Oxidation of Ti_3C_2 MXene probed by soft X-ray absorption spectroscopy

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MXenes are a new class of 2D materials consisting of transition metals carbides and nitrides that demonstrate extraordinary properties for electrochemical energy storage [1]. However, the electronic structure of MXenes is largely unexplored till date, although it could provide more insights into the energy storage mechanism [2]. Several reports have shown that MXenes can oxidize naturally in various environments [3]. Understanding the possible origins of the oxidation of MXene and its impact on the electronic state of MXene is fundamental to better estimate aging behavior of MXenes in relevant conditions.

In this work, the MXenes $Ti_3C_2T_x$ have been investigated in water using soft X-ray absorption spectroscopy (XAS) at the O K-edge and Ti L-edge before and after urea intercalation. The samples have been characterized both in the dried state under vacuum conditions and also in aqueous dispersions using a flow cell system to evaluate the impact of the solvation on the MXene oxidation state. For dispersed samples, total fluorescence yield (TFY) measurements have been performed whereas total electron yield (TEY) measurements have been used to study the dry samples.

Furthermore, X-ray photoemission electron microscopy (X-PEEM) has also been applied to pure MXene and urea-intercalated MXene to record spatially resolved XAS spectra at the Ti L-edge and O K-edge on individual MXene flakes.

In the present work, combining XAS and X-PEEM, different oxidation levels of the Ti atoms before and after urea intercalation will be shown. The impact of MXene dispersion in aqueous solution on the electronic structure will also be discussed.

This study provides new insights toward the understanding of MXene modification after urea intercalation and solvation. It demonstrates that XAS performed directly in aqueous media is a powerful method for the characterization of energy-related materials. Future experiments related to *in operando* characterization of MXene-based supercapacitors will also be mentioned.

References

[1] B. Anasori et al., Nature Reviews Materials, 2, (2017) 16098
[2] M. R. Lukatskaya et al., Adv. Energy Mater, 5, (2015) 1500589
[3] C. J. Zhang et al., Chem. Mater, 29 (11), (2017) 4848-4856