

Optimisation Study of Co Deposition on Chars from MAP of Waste Tyres as Green Electrodes in ORR for Alkaline Fuel Cells

Co Electrodeposition

The process of deposition was investigated on GC electrode via cyclic voltammetries following the procedure seen in [1].

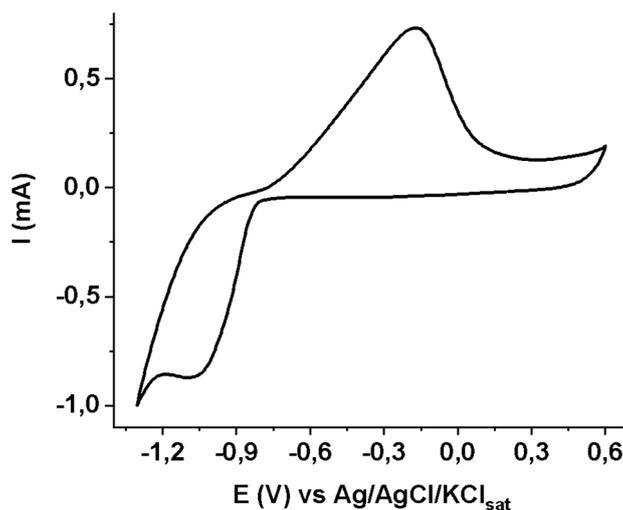
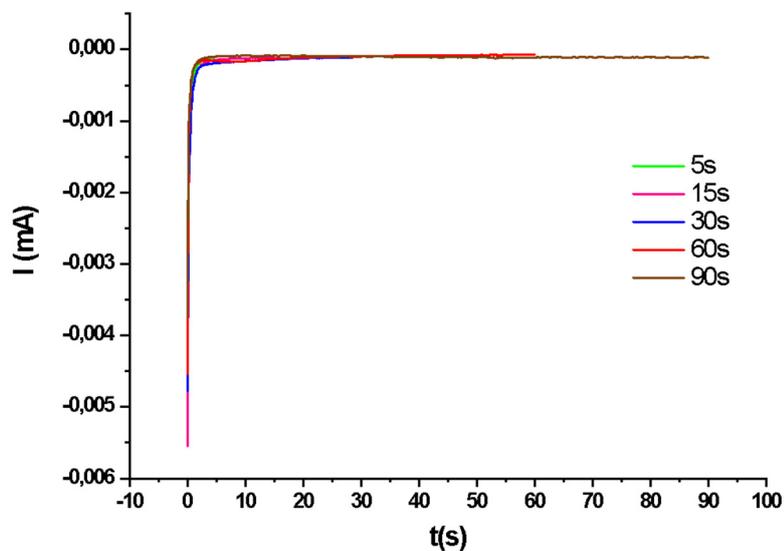


Figure S1. Co electrodeposition on GC electrode, scan in a potential range from 600 to -1000 mV vs. Ag/AgCl in KCl sat. with a scan rate of 5 mV/s.

Chronoamperometry with a constant potential of -850 mV (vs. Ag/AgCl in KCl sat.) under constant magnetic stirring is reported in Figure S2.



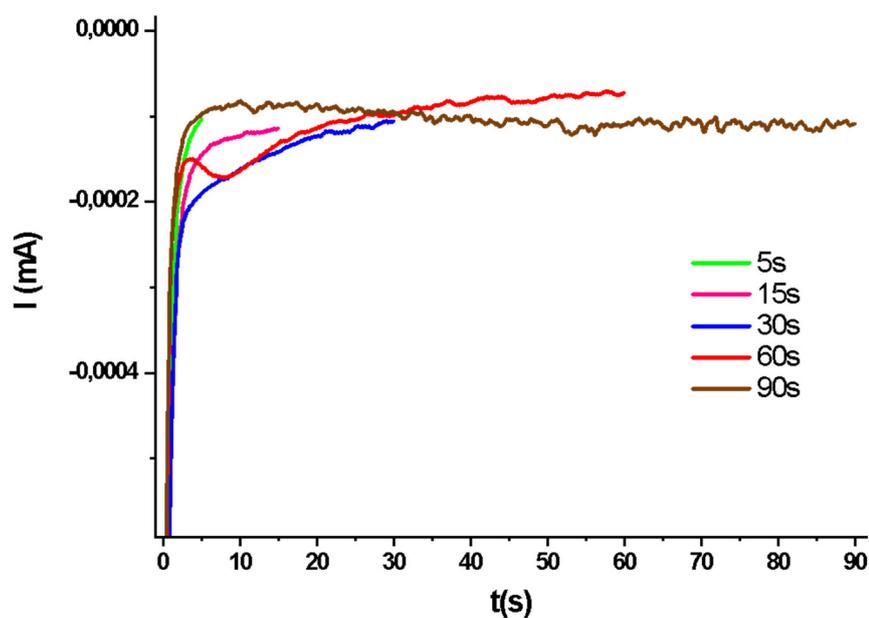


Figure S2. Chronoamperometry under constant magnetic stirring for deposition of Co on CHraw at different times in two different scales.

Figure S3 shows the electrochemical check of Co electrodeposited: the absence of anodic stripping peak is attributable to the formation of $\text{Co}(\text{OH})_2$ on the electrode surface as seen in [1].

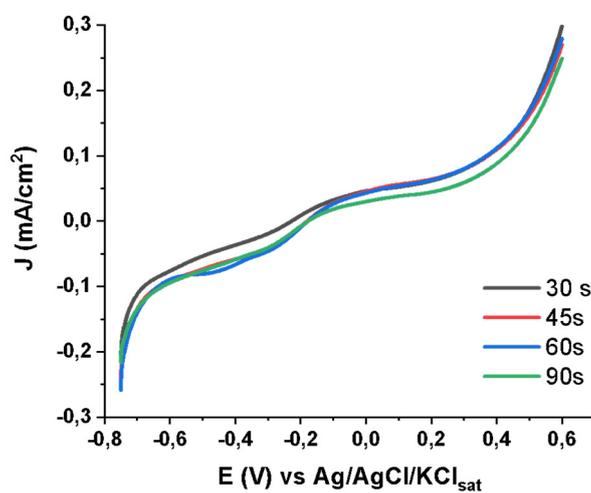
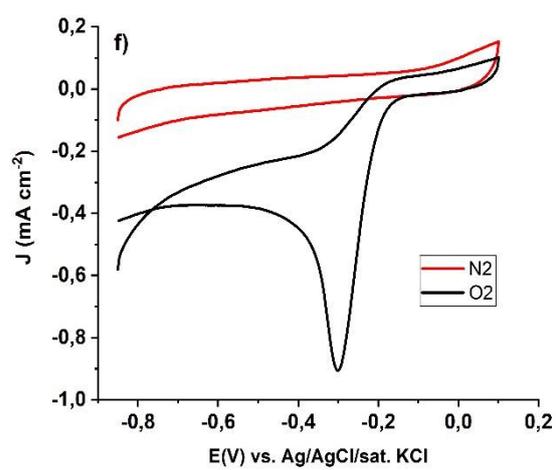
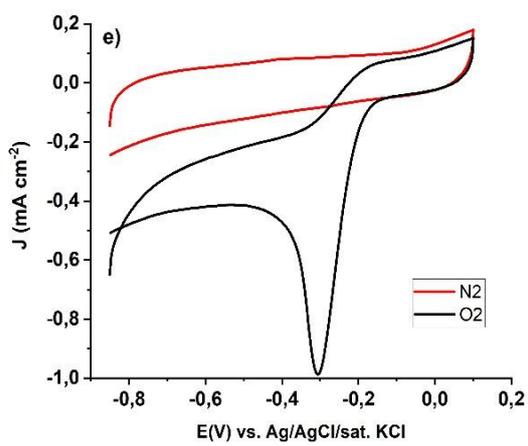
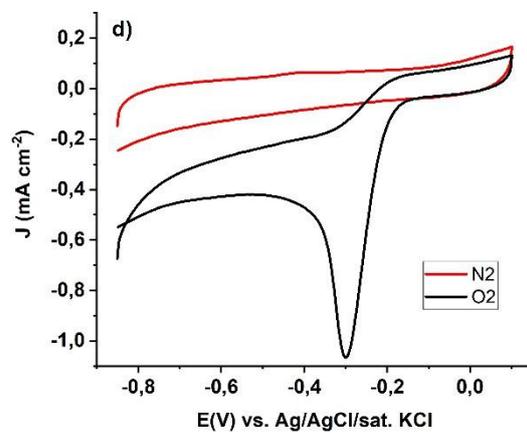
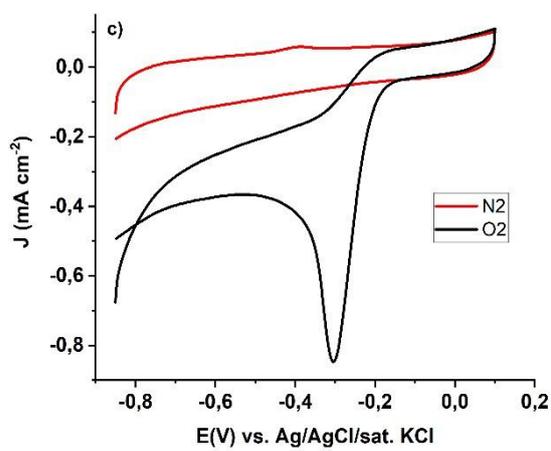
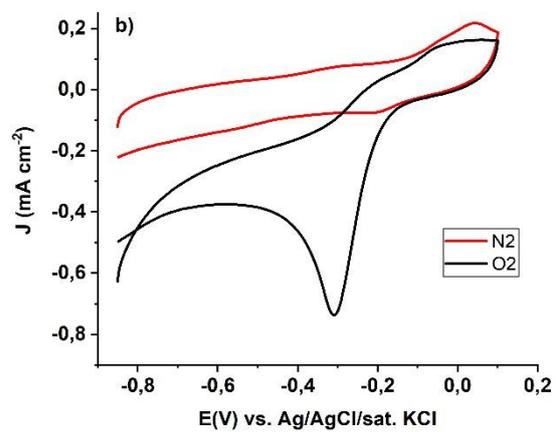
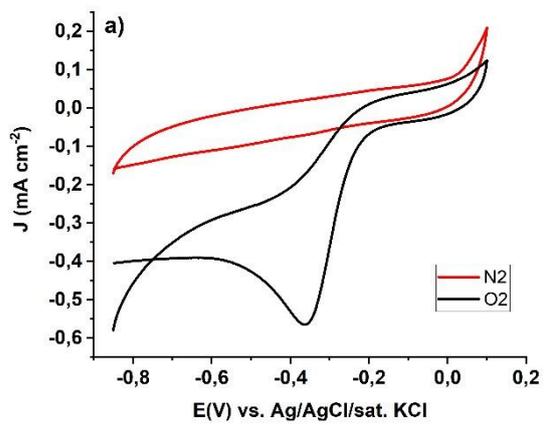


Figure S3. Stripping voltammeteries of Co electrodeposited on CHraw as a function of time. The analyses were conducted from -0.750 to +0.6 V at a scan speed of 10 mV/s in a buffer solution of $\text{NH}_4\text{OH}/\text{HClO}_4$, pH = 9.2.

Electrocatalytic Characterisation



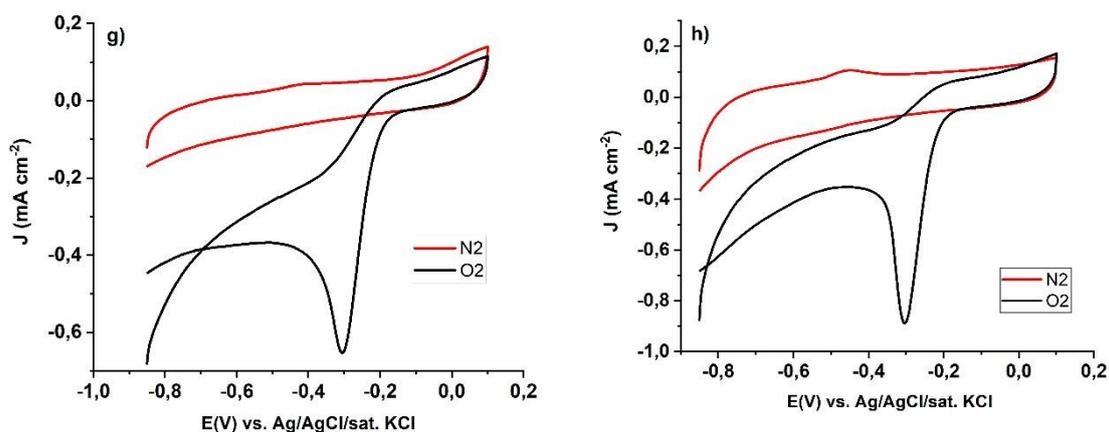


Figure S4. CVs in quiescent 0.1 M KOH aqueous solution with either saturated O₂ (black) or N₂ (red), with scan rate of 5 mV/s. Samples underwent different deposition techniques, sonochemical deposition: (a) Co; and (b) Cu. Electrochemical deposition of Co at different times: (c) 05 s; (d) 15 s; (e) 30 s; (f) 60 s; and (g) 90 s. (h) The last graph shows *CHraw* behaviour.

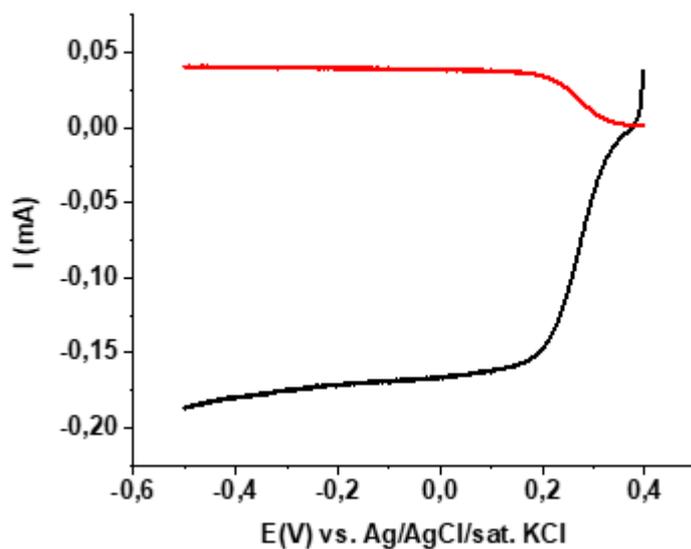
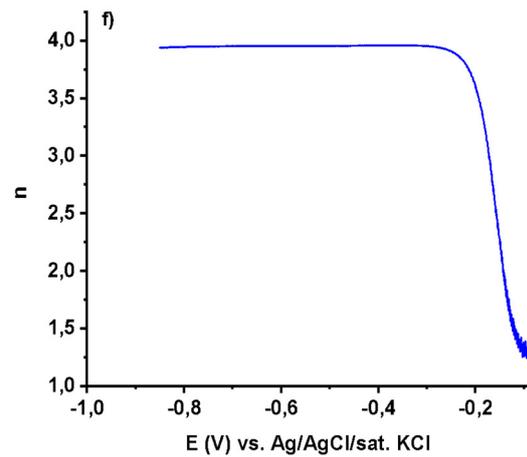
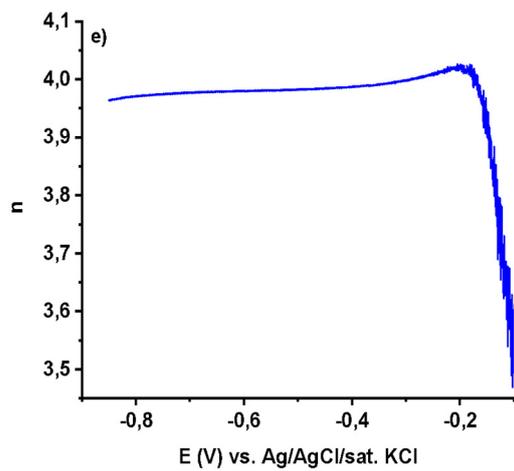
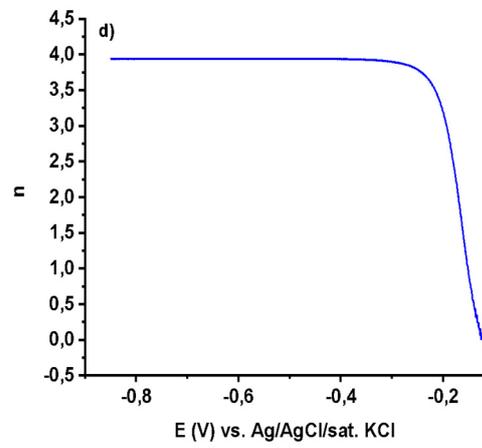
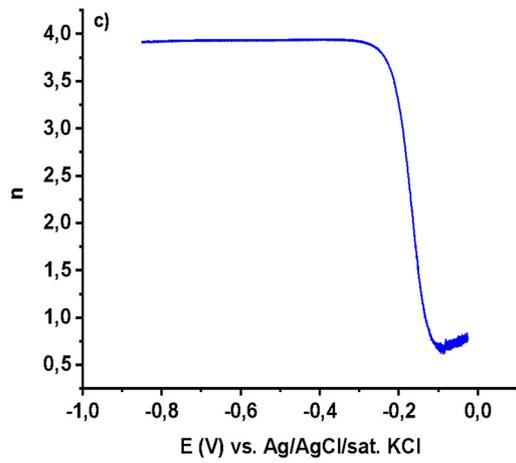
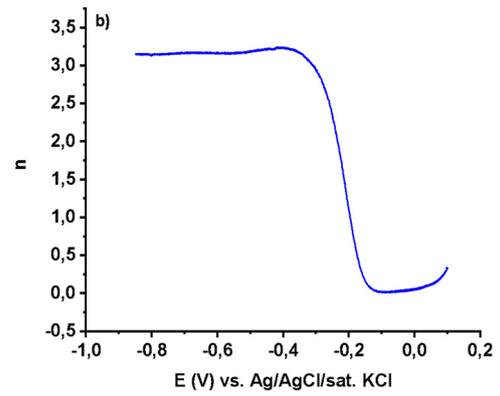
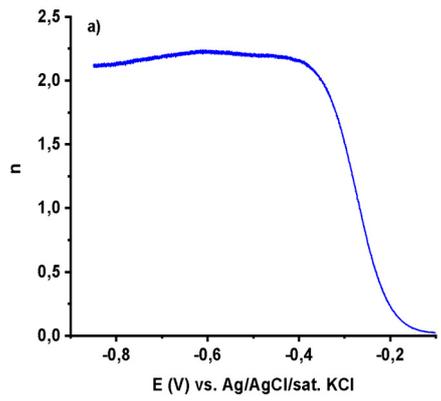


Figure S5. Typical LSV obtained from sample *CHecCo30s* by RRDE with a solution of K₃Fe(CN)₆ to calculate the collection efficiency (N) (Black line I_d , red line I_r).



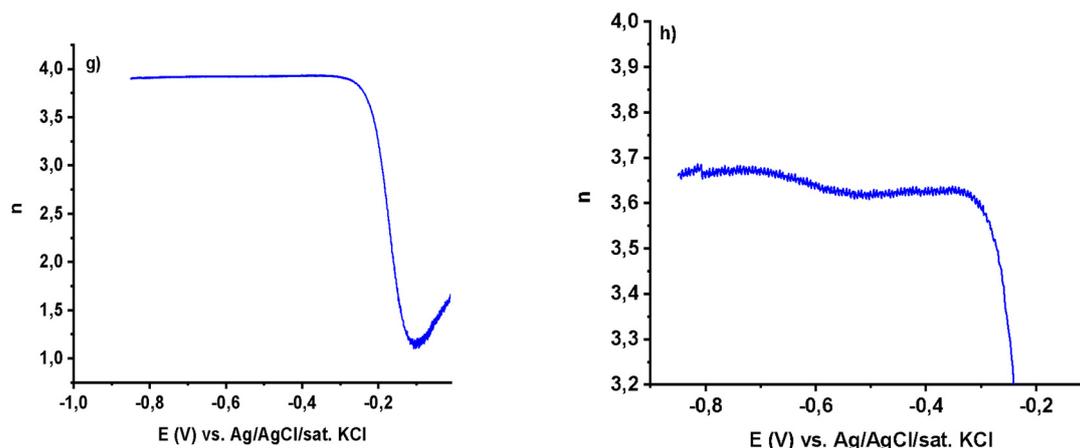


Figure S6. Number of exchanged electrons vs. potential values for different samples obtained by the two deposition techniques, sonochemical deposition: (a) Co; and (b) Cu. Electrochemical deposition of Co at different times: (c) 05 s; (d) 15 s; (e) 30 s; (f) 60 s; and (g) 90 s. (h) The last graph shows *CHraw* behaviour.

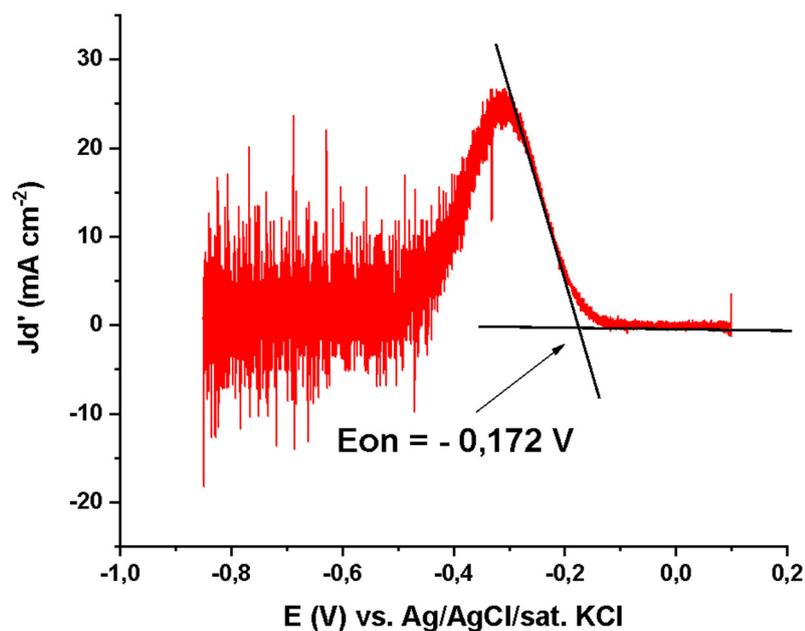


Figure S7. Example of first derivative curve of disk current to determine onset potential value on *CHec30s*.

EDX analyses of the raw char material confirmed nanoparticles are made of Zn and Sulphur. Quantification of Zn and S signal using Cliff–Lorimer method in thin film approximation show 1:1 ratios within the experimental error.

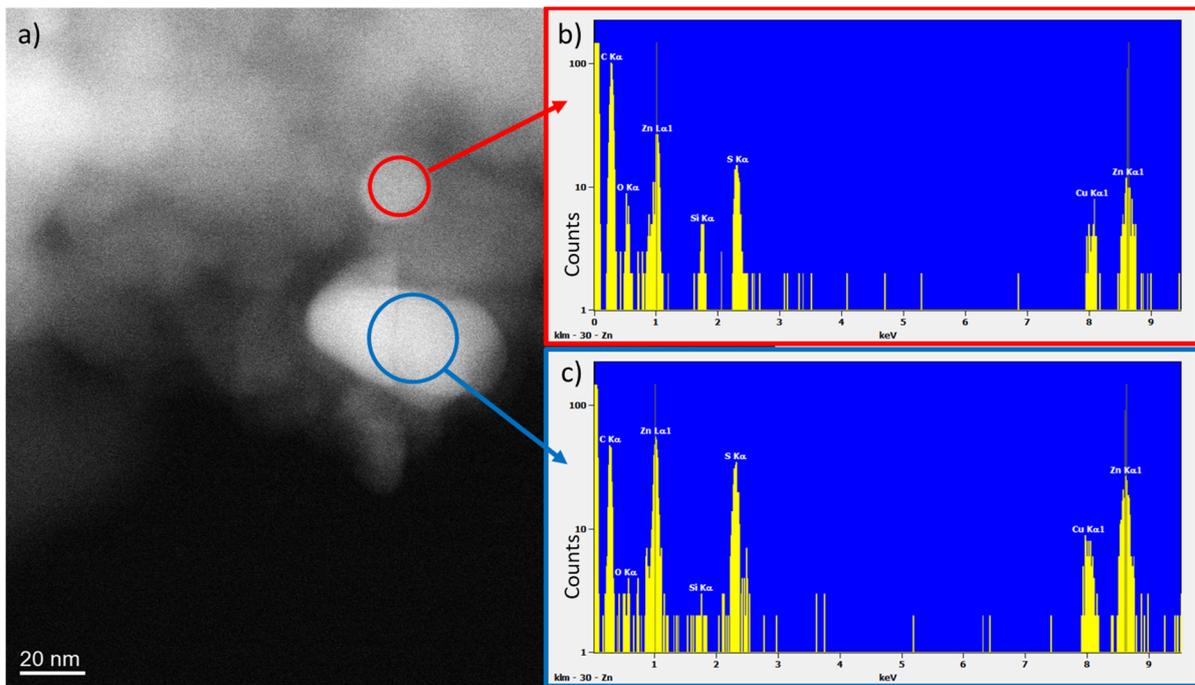


Figure S8. HAADF STEM image from *Chraw* sample (a) showing the two nanoparticles, and their relative EDX spectra from the area encompassed by the red circle (b) and the blue circle (c).

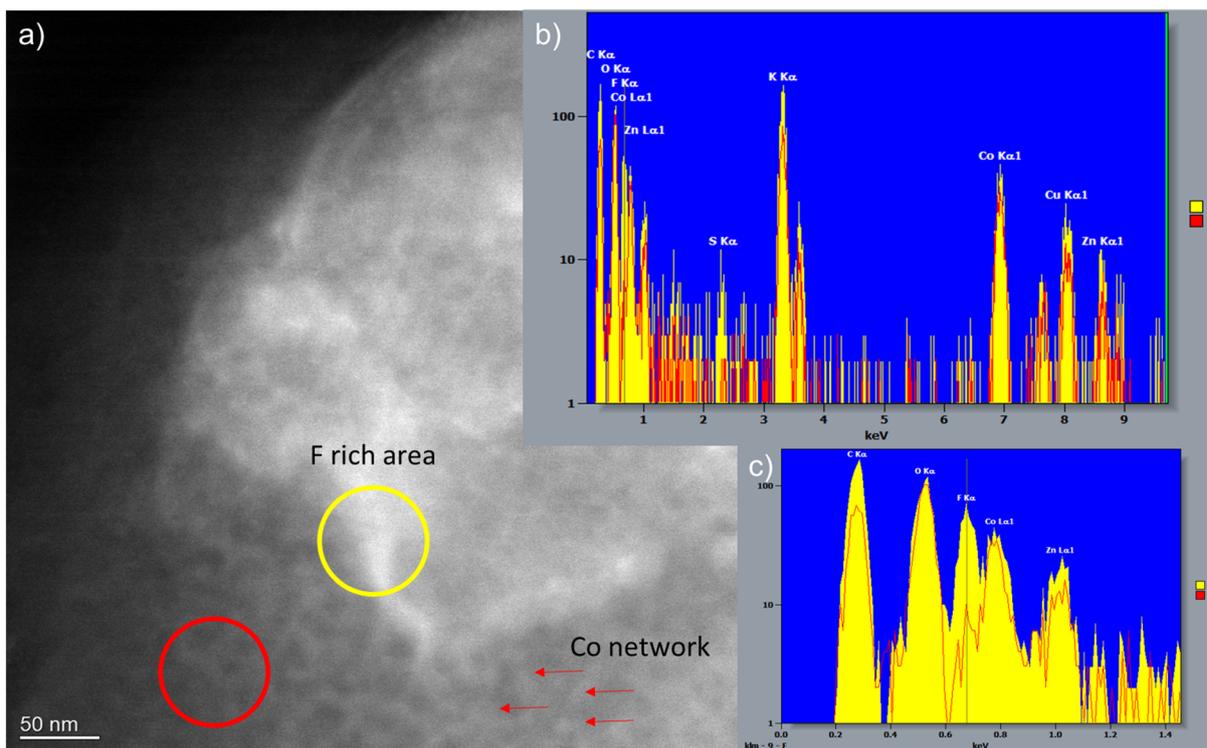


Figure S9. HAADF STEM image (a) showing a grain of material after Co electrodeposition. Yellow and red circles show the areas of EDX analysis relative to spectra in (b) (energy range 0–10 keV) and (c) (energy range 0–1.5 keV). Similar amounts of Co are found in both areas. The whiter regions in (a) contains higher concentration of Fluorine associated with Nafion ($C_7HF_{13}O_5S$, C_2F_4) used to treat the sample. The red arrows point to the uniform deposited Co network.

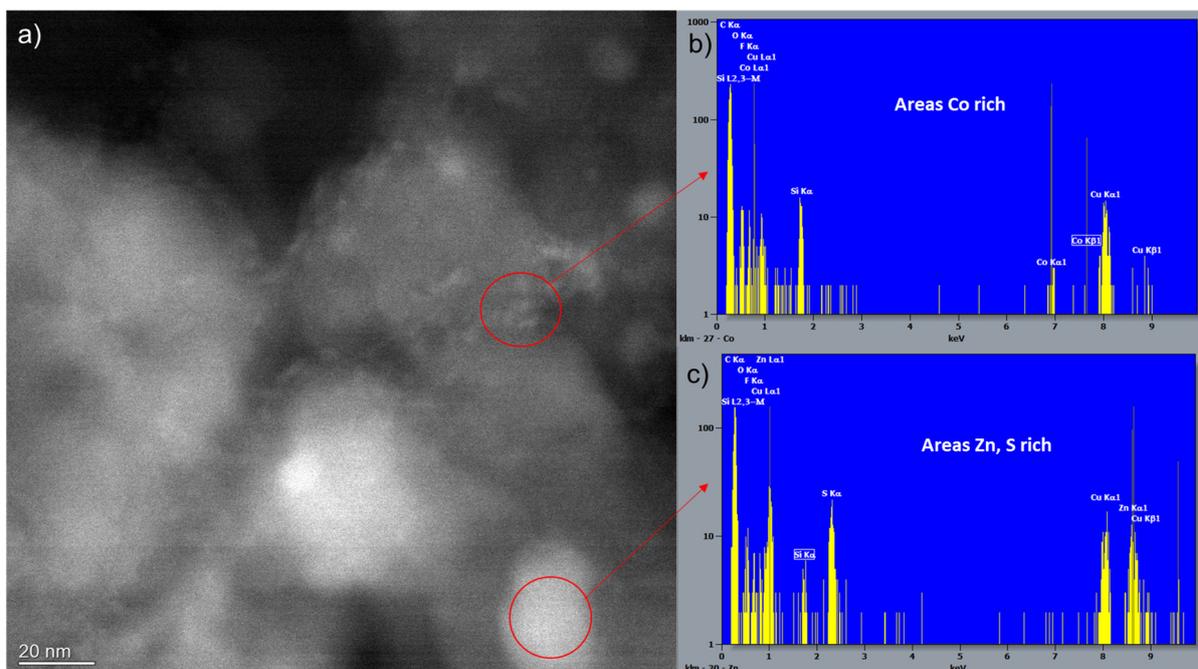


Figure S10. HAADF STEM image (a) showing details of a grain of material after Co electrodeposition and KOH treatment. (b) EDX spectrum confirming that brighter areas in the grain are due to the left cobalt on the surface. (c) EDX analysis of a nanoparticle confirming they are made of Zn and S.

References

1. Zafferoni, C. *et al.* Synergy of cobalt and silver microparticles electrodeposited on glassy carbon for the electrocatalysis of the oxygen reduction reaction: An electrochemical investigation. *Molecules* 2015, 20, 14386–14401 (2015).