

# Preparation of Cu-O-Zn superoxide by sol-gel technique

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## ABSTRACT

A general sol-gel method was successfully applied to the synthesis of homogeneous 1-Copper, 2-Zinc Superoxide (Cu-O-Zn) material synthesized in different ratios viz. 5:2 and 5:4. These metal oxide were prepared using solutions of respective metal chlorides without the need for alkoxides, polymeric gel agents or elaborate reaction schemes. The gels were dried in ambient condition. The formed material was then characterized using XRD and SEM. The SEM image reveals that the particles are formed in oval shape and XRD data suggests that these samples of particle size  $\mu\text{m}$  range are highly crystalline.

**Keywords-** Metal-O-Metal, Cu-O-Zn, sol-gel technique.

## 1. INTRODUCTION

Cu-O-Zn Mixed oxide is a catalyst having wide applications and has been synthesized here using sol-gel method. Among the various techniques adopted for synthesis of crystalline material like sol-gel method, sonochemical method, co-precipitation method, solvo-thermal technique, microwave synthesis etc., we have chosen sol-gel process, a wet chemical technique, as it has the advantage of synthesizing the mixed metal oxides (MMO) with better homogeneity, defined pore sizes and a better control over the structural properties. [1]. Due to this high and rapid productivity of fine homogeneous powder, cost effective processing, good atomic efficiency and rapid response to changing condition in catalyst system, this technique is widely used as an effective technique for synthesis of materials by material scientists, engineers and researchers [2]. Other advantages of this method are its versatility and the possibility to obtain high purity materials, the provision of an easy way for the introduction of trace elements, allowance of the synthesis of special materials and energy savings by using low processing temperature [3]. The material obtained in this process is largely amorphous in phase but on heating produces the crystalline phase.

The present work focuses on the synthesis of metal-oxide-metal Cu-O-Zn by sol-gel method. The crystalline phase of these obtained superoxide materials were characterized by XRD and morphology was investigated by scanning electron microscopy (FESEM).

## 2. EXPERIMENTAL WORK

For this purpose we used the respective metal chlorides as precursor and reacted them with acid and alcohol to obtain the respective mixed metal oxide in gel form. The preparation is explained below:-

**2.1 Chemicals involved:** Zinc Chloride ( $\text{Zn Cl}_2$ ) was purchased from Merck Life Science Pvt. Ltd, Mumbai, Copper Chloride ( $\text{CuCl}_2$ ) was purchased from Merck Specialties Pvt. Ltd, Mumbai. Hydrochloric

acid (HCl) and ethyl alcohol (C<sub>2</sub>H<sub>5</sub>OH) were purchased from Himedia Laboratories, Mumbai. The chemicals of analytical grade were received and used without further purification.

**2.2 Synthesis of 1-copper, 2-zinc superoxide:** These mixed metal oxide samples were prepared for different ratio of precursor salts viz 5:2 and 5:4. For preparation of Sample 1 i.e. 1-copper,2-zinc superoxide in ratios of constituents metals as 5:2, we took – 4 gm zinc chloride and 10 gm of copper chloride. Further for Sample 2, we again took 8 gm zinc chloride and 10 gm of copper chloride to obtain 1-copper, 2-zinc superoxide in ratio 5:4.

The white coloured Zinc chloride anhydrous powder and blue coloured copper chloride powder in their fixed ratios were made to react with 20 ml of hydrochloric acid (pure 35%) in a beaker kept over a magnetic stirrer. After stirring for almost an hour we added ethanol (pure 99%) in to this solution. The solution was of yellowish colour with a tinge of blue. We kept stirring it for another four hour at room temperature till the solution turn into a denser gel of blue colour. In this process the precursors react with the OH functional group of ethanol. When stirring was stopped and evaporation of solution was allowed to occur at room temperature, a reaction occurred within several minutes, followed by a rapid formation of a rigid bluish green coloured gel form of 1-Copper,2-Zinc Superoxide. Then for purpose of characterization we heat this gel at 60 degree centigrade in an oven to get a dry powder.

### 3. RESULT AND DISCUSSION

The powders obtained as **Sample 1** and **Sample 2** were characterized by XRD and FESEM to understand the orderly arrangement and morphological structure.

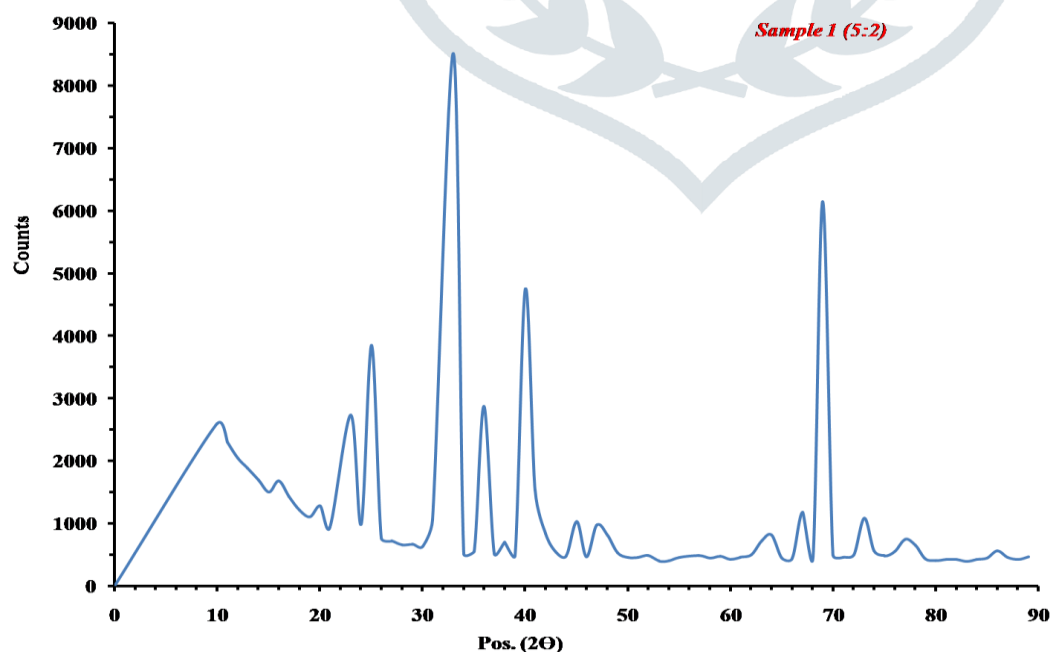
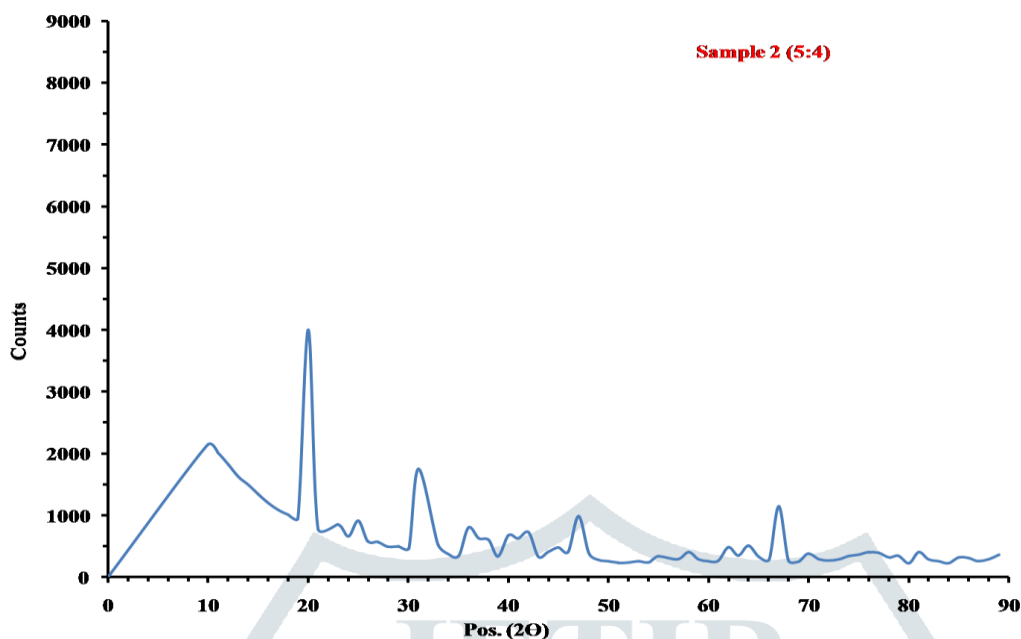
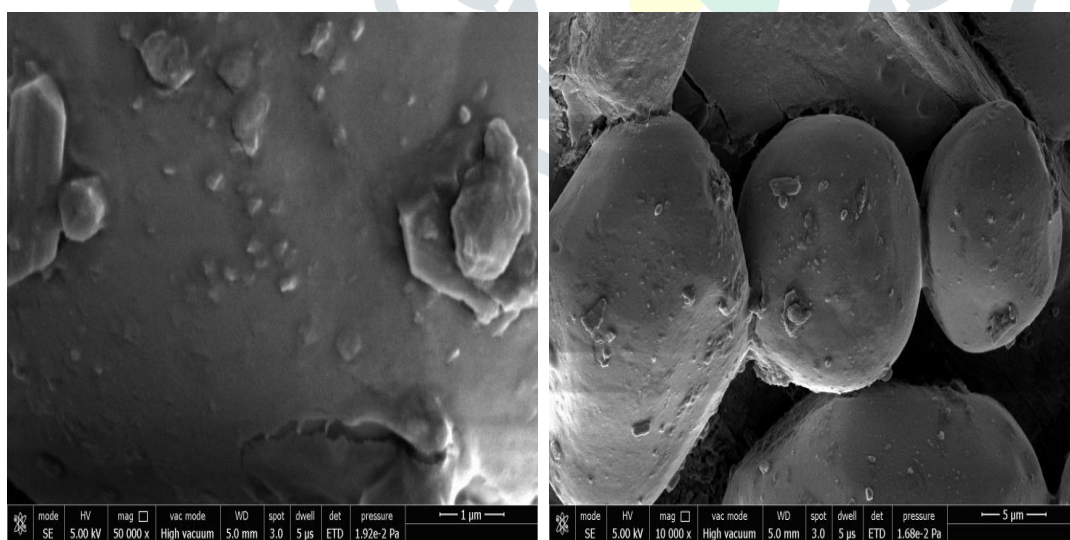


Figure 1: XRD of Mixed Metal-Oxide [Cu-O-Zn] in the ratio 5:2 ratio.

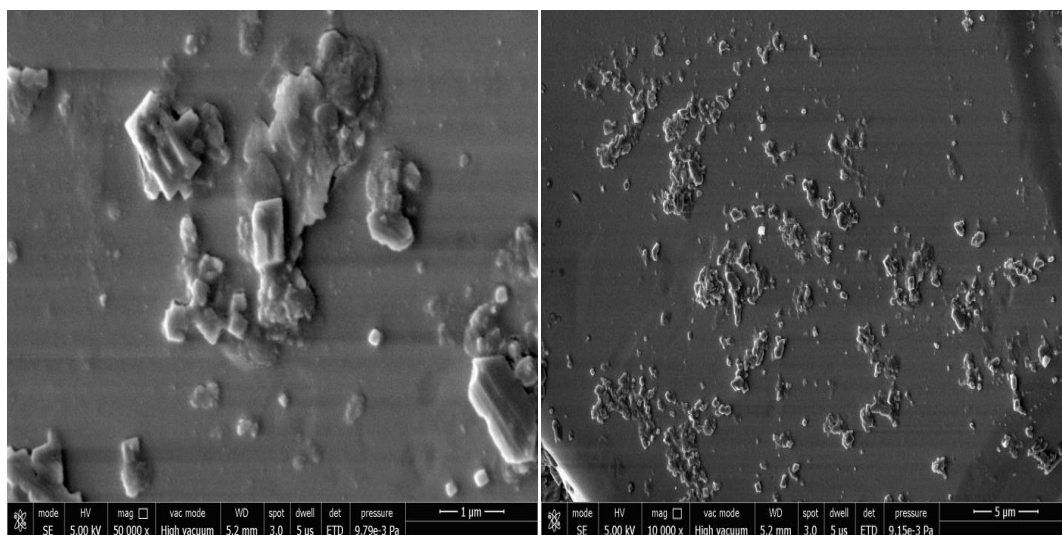


**Figure 2: XRD of Mixed Metal-Oxide [Cu-O-Zn] in the ratio 5:4 ratio.**

**XRD Patterns of the Samples:** Figure 1 and 2 show the XRD pattern of the synthesized mixed metal oxide material. All the diffraction peaks are sharp which indicate the Cu-O-Zn Sample1 and Sample 2 to be having high degree of crystallinity. The comparison of both the diffraction patterns shown in figure 1 and 2 makes it explicit that on insertion of higher amount of Zn metal into the mixed metal oxide Sample 2 results in decrease of intensity and disappearance of certain characteristic peaks clearly visible for Sample 1. This clearly suggests significant interaction between the both the metals forming superoxide.



**Figure 3: FESEM images of Metal-Oxide-Metal [Cu-O-Zn] of 5:2 ratio.**



**Figure 4: FESEM images of Metal-Oxide-Metal [Cu-O-Zn] of 5:4 ratio.**

**SEM images of the Samples:** Figure 3 and 4 depict the SEM images at different magnifications for the synthesized mixed metal oxide material. These images reveal the significant difference in surface morphology on change in ration of Zinc precursor. These images also reveal the particles to be oval shaped and of higher diameter for sample 1 whereas random shaped and lesser diameter in sample 2.

#### 4. CONCLUSION

1-Copper, 2-ZincSuperoxide formation by using different ratios of precursor metal halides by sol- gel method has been presented. The method is quite simple and effective in governing the structural variations. XRD patterns and SEM images clearly show the difference in crystallinity and structural behaviour due to interactions between metal precursors when taken in different ratios.

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