Synthesis of 1-Copper, 2-Lead superoxide [Pb-O-Cu] Mixed Metal Oxide By Sol- Gel Technique

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ABSTRACT:

The easy, fast, simple and cost effective sol-gel technique to produce gels, and powders has been used to synthesis a novel metal-oxide-metal 1-Copper , 2-Lead Superoxide [Pb-O-Cu] in different ratios viz. 2:5 and 4:5. For the purpose of synthesis of [Pb-O-Cu]; the respective metal chlorides i.e. Lead chloride and copper chloride have been taken as the starting precursors dissolved in hydrochloric acid followed by stirring on addition of ethanol to produce a gel form of the mixed metal oxide material. These after drying them at room temperature were used for structural characterization using XRD and FTIR. The FTIR spectra reveals that the and XRD data suggests that these samples of particle size μm range are highly crystalline

Keywords: Metal-Oxide-metal, [Pb-O-Cu], Sol-Gel Method.

1. INTRODUCTION:

Mixed Metal oxides are that novel class of material that have a wide application due to fascinating electronic, chemical, and physical properties that are often highly sensitive to changes in their chemical environment. The mixed oxides systems are such structures that possess unique properties either in combinations with the properties the individual oxide have or even those that neither of these individual oxides has. So a lot of interest has been generated in the synthesis of these novel materials. Further these materials can be easily synthesized by sol gel technique. In the sol-gel method, the sol which is a solution of any of the metallic chlorides, metallic nitrates or organo-metallic precursors, has suspended that are polymerized at room temperature to undergo a phase transformation in order to form a wet gel. In the gel state, the particles are prevented from agglomeration by mutual repulsion of similar charges at the particle surface using alcoholic media. The nature and kinetics of these reactions determine the properties of the gel. The solvent is removed by drying the gel and the next step is a proper heat treatment to obtain the desirable coating or thin film or the powder state. Apart from being easier and simpler this method has and added advantage of producing highly pure materials.

In this present work, the authors have used this simple sol gel technique to prepare 1-lead, 2-copper superoxide [Pb-O-Cu] starting with respective metal chloride precursors. The structural analysis of these obtained superoxide materials were done using Powder X'Pert Version 3 X Ray Diffractometer and Perkin Elmer Spectrum Version 10.4.00 FTIR Spectrophotometer. It is expected that the obtained [Pb-O-Cu] material can be utilized for wide variety of applications.

2. METHODS AND MATEIRALS

For the synthesis purpose we took the metal chlorides as precursor, reacted them with hydrochloric acid and ethanol to obtain the respective metal-oxide-metal. The preparation is as explained below:-

a. **Materials :** Lead Chloride was purchased from Merck Life Science Pvt. Ltd, Mumbai , Copper Chloride & Dichloromethane was purchased from Merck Specialties Pvt. Ltd, Mumbai, Hydrochloric acid & Ethyl alcohol was purchased from Himedia Laboratories, Mumbai.

b. **Synthesis of 1-Copper, 2-Lead superoxide :** These mixed metal oxides or metal-oxide-metal have been prepared for different ratio of precursor salts viz 2:5 and 4:5. For preparation of 1-Copper , 2-Lead superoxide in different ratios of constituents metals we took - 4gm lead chloride in 10gm copper chloride to obtain 1-Copper , 2-Lead superoxide ratio 2:5. The gel product obtained due to this shall be termed as SMOM'-1. Again we took 8 gm lead chloride in 10gm copper chloride to obtain 1-Copper , 2-Lead superoxide ratio 4:5. The gel product so obtained shall be now referred as SMOM'-2.

The precursors lead chloride and copper chlorides were made to dissolve in 20 ml of hydrochloric acid in a beaker kept on a magnetic stirrer for thorough stirring for an hour. This solution was then made to react with ethyl alcohol added in to this solution and was allowed to continue stirring for another four hour at room temperature. The OH molecule of ethyl alcohol, prevent the agglomeration of particles resulting in phase change of the material from solution to gel phase. Then we heated this solution at room temperature so that the remnant solvents evaporates leaving behind green colored gel form of 1-Copper , 2-Lead Superoxide (i.e. mixed metal oxide). For purpose of characterization we dried this gel in an oven at 60° C temperature. We notice that if drying process was performed at temperatures higher than 60° C then the gel properties changes in appearance varying from green to black colour.

3. RESULT AND DICUSSION

The powders obtained as **S-1** and **S-2** were characterized by XRD and FTIR to understand the orderly arrangement and structure within.

3.1 XRD Patterns of the Samples: Figure1shows the XRD pattern of the synthesized Pb-O-Cu superoxide material. All the diffraction peaks are sharp which indicate the Pb-O-Cu samples S-1 and S-2 to be having high degree of crystallinity but a broad peak at $2\theta = 15.8^{o}$ shows formation of some amorphous regions.

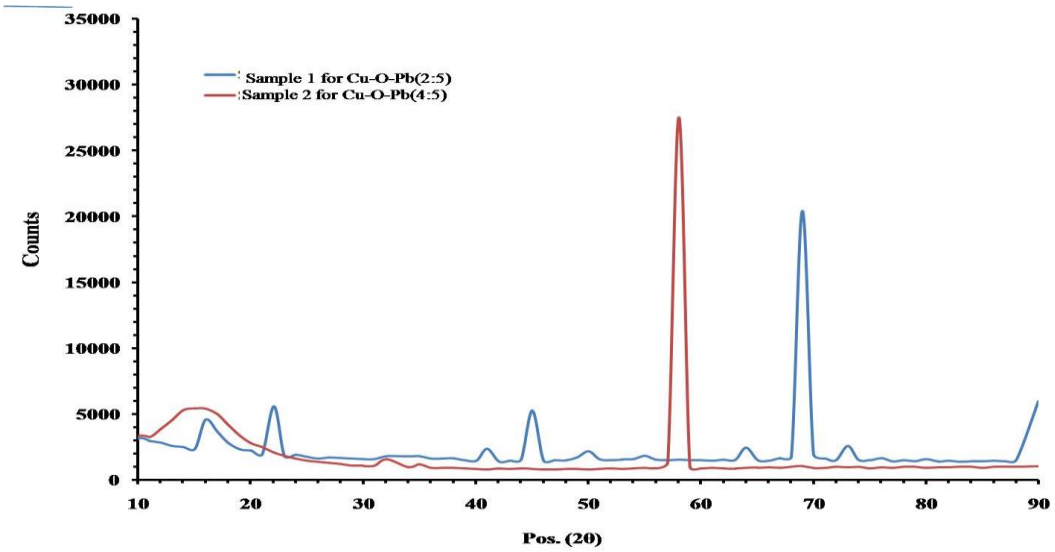

Figure 1: XRD pattern of Pb-O-Cu superoxide for SMOM'-1and SMOM'

The comparison of both the diffraction patterns makes it explicit that on insertion of higher amount of Cu metal into the S-2 results in decrease of intensity but increase in characteristic peak intensity; disappearance and shifting of significant characteristic peaks of Pb clearly visible in S-1. These also strengthen the significant interaction between the both the metals forming superoxide. The characteristic peak observed at $2\theta = 68^{o}$ for sample S-1 shifts to $2\theta = 59.2^{o}$ for sample S-2 indicating significant structural variations.

-2.

3.2 FTIR Pattern for 1-copper,2-lead superoxide metal-oxide-metal :

As XRD patterns revealed a change in structure so to investigate those variations we performed FTIR spectroscopy. FTIR spectrum represents the molecular absorption, reflection and transmission by the sample material, creating a molecular fingerprint of the sample and helps analyze the consistency of the sample.

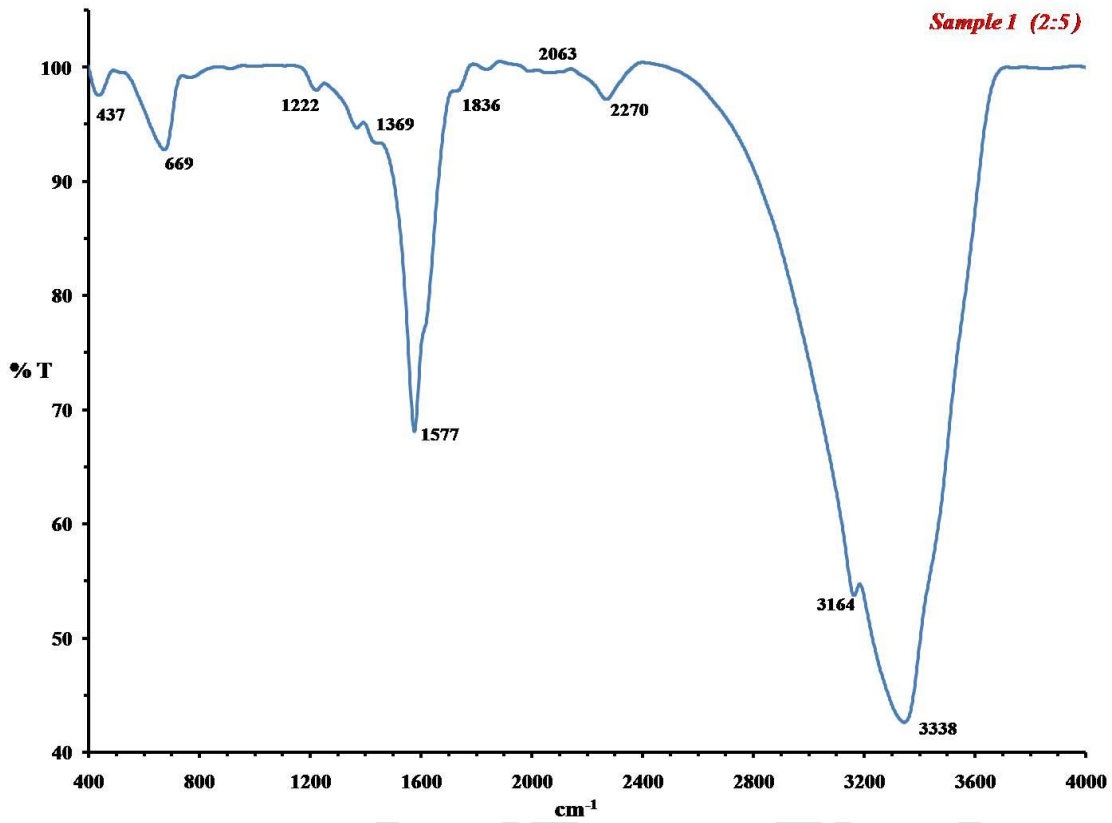

Figure 2: XRD pattern of Pb-O-Cu superoxide for sample S-1

A close inspection of FTIR patterns shown for sample S-1 in Figure 2 and S-2 in figure 3 respectively shows the variations that occur on increasing the lead content in sample 2. One can notice the origin of new peak at 774 cm^{-1}. We also observe the significant shifting of three peaks i.e from 669 cm^{-1} to 674 cm^{-1}, from 1836 cm^{-1} to 1622 cm^{-1} and 2063 cm^{-1} to 1739 cm^{-1}. The broadening of peak at 3338 cm^{-1} clearly reflects the occurrence of interactions within the material.

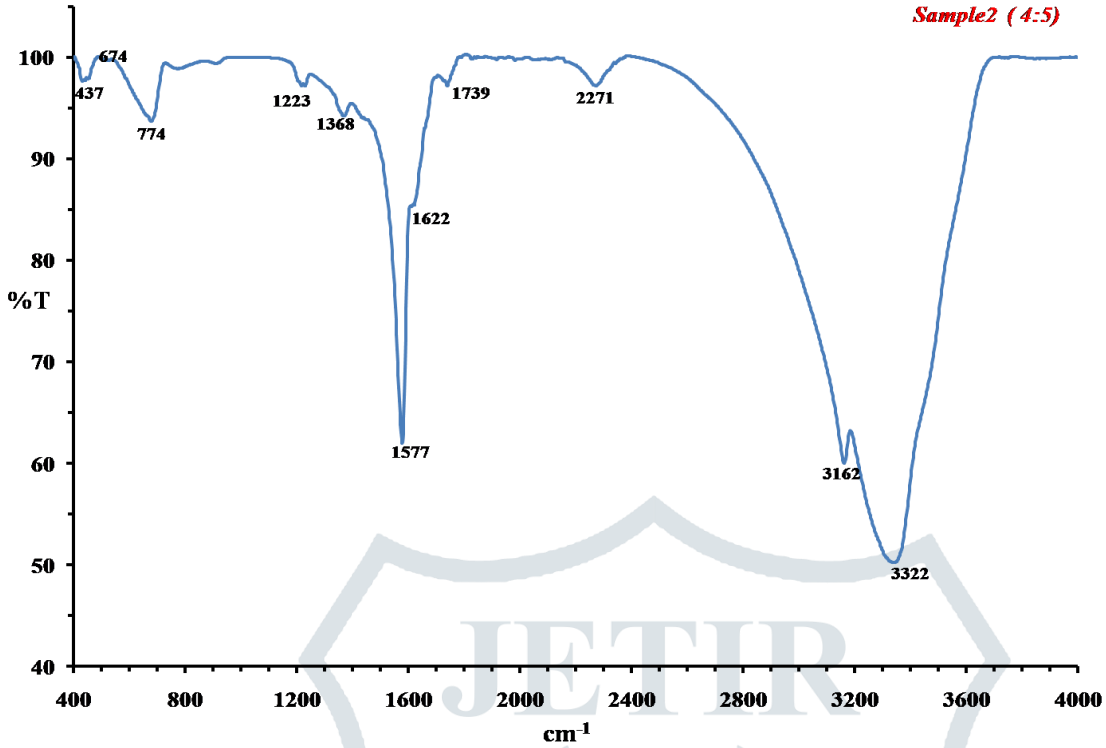

Figure 3: FTIR spectrum for Pb-O-Cu superoxide for sample S-2.

4. CONCLUSIONS :

1-lead,2-copper superoxide material were prepared by sol- gel method using different ratios of precursor metal halides has been presented. The method is quite simple and effective in governing the structural variations. XRD patterns clearly show the difference in crystallinity and FTIR spectra reveal the structural behaviour due to interactions between metal precursors when taken in different ratios.

REFERENCES

1. N.K.Javir, S.R.Bamane Synthesis of nanocrystalline $ZnCeO_2$ by sol-gel method International Journal of Scientific Engineering and Applied Science, 2(3), 424-426, 2016.

2. R. Madhusudhana1, M.A. Sangamesha, R. Gopal Krishne Urs, L. Krishnamurthy, G.L. Shekar Synthesis and Characterization of Zirconia (ZrO2) by simple Sol-Gel Route International Journal of Advanced Research 2(4), 433-436, 2014.

3. Giable George, Nibu PG Non-Alkoxide Sol–Gel Syntheis of Rhombohedral $CEZRO_{3.5}$Nanopowder and its Nanocomposites, International Journal of Multidisciplinary Research Review, 1(9), 255-259, 2015.

4. Nisha Bayal, P Jeevanandan , Synthesis of TiO_2-MgO mixed metal oxide nano particles via a sol gel method and studies of their optical properties, Ceramics International, 40 (10A), 15463-15477, 2014.