



## EXPLORATION OF STRUCTURAL AND ELECTROCHEMICAL PROPERTIES OF BINARY (Ni: Cu) METAL OXIDE ELECTRODE FOR SUPERCAPACITOR APPLICATION

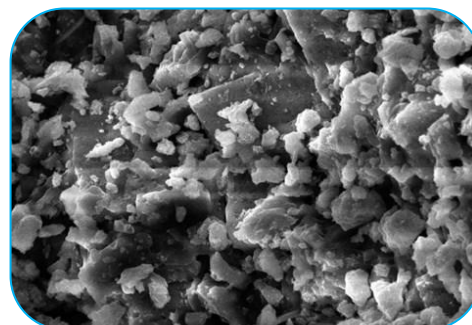
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### ABSTRACT:

The Binary (Ni : Cu) Metal oxide electrode material was synthesized by Sol-gel spin coat method from  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{CuCl}_2 \cdot 6\text{H}_2\text{O}$  solutions. The obtained products were characterized using X-ray Diffractometry, Scanning Electron Microscopy (SEM), Cyclic Voltametry (CV) and Galvanostatic Charge-Discharge (GCD). XRD revealed the crystalline structure of thin film electrode by exhibiting sharp peaks of  $\text{NiO}_2$  and  $\text{CuO}_2$  corresponding to different planes. Deep porous and rough surface morphology has been observed from SEM micrographs. Electrochemical studies were carried out to examine the specific capacitance of binary (Ni-Cu) Metal oxide electrodes in 0.1 M KOH electrolyte at various scan rates.



**KEY WORDS:** (Ni:Cu) oxide; Spin Coating; XRD; SEM; CV; GCD;

### INTRODUCTION

Electrochemical capacitors (ECs) also known as supercapacitor or ultracapacitor, is one of the emerging class of energy storage devices. Electrode materials, which have dominant effect on the electrochemical capacitor performance, have become the research focus for several years [1]. Among them pseudocapacitive-based materials such as transition metal oxides (TMOs) have become judicious choices due to their robust crystal structure ultrahigh capacitances and energy densities [2,3]. But they suffer from low rate capability and poor cyclic stability.

To enhance the performance of transition metal oxides with unitary component, nickel oxide for example, binary transition metal oxides (BTMOs) have attracted worldwide interest as supercapacitor materials since cationic substitutions in binary oxides have shown dramatic improvements over their binary analogues [4,5]

More concretely, copper is considered to be fascinating candidate for electrode materials due to its low cost, abundant resources, good conductivity, high chemical stability and green environmental protection [6]. Moreover copper ions into nickel oxide lattice can accelerate the charge transfer and enhance the electrode material conductivity showing a good synergistic effect between copper and nickel [1]. Many binary oxides have been proposed for nickel oxide based electrochemical capacitors. It has discovered that nickel copper oxide ( $\text{Cu}_{0.2}\text{Ni}_{0.8}\text{O}$ ) is promising binary metal oxide candidate to enhance the performance of bare nickel oxide and 30% increment in specific capacitance is achieved [7].

In the present work, nickel-copper oxide was successfully prepared by sol-gel spin coating deposition technique. A systematic study of its structural and electrochemical properties is reported.

### EXPERIMENT:

Binary metal (Ni: Cu) oxide thin films were grown on stainless steel substrate by sol-gel spin coating method using 0.0125 M Nickel chloride ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ) and 0.2 M Cupric chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ) precursors. Initially the substrates were cleaned with detergent, washed with double distilled water, then rinsed with acetone and dried. The solution was prepared with percentage of Cu as 50%. Ethanol was then added to the solution for the gel formation and stirred for 6 hours by keeping magnetic stirrer temperature at  $60^\circ\text{C}$ . Then solution was settled for 24 hours for the formation of gel. After aging we got a clear and viscous solution which was light blue in color.

The above solution was deposited on to the stainless steel substrate (SS) by spin coating technique. Before deposition, stainless steel substrate were polished with zero grade polish paper and then degreased with water and acetone, impressed in 0.1M  $\text{H}_2\text{SO}_4$  solution and kept in ultrasonic bath for 20 minute, after that rinsed with double distilled water and dried up in the air [8].

### RESULT AND DISCUSSION:

#### Structural Analysis by XRD

The structural analysis of as deposited film was carried out using X-ray diffractometer. The XRD pattern of binary (Nickel : Copper) oxide thin films annealed at  $400^\circ\text{C}$  is shown in Fig. 2. The samples are crystalline in nature.

XRD patterns were obtained with source  $\text{CuK}\alpha$  with  $\lambda = 1.54060$ , the  $2\theta$  angle is varied from  $20^\circ$  to  $90^\circ$ . The XRD pattern implies that as deposited films are crystalline in nature with monoclinic structure. XRD pattern showed the peaks of both oxides but dominating peak is observed for CuO phase. Nickel-Copper ratio that has the synergistic effect of optimal nickel-copper results in the best electrochemical performance of the candidate.

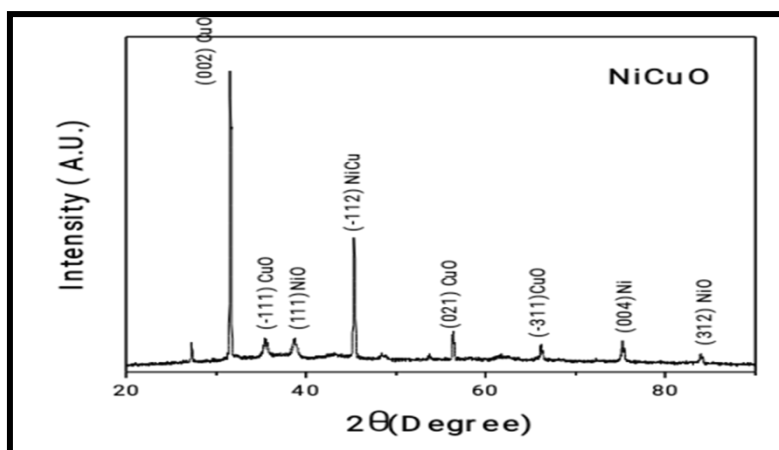
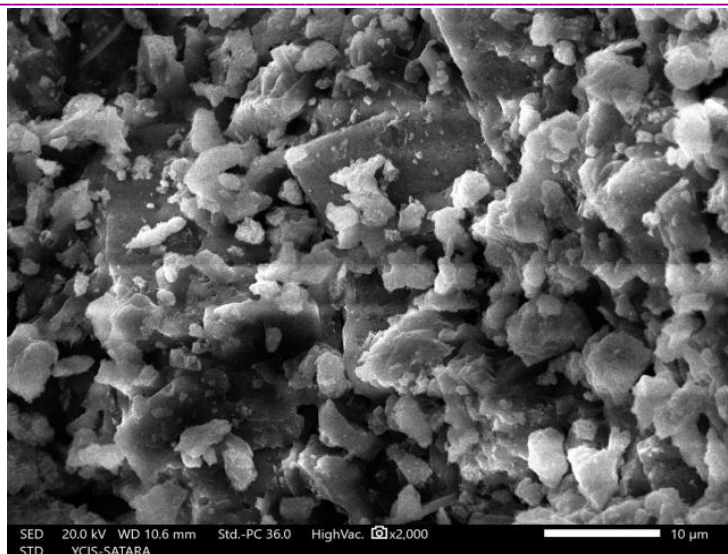


Fig.1 : XRD pattern of as deposited binary metal (Ni:Cu) oxide thin film.

#### Morphological Analysis by SEM

The surface morphological study has been carried out by Scanning Electron Microscopy (SEM) using JEOL JSM-IT200 instrument.

The SEM images revealed the formation of thin film which is well adherent to the substrate. The SEM image of binary metal (Ni: Cu) oxide at  $\times 2,000$  magnification is as shown in the Fig.2 From the SEM image the irregularly arranged agglomerates forming a rough surface with porous morphology is observed. The porosity prompts possibility of better electrochemical supercapacitor behavior of binary metal (Ni: Cu) oxide thin film electrode [9].



**Fig. 2 : SEM Morphology of as deposited binary metal (Ni: Cu) oxide thin film.**

### Electrochemical Analysis by Cyclic Voltammetry

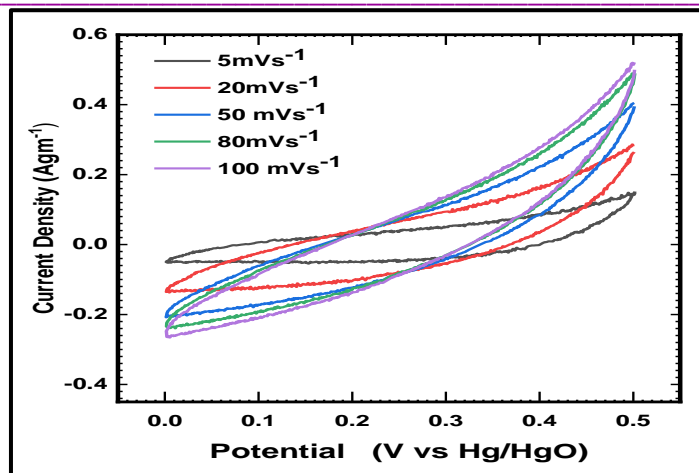
The CV is an important technique in electrochemistry which provides the qualitative information about the electrochemical process, whether the process is Faradic or non-Faradic, that takes place in the material. The electrochemical analysis of binary metal (Ni: Cu) oxide thin film was done with Cyclic Voltammetry (CV) measurements was subjected at various scan rates from 5 mV/s to 100 mV/s in 0.1M KOH electrolyte with potential window of 0V to 0.5 V. The as deposited binary metal (Ni: Cu) oxide thin film electrode is used as working electrode, Platinum as counter electrode and Hg/HgO (Mercury/Mercury Oxide Electrode) as reference electrode. During the different scan rate, it was observed that the current under the curve gradually increased with scan rate. From this we can conclude that voltammetric current is directly proportional to the scan rates of CV and is a good sign of supercapacitive behavior [10].

To calculate the specific capacitance (SC) of the electrode from the CV curves following formula was used.

$$SC = \frac{c}{m} = \frac{\int_{V1}^{V2} I dV}{m(V) \frac{dV}{dt}}$$

Where  $m$  is the mass of active material,  $V1$  and  $V2$  are the potential limits,  $\frac{dV}{dt}$  is the scan rate potential [11].

Fig. 3 shows cyclic voltammograms with potential window of 0 V to 0.5 V at various scan rates 5, 20, 50, 80 and 100 mVs<sup>-1</sup>. From CV analysis, the electrode exhibited maximum specific capacitance of 781 F/g at 5mV<sup>-1</sup> scan rate. As current under curve slowly increased with scan rate. we conclude that the voltammetric current is directly proportional to scan rate and this is a good indication of supercapacitive behavior [12].



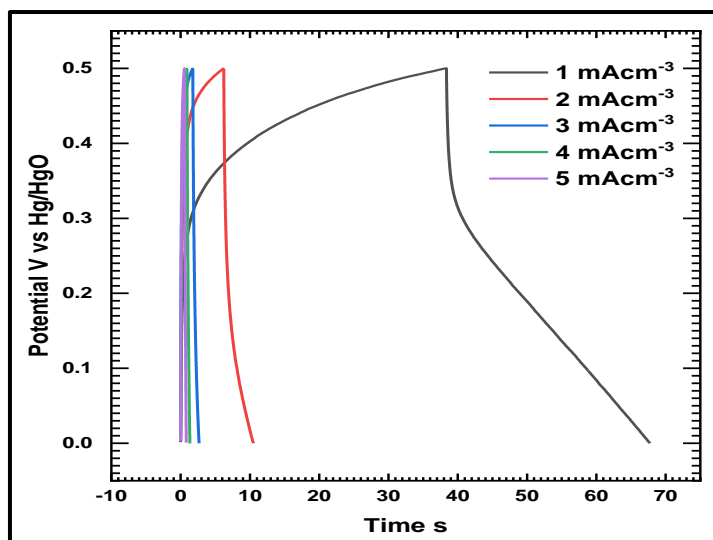
**Fig. 3 : CV curves of as deposited binary metal (Ni: Cu) oxide thin film.**

**Galvanostatic Charge-discharge**

Galvanostatic charge-discharge curves at various current densities of binary metal (Ni: Cu) oxide is as shown in the Fig. 4. The specific capacitance, can also calculated from charging/discharging curves according to the equation

$$C_s = \frac{I \cdot t}{m \cdot \Delta V}$$

Where I is the applied charging/discharging current, t is the discharge time, m indicates the mass of active electrode material and ΔV is the potential range of scanning segment [13]. The specific capacitance is calculated according to discharging curve using above formula. A high specific capacitance of 248 F/g is obtained at current density of 1 mA/cm<sup>2</sup>.



**Fig.4 : Charge Discharge curves of as deposited binary metal (Ni: Cu) oxide thin film.**

**CONCLUSION:**

In the present work, Binary Nickel-Copper metal oxide is successfully grown on stainless steel (SS) substrate using sol-gel spin coating deposition technique. The structural investigation suggests

crystalline nature and monoclinic structure of binary (Ni: Cu) metal oxide. XRD pattern include dominating peaks of both metal oxides. SEM images exhibited irregularly arranged agglomerates forming rough surface with porous morphology. From CV analysis the maximum specific capacitance obtained is 781 F/g at  $5\text{mV}^{-1}$  scan rate. From GCD a high specific capacitance of 248 F/g is obtained at current density of  $1\text{ mA/cm}^2$ . This study corroborates that nickel copper oxide is a promising material as electrochemical capacitor electrode material in future.

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