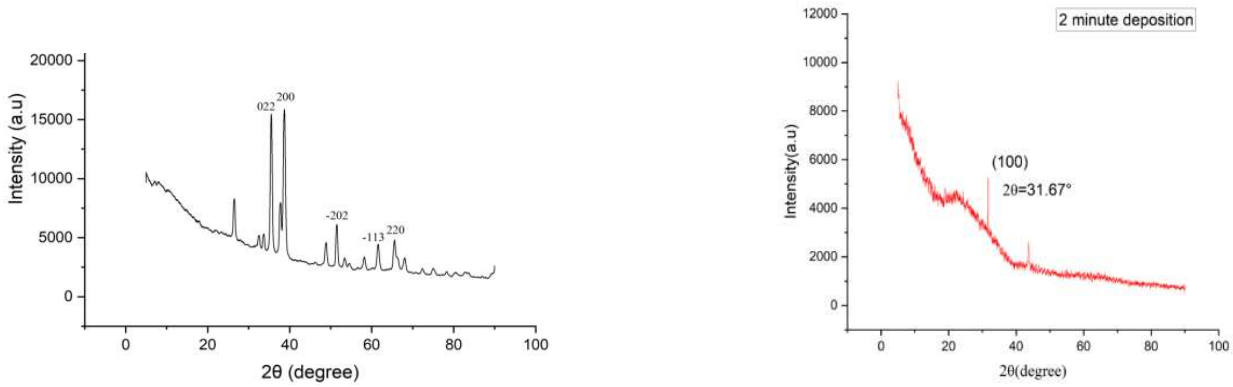
# Ethylene Glycol Electrolyte-Based Electrodeposition and Characterization of Nanostructured Earth-Abundant Metal Oxides on FTO Substrate

## Electrodeposition method for $Cu_xO$-ZnO heterustructure:

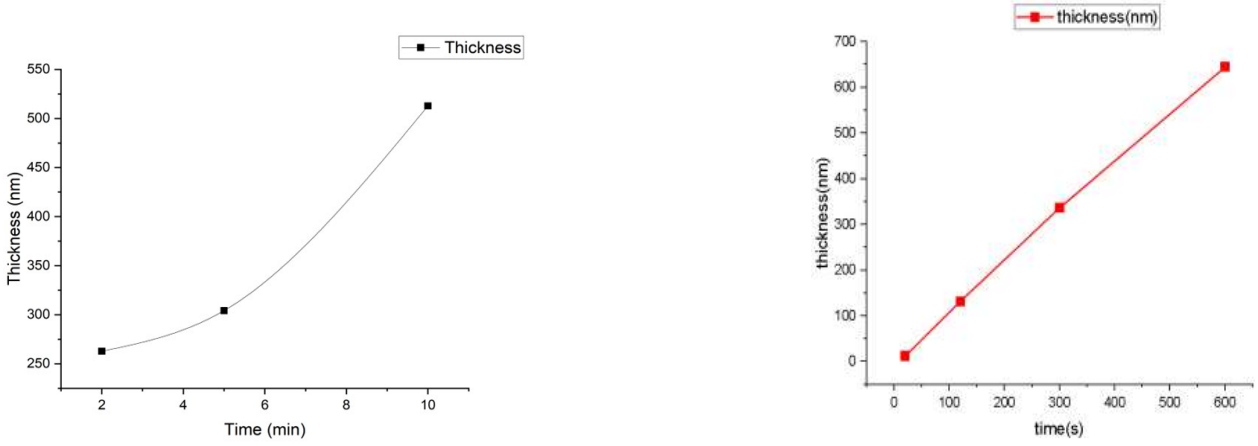
The nanostructure ZnO-$Cu_xO$ heterustructure films were fabricated employing by electrochemical deposition method on precleaned fluorine-doped tin oxide (FTO) coated glass substrate. Three-electrode cell system (Admiral Instruments Squidstat Plus Potentiostat operate by Squidstat User Interface (SUI)) has been adopted; FTO was utilized as working electrodes (WE), a Pt grid as counter electrode (CE) and a silver/silver chloride (Ag/AgCl) as reference (RE). The cell was contacted with an 0.3 M acetate solution (zinc acetate, or copper acetate) and 0.2 M Sodium acetate trihydrate, dissolved in 40 ml ethylene glycol at fixed pH (4.68 for ZnO, 5.68 for $Cu_xO$). The bath temperature was maintained at 70˚C. Deposition potential of -1.325 V for ZnO and -0.63 V for $Cu_xO$ were applied and controlled by in-situ Cyclic Voltammetry (C-V) observation. Electrodeposition was executed for varying period of 2min, 5min, 10 min for both of ZnO and $Cu_xO$ films. For ZnO/$Cu_xO$ heterostructure films, two methodologies were employed: (i) separate electrodeposition of ZnO and $Cu_xO$ from distinct electrolyte solutions of 0.3M zinc acetate, 0.3M copper acetate, and 0.2M sodium acetate trihydrate, while (ii) simultaneous deposition performed from a common buffer solution containing both precursors. The overall process was executed under computer program controlled in-situ cyclic voltammetry observations.

**Structural analysis and deposition rate of ZnO and CuO films on FTO substrate:**



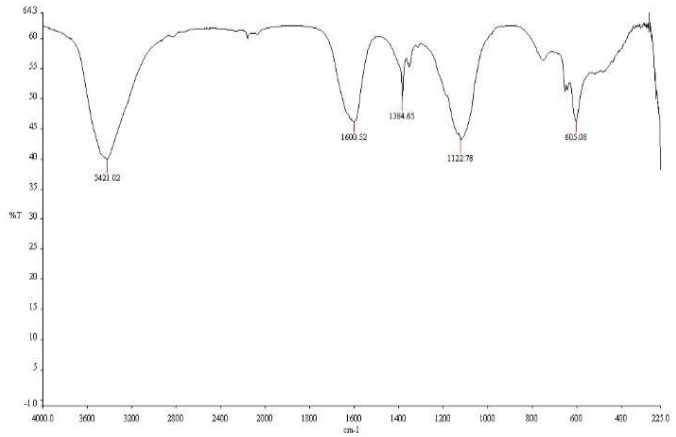

(a) (b)

XRD of (a) $Cu_xO$ and (b) ZnO thin films deposited on transparent FTO substrate.

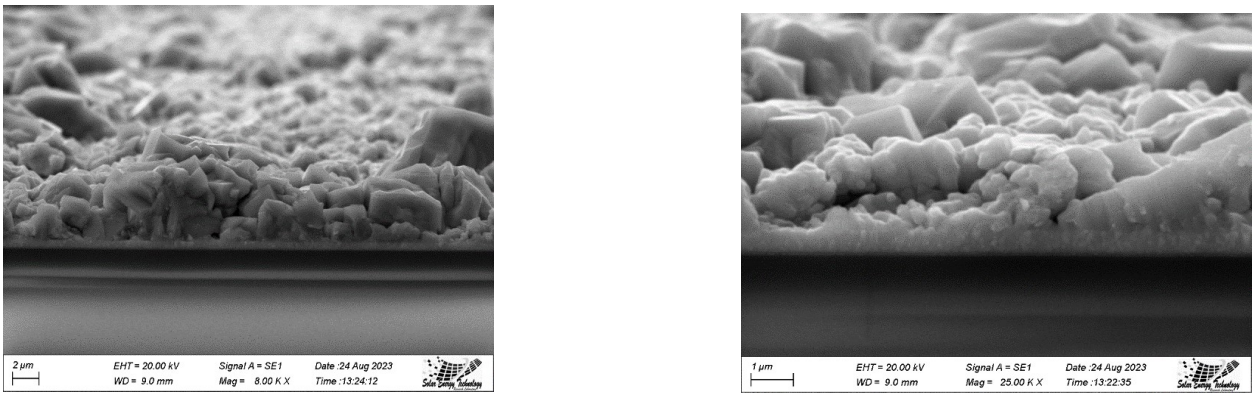



(a) (b)

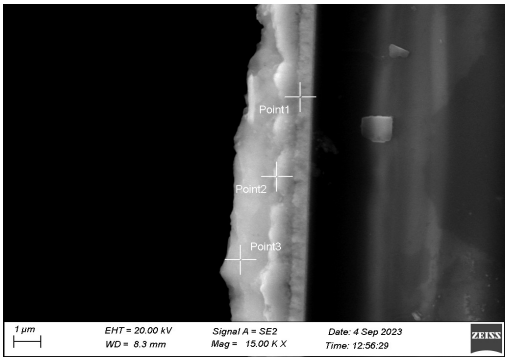
Deposition rate of (a) $Cu_xO$ and (b) ZnO thin films, both were deposited on transparent FTO substrate.




FTIR spectrum of electrodeposited bulk FTO/$Cu_xO$-ZnO heterojunction films from a common Zn-acetate and Cu-acetate precursors containing buffer solution.




SEM images of electrodeposited FTO/$Cu_xO$-ZnO heterojunction films from binary electrolytic (Zn-acetate and Cu-acetate) precursors solution.




Crossectional image of the formation of $Cu_xO$-ZnO heterostructure

A sustainable and efficient acetate-derived electrodeposition method for the scalable synthesis of ZnO and $Cu_xO$ and their heterostructures has been studied and analyzed systematically. The structural and compositional integrity, as evidenced by XRD, EDS, FTIR, and SEM analyses, ensures superior optoelectronic properties, making these heterojunctions a compelling candidate for both energy harvesting and photocatalytic applications.