JOINT MEETING OF POLISH SYNCHROTRON RADIATION SOCIETY MEMBERS
AND SOLARIS CENTRE USERS

Autonomous Sampling α-Fe_2O_3 Hollow Microspheres with Carbon-Stabilized Defects: Calcination-Tuned Humidity Sensor Performance

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Control of defects and surface chemistry in metal-oxide sensors remains a significant challenge for achieving ultrahigh sensitivity and rapid response in humidity monitoring. Herein, we present a scalable mechano-thermal route to synthesize carbon-doped α-Fe_2O_3 hollow microspheres with built-in voids acting as autonomous sampling chambers. By systematically varying calcination temperature (500–900 °C), we tune crystallite size (38.2–87.6 nm), lattice strain (0.10–0.77%), dislocation density (1.4×10^{-4}–3.07×10^{-3} nm^{-2}), and C content (21→14 wt.%), thereby carbon-oxygen moiety that govern water adsorption and proton-hopping conduction. XPS/XAS confirm unusual stable Fe^{2+}/Fe^{3+} surface ratio and C–Fe–O interactions and hydrophilic sites, facilitating a dual-regime sensing mechanism transitioning from ionic conduction (11–75%RH) to Grotthuss-mediated proton transport (>75%RH), with unpredicted non-monotonic structural rearrangements at 800 °C underscore the critical role of transient Fe_3O_4 nucleation in defect activation. The Fe-500 sensor exhibits exceptional performance of 0.75 kΩ/%RH, response time of 40 s, and recovery time of 85 s, outperforming benchmark hematite-based devices..

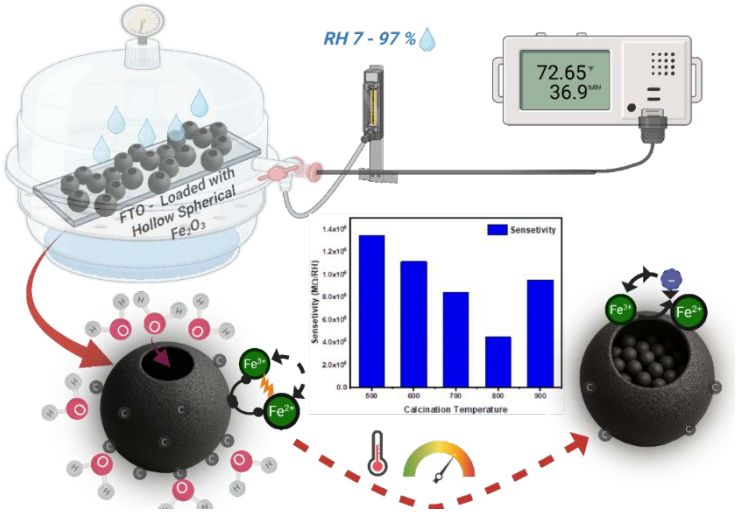

Figure 2: X-ray absorption spectra at the O K-edge (c), Fe $L_{2,3}$-edge (d), and C K-edge (e) for Sphere.