Insights into carbon electrode/aqueous electrolyte interface from *the operando* approach

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The lecture aims at providing a comprehensive insight on 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 aging factors in activated carbon-based supercapacitors.

In-situ Raman investigation for activated carbon electrodes operating in neutral aqueous media like Li_2SO_4 or $LiNO_3$ solutions indicated that there is mild oxidation of positive electrode during cycling (vibration modes from oxygen-based functionalities found) whereas the surface chemistry of negative electrode appears to be stable. EQCM study confirmed significant frequency/mass variation on the positive side, whereas the negative electrode remained stable. However, SECM demonstrated that during positive and negative polarization, the thickness (and volume) of the electrode changed remarkably. Additionally, it has been found that charge/discharge process, even at the 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 iodide anion undergoes several redox processes and actively interacts with activated carbon surface. Oxidation of carbon surface has been identified near the iodide/iodine redox activity potentials. EQCM study confirmed the presence of various iodine species in the electrolyte. Carbon 'corrosion' has been observed especially for more concentrated iodide solution. However, we proved that IO_3^- anion does not contribute significantly to this process.

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