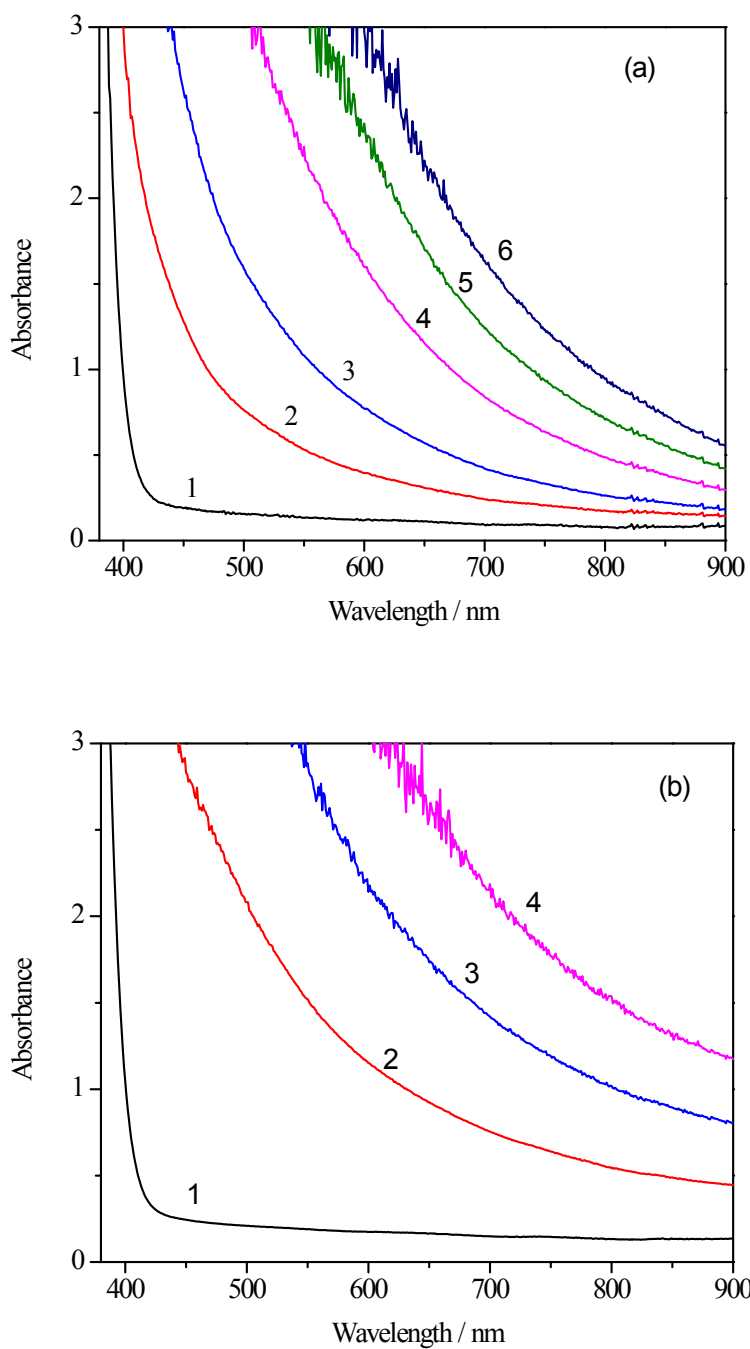
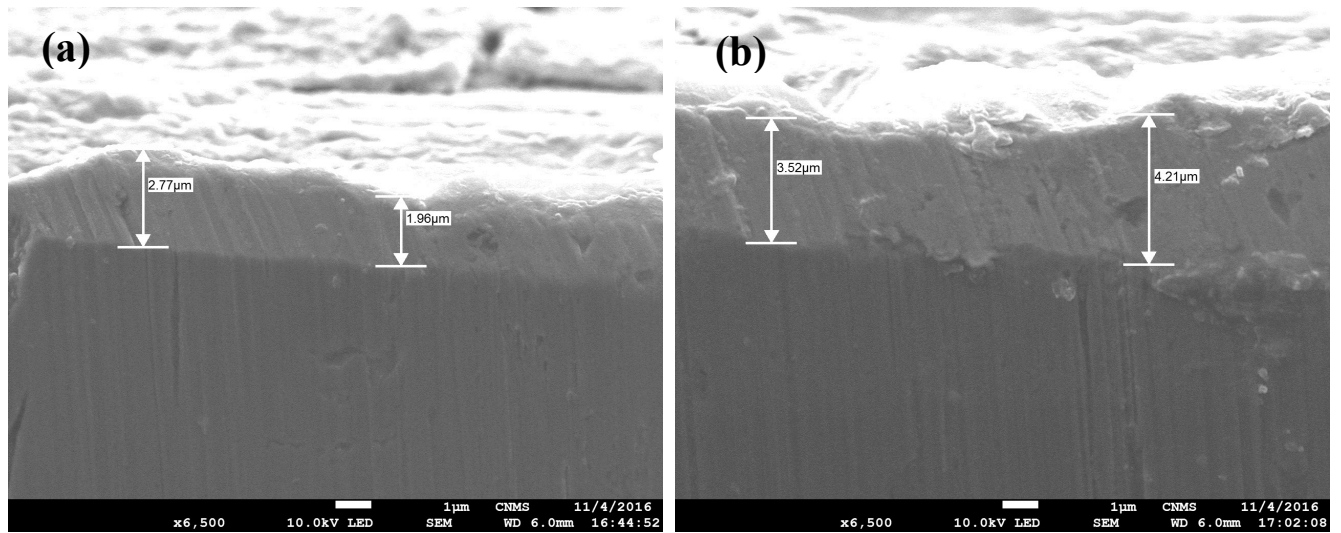


**Figure S1**



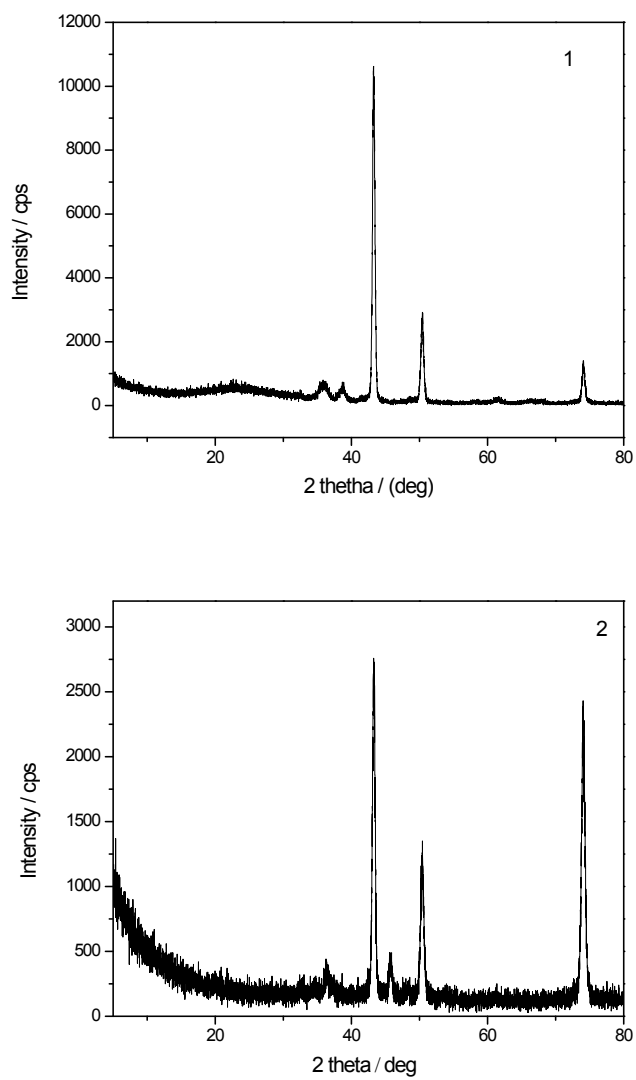
**Figure S1: (a)** UV-Visible spectra of (Cu-NiTCA) 7% TCA consists of 5 (1), 10(2), 15(3), 20(4) and 25  $\mu\text{M}$  NiSO<sub>4</sub> (5). **(b)** UV-Visible spectra of (Cu-CoTCA) 7% TCA consists of 5(1), 10 (2), 15(3), and 20  $\mu\text{M}$  CoSO<sub>4</sub>.

**Figure S2**



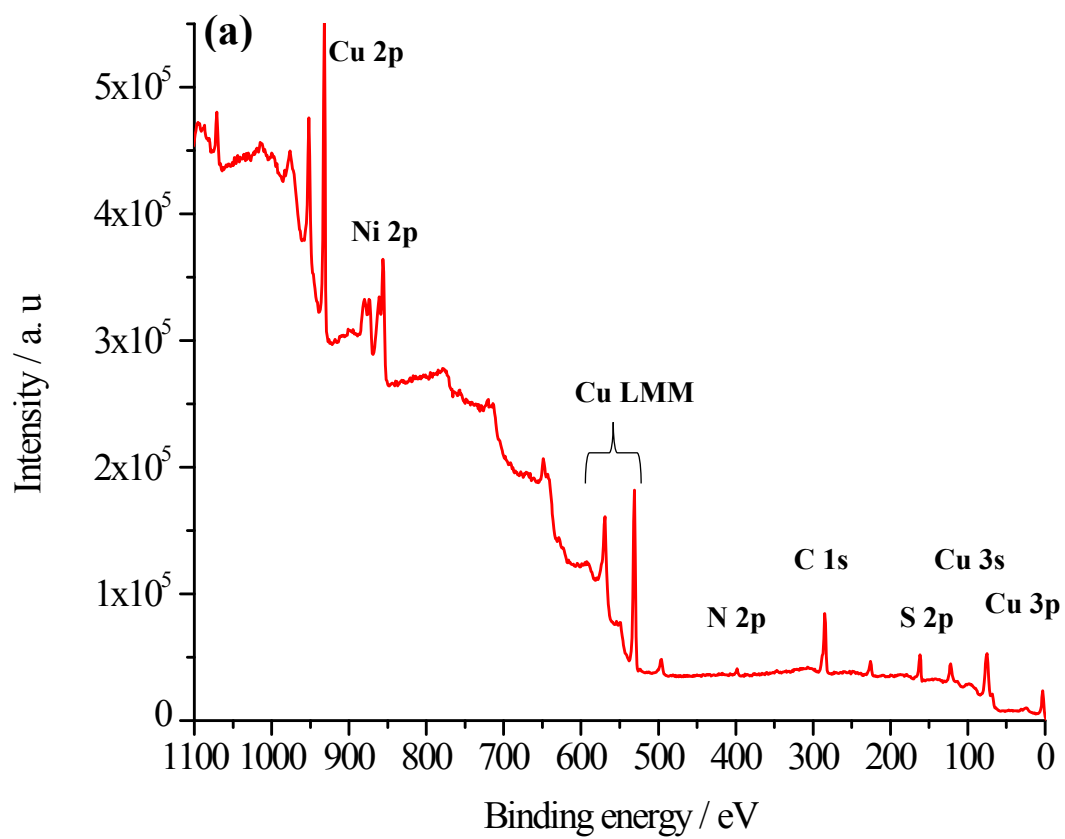
**FigureS2:** Cross section SEM ofCuNiTCA (a) and CuCoTCA (b).

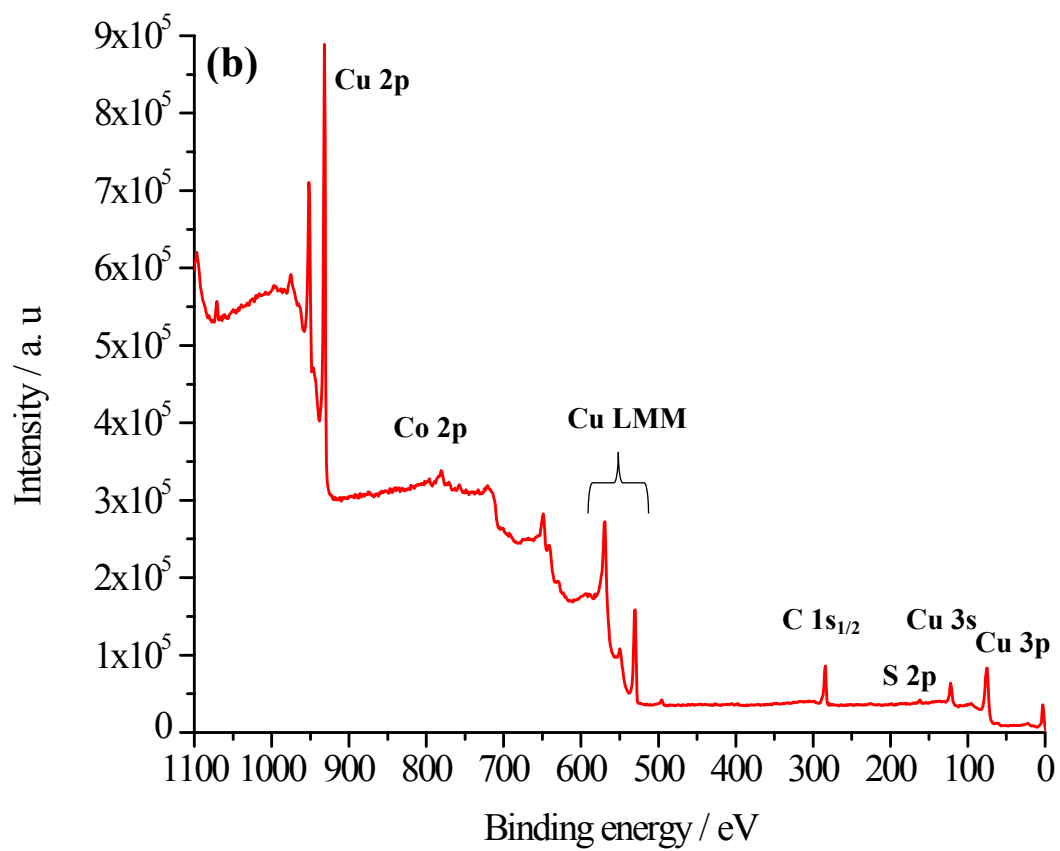
**Figure S3**

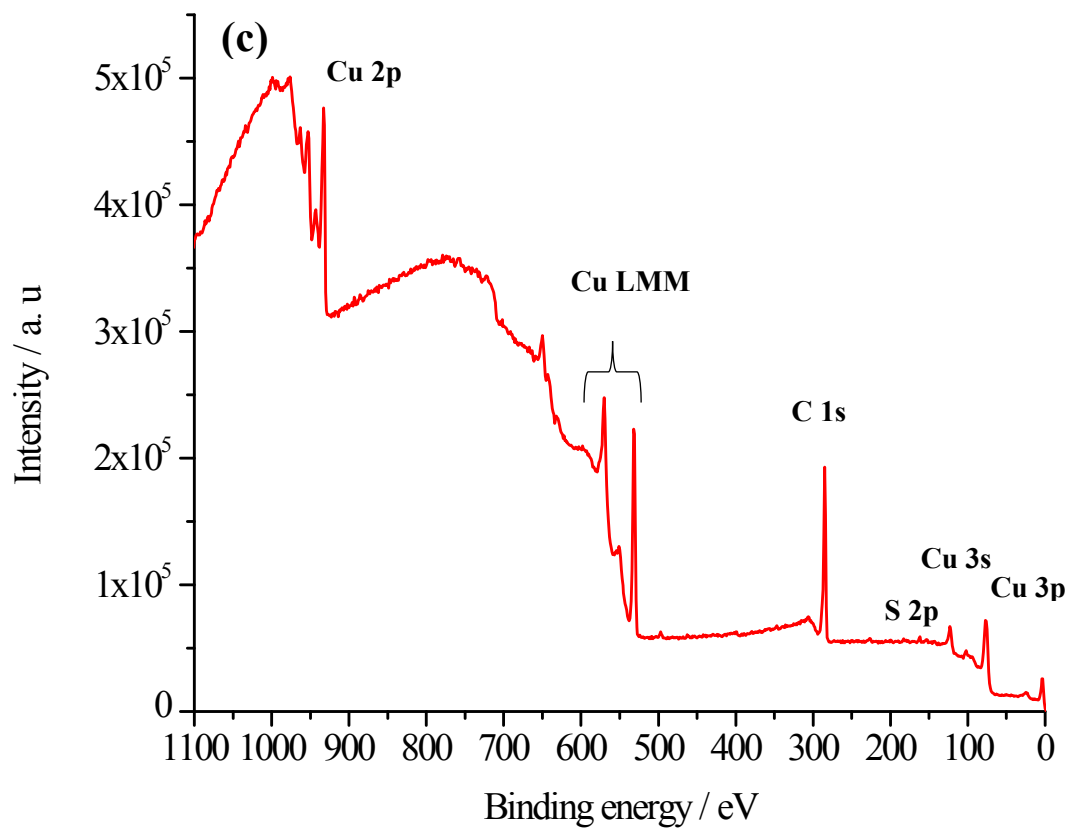


**Figure S3.** X-diffraction pattern of CuNiTCA (1) and CuCoTCA (2) thin films on Copper substrate.

Figure S4

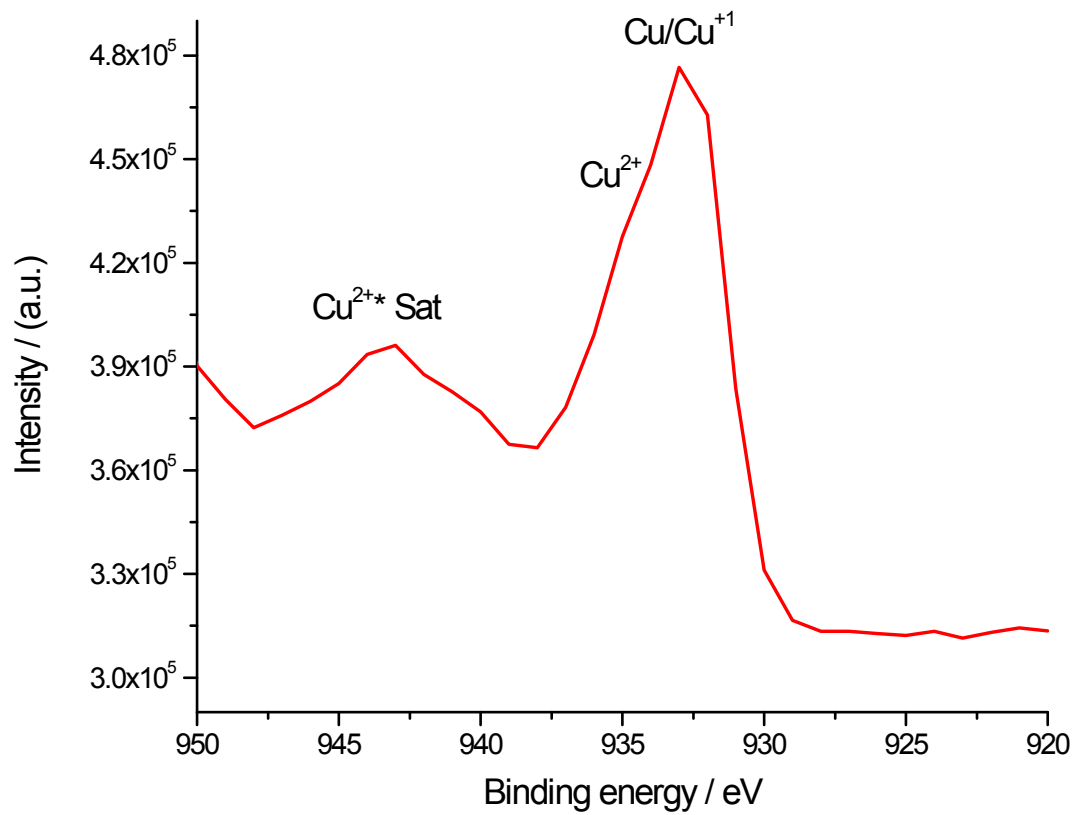






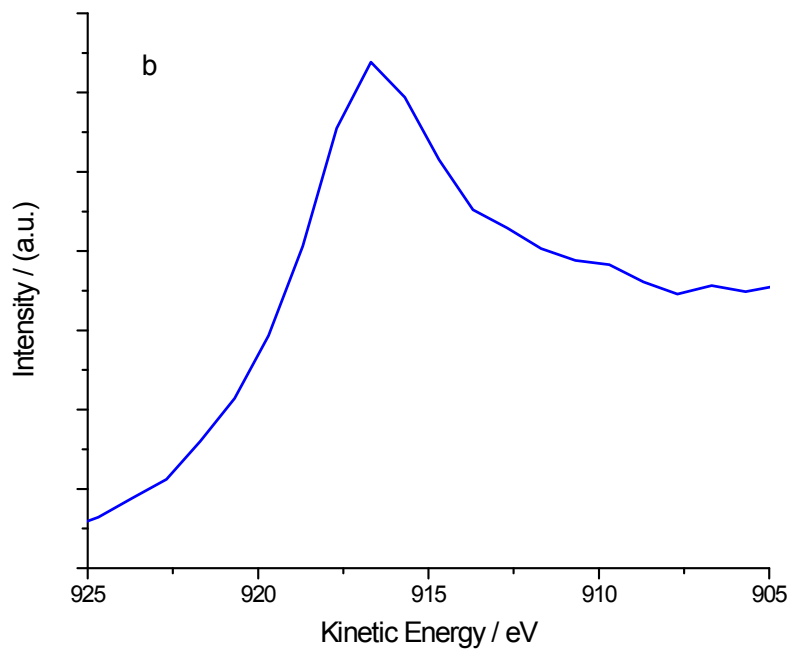
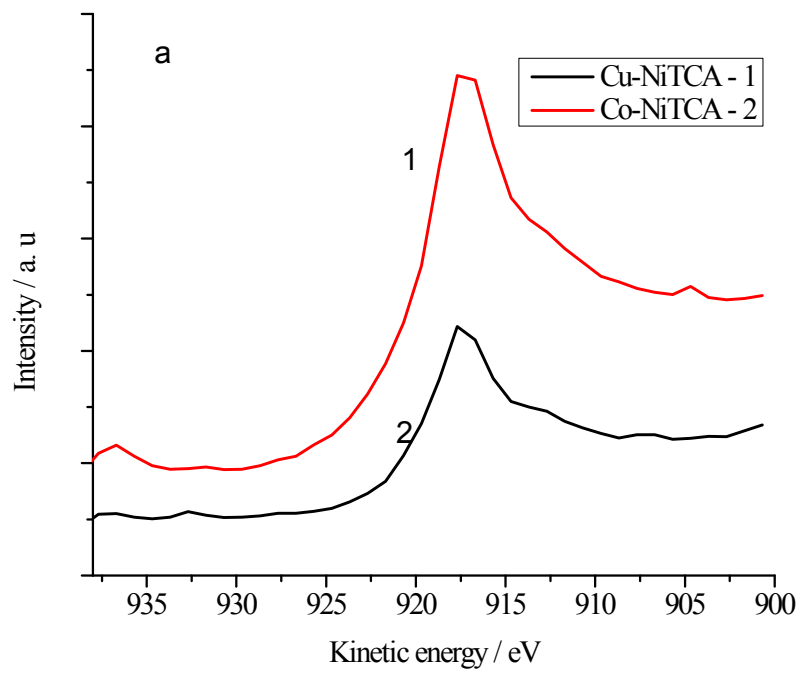
**Figure S4:** Wide scan XPS spectra's of Cu-NiTCA (a), Cu-CoTCA (b), and CuTCA (c).

**Figure S5**



**Figure S5.** XPS spectrum of Cu<sub>2</sub>p region of CuTCA electrode. The satellite peak of Cu<sup>2+</sup> shown along with Cu<sup>+</sup>.

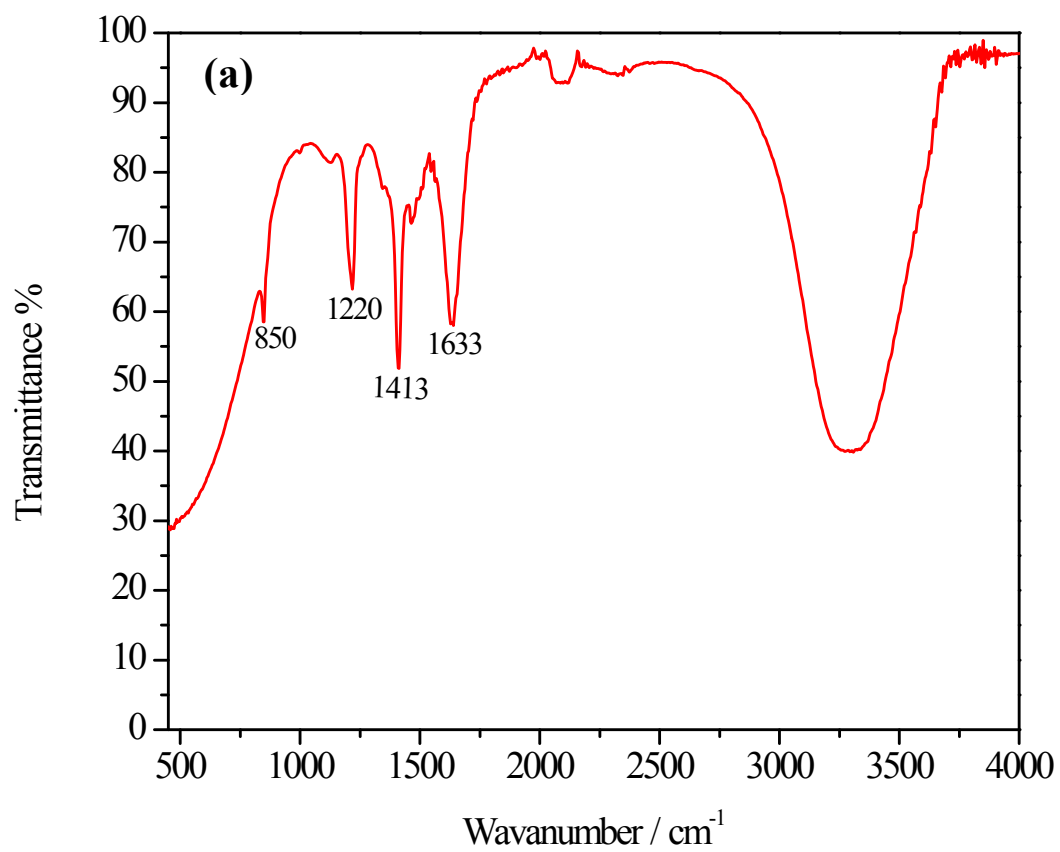
**Figure S6**

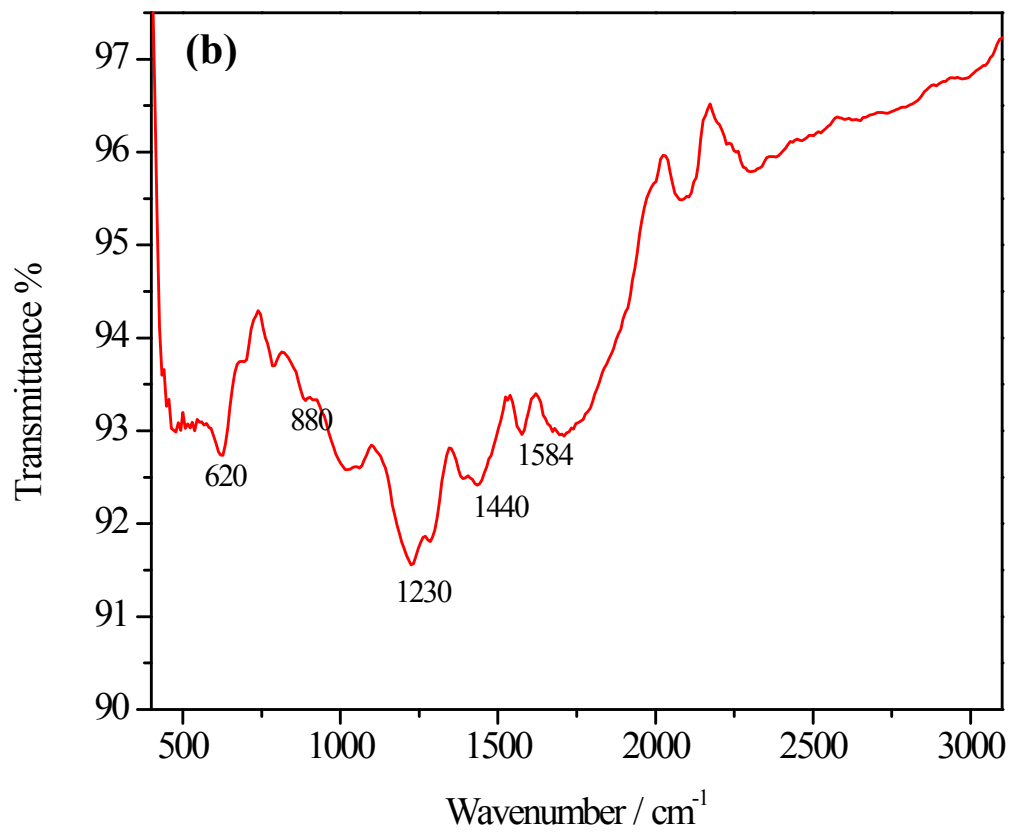


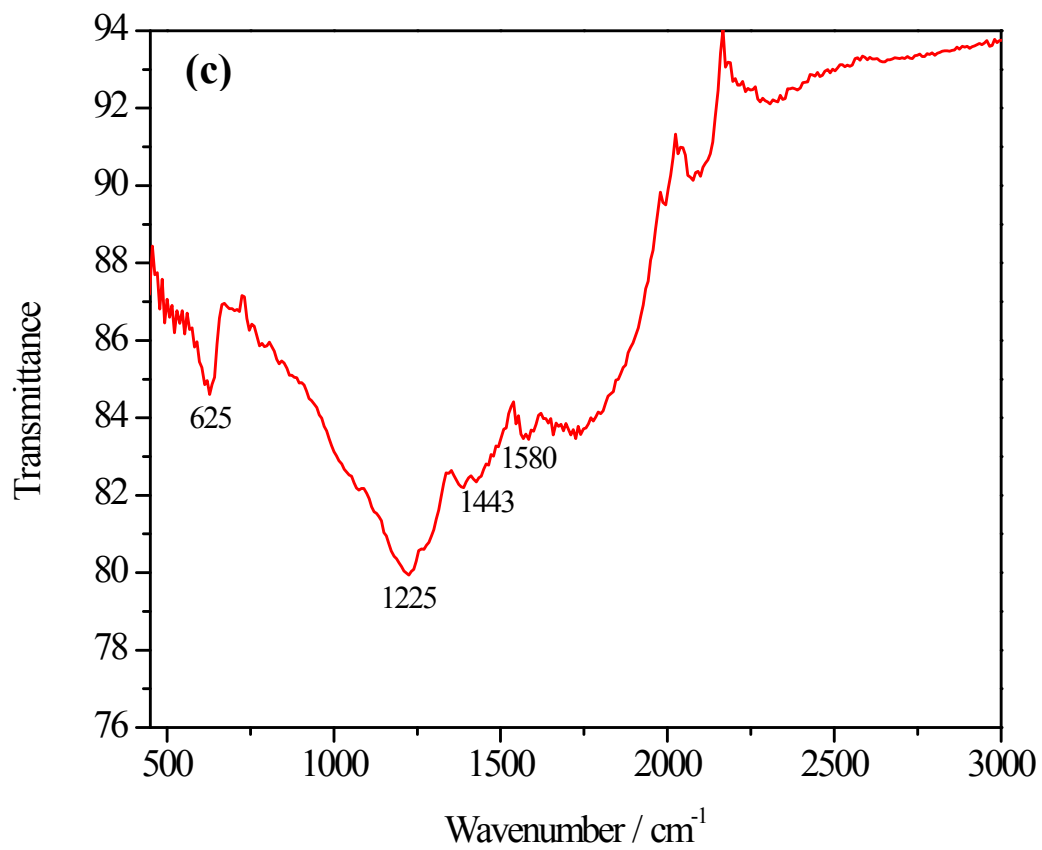
**Figure S6:** Auger Spectra of Ni TCA & CoTCA (a), CuTCA (b)



Figure S7

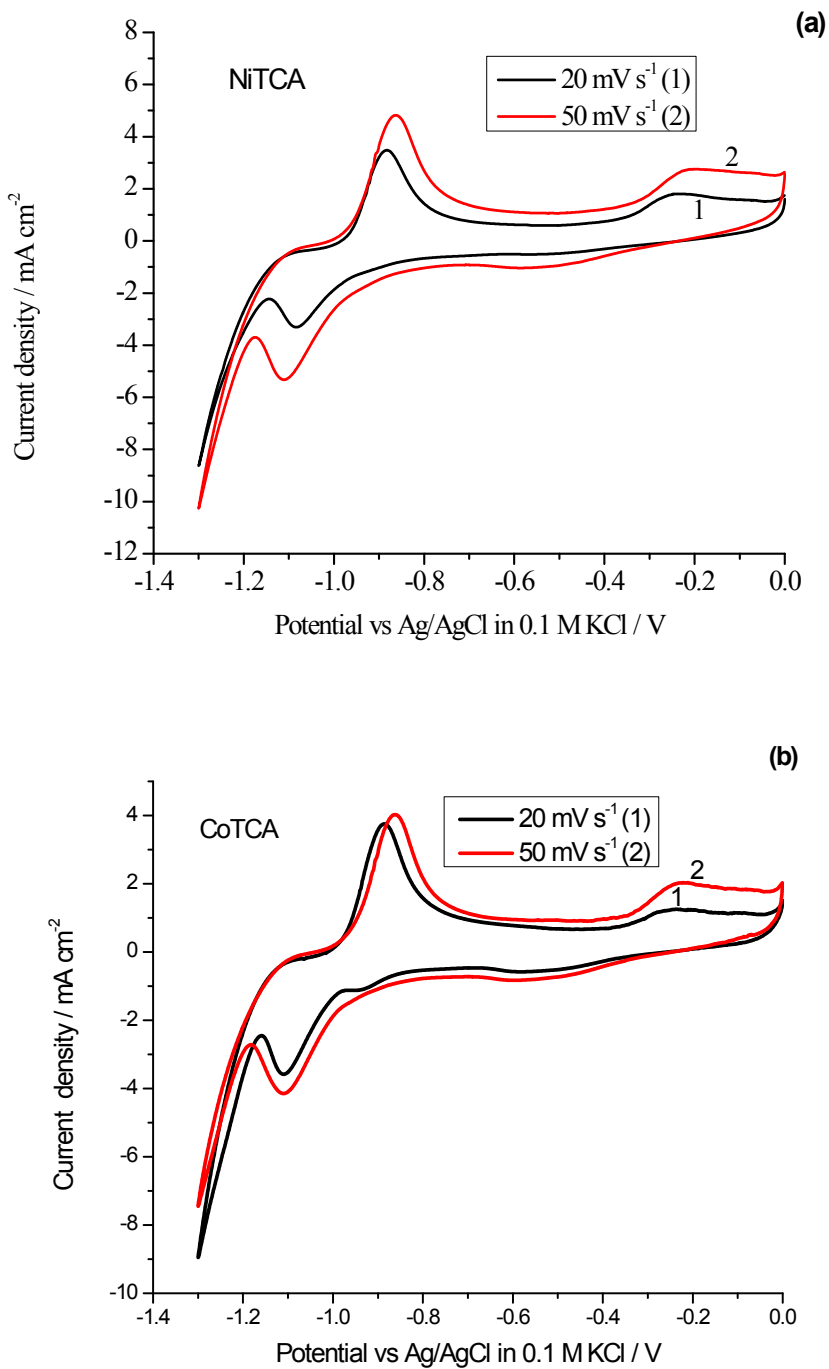




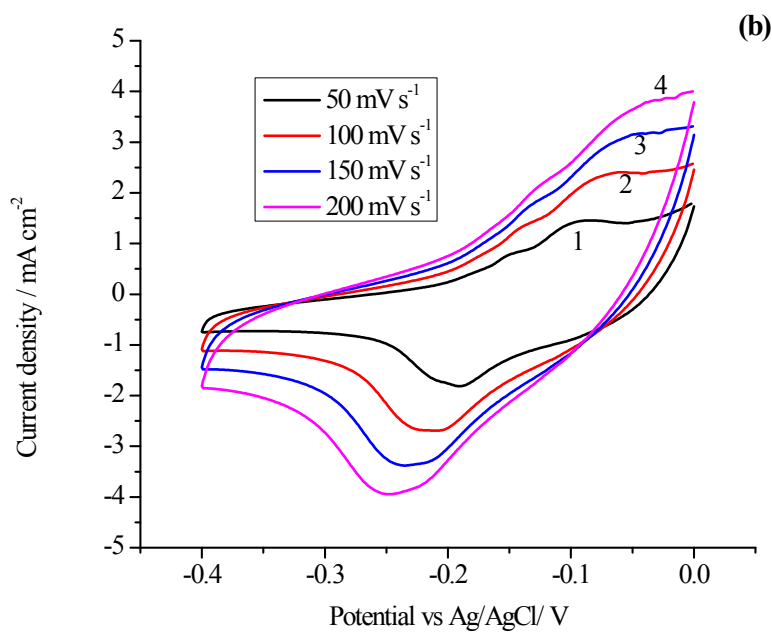
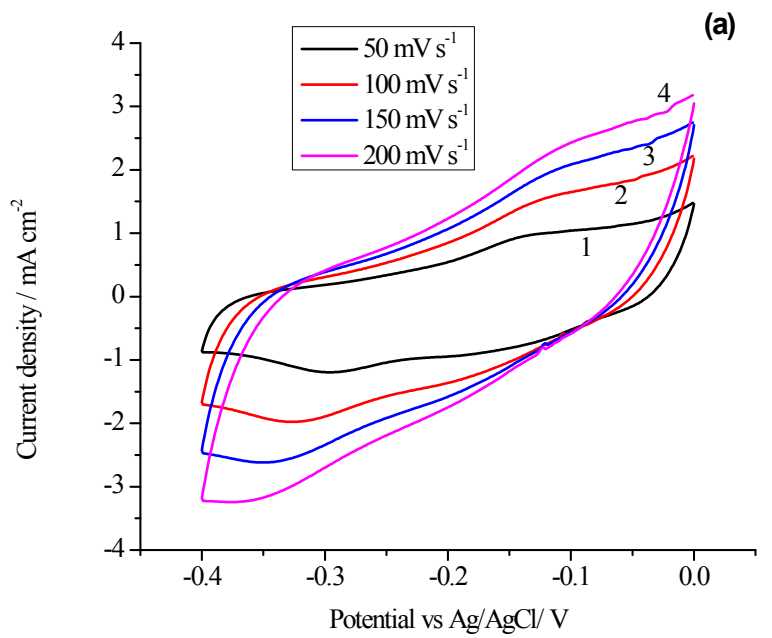


**Figure S7.**(a) FT-IR spectrum of TCA salt solution. (b) FT-IR spectrum of Cu-NiTCA thin film on Cu. (c) FT-IR spectrum of Cu-CoTCA thin film on Cu.

**Figure S8**

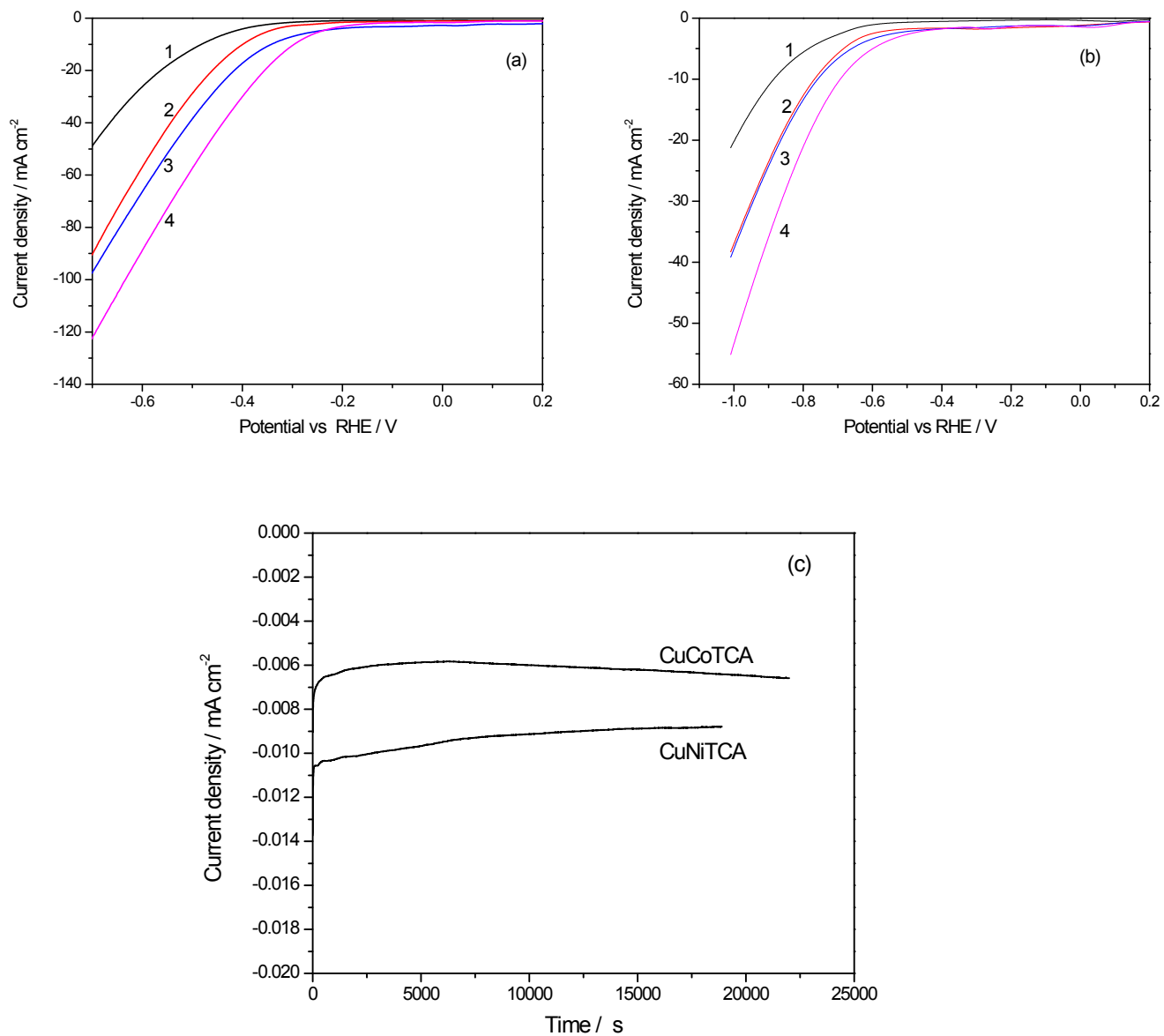


**Figure S8:** CVs of Cu-NiTCA(a), Cu-CoTCA(b) electrodes in 0.5 M Na<sub>2</sub>SO<sub>4</sub> at the sweep rates of 20 (1), 50(2).



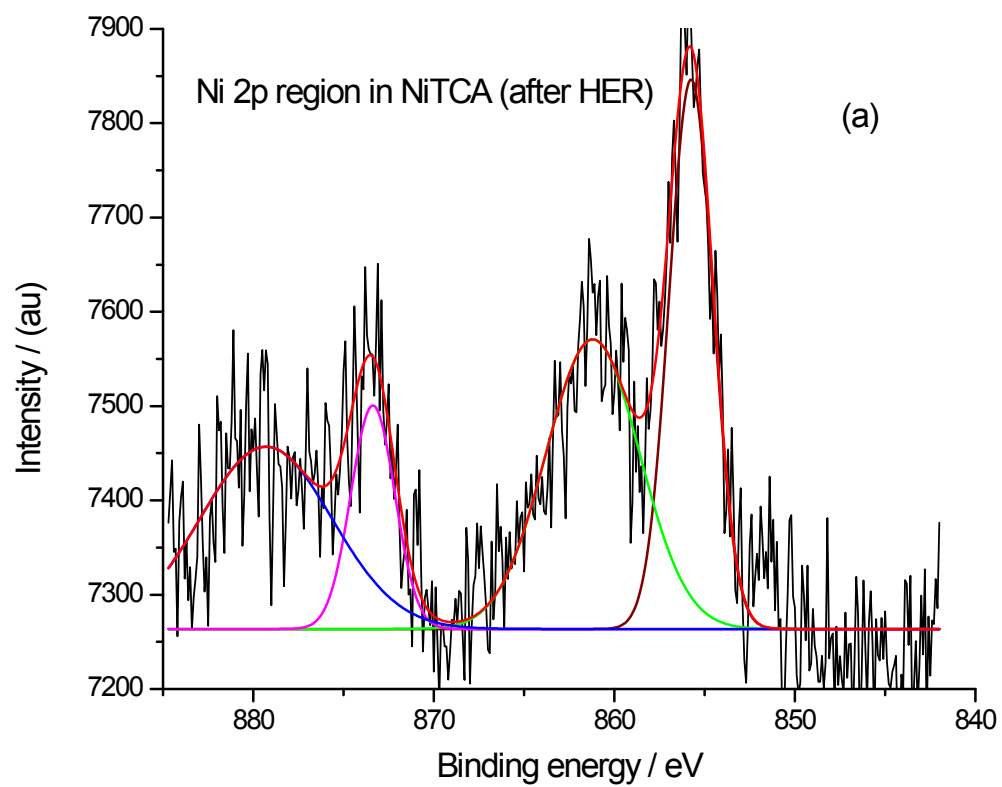
**Figure S9:** CVs of Cu-NiTCA (a), Cu-CoTCA(b) electrodes in 0.1 M HCl at the sweep rates of 50 (1), 100(2), 150 (3) and 200 mV s<sup>-1</sup> (4).

**Figure S10**

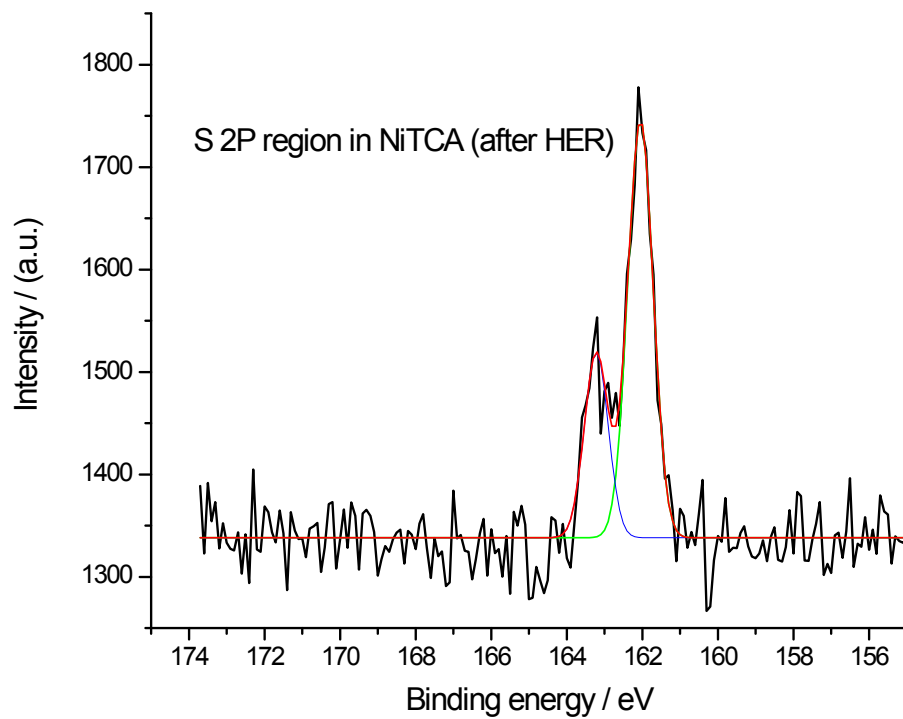


**Figure S10.** **(a)** LSVs of Cu (1), Cu-TCA (2), Cu-CoTCA (3) and Cu-NiTCA (4) in 0.5 M NaOH solution a sweep rate of 10 mV S<sup>-1</sup>. **(b)** LSVs of Cu (1), Cu-TCA (2), Cu-CoTCA (3) and Cu-NiTCA (4) in 0.5 M Na<sub>2</sub>SO<sub>4</sub> solution a sweep rate of 10 mV S<sup>-1</sup>. **(c)** Chronoamperometric curves of Cu-NiTCA and Cu-CoTCA at -0.35 V vs RHE in 0.5 M NaOH.

**Figure S11**

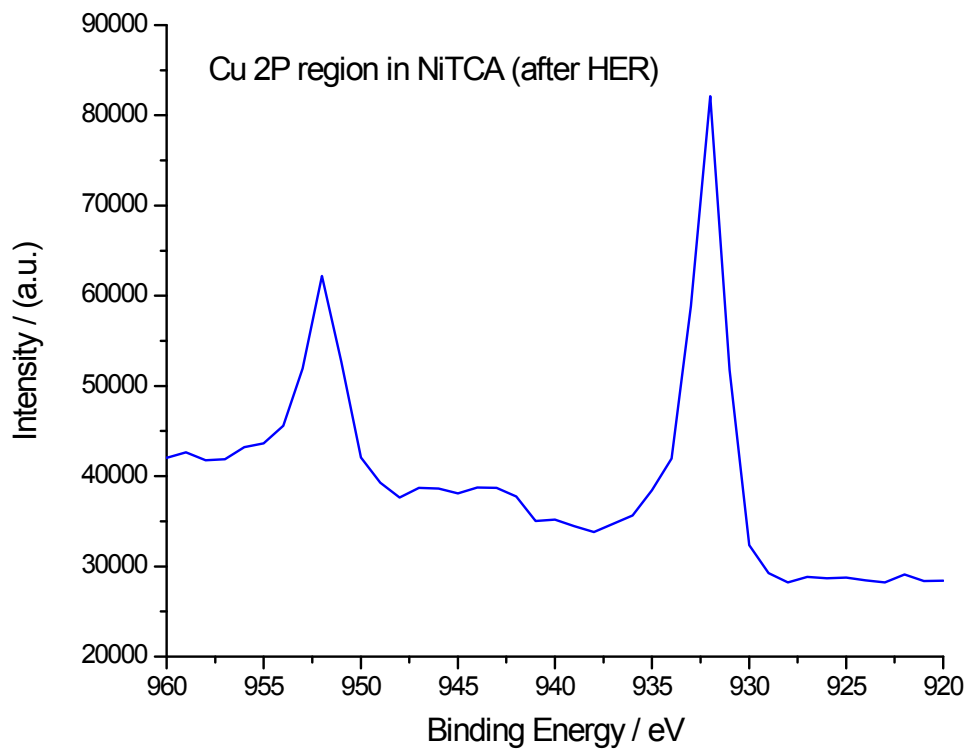


**Figure S11a.** Post HER XPS spectrum of Ni2p region of Cu-NiTCA electrode.



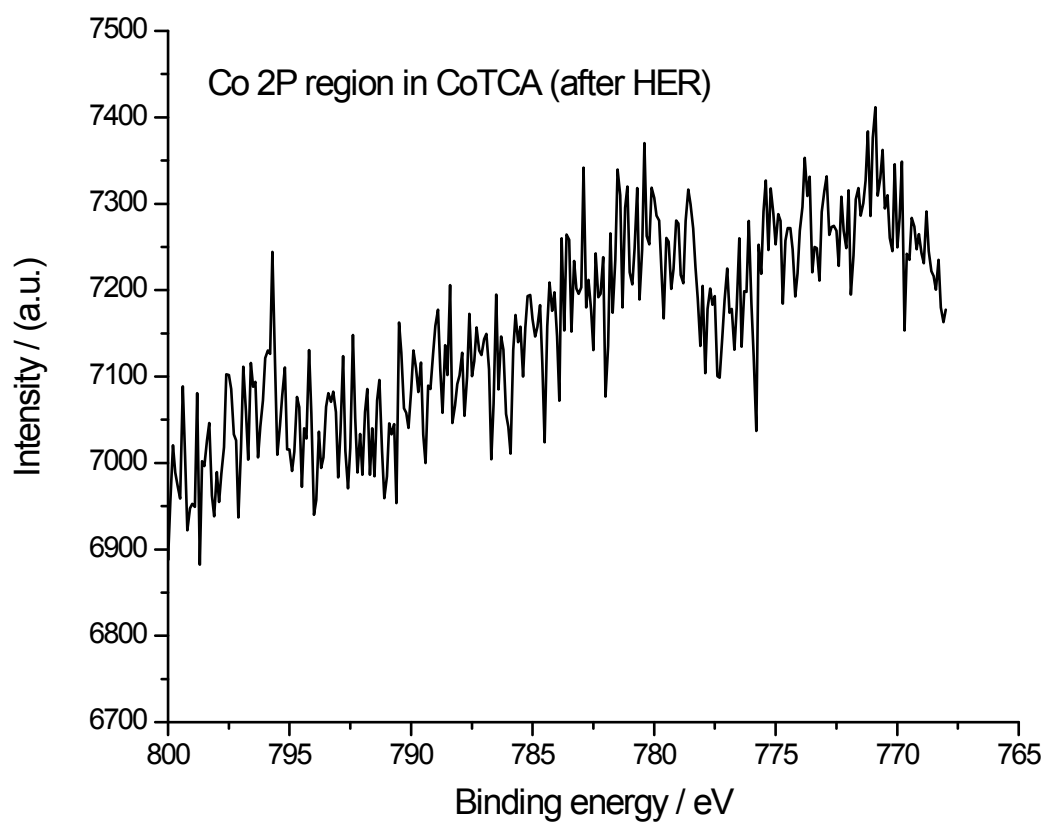
**Figure S11b.** Post HER XPS spectrum of S2p region of Cu-NiTCA electrode.



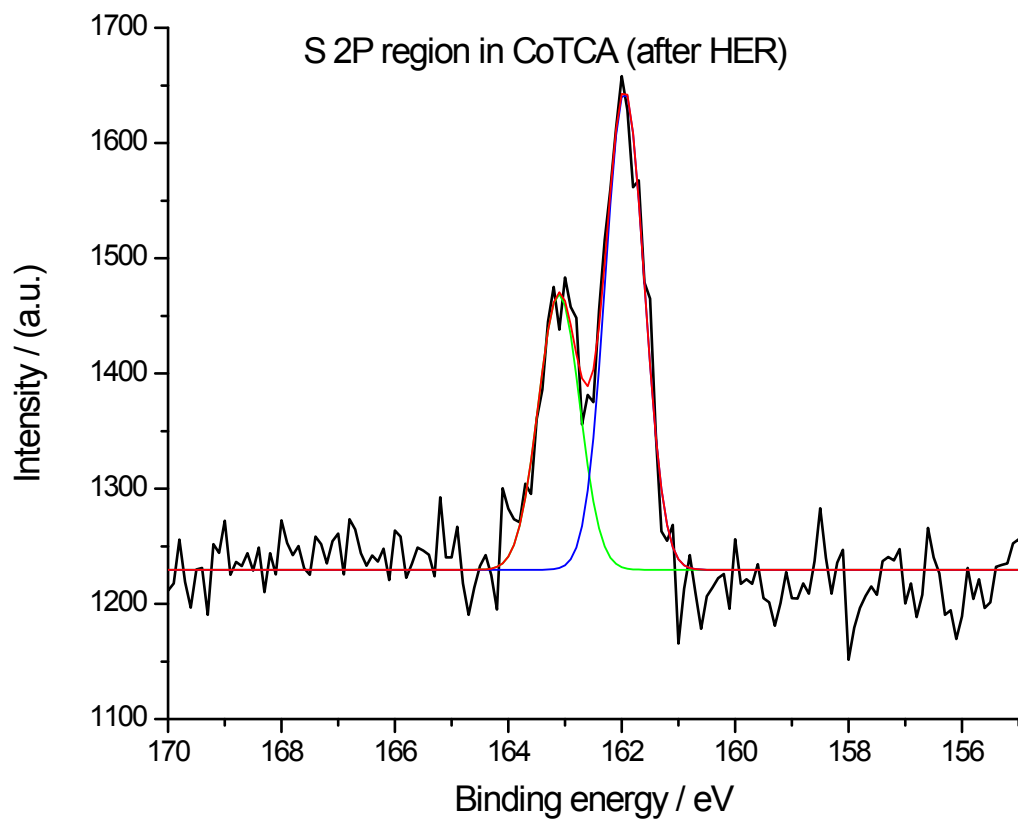


**Figure S11c.** Post HER XPS spectrum of Cu<sub>2p</sub> region of Cu-NiTCA electrode.

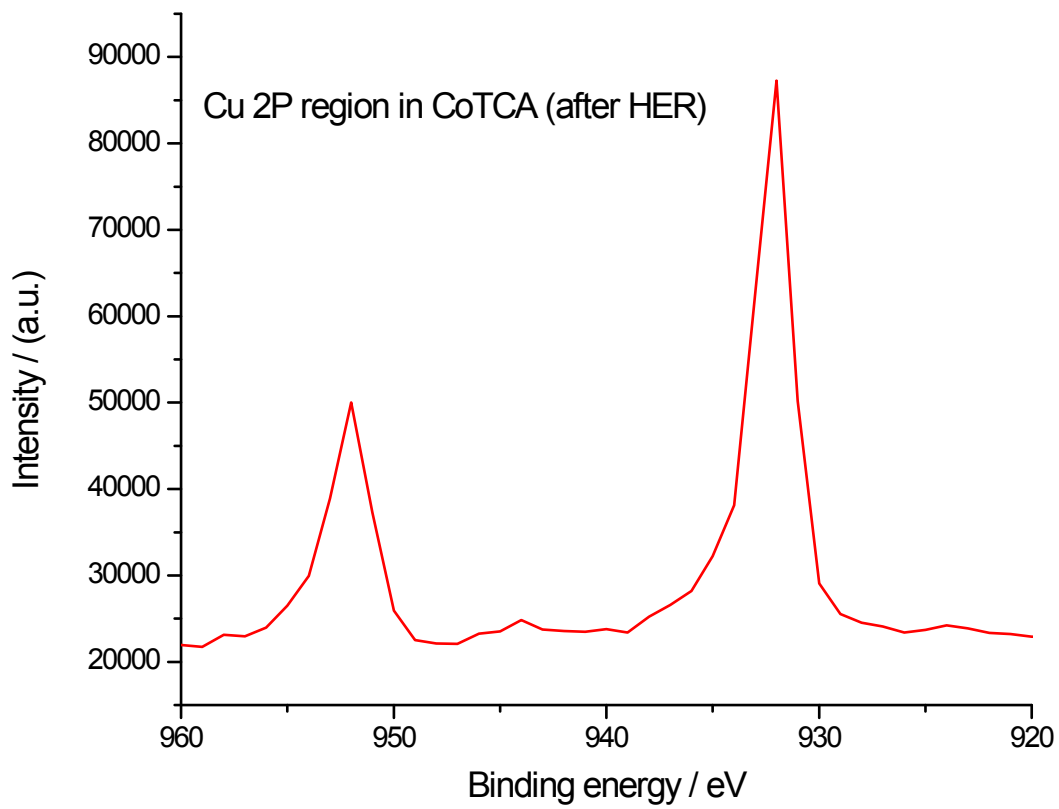
**Figure S12**



**Figure S12a.** Post HER XPS spectrum of Co2p region of Cu-CoTCA electrode.

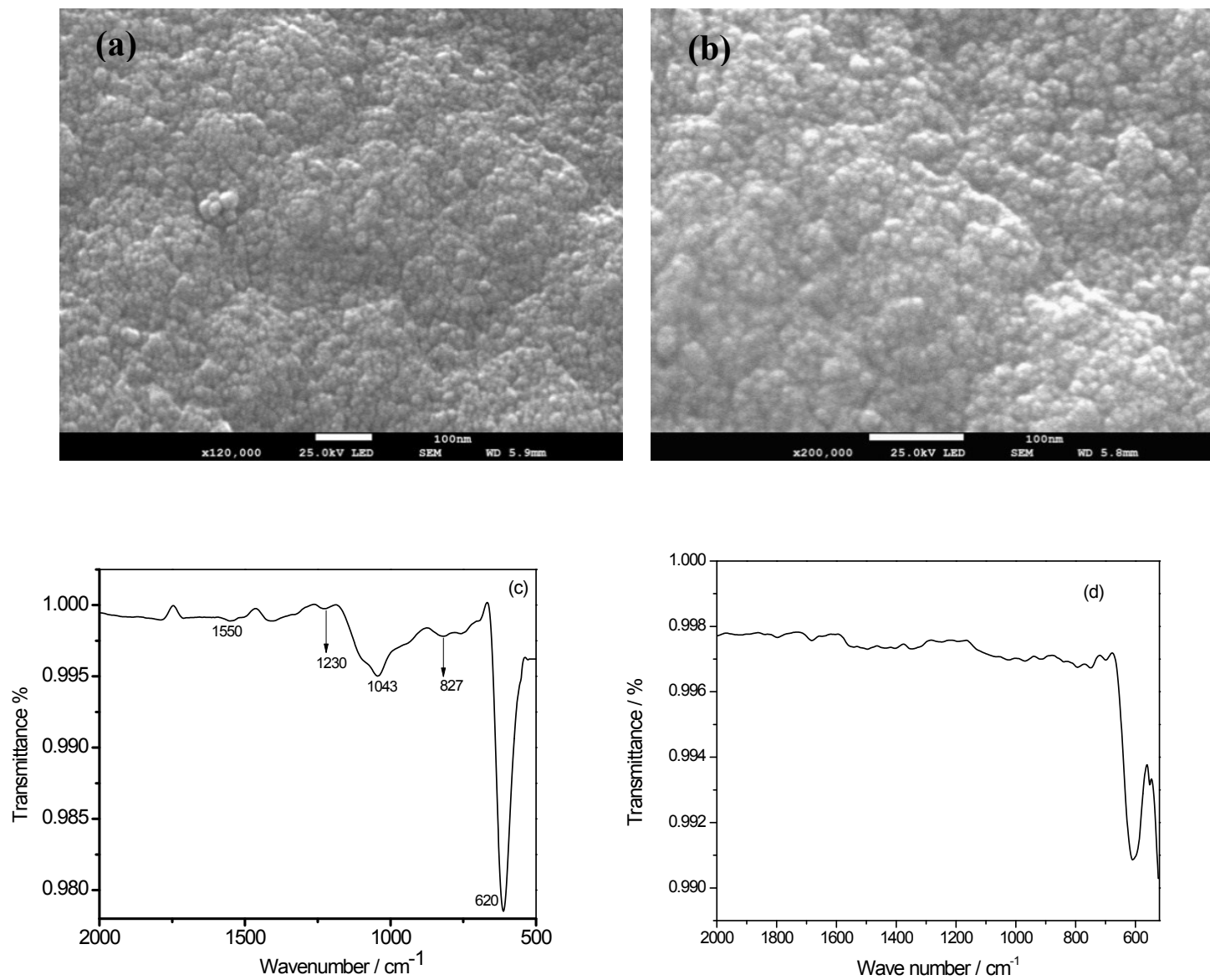


**Figure S12b.** Post HER XPS spectrum of S2p region of Cu-CoTCA electrode.



**Figure S12c.** Post HER XPS spectrum of Co2p region of Cu-CoTCA electrode.

**Figure S13**



**Figure S13.** Post – HER Scanning electron micrograph of CuNiTCA (a) and CuCoTCA(b), Post IR Spectra of CuNiTCA(c) and CuCoTCA(d).