Electronic Supplementary Information

Potentiostatic Phase Formation of β-CoOOH on Pulsed LASER Deposited Biphasic Cobalt Oxide Thin Film for Enhanced Oxygen Evolution

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EXPERIMENTAL

Characterization Techniques

The phase formation and crystal structure Co_3O_4 thin films coated on silicon substrates were investigated using X-Ray Diffraction technique (panalytical) using Cu k_α radiation of wavelength 1.5418 Å with the scanning rate of 0.020 s^{-1} in the 2θ range of 20 to 80°. The crystallite size was measured using Scherrer formula. The surface morphology of thin film was obtained by SEM and Zeiss ultra-Field Emission Scanning Electron Microscope (FE-SEM) Instrument and Transmission Electron Microscope TEM (Make: FEI, The Netherlands.Model: Tecnai 20 G2). The chemical composition was also obtained from Energy Dispersive X-ray spectroscopy technique (EDAX) analysis was performed on the same FESEM instrument with a separate EDS detector connected to that instrument. The electrochemical characterization was done in the CH Instrument model CHI6048c. A platinum foil counter electrode and Hg/HgO reference electrode was used along with the fabricated thin films of spinel Co_3O_4 on FTO.

Sample Preparations for Various Material and Electrochemical Characterizations.

For XRD, SEM, FESEM and XPS analysis, the fabricated Co_3O_4/FTO thin films were directly used without any further treatment. For TEM and HRTEM analysis, the thin film was gently scratched off from FTO surfaces and dispersed in DI water by ultrasonic treatment for 5 min. the resultant dispersion was used to fabricate the TEM specimens. For RuO_2/FTO electrode fabrication, 3 mg of commercial RuO_2 procured from Sigma Aldrich was dispersed by sonication for 15 min in a 1 mL solution of DI water, isopropyl alcohol and Nafion in the volume proportion of 0.75:0.20:0.05. About 68.5 µL of the prepared ink was carefully casted on the conducting side of a FTO substrate electrode of geometrical area of 1 cm^2 which correspond to a benchmarking loading of 0.205 $mgcm^{-2}$.

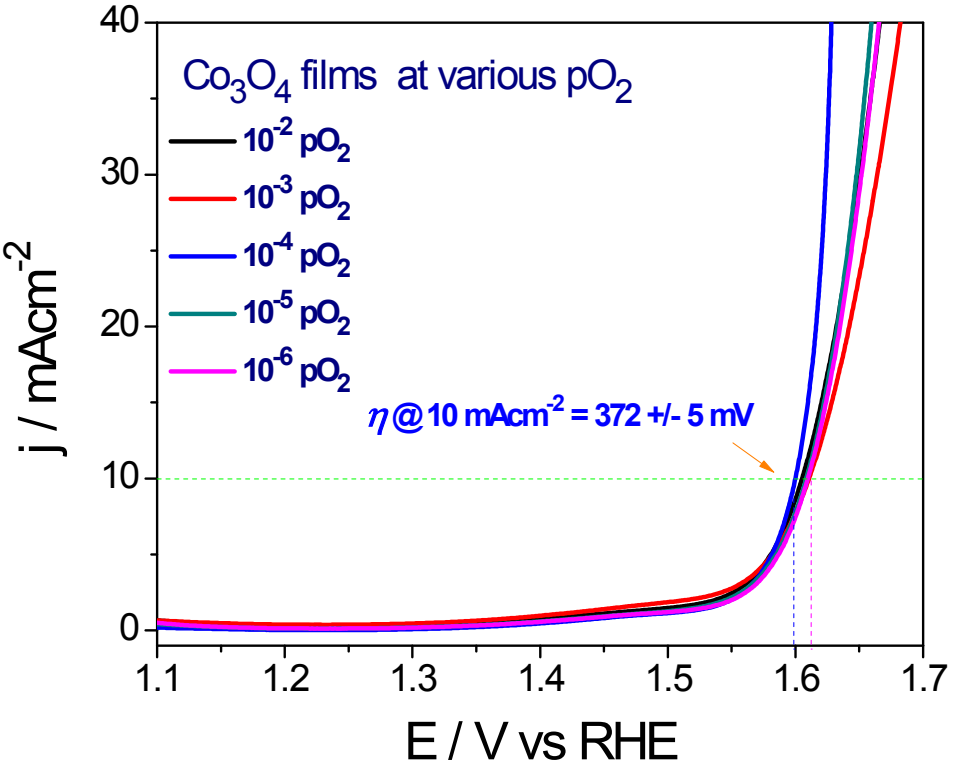

Figure S1: LSVs of Co_3O_4/FTO thin films fabricated at various pO_2.

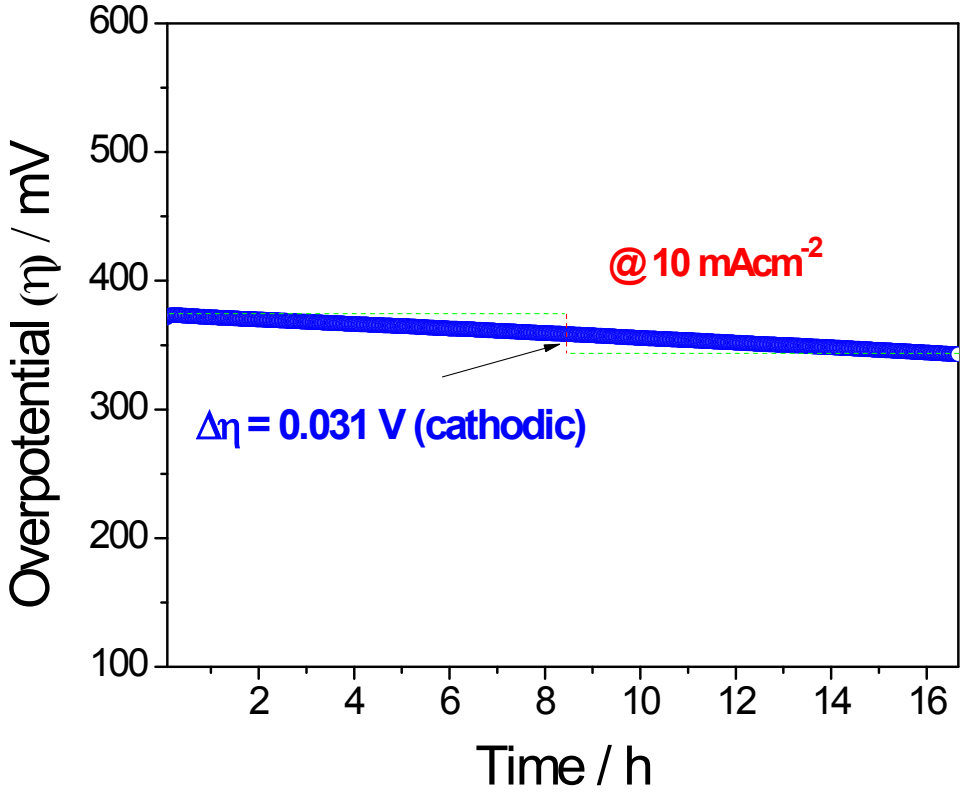

Figure S2: The chronopotentiometric response of Co_xO_y interface for a fixed current density of 10 $mAcm^{-2}$.

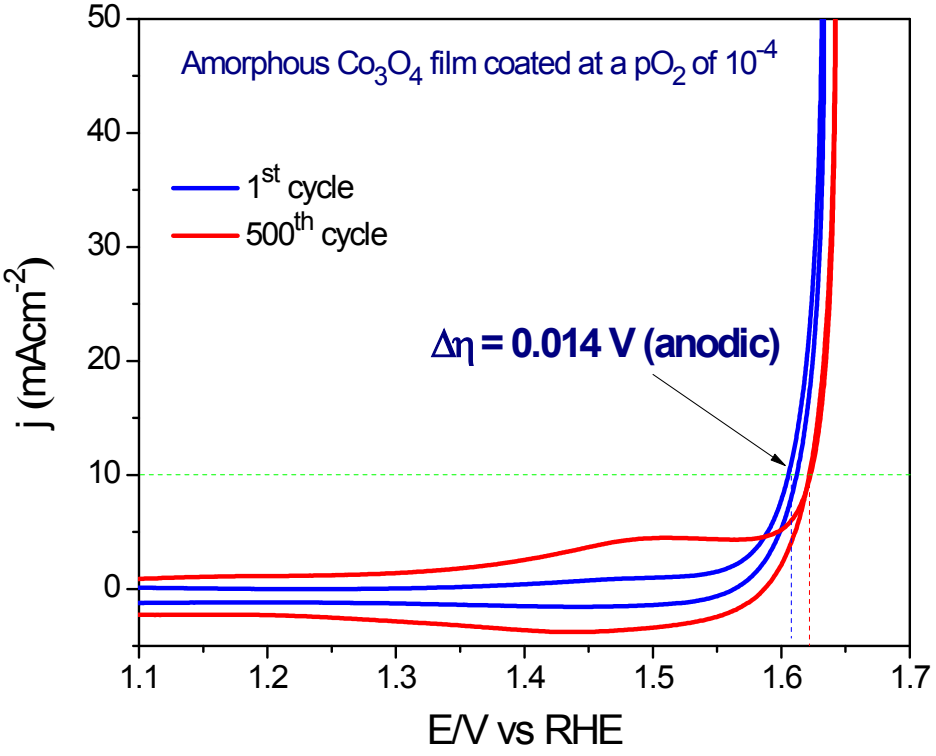

Figure S3: CVs acquired before and after AD test on Co_xO_y interface fabricated at a pO_2 of 10^{-4} Pa.

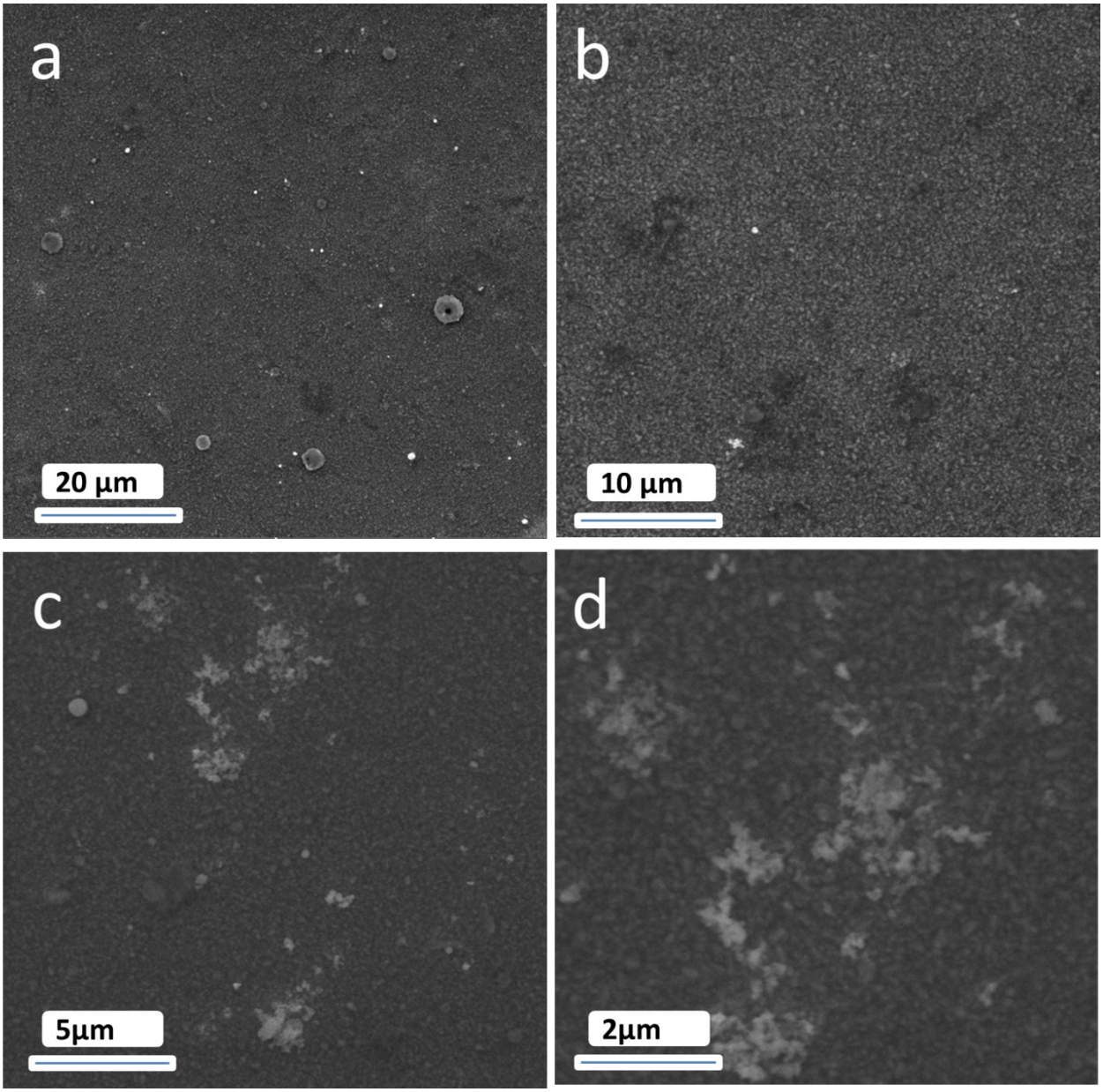

Figure S4: (a-d) SEM micrographs of Co_xO_y thin film before PSTAT activation with increasing magnification.

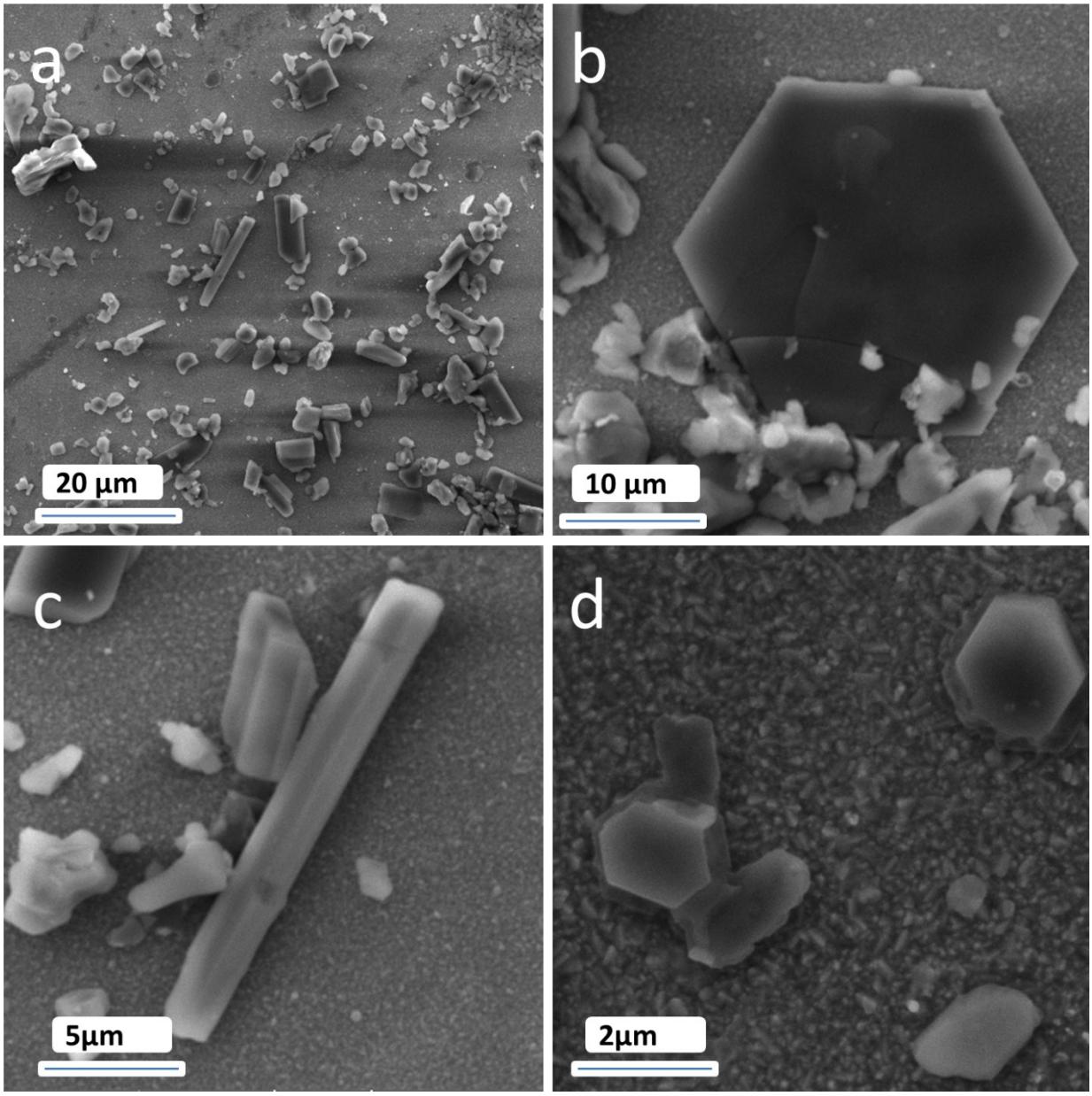

Figure S5: (a-d) SEM micrographs of Co_xO_y thin film after PSTAT activation with increasing magnification.

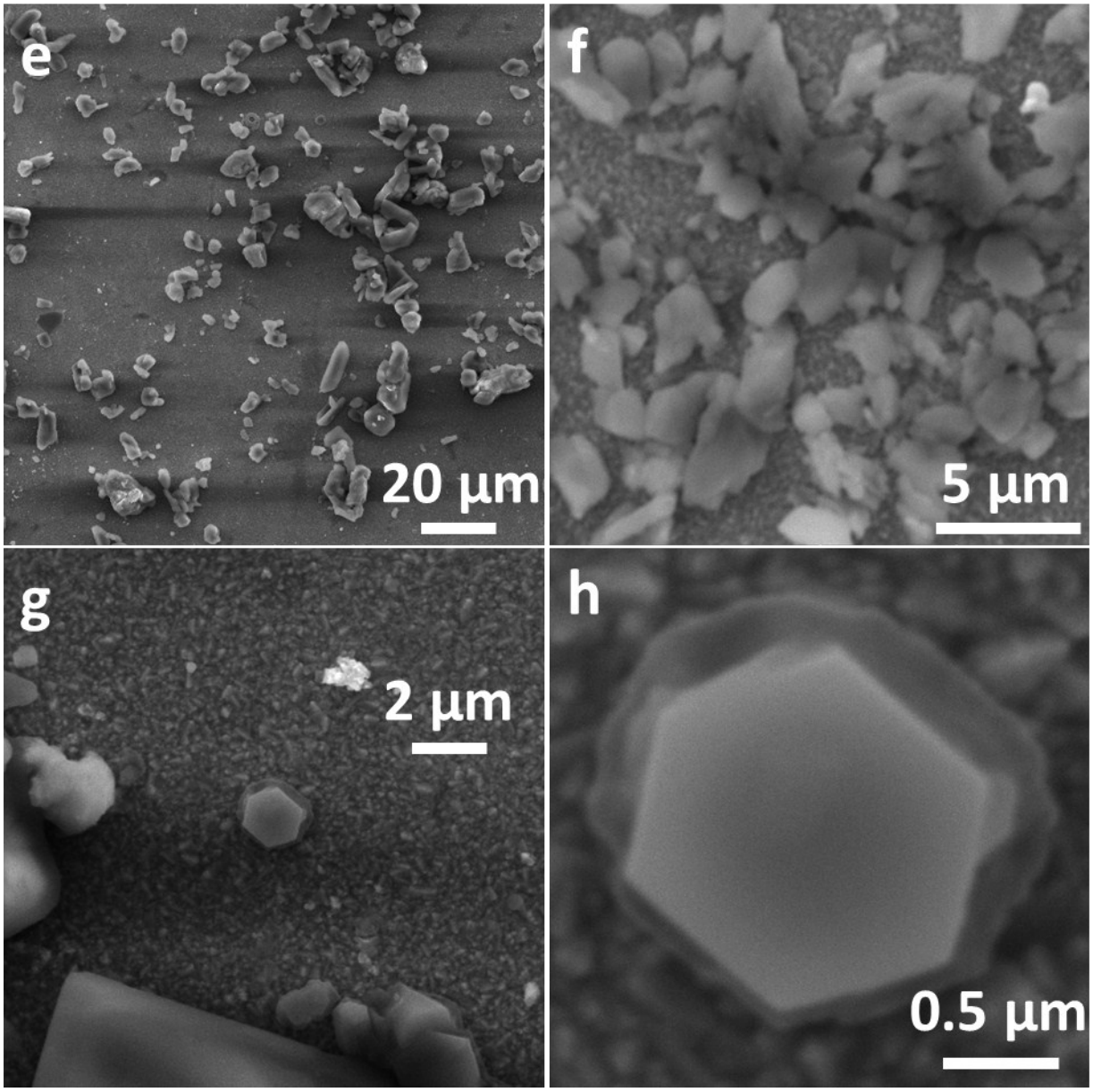

Figure S5: (e-h) SEM micrographs of Co_xO_y thin film after GSTAT activation with increasing magnification.

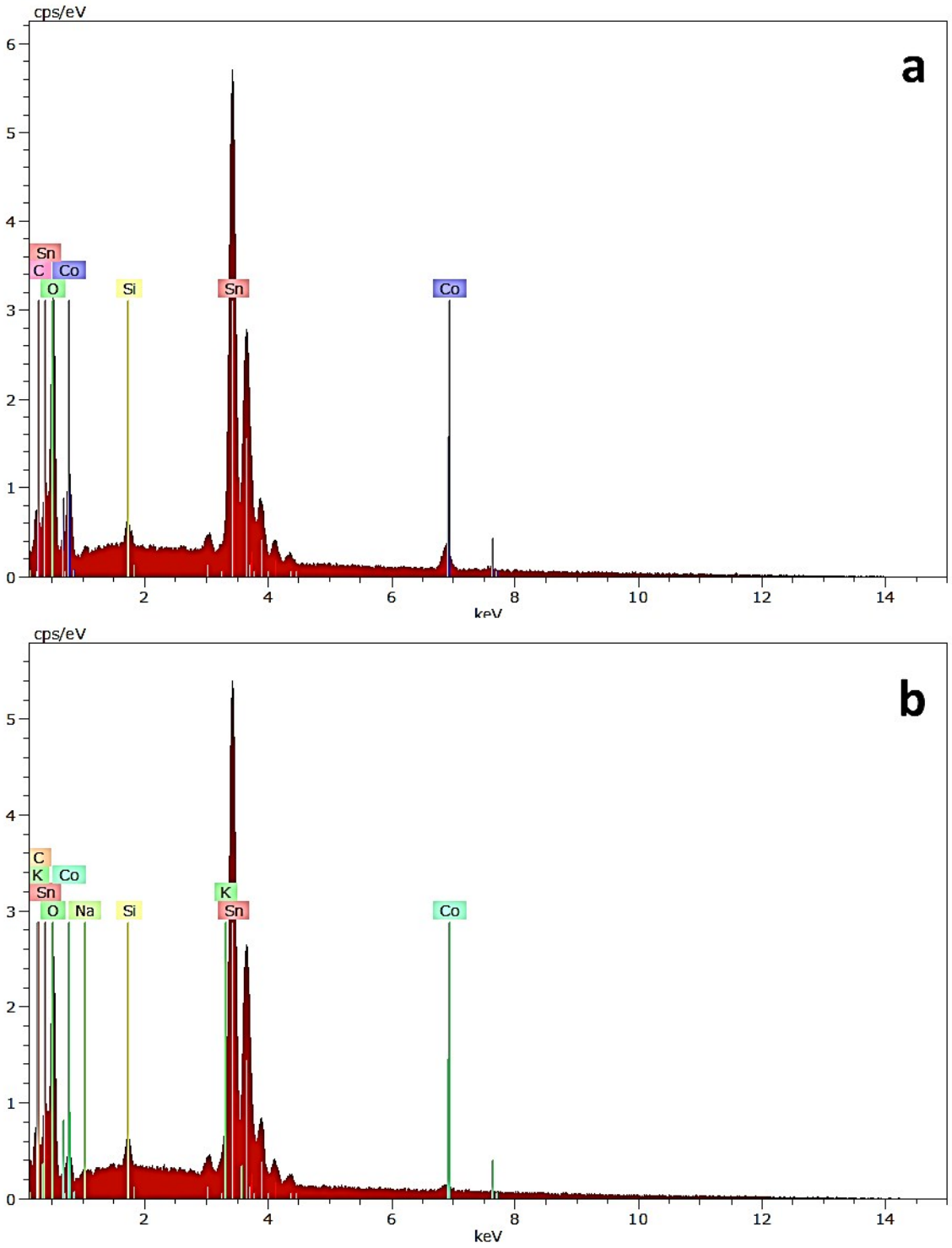

Figure S6: EDAX spectra of Co_xO_y thin film before and after PSTAT activation.

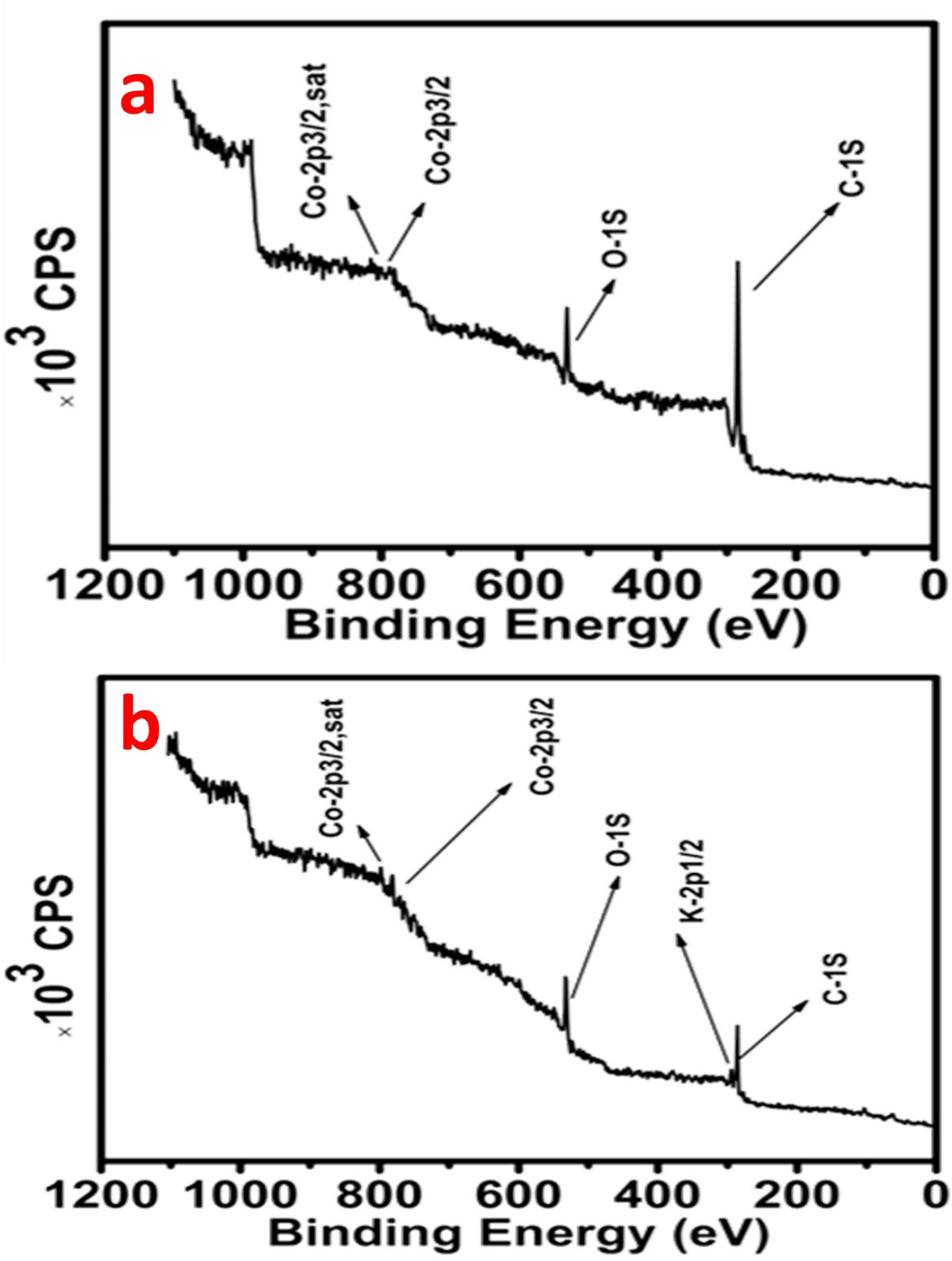

Figure S7: XPS survey spectra of Co_xO_y thin film before and after PSTAT activation.

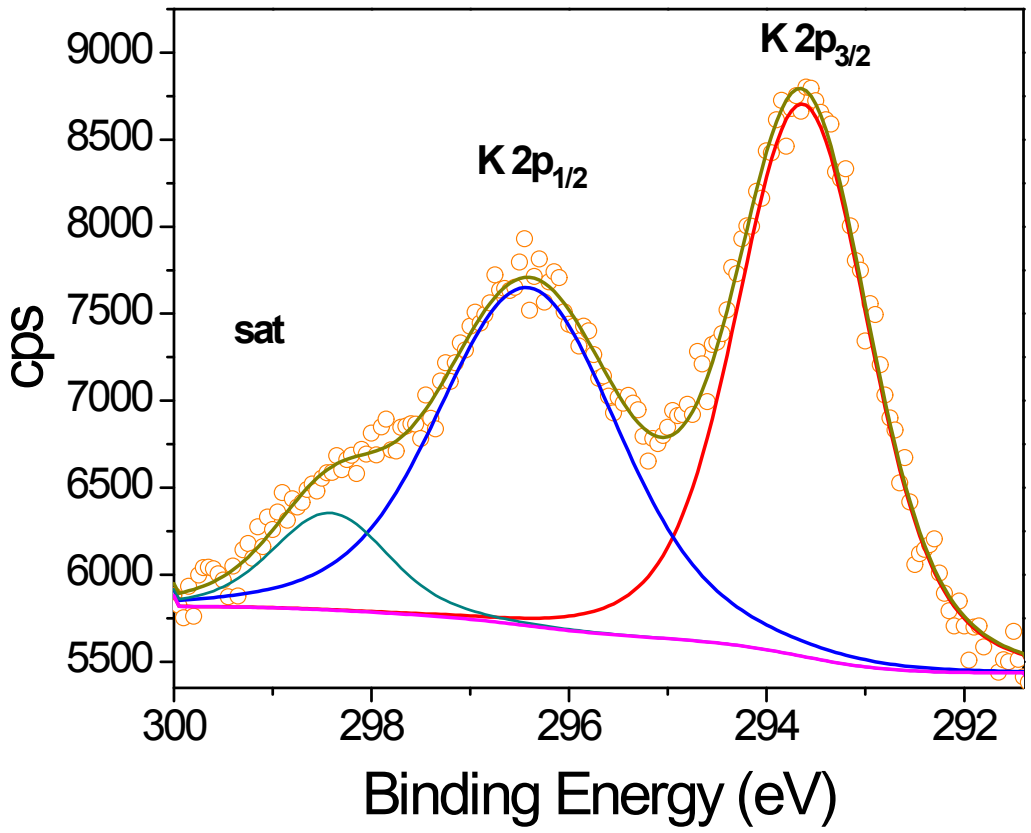

Figure S8: XPS high resolution spectrum of K 2p stated acquired on Co_xO_y thin film after PSTAT activation which proves the intercalation of K^+ ions.

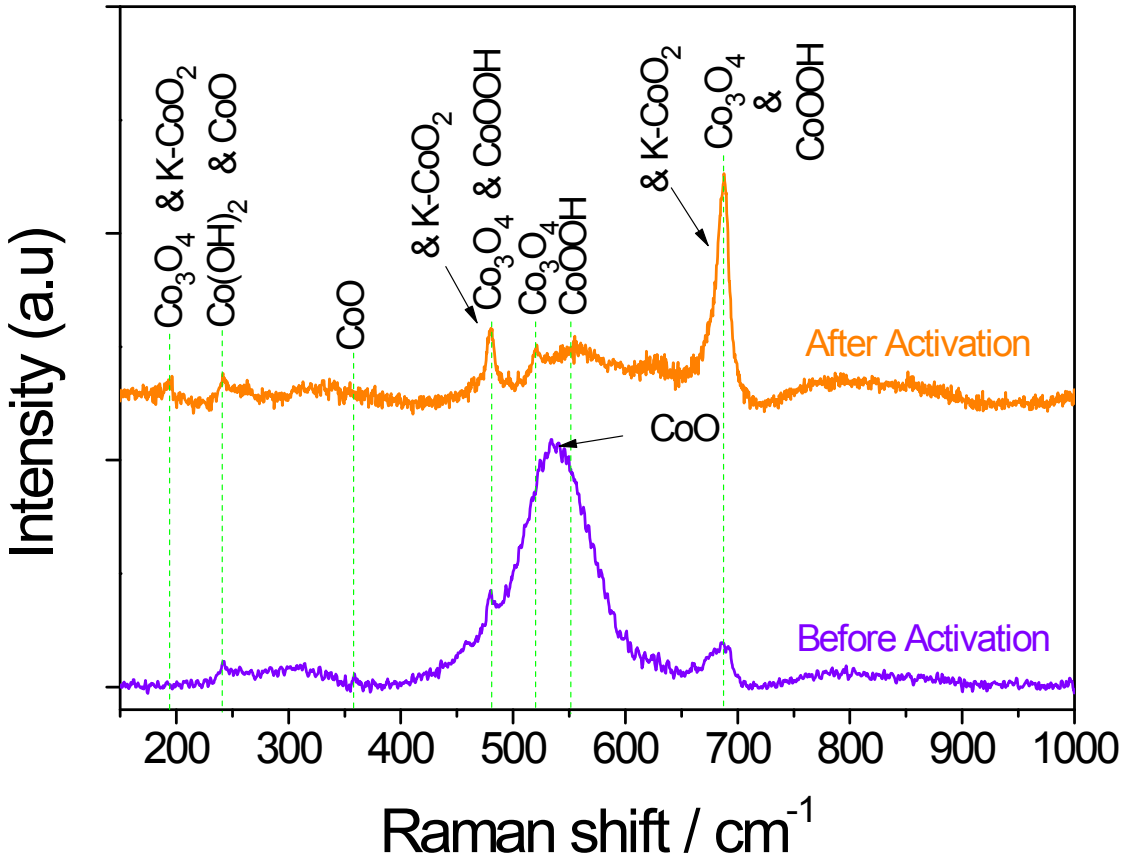

Figure S9: Raman spectra of as fabricated biphasic Co_0O_y thin film and the Raman spectra of the same after activation.