Scanning Electrochemical Microscopy of Graphene-based Hybrids: Insights into Physicochemical Interfacial Processes and Electroactive Site Density Distribution

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ABSTRACT

Surface (and interfacial) chemistry is found in various environments of scientific significance including biomembranes, ocean and atmospheric chemistry and applied electrochemistry. Molecular redox behavior on the surface and at the interface is drastically different than their bulk counterpart. Scanning electrochemical microscopy (SECM) is a powerful tool to investigate interfaces determining ion transfer kinetic rate, diffusion coefficient, imaging topography and electrochemical redox reactions. The significant advantage offered by SECM is its capability of probing chemical information of interfacial electron and ion transfer processes at solid/liquid interface irrespective of substrates. A constant potential is applied to the tip and electrochemical working electrode (*i.e.* the substrate in electrolyte) to drive reaction in bulk electrolyte solution of redox species (or mediator) to probe the surface of certain thickness of graphene-based hybrids. The microscaled cyclic voltammograms, probe approach (current *versus* tip–substrate distance) curves, 2D and 3D micrographs in feedback mode, were chosen for graphene/CNT, graphene/transition metal oxide as supercapacitors to probe ion adsorption and to map highly electroactive ('hot spots') sites. The SECM setup has a resolution of ~40 nm and can locate and relocate areas of interest precisely after a coarse image. We present our findings from viewpoint of reinforcing the roles played by heterogeneous electrode surfaces comprised of graphene nanosheets (conducting)/nanomaterials (semiconducting) via higher/lower probe current distributions. SECM approach curves as well as two dimensional scans elucidated the existence of regions of different conductivity and the data is analyzed in terms of edge plane defects distribution within the probes regions, determining diffusion coefficient and heterogeneous rate constant.