

The Effect of Aqueous Solution Treatment on The Li⁺ Storage Characteristics of Ti₃C₂Cl_x MXene Produced from Molten Salt

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Introduction

Due to the distinctive chlorides and -O surface groups, molten salt (MS) produced MXenes have been reported to have remarkable pseudocapacitive Li⁺ storage performance. We study the effect of different aqueous solutions (including H₂O, 0.5 M H₂SO₄, 0.5 M NaOH, and 0.5 M ammonium persulfate (APS)) washing on the Li⁺ storage characteristics of Ti₃C₂Cl_x MXene. Ti₃C₂Cl_x-H₂O, Ti₃C₂Cl_x-H₂SO₄, Ti₃C₂Cl_x-NaOH, and Ti₃C₂Cl_x-APS have maximum specific capacities of 137, 140, 103, and 204 mAh g⁻¹, respectively. The APS treatment, it is believed, will raise the -O termination content, resulting in increased lithium-ion storage capacity. NaOH treatment, on the other hand, causes partial conversion of Ti₃C₂Cl_x to TiO₂, lowering the lithium-ion storage capacity. The importance of aqueous solution washing on Li is highlighted in this study.

MXene is a 2D material made up of transition metal carbide/nitride that is made by selectively etching the A layer of the MAX phase precursor. Mn+1XnTx is the generic formula for MXene, where M stands for early transition metal elements, X for carbon or nitrogen, and Tx for surface functional groups (-O, -F, -Cl, and so on). MXene has demonstrated vast application potential in energy storage catalysis, electromagnetic shielding, and other fields due to its strong electrical conductivity and variable surface chemistry. MXene is traditionally made by chemically etching the A elements in MAX phase precursors with HF-containing solutions as etchants. MXenes etched with HF are typically hydrophilic and terminated on the surface with a combination of -F, -O, and -OH functional groups

The toxicity and corrosiveness of HF, on the other hand, raises the potential of contamination throughout the preparation process. To produce MXenes with chlorides terminations, a generic technique using chlorides Lewis acid salts (CuCl₂, NiCl₂, ZnCl₂, etc.) as etchants has been developed in recent years. APS acidic aqueous solution is used to remove solidified salts and metal particles (reduction of metal ions from Lewis acidic salt, Cu²⁺ in CuCl₂), which is an essential step in generating MXenes via the molten salt etching process. The -O surface groups produced during aqueous solution washing are thought to be important in increasing Li⁺ storage characteristics.

Indeed, MS-derived MXenes with high Li⁺ storage capacities and high rate performance have shown significantly different electrochemical signatures than HF-derived MXenes. The enhanced Li⁺ affinity of MXenes with -O surface groups has been suggested by certain theoretical investigations. However, there is no direct evidence from experimental studies.

Given the rising interest in MS-derived MXenes, it's important to understand how aqueous solution treatment affects the Li⁺ storage properties of MS-derived MXenes. The molten salt etching process was used to create Ti₃C₂Cl_x-MXene, which was then washed with H₂O, H₂SO₄, NaOH, and APS solutions. The produced MXenes' surface terminations and Li⁺ storage characteristics were studied

The molten salt approach was used to make Ti₃C₂Cl_x, as previously reported. In a nutshell, Ti₃AlC₂ (400 mesh) was used as the

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precursor, with a eutectic mixture of NaCl and KCl serving as the salt bed. Due to the use of NiCl₂ as an etchant, the nickel metal in the reaction products can be extracted with a magnet without the use of an acidic solution. Ti₃AlC₂, NiCl₂, NaCl, and KCl have a molar ratio of 1:6:10:10. The Ti₃AlC₂ powder was placed in the bottom of the crucible, followed by a uniformly mixed etchant and the salt bed on top of the Ti₃AlC₂ powder, and the crucible was then placed in a muffle furnace.

An X-ray diffractometer was used to measure the samples' X-ray diffraction pattern using Cu K radiation (= 0.15406 nm) and a measurement angle range of 5°-90°. The morphology and element analysis of the sample are seen using a scanning electron microscope (SEM, JSM-7900F) and an energy dispersive spectrometer (EDS, Ultima MAX 65). X-ray photoelectron spectroscopy (XPS-UltraDLD) was used to examine the composition and chemical state of samples, with the binding energy referring to the C 1s of 284.6 eV.

A CR2030 coin-type half-cell was used to examine the electrochemical characteristics of freshly produced powders. The working electrode was made by coating a slurry of MXene powder, acetylene black, and PVDF binder on Cu foil in an 8:1:1 mass ratio, then drying it for 10 hours in a vacuum oven at 80°C. The counter and reference electrodes were made of lithium foil, while the separator and electrolyte were made of glass fibre (Whatman, GF/A) and 1 M LiPF₆ (in 1: 1 v/v EC/DMC). The coin cells were built in a glove box with a water and oxygen concentration of less than 0.01 parts per million. Within the potential range of 0.1–3 V, the galvanostatic charge–discharge curves and cyclic voltammetry were tested using the Neware battery testing equipment and Autolabm204 electrochemical workstation.

In conclusion, the effects of H₂O, H₂SO₄, NaOH, and APS solutions on Ti₃C₂Cl_x MXene Li⁺ storage characteristics were examined. The Ti₃C₂Cl_x-APS electrode provided the best Li⁺ storage performance, while the Ti₃C₂Cl_x-NaOH electrode provided the worst Li⁺ storage performance. The increased Li⁺ storage performance of the Ti₃C₂Cl_x-APS electrode is explained by an increase in -O terminations on the MXene surface. NaOH treatment, on the other hand, causes partial conversion of Ti₃C₂Cl_x-MXene to TiO₂, lowering Li⁺ storage performance. This study underlines the importance of aqueous solution treatment on MXene surface chemistry and demonstrates how solution washing treatment may be used to tune the Li⁺ storage characteristics of MS-derived MXenes.