

## Supporting Information

### **Development of Self-Supported 3D Micro-Porous Solder Alloy Electrodes for Scalable CO<sub>2</sub> Electroreduction to Formate**

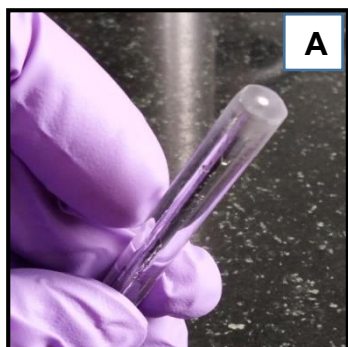
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Department of Chemistry, Savitribai Phule Pune University Ganeshkhind Pune 411007,  
India.

New Journal of Chemistry (RSC)

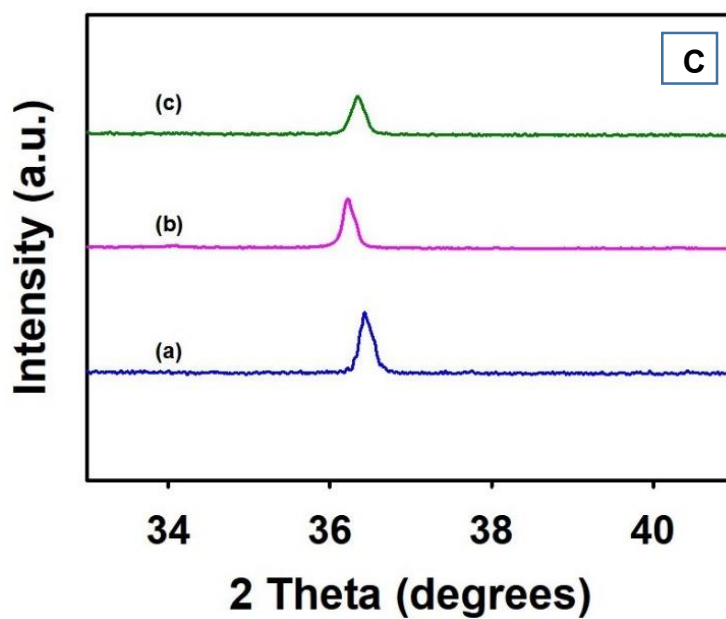
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<sup>\*</sup>Corresponding Author E-mail: [haram@chem.unipune.ac.in](mailto:haram@chem.unipune.ac.in)

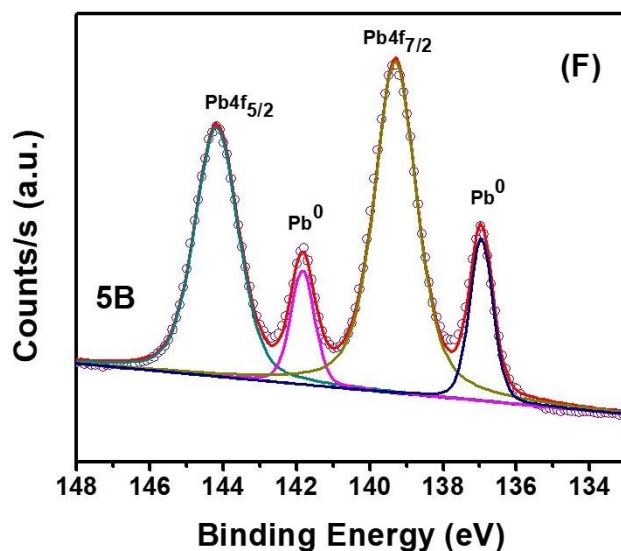
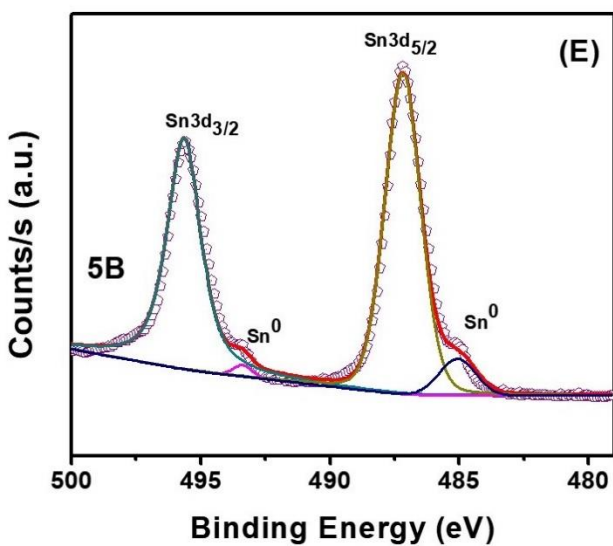
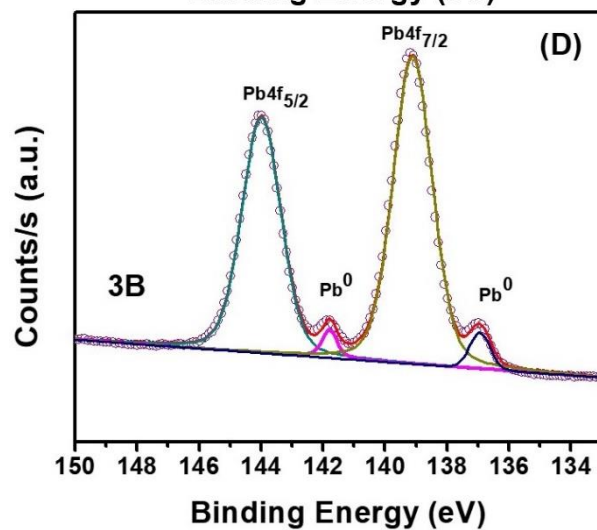
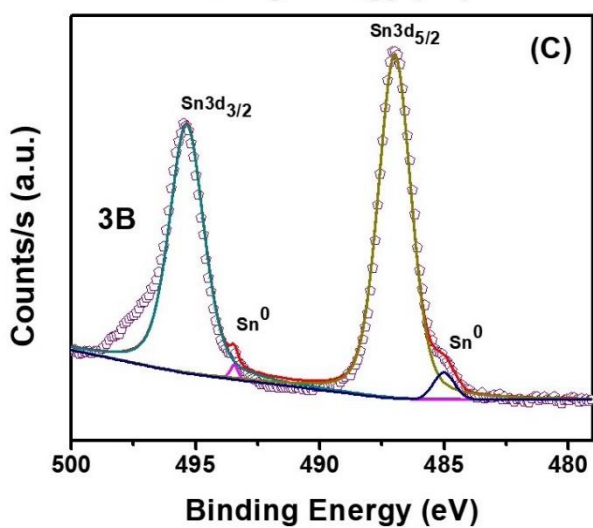
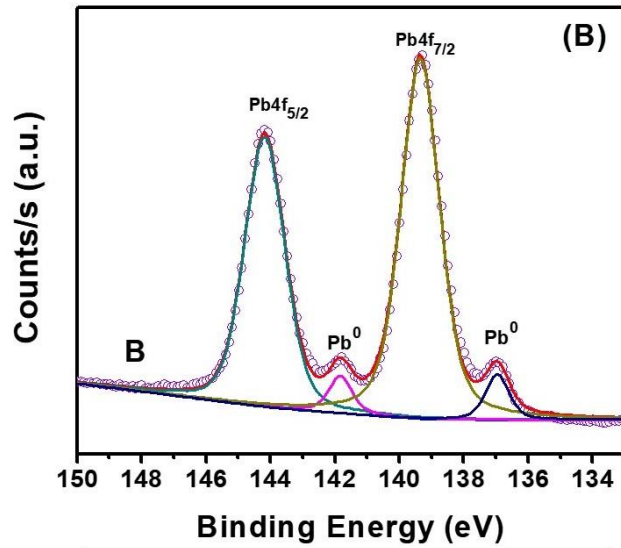
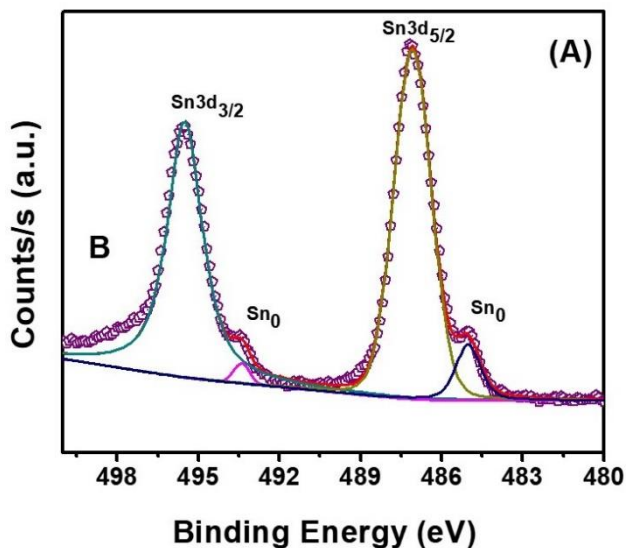
## Solder wire disk electrode



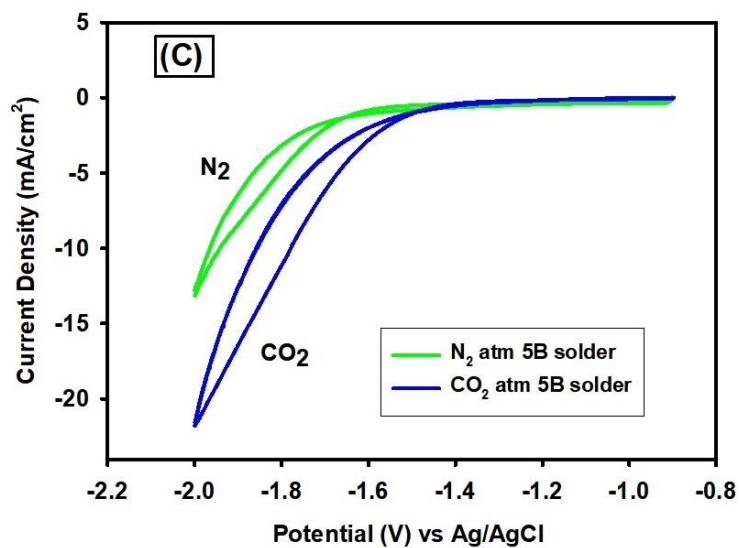
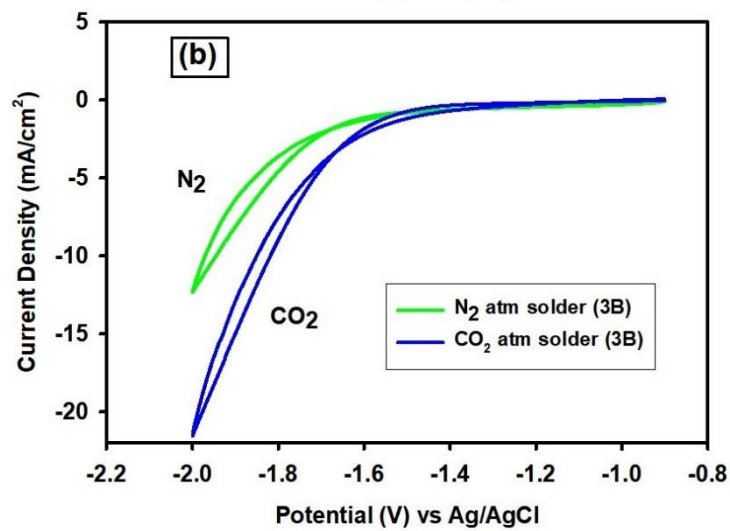
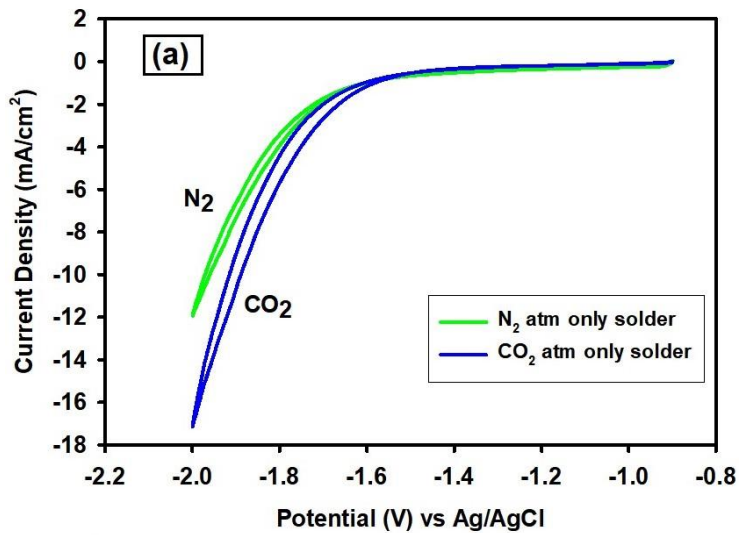
## Solder plate electrode



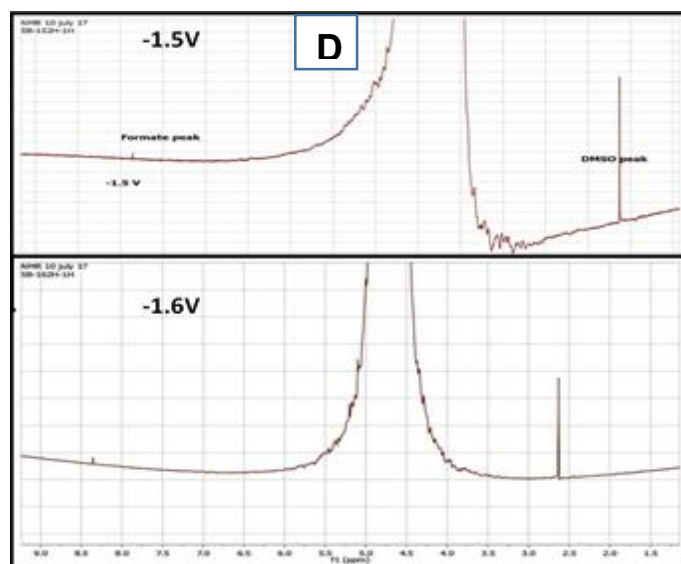
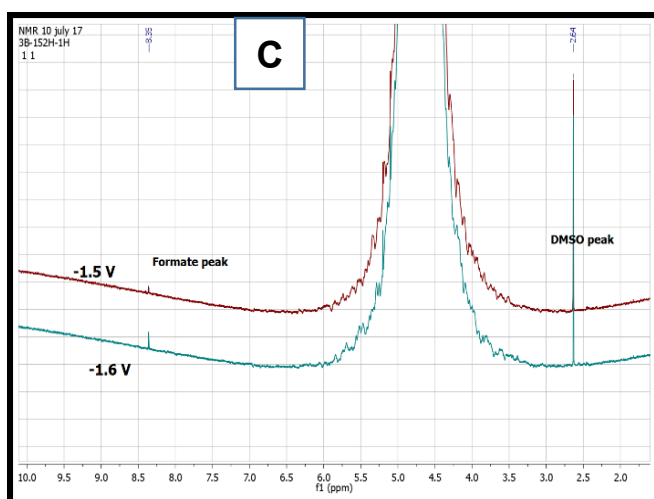
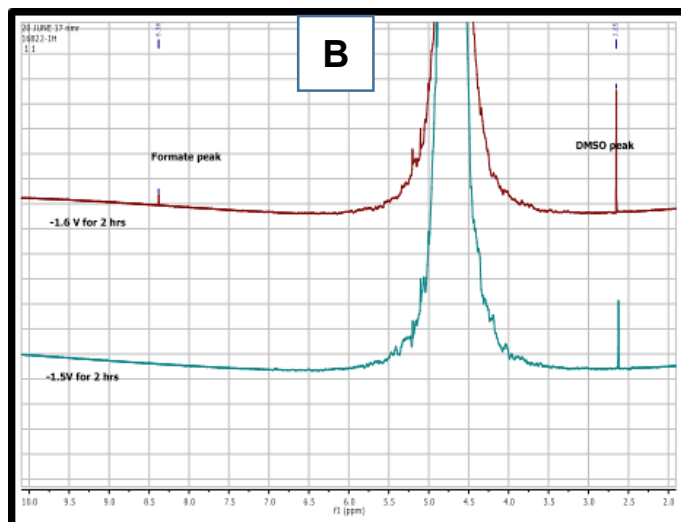
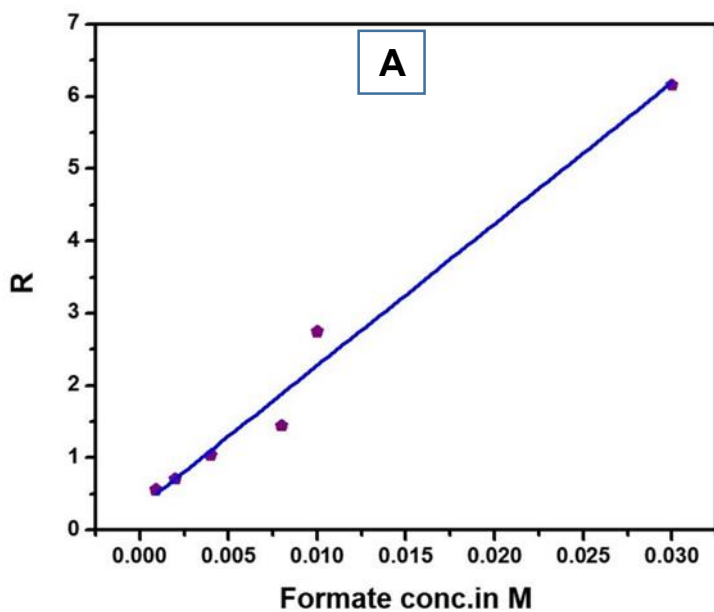
**S1:** (A) Photo of the prepared solder disk electrode from commercially available solder wire. (B) Photo of the solder plate prepared (C) Enlarged XRDs of (a) commercial solder alloy electrode, (b) solder alloy electrode polarized at -3V vs SCE for 50 s in 2 M NaOH and (c) at -5 V vs SCE for 50 s in 2 M NaOH + 1M glycerol + 1M TX-100 .



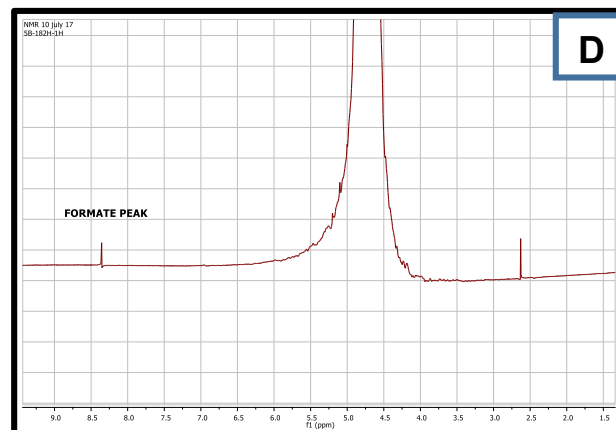
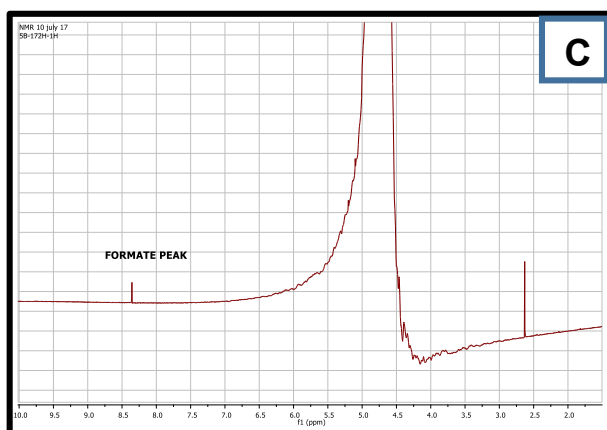
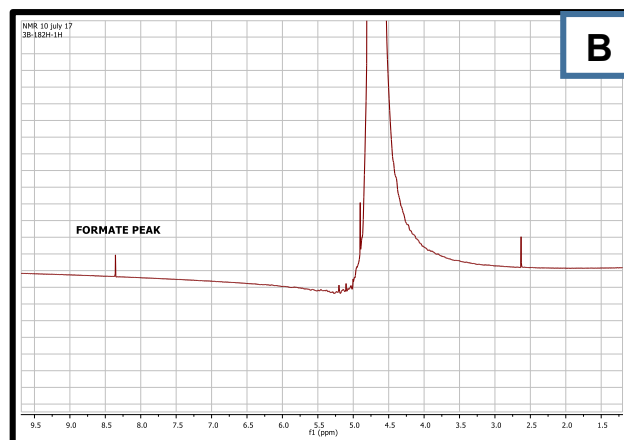
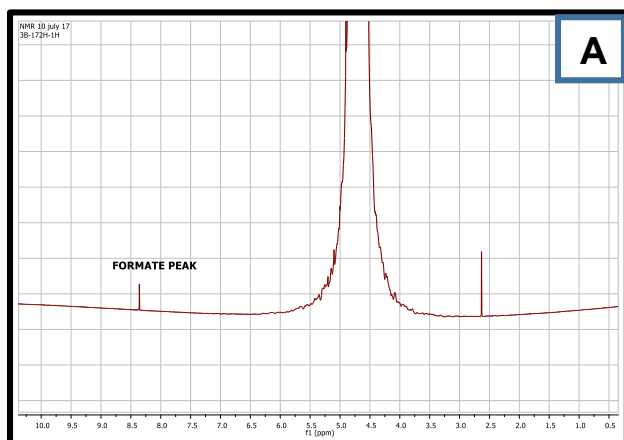
**S2:** XPS Spectra fitted (A) Sn3d for electrode B, (B) Pb4f for electrode B, (C) Sn3d for electrode 3B, (D) Pb4f for electrode 3B, (E) Sn3d for electrode 5B and (F) Pb4f for electrode 5B



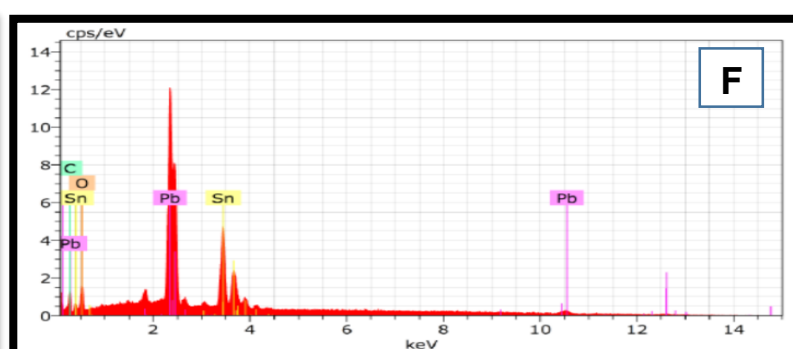
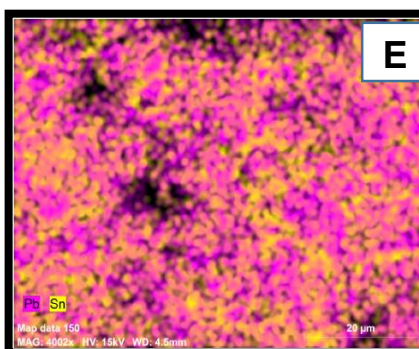
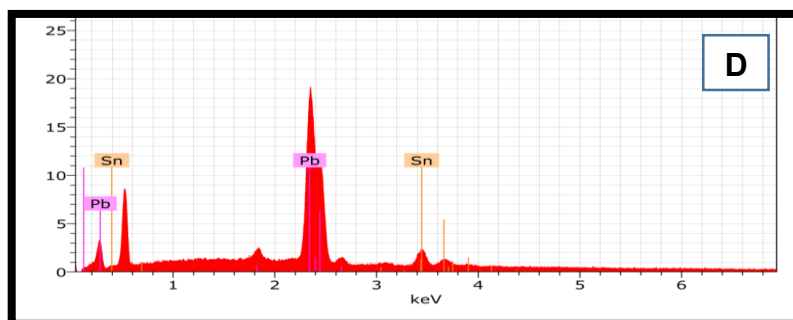
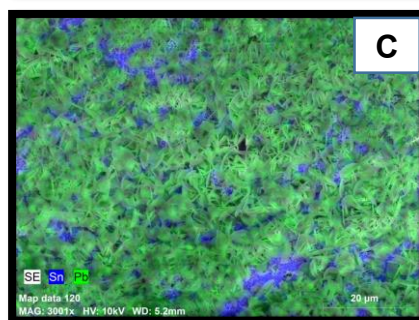
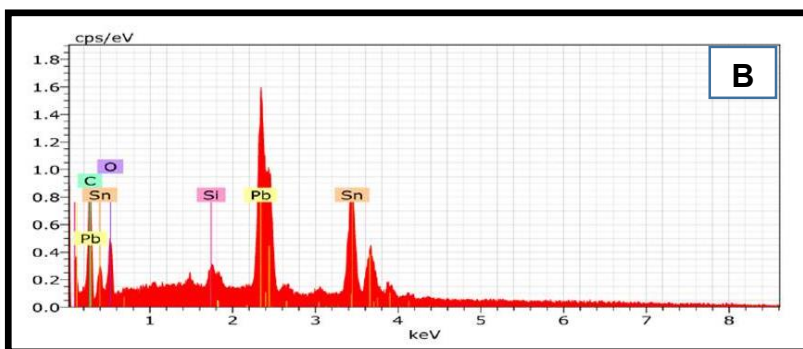
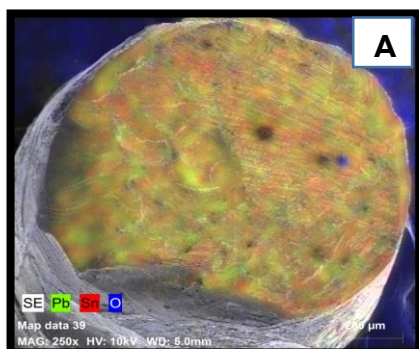
S3 (a) Cyclic voltammograms of solder disk electrode (electrode B), (b) Cyclic voltammogram of the solder disk electrode after applying potential of  $-3\text{V}$  for  $50\text{ s}$ , (electrode 3B) (c)  $-5\text{ V}$  for  $50\text{ s}$  (electrode 5B)



**S4:** (A) Calibration curve of standard different concentrations for formate obtained from NMR. (B) NMR spectra of product obtained after electrolysis for 2 hours at -1.5 V and -1.6 V vs. Ag/AgCl during electrolysis only solder plate used (C) porous solder electrode prepared at -3V 50 s (electrode 3B) (D) porous solder electrode prepared at -5V 50 s (electrode 5B).

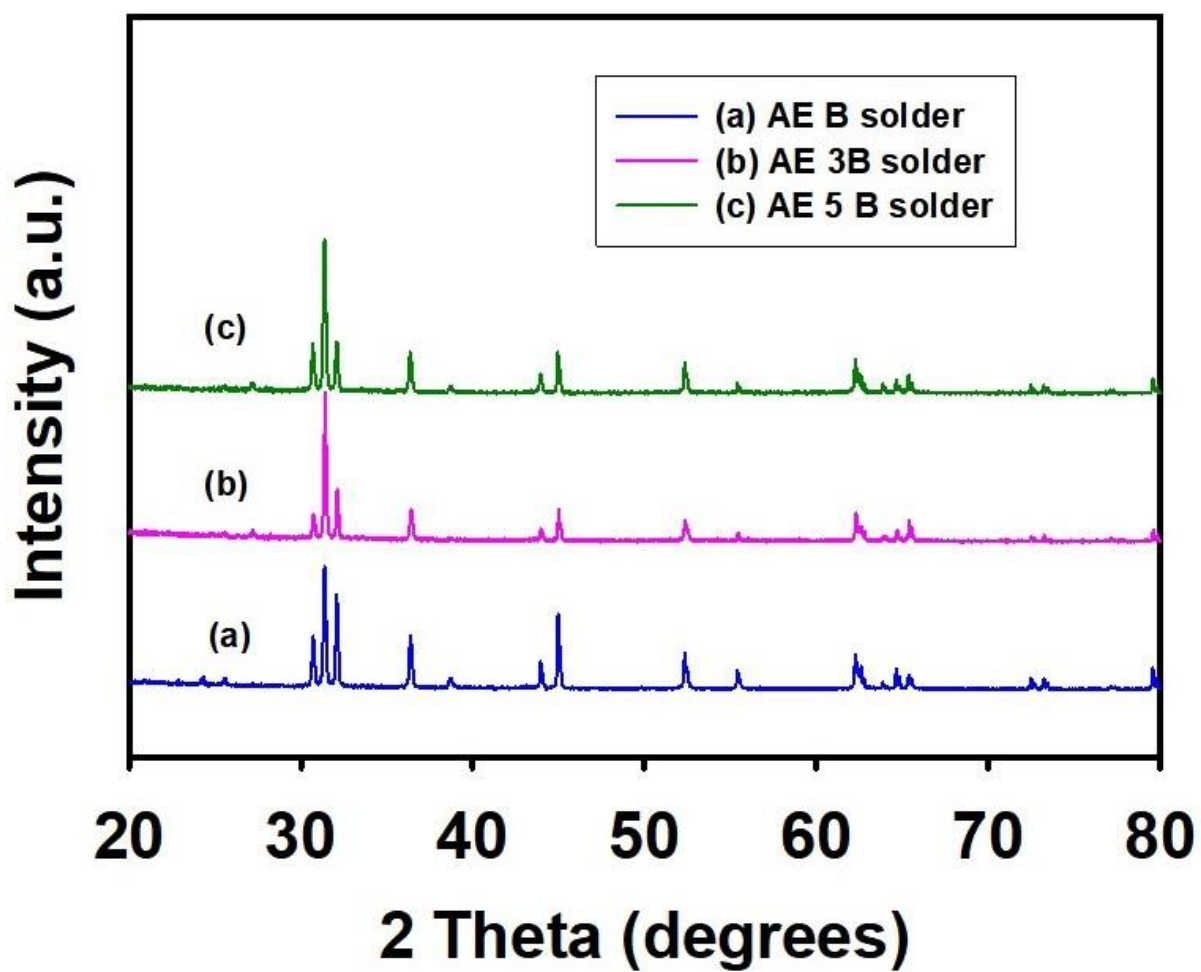


**S5:** NMR spectra of product obtained after electrolysis for 2 hours (A) at -1.7 V for electrode 3B (B) at -1.8 V for electrode 3B (C) at -1.7 V for electrode 5B (D) at -1.8 V for electrode 5B



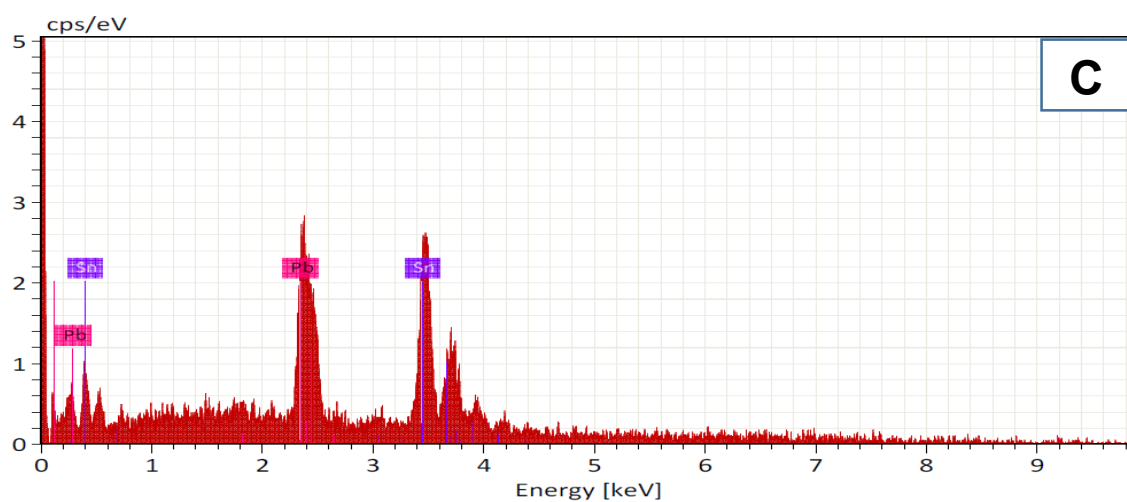
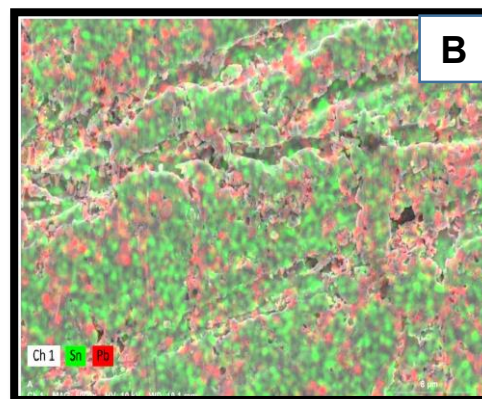
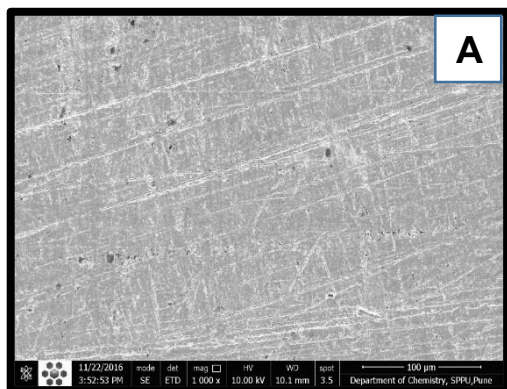
**S6:** (A) Elemental mapping and (B) EDS of the solder wire electrode (C) Elemental mapping and (D) EDS of the solder electrode polarized at -3V vs SCE for 50 seconds in 2 M NaOH (E) Elemental mapping and (F) EDS of the solder electrode polarized at -5V vs SCE for 50 seconds in 2 M NaOH + 1M glycerol + 1M TX-100



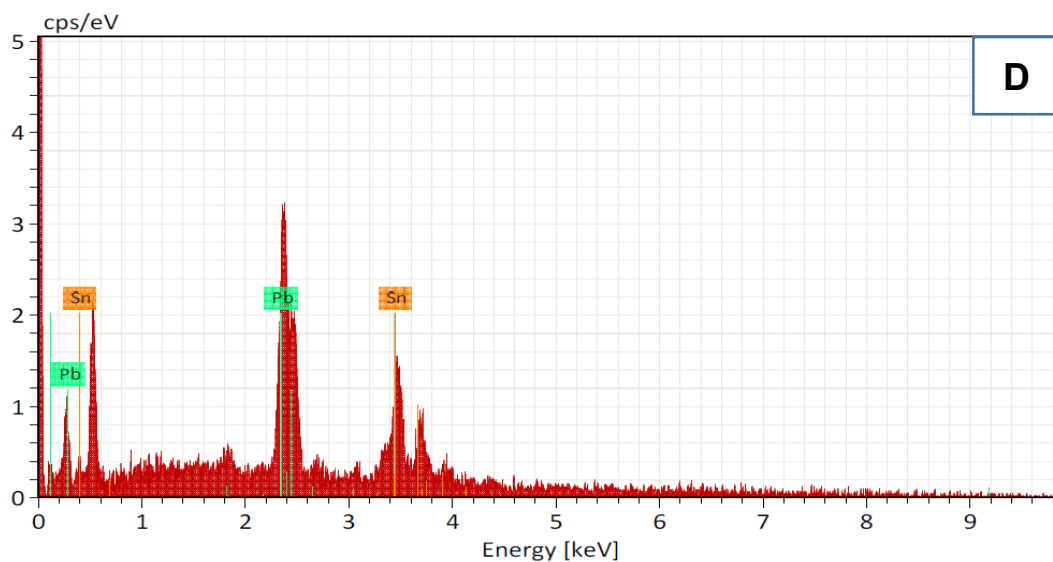
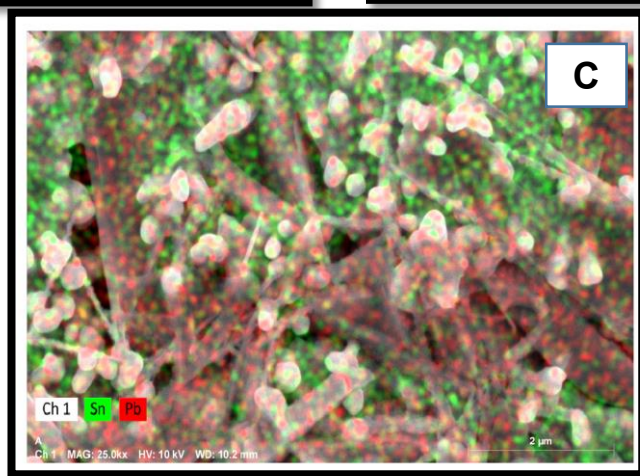
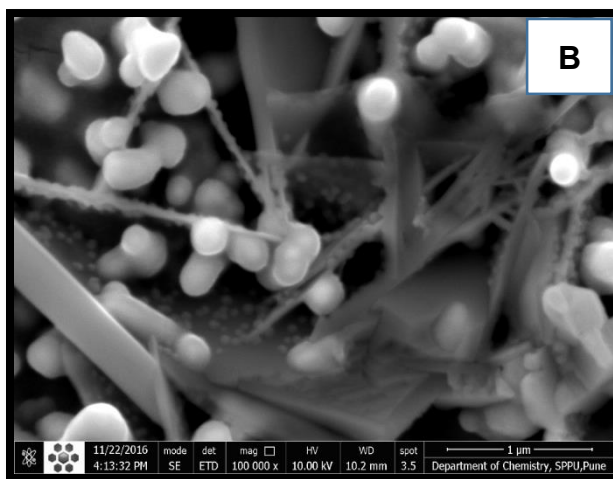
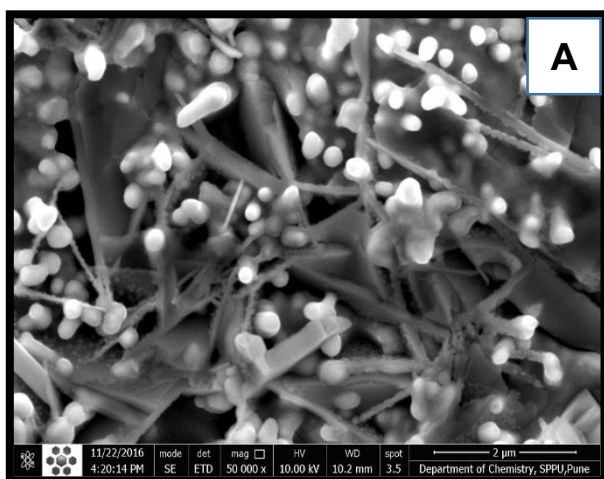


S7: XRD of B, 3B, 5B electrode after CO<sub>2</sub> electroreduction reaction for 2 hours in 0.1 M KHCO<sub>3</sub> (saturated with CO<sub>2</sub> for 30 minutes) at -1.7 V vs Ag/AgCl.

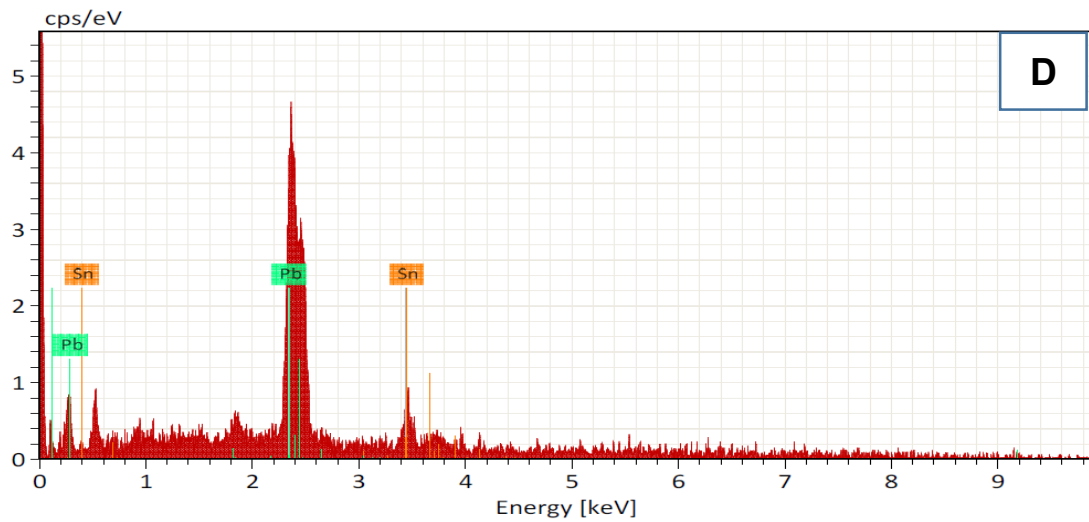
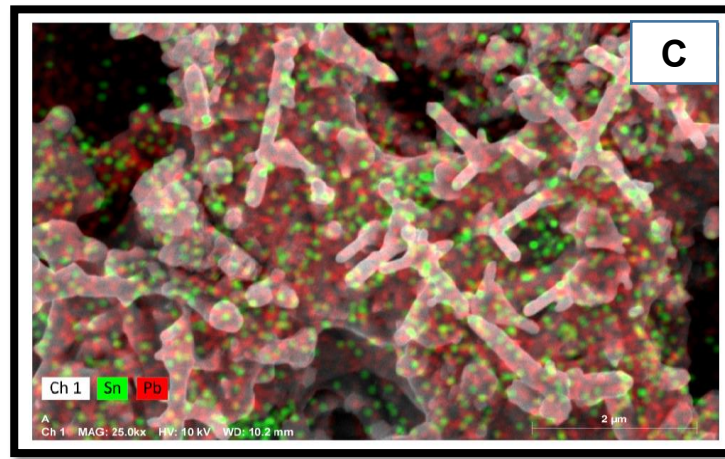
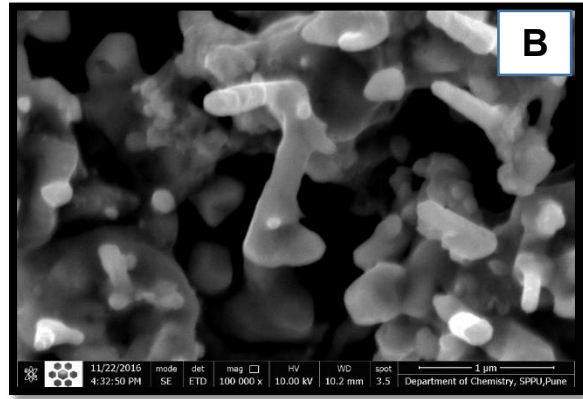
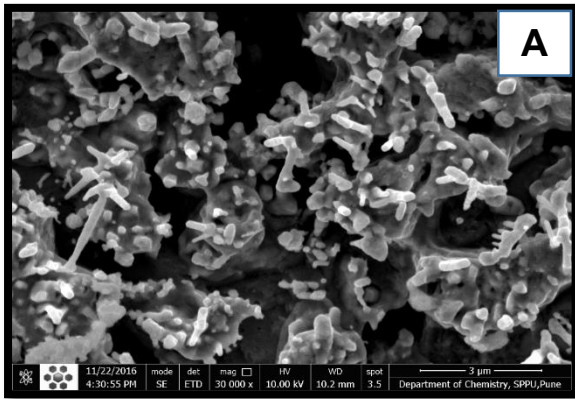




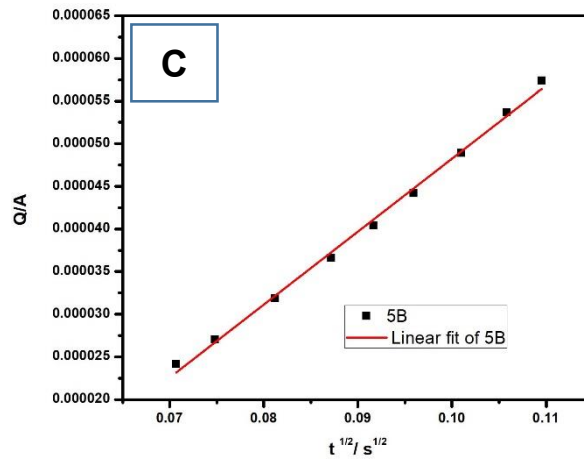
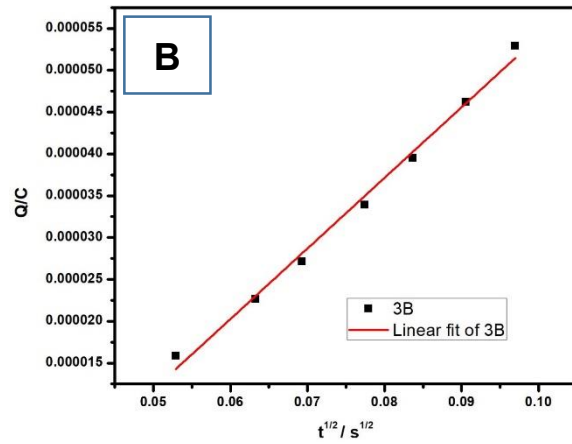
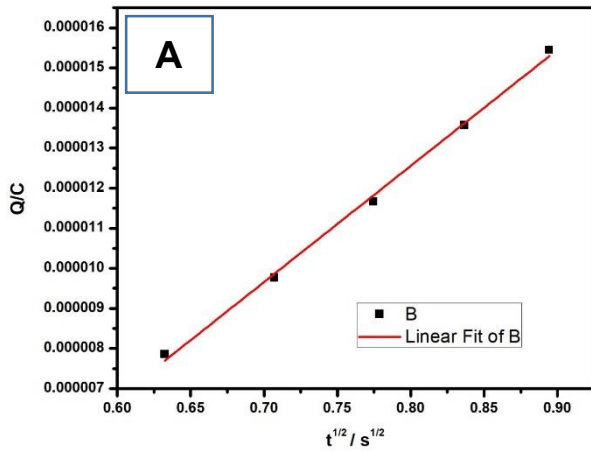
**S8:** (A) FESEM image of solder plate, (B) Elemental mapping of solder plate electrode and (C) EDS of solder plate after CO<sub>2</sub> electroreduction reaction for 2 hours in 0.1 M KHCO<sub>3</sub> (saturated with CO<sub>2</sub> for 30 minutes) at -1.7 V vs Ag/AgCl.



**S9:** (A and B) FESEM images of 3B electrode, (C) elemental mapping (D) EDS of 3B electrode after CO<sub>2</sub> electroreduction reaction for 2 hours in 0.1 M KHCO<sub>3</sub> (saturated with CO<sub>2</sub> for 30 minutes) at -1.7 V vs Ag/AgCl



**S10: (A and B) FESEM images of 5B electrode, (C) elemental mapping (D) EDS of 5B electrode after CO<sub>2</sub> electroreduction reaction for 2 hours in 0.1 M KHCO<sub>3</sub> (saturated with CO<sub>2</sub> for 30 minutes) at -1.7 V vs Ag/AgCl**



**S11: (A), (B), and (C) Plot of Q vs  $t^{1/2}$  of electrode B, 3B and 5B for ECSA calculation**

Electrochemical surface area for the 3 electrodes B, 3B and 5B were calculated using the following equation <sup>1,2</sup>

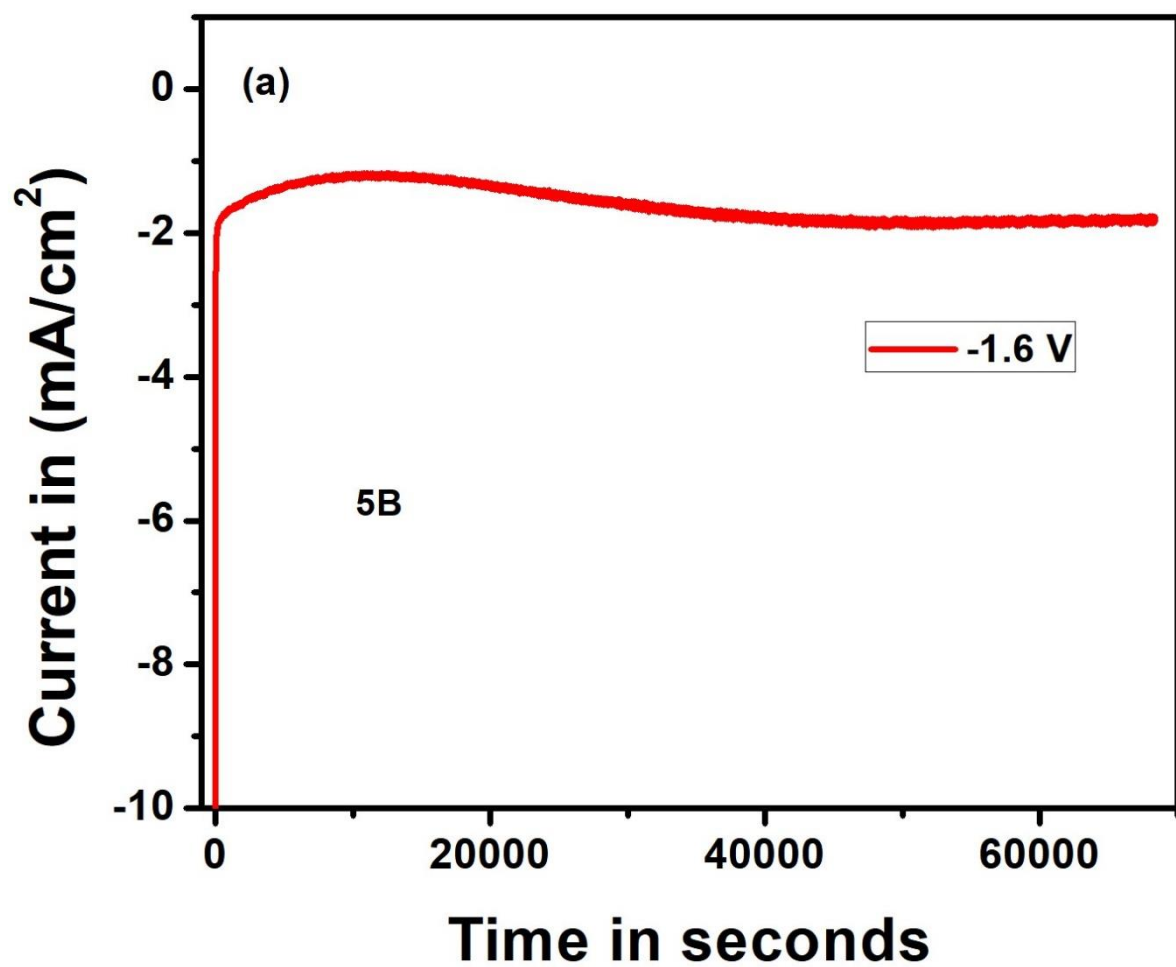
$$Q = 2nAFC_0D^{1/2}t^{1/2} / \pi^{1/2}$$

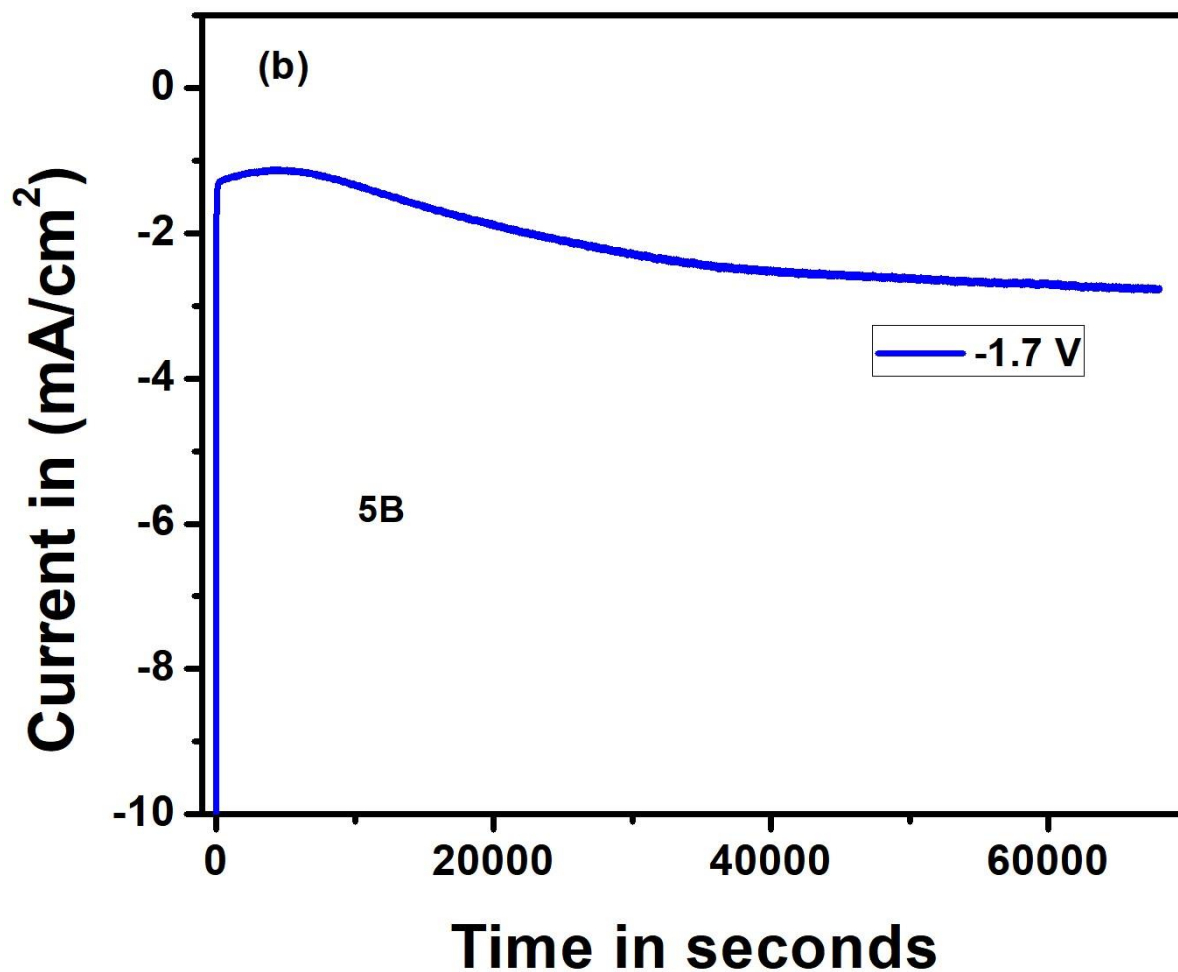
Where Q = charge in coulombs

n = Number of electrons for  $K_4[Fe(CN)_6]$

C = concentration of  $K_4[Fe(CN)_6]$

D = Diffusion coefficient for  $K_4[Fe(CN)_6]$





S12: (a) Stability testing for 5B electrode at -1.6V for 19 hours and (b) at -1.7V vs Ag/AgCl for 19 hours in 0.1 M KHCO<sub>3</sub> saturated with CO<sub>2</sub> for half hour only.

### References

- 1 Y. Xue, J. Xie, M. He, M. Liu, M. Xu, W. Ni and Y. Yan, 2018, 19210–19220.
- 2 L. Fotouhi, M. Fatollahzadeh and M. M. Heravi, 2012, **7**, 3919–3928.