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Towards better materials for energy storage: insights into electrode/electrolyte interface from *operando* techniques

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The lecture aims at providing a comprehensive insight into properties of the materials suitable for electrochemical energy conversion and storage by the application of the *in-situ* and operando techniques such as Raman spectroscopy, Quartz Crystal Microbalance (EQCM) or Scanning Electrochemical Microscopy (SECM) for determination of charge storage phenomena and ageing factors in activated carbon-based supercapacitors. Before going into technical details, the attendees will become familiar with the most common electrochemical energy/conversion and storage systems, their properties and challenges.

In situ Raman investigation for activated carbon electrodes operating in neutral aqueous media such as Li_2SO_4 or $LiNO_3$ solutions indicated that there is a mild oxidation of the positive electrode during cycling (vibration modes from oxygen-based functionalities found) while the surface chemistry of the negative electrode appears to be stable. The EQCM study confirmed significant frequency/mass variation on the positive side, while the negative electrode remained stable. However, the SECM demonstrated that during positive and negative polarization, the thickness (and volume) of the electrode changed remarkably. Additionally, it has been found that the charge/discharge process, even at cell voltages well below the electrolyte decomposition values, induces quasi-reversible changes of cell pressure.

Interesting results were obtained for carbon electrodes operating in KI solutions. It has been confirmed that the iodide anion undergoes several redox processes and strongly interacts with the activated carbon surface. An oxidation of the carbon surface has been identified near the iodide/iodine redox activity potentials. The EQCM study confirmed the presence of various iodine species in the electrolyte. Carbon 'corrosion' has been observed especially for a more concentrated iodide solution. However, we proved that the IO_3^- anion does not contribute significantly to this process.

Finally, operando studies allowed for elucidation on the charge storage mechanisms in water-based capacitive systems and identification of the tentative reasons for their performance fade.

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