Fabricação e caracterização de um duplo sensor eletroquímico combinando SECM-SICM

Fabrication and characterization of dual-functional probe combining Calcium Ion Selective Potentiometric Microscopy and Ion Conductance Microscopy (SECM-SICM)

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Abstract: Herein, we report the fabrication and use of a new dual function probe, combining the imaging and localized delivery capabilities of a scanning ion conductance microscopy (SICM) probe with an ion selective microelectrode (ISME). Applications on dynamic processes at calcite microcrystals interfaces were performed in an attempt to contribute to develop nanoscale electrochemical imaging techniques. These probes were fabricated from a laser pulled sharp-ended glass theta pipet, where one of the barrels was filled with electrolyte solution and a quasi-reference counter electrode (QRCE) inserted in it to produce the SICM part. The other barrel was a potentiometric ISME based on liquid membranes that after silanization procedure was filled with a calcium ionophore to form a potentiometric SECM part. Two types of electrical contact were constructed and tested for this part of the multifunctional probe. The first was a *liquid-based contact* employing a QRCE inserted in the reference inner filling solution that contains a certain concentration of the ion of interest in contact with the calcium ionophore membrane. In the second type, a conducting polymer based on 4,3-ethylenedioxythiophene (EDOT) coated carbon microfiber forming a *solid-contact* was inserted directly into the calcium ionophore membrane. The *solid-contact* microsensors showed a higher stability with time, making them suitable for long duration electrochemical imaging. The capabilities of these dual functional probes were demonstrated by employing them to investigate the dynamics of chemical processes at calcite microcrystal interfaces during the acid induced dissolution reaction. This study enabled the extraction of kinetics information related to the dissolution fluxes at different pH values through coupling probe measurements with finite element method modeling. The obtained values were in good agreement with the literature reported ones. The difficulties in fabricating nano-scale probes with multifunctional electrochemical detection restricted the technique to microscale and measurements of chemically-specific fluxes on the nanoscale using potentiometric ISME based on liquid membrane continues to be a challenge in analytical instrumentation research.

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