The high μ_{Zn} refers to a highly zincated $Zn_xVOPO_4{\bullet}nH_2O$, while decreasing μ_{Zn} corresponds to the de-zincation. In region 1 with α_{H2O} above 10^{-1}, the reversible zincation process happens between the two hydrated phases through route a. In region 3 with low α_{H2O}, the reversible zincation could not

proceed. In the intermediate region 2 with moderate α_{H2O}, the four-phase coexistence makes the process rather complicated, where the de-zincation could proceed through route b. As one could predict from the phase diagram, the de-zincation of $Zn_{0.5}VOPO_4{\cdot}2H_2O$ along with the release of H_2O into the electrolyte increases α_{H2O} in the electrolyte. If the increased α_{H2O} is not sufficiently high to support the hydration of $VOPO_4$, the capacity decay will happen. Therefore, this region should be circumvented to achieve desirable cyclability.

Although the quantitative relation between α_{H2O} and the H_2O content in the electrolyte needs further investigation, the phase diagram shows that once H_2O content is high enough to maintain the electrochemical process in region 1, a reversible and facile Zn^{2+} diffusion could happen, as confirmed by cyclability of $VOPO_4{\cdot}2H_2O$ in different electrolytes (Figure 4c). When the water content increased from 0 to 5%, the capacity retention improved significantly, although excessive water content above 5% will not further enhance the cycle stability. Certainly, $VOPO_4{\cdot}2H_2O$ could cycle well in the pure aqueous electrolyte and delivered a discharge capacity of 147 mAh/g (Figure 4d), with excellent cycling stability at a decay rate of 0.027% per cycle at the high rate of 1 A/g. On the other hand, similar cycling stability is achieved by VOPO4 (Figrue S7), if sufficient time is given to allow the interface as well as the electrode lattice to be fully hydrated. It is noteworthy that even in the aqueous electrolyte the water exchange still happens and the dynamic equilibrium could be easily achieved, as indicated by the reversible structural evolution (Figure S8).

In conclusion, we demonstrate that $VOPO_4$ could be utilized as a Zn^{2+}-intercalation host, and that H_2O molecule plays a critical role in enabling its electrochemical activity and reversibility. A hydrated electrode/electrolyte interface assists facile Zn^{2+} diffusion, while the lattice H_2O dictates Zn^{2+}-insertion potential. It is proposed that only H_2O molecules with enough activity could shuttle between the electrolyte and electrode, and a dynamic H_2O equilibrium is eventually established between the the electrode lattice and the electrolyte. To achieve a high cycability, α_{H2O} should be above 10^{-1}. Although the present work selects Zn^{2+} and $VOPO_4$ host as candidates of thorough investigation, the fundamental mechanism established herein should universally apply to electrode/electrolyte systems of other bivalent ion chemistries.

Experimental Section

Experimental Details could be found in the supporting information.

Acknowledgements

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Keywords: Aqueous battery • Zn-ion battery • Multivalent battery • H_2O diffusion • Kinetics

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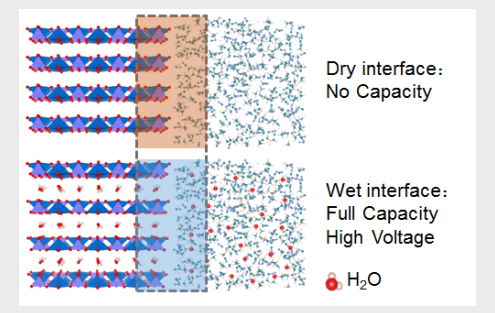

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