Electronic Supplementary Material (ESI) for Energy & Environmental Science
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Supporting Information for:

Highly Active Nickel Oxide Water Oxidation Catalysts Deposited from Molecular Complexes

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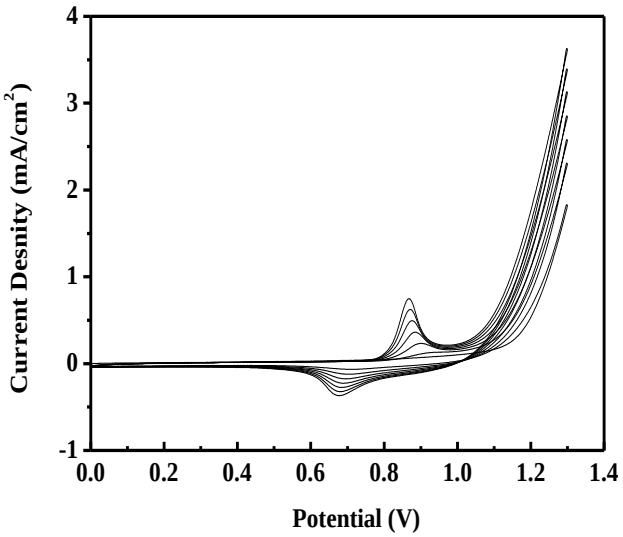

Fig. S1: Continuous CV scans of a 1.0 mM solution of $[Ni(OH_2)_6](NO_3)_2$ recorded using glassy carbon electrode at a scan rate of 100mV/s in 0.10 M NaBi buffer (pH=9.2).

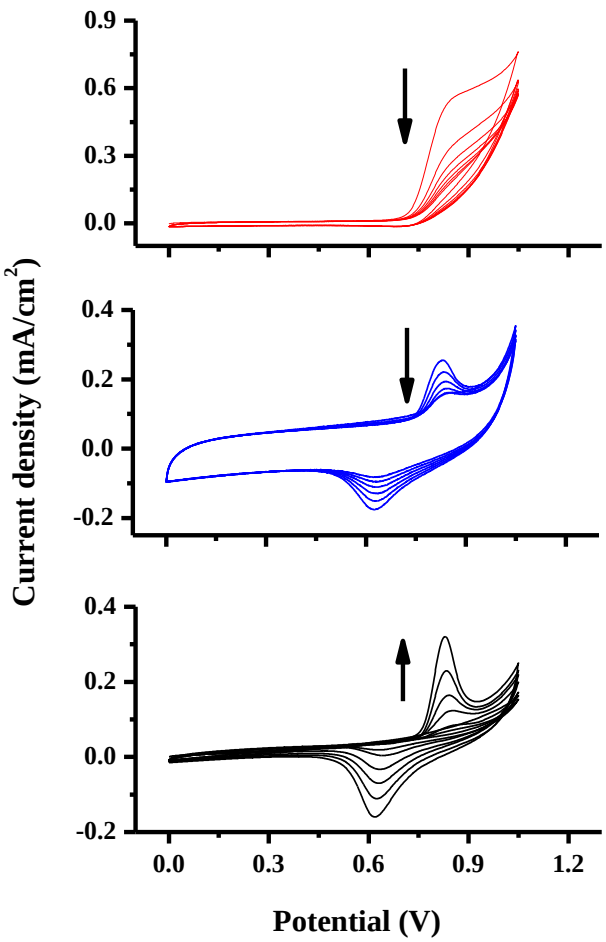

Fig. S2: Continuous CV scans recorded at 100mV/sec for 1.0 mM solutions of $[Ni(en)_3]^{2+}$ (red trace), $[Ni(NH_3)_6]^{2+}$ (blue trace) and $Ni[(OH_2)_6]^{2+}$ (black trace) recorded in 0.10 M NaBi buffer with the scans reversed before onset of water oxidation catalysis (arrow shows the change in anodic current density for different precursors on increasing number of consecutive scans).

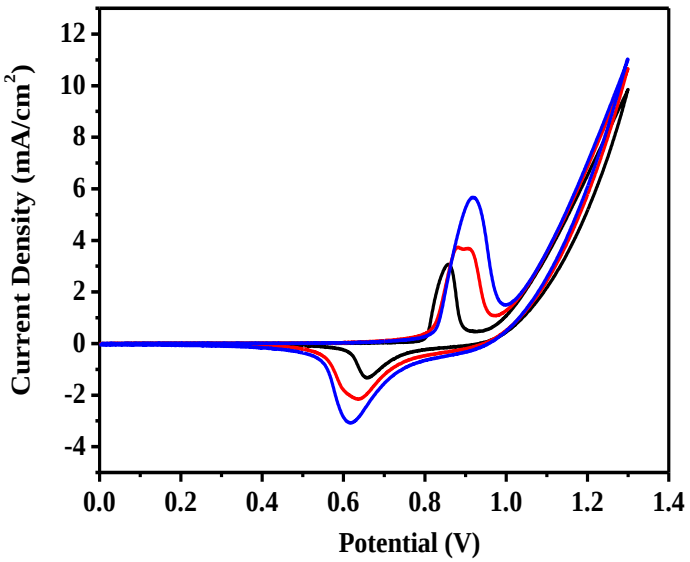

Fig. S3: CV scans of a NiO_x-en film in 0.60 M NaBi buffer in the absence of Ni(II) ion at 25mV/s (black), 50mV/s (red) and 75mV/s (blue) scan rate.

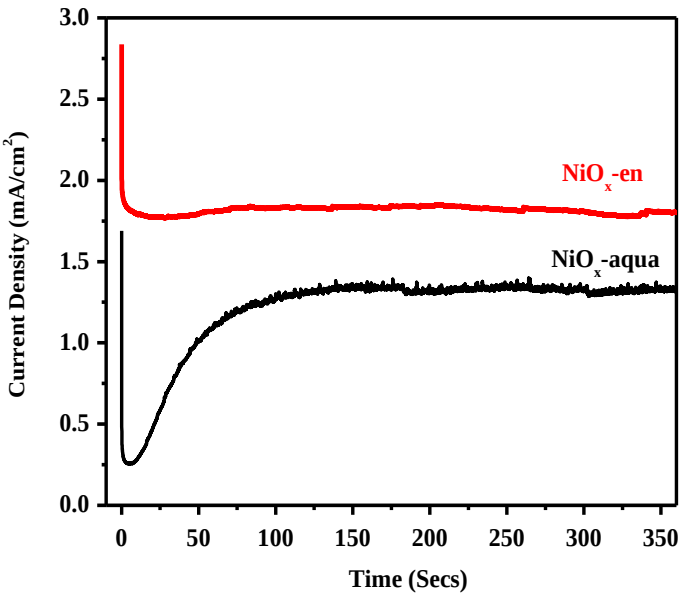

Fig. S4: Controlled potential electrolysis experiment on a NiO_x-en film (red trace) and NiO_x-aqua film (black trace), deposited on FTO, carried out over a period of six hours at 1.1V (vs Ag/AgCl) in 0.60 M NaBi buffer electrolyte in the absence of a Ni(II) source.

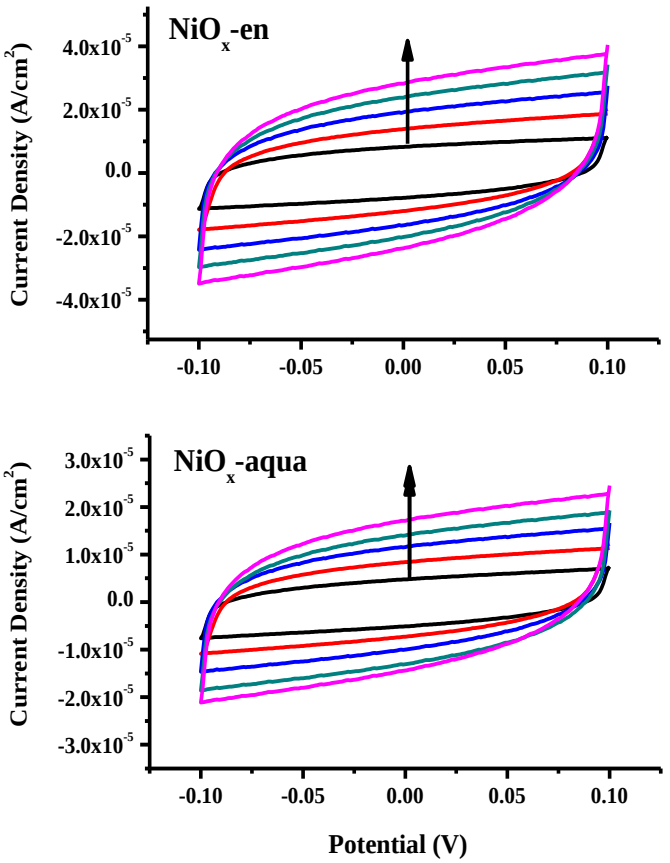

Fig. S5: CVs of NiO_x-en (top) and NiO_x-aqua (bottom) films deposited on glassy carbon electrodes recorded at scan rates ranging from 10mV/sec to 200 mV/sec in 0.60 M NaBi buffer (arrow shows the direction of increase in current on increasing scan rate).

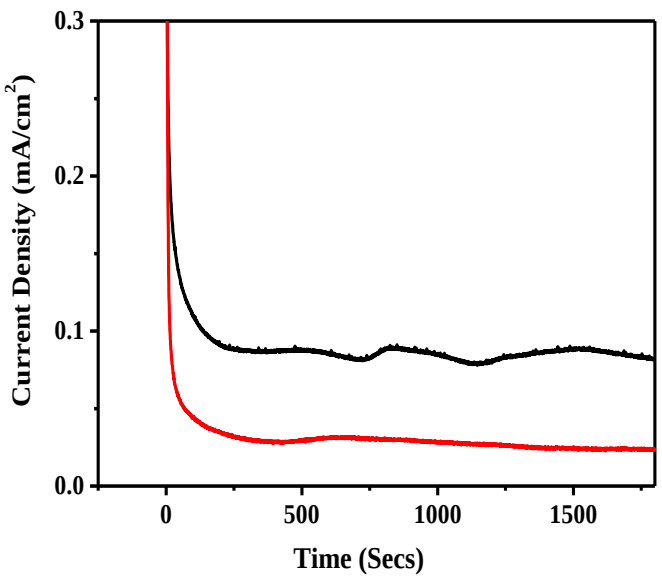

Fig. S6: CPE of the NiO_x-en film at 1.1V in Na_2SO_4 (pH 9.2) electrolyte solution with 1.0 mM $[Ni(en)_3]^{2+}$ (black trace) and without $[Ni(en)_3]^{2+}$ (red trace).

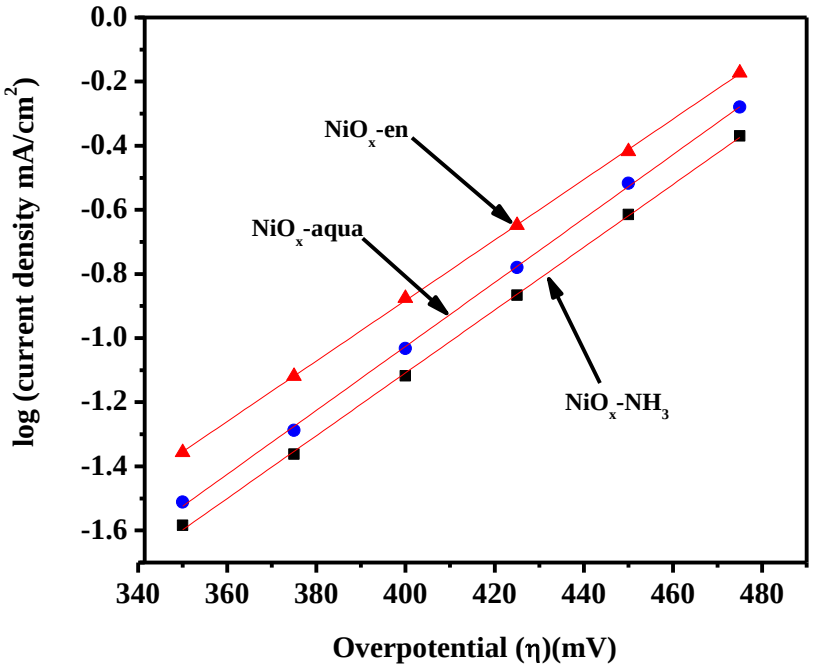

Fig. S7: Tafel plots for the NiO_x-en, NiO_x-NH_3 and NiO_x-aqua films calculated from data measured in 0.60 M NaBi buffer.

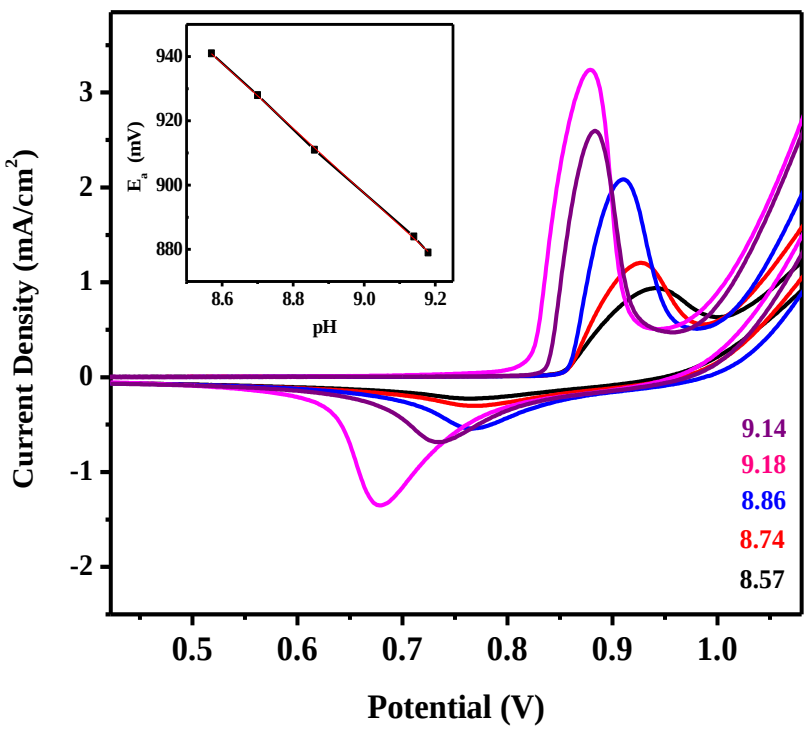

Fig. S8: CV scans of an NiO_x-en film recorded at scan rate of 25 mV/s, at different pH values in absence of Ni(II) ion in the electrolyte. Inset shows the linear plot obtained for pH *vs* anodic potential of the NiO_x-en films.

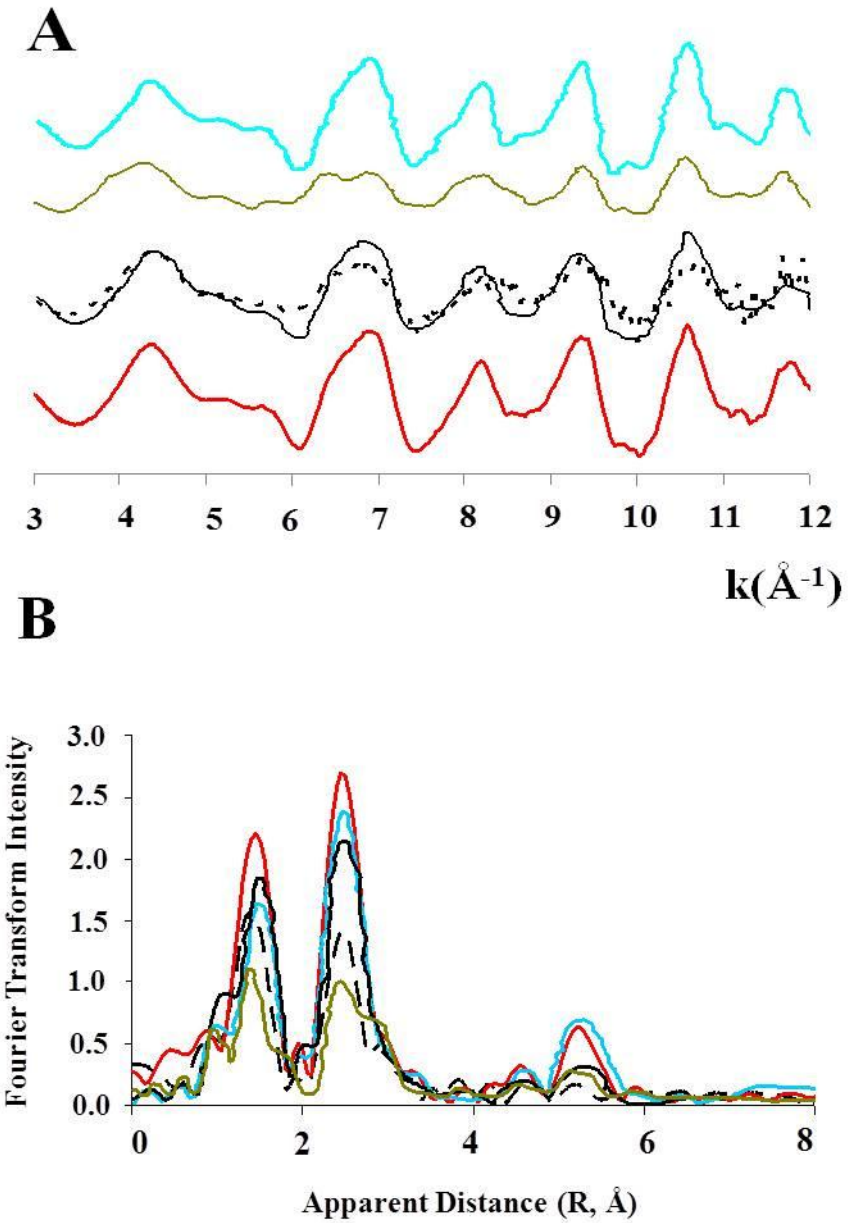

Figure S9. XAS data on films taken ex-situ compared to the data of Bediako (Reference 25). A. EXAFS spectra and B. Fourier Transform data of γ-NiOOH from reference 25 (blue trace), β-NiOOH (green trace), NiO_x-aqua; in situ from reference 25 (black trace in bold line), NiO_x-aqua; ex situ (black dashed line) and NiO_x-en; ex-situ (red trace). The results indicate the films to be predominated by the γ-NiOOH phase. Fourier Transforms are directly taken from Ref 25 without re-calculation (which was not possible as limited EXAFS range was shown) so care should be taken in comparisons as different algorithms may have been used.

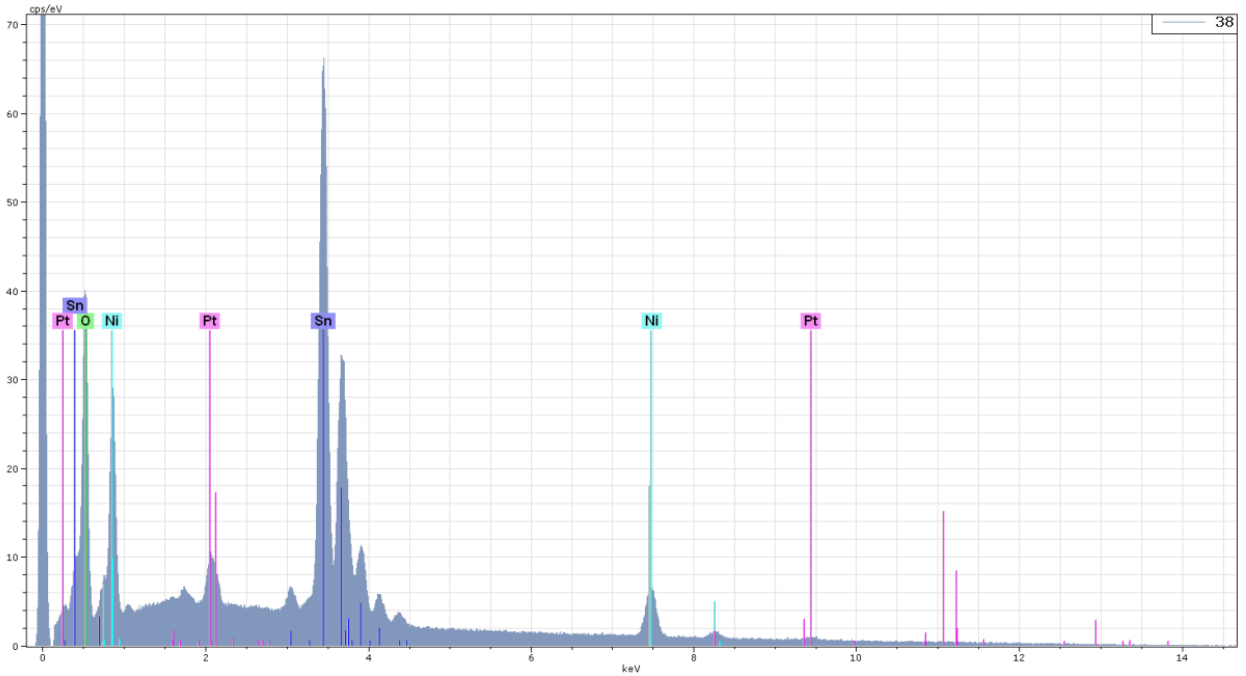

Fig. S10: EDX spectrum of NiO_x-en film deposited on FTO conducting glass. The samples were coated with platinum and the spectrum was acquired at 15kV (Pt is due to the coating of the substrate; Sn, F and O from the substrate).

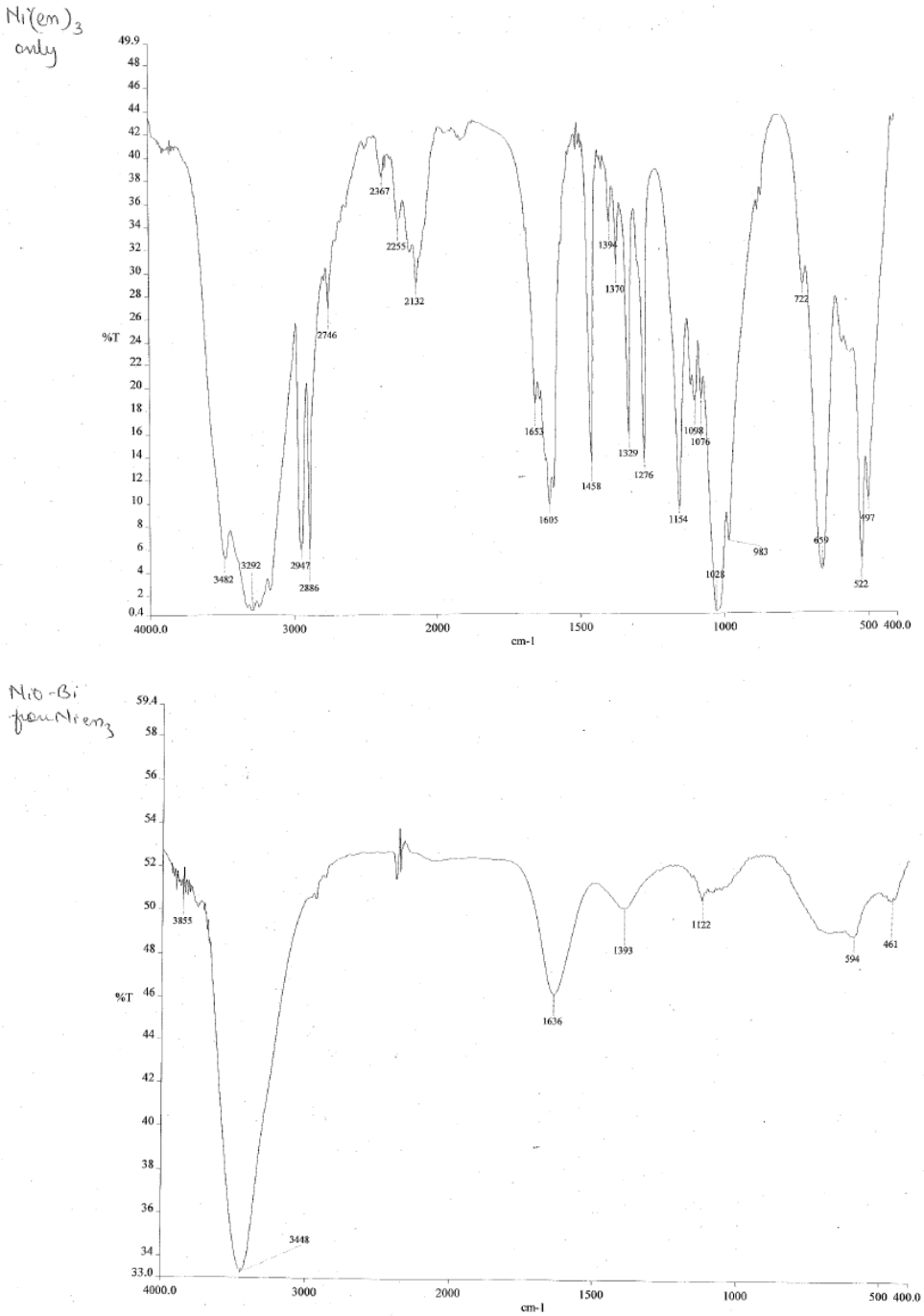

Fig. S11: IR spectrum of $[Ni(en)_3]Cl_2$ (top) and a NiO_x-en film (bottom) recorded using KBr discs.