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Research Article

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Simple methods of synthesis of copper oxide, zinc oxide, lead oxide and barium oxide nanoparticles

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ABSTRACT

Nano meter sized metal oxide draws much attention because of their unusual physical and chemical properties and extremely large specific area. While most micro structural materials have similar properties to the corresponding bulk materials, the properties of materials with nano meter dimension are significantly different from those of atoms and bulk. This is mainly due to the nano-meter size of the materials which render them (i) large fraction of surface atoms; (ii) high surface energy; (iii) spatial confinement; (iv) reduced imperfections, which do not exist in the corresponding bulk materials. Nano metal oxides have a wide range of application in the field of electronics, fuel cells, batteries, agriculture, food industry medicines etc. Various methods have been reported for the synthesis of metal oxide nano particles in which stabilizers such as polymers and surfactants have been used to prevent the particles from aggregation. The present study aimed at the synthesis of CuO, ZnO, BaO and PbO nano particles using simple chemical precipitation and hydrothermal techniques and their characterization using XRD and SEM.

Keywords: Nano metal oxides, Chemical Precipitation, Hydrothermal, XRD, SEM

INTRODUCTION

Nano particles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures [1]. The interesting and sometimes unexpected properties of nano particles are therefore largely due to the surface area of the material, which dominates the contribution made by small bulk of the material. Metal oxides are crystalline solids that contain a metal cation and oxide anion. They typically react with water to form bases or with acids to form salts [2-3].

Metal-oxides are emerging as technically important materials because of the wide variety of physical properties they possess, which make them attractive for applications such as photovoltaic devices, gas sensors, micro-electronics and corrosion protection devices. However, the production of high quality metal oxide films with a desired chemical composition has been costly and challenging. The synthesis of metal and metal oxide nanoparticles has attracted considerable attention in physical, chemical, biological, medical, optical, mechanical and engineering sciences where novel techniques are being developed to probe and manipulate single atoms and molecules [4-7]. Metal and metal oxide nanoparticles have high surface area and high fraction of atoms which is responsible for their fascinating properties of nanoparticles which depend on size, shape, composition, morphology and crystalline phase [8]. The various metal oxide nanoparticles have wide applications in air and water purification, due to their potential oxidation strength, high photo stability and non-toxicity [9]. Some of the commonly used synthetic methods are non-sputtering, solve-thermal, reduction, sol-gel technique and electrochemical technique. But these methods are costly, toxic, and involve high pressure, high energy requirement, difficult separation and potentially hazardous [10].

EXPERIMENTAL SECTION

Chemical Precipitation Method of Synthesis of Copper Oxide, Zinc Oxide, Lead Oxide and Barium Oxide Nano particles

0.01M Metal nitrate solution (Metal = Copper, Zinc, Lead and Barium) and 0.01M sodium bicarbonate solution are prepared in distilled water separately. The sodium bicarbonate solution is added drop wise under constant stirring to the Metal nitrate solution and the reaction is allowed to proceed for 2hours until addition of sodium bicarbonate solution is complete. After the completion of reaction, the precipitate is allowed to settle overnight. It is then filtered off and the precipitate is washed several times with distilled water, until free from excess bicarbonate which may be present. A blue colour precipitate for Copper and white precipitate for Zinc, Lead and Barium are obtained and the supernatant solution is then discarded carefully. After washing, the paste cake shapes were dried at 80^0C for one hour, and then calculated at 150^0C for 3hours [11].

$M(NO_3)_2 + NaHCO_3$

↓ Drop wise addition for 2 hours

$M(HCO_3)_2$
Metal bicarbonate precipitate
(Allowed to settle over night)

↓

Paste cakes are dried at 80^0C for 1 hr

↓

Calculated at 150^0C
Blue precipitate of CuO and
White precipitate of ZnO, PbO and BaO

2) Hydrothermal Method of Synthesis of Copper Oxide and Zinc Oxide [12]

Copper Oxide and Zinc Oxide are also prepared by Hydrothermal Method in two stages.

Stage 1: Preparation of the Precursor Copper Oxalate or Zinc Oxalate. [13]

Equimolar proportions say, 0.25g of $CuSO_4.5H_2O$ or $ZnSO_4.7H_2O$ and Oxalic acid is dissolved in minimum volume of water and stirred for 15 minutes on a magnetic stirrer.

A blue colour precipitate for Copper Oxalate and white precipitate of Zinc Oxalate are obtained at pH 6. The precipitate is washed with water and finally dried with acetone.

$CuSO_4.5H_2O$ / $ZnSO_4.7H_2O$
+
$H_2C_2O_4$

↓ Added with Stirring for 15 minutes on a magnetic stirrer

Copper Oxalate (Blue) / Zinc Oxalate (White)

Stage 2: Preparation of Copper Oxide and Zinc Oxide Nanoparticles [14]

The prepared metal oxalate precursors are mixed with polyvinyl alcohol in the weight ratio 1:5, powdered in a mortar, mixed in a crucible and ignited in an electric furnace. The temperature should not exceed 300^0C. The CuO and ZnO sample obtained are cooled and the samples are then kept in an air oven at 110^0C, for 4 hours and then cooled to room temperature. The dried samples were separated and powdered.

Copper Oxalate (Blue)/Zinc Oxalate (White)
+
PVA (weight ratio is 1: 5)

↓ Powdered in a mortar, ignited in an electric furnace not more than 300^0C

Blue CuO/White ZnO, PbO and BaO,
(Dried with 1100c for 4 hours)

RESULTS AND DISCUSSION

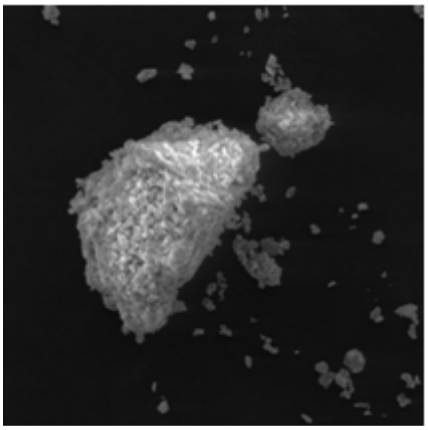


Fig.1 SEM Image of CuO Synthesised by Chemical Precipitation Technique

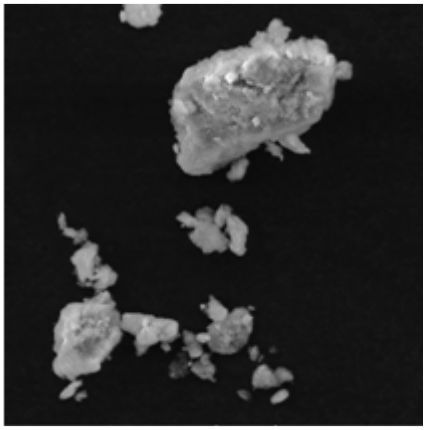


Fig.2 SEM Image of CuO Synthesised by Hydro - Thermal Technique

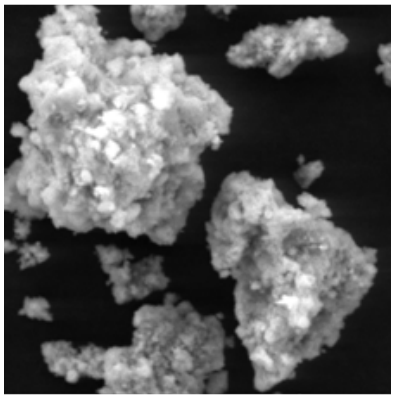


Fig.3 SEM Image of ZnO Synthesised by Chemical Precipitation Technique

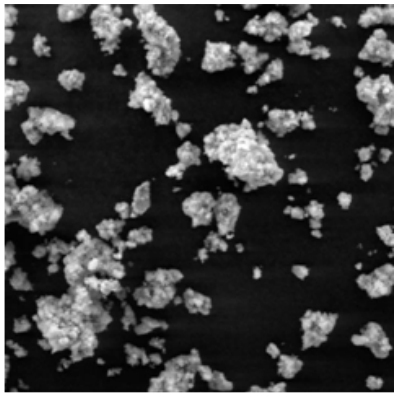


Fig.4 SEM Image of ZnO Synthesised by Hydro - Thermal Technique

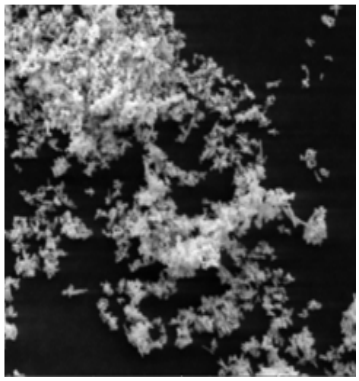


Fig.5 SEM Image of PbO Synthesised by Chemical Precipitation Technique

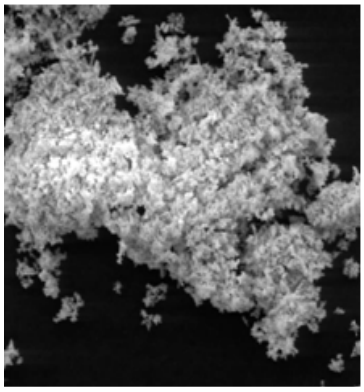


Fig.6 SEM Image of PbO Synthesised by Hydro - Thermal Technique

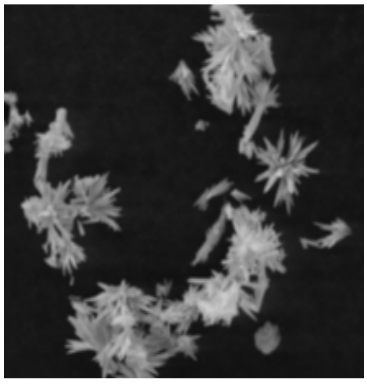


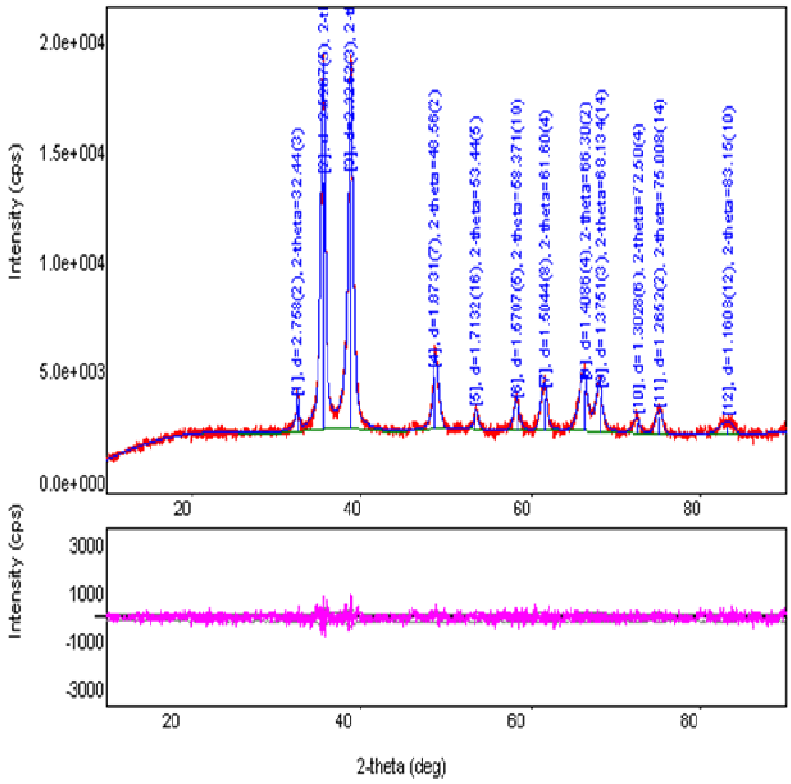
Fig.7 SEM Image of BaO Synthesised by Chemical Precipitation Technique

Fig.8 SEM Image of BaO Synthesised by Hydro - Thermal Technique

Fig.1&2 shows the SEM images of Copper Oxide by chemical precipitation and hydrothermal method, similarly Fig.3&4 Shows SEM images Zinc Oxide of Nanoparticles. The SEM image shows morphology of the synthesised Copper Oxide and Zinc Oxide nanoparticles and is found to be of spherical shaped. Two different sizes of ZnO & CuO Nanoparticles are noted, (1) CuO of 61nm and (2) ZnO of 92nm as seen. Other particles joined as group and form bigger particles. Fig. 5&6 shows the SEM images of Lead Oxide nanoparticles by Chemical–Precipitation method. The SEM image shows morphology of the synthesised Lead Oxide nanoparticles and is found to be grain-sized particles. Fig. 7&8 shows the SEM image of Barium oxide nanoparticles. The SEM image shows morphology of the synthesised Zinc oxide Nanoparticles and is found to be needle shaped structure [15-17].

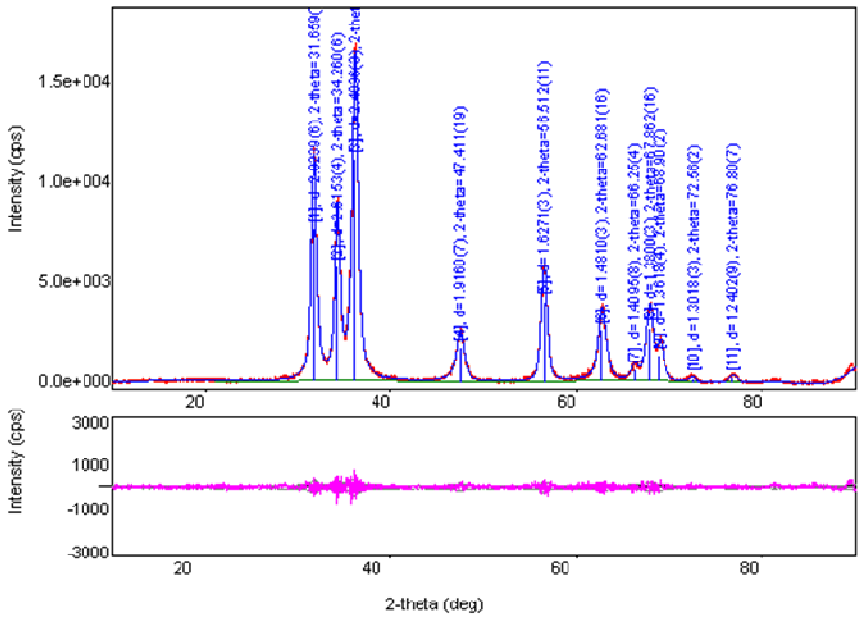
It is confirmed by the XRD data given in the table and it is also confirmed by the calculation using Debye equation [18,19].

$D = 0.94\lambda/\beta\cos\theta$
Where,
D = the particles sizes,
Λ = wavelength of the x-ray,
β = width of the measured peak after correcting instrument with,
θ = XRD peak position



XRD of pattern copper oxide

S. no	2-theta	D (ang.)	FWHM(g)	Int.W(deg)	Size (ang.)	Rel.int.I(a.u)	Rel. height(a.u)
1	66.30(2)	1.4086(4)	1.05(3)	1.36(12)	94(3)	24.23	16.56
2	83.15(10)	1.1608(12)	1.83(9)	2,0(4)	61(3)	7.18	3.38



XRD pattern of Zinc Oxide

S. no	2-theta	D (ang.)	FWHM(g)	Int.W(deg)	Size (ang.)	Rel.int.I(a.u)	Rel. height(a.u)
1	47.411(19)	1.9160(7)	0.0984(19)	1.41(11)	92.1(18)	20.86	15.04

CONCLUSION

Development of synthesis of nanomaterials over a range of sizes, shapes and chemical composition is an important aspect of nano technology. The size-development and physical-chemical properties of nanoparticles have fascinated and inspired research activities. CuO, ZnO, PbO and BaO Were synthesised characterisation which and further studied may be done for studying its applications.

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