International Journal of Applied Engineering Research ISSN 0973-4562 Volume 13, Number 16 (2018) pp. 12513-12515

Synthesis and Characterization of Mixed Oxides of Cerium, Copper & Zinc

[1]Jeyaparatha J. and [2]Clara Jeyageetha J.*

[1]Department of Chemistry, Kamaraj College, Tuticorin, Tamil Nadu, India.
*[*2] Department of Chemistry, A.P.C Mahalaxmi College for Women, Tuticorin, Tamil Nadu, India.*
Manonmaniam Sundaranar University, Tirunelveli, Tamil Nadu, India.
**Corresponding author*

Abstract

Cerium-copper-zinc mixed oxides has been prepared by adopting the sol gel method using ammonium cerium (IV) nitrate, copper sulphate, zinc sulphate and ammonium hydroxide. During the course of preparation the pH was maintained 8 and subjected to magnetic stirring for about 5 hours. The structure was studied by the following characterization methods like scanning electron microscope (SEM) attached with EDAX, Fourier transform infra red spectroscopy (FTIR), ultraviolet visible spectroscopy (UV-VIS), and X- ray diffraction studies (XRD). The UV spectrum showed absorption at 349 nm, 231 nm and a band below 300 nm which was due to the presence of zinc oxide, copper oxide and cerium oxide respectively. SEM images showed the samples consisted of typical agglomerates of homogeneous morphology. The required composition of the mixed oxides was confirmed by EDAX. Hence, Zn, Cu, Ce-oxides were present in the mixed oxides.

Keywords: Mixed oxides, SEM, EDAX, FTIR, UV-VIS, XRD

INTRODUCTION

Metal oxides play a very important role in many areas of chemistry, physics and materials science[1]. The metal elements are able to form a large number of oxide compounds[2]. Most oxides have significant degree of ionic bonding because of the most electronegative divalent oxygen atoms, which results in characteristics of ionic crystals, i.e., optical transparency, high electrical resistivity, low thermal conductivity, diamagnetism and chemical stability [3]. In technological applications, oxides are used in the fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings for the passivation of surfaces against corrosion, and as catalysts [4]. Oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites.

Zinc Oxide : Zinc oxide, ZnO is an inorganic compound also known as zincite and occurs rarely in nature [5]. ZnO is actually a wide band gap semiconductor of the II-VI semiconductor group [6]. This has several favourable properties like high electron mobility, good transparency, wide band gap for semi-conductivity, high room-temperature luminescence, etc.

Cerium Oxide: Cerium oxide has been extensively used in catalytic converters for automobile exhaust systems, as an ultraviolet absorber, and as an electrolyte for fuel cells [7]. Ceria based materials are famous for their redox properties. Cerium oxide has found use in Infrared filters, as an oxidizing species in catalytic converters and as a replacement for Thorium dioxide in incandescent mantles [8].

Copper oxide: Interest in copper oxide nanoparticles arises from the useful properties of this metal such as the good thermal and electrical conductivity at a cost much less than silver. [9]. It also plays an important role in the optoelectronics and solar cell. In this paper, we prepared and characterized the mixed oxide containing Ce, Cu, Zn.

MATERIALS AND METHODS

Mixed oxide containing Ce, Cu, Zn was prepared by adopting the co-precipitation method [10] using ammonium cerium (IV) nitrate, cupric acetate, zinc acetate as their precursors. The ammonium cerium (IV) nitrate solution (0.2 M) was prepared by dissolving 2.741 grams in 25mL of deionised water, cupric acetate solution (0.1M) was prepared by dissolving 0.025 grams in 25 mL of deionised water, zinc acetate (0.1M) was prepared by dissolving 0.4588 grams in 25mL deionised water and finally all the three solutions were mixed together. Ammonium hydroxide was added drop wise to maintain pH 8. Then it was subjected to magnetic stirring for about 5 hours. The resulting slurry was filtered off and thoroughly washed with distilled water in order to remove the anion impurities. The mixed oxide paste was allowed to dry overnight in a hood. It was then oven dried to remove water molecules. Finally, a pale green color mixed oxide was obtained.

RESULTS AND DISCUSSION

UV-VIS Spectroscopy

The UV-VIS spectrum was measured in the wavelength range of 200-400 nm and shown in the Fig. 1. A strong absorption band below 350 nm may be due to the presence of cerium oxide. A strong absorption peak at 349 nm may be due to the excitation of Zinc oxide. The weak absorption band at 231 nm may find copper oxide presence.

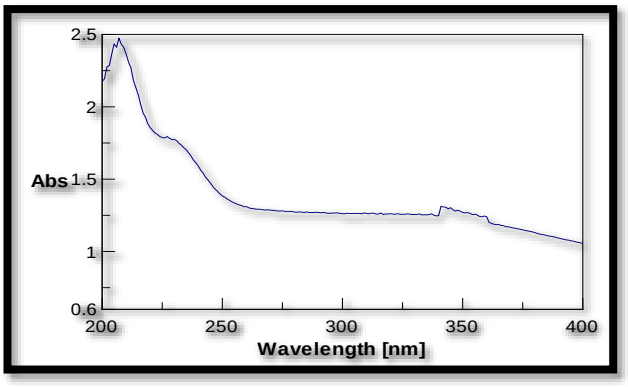

Figure 1. UV-VIS Spectra of mixed oxides

Fourier Transform Infrared Spectrum (FTIR)

Fig. 2 shows several shoulders and absorption bands. The absorption band at 3406.05 cm^{-1} may be due to –OH group. This indicates the presence of residual water. The absorption band at 2977.89 cm^{-1} was due to the stretching mode of –CH group which may be present as impurity during processing. The band at 1670.25 cm^{-1} was due to asymmetric and symmetric vibration of zinc oxide. Further bond between zinc oxide may be confirmed by band at 601.75 $cm^{-1.}$ Small band at 550.8 cm^{-1} was due to Ce-O stretching vibration.

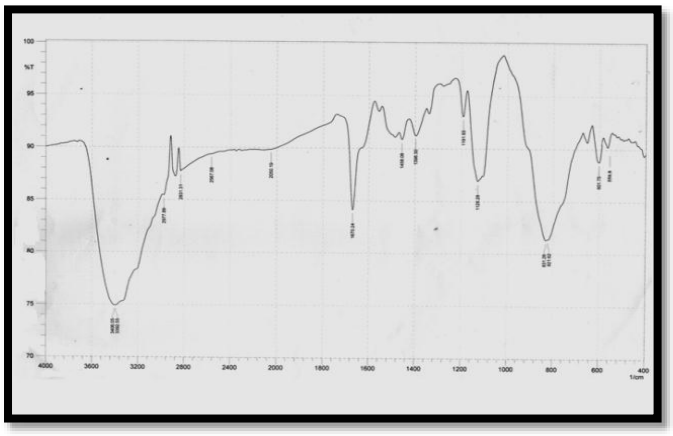

Figure 2. FTIR spectrum of mixed oxides

X-Ray Diffraction studies (XRD)

The synthesized mixed oxide was confirmed by the characteristic peaks observed in XRD images. By using Scherrer's formula i.e., Particle size = 0.94 Xλ/ β cos θ , the particle size was calculated and in the range of 73-87 nm. The diffraction lines observed at 2θ angle 20.076, 22.301, 26.972, 29.822, 35.844, 40.702, 47.312, 51.874, 59.158, 66.553 have been indexed to (100),(100),(110),(110),(111),(200) (210),(211),(220),(310) respectively. As shown in the Table 1 the mixed oxide was in the nanometer range. The average crystal cell parameter was calculated 4.42Å. The planes (100),(110) corresponds to ZnO, the planes (111),(200) corresponds to cerium (JCPDS No: 34-0394) and copper oxide.

Table 1. XRD of mixed Oxides

Position [2 theta]	d-spacing [Å]	Particle size (nm)	Lattice parameter (Å)	plane
20.076	4.41928	73	4.42	100
22.301	3.98327	73	4.42	100
26.972	3.30309	74	4.42	110
29.822	2.99352	74	4.41	110
35.544	2.52365	74	4.41	111
40.702	2.21499	74	4.42	200
47.312	1.91976	80	4.42	210
51.874	1.76115	80	4.42	211
59.158	1.5605	84	4.42	220
66.553	1.40382	87	4.42	310

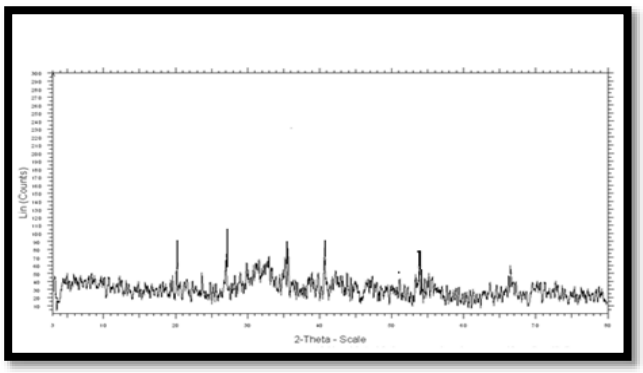

Figure 3. XRD pattern of mixed oxides

Scanning Electron Microscope (SEM)

The SEM measurements were performed to assess the external morphologies of the mixed oxides. As observed from SEM images (Fig. 4) the samples consisted of typical agglomerates of homogeneous morphology . The agglomeration of particles may be due to the high surface energy. Due to this agglomeration there happens to be a discrepancy in the particle size, that was from XRD, the particle size was found to 74-87 nm , whereas, SEM data revealed the particle size was 160-180 nm.

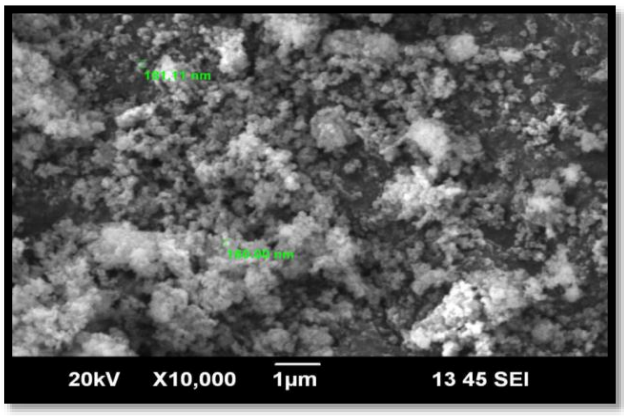

Figure 4. SEM image of metal oxides

EDAX

The EDAX reading (Fig. 5) proved that the required phase of Cu,Ce,Zn Oxide was found in the sample. The graph also shows the presence of Cu, Ce and Zn in the EDAX picture which was probably due to the presence of substrate over which the sample was held during SEM microscopy.

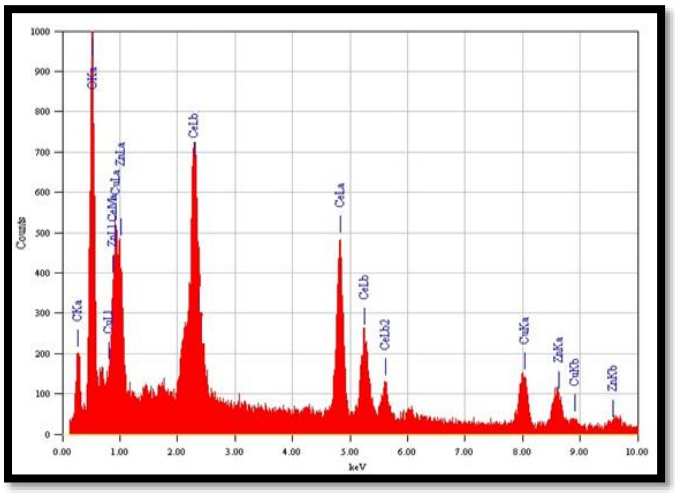

Figure 5. EDAX of mixed oxides

CONCLUSION

The UV spectrum showed absorption at 349 nm, 231 nm and a band below 400 nm which was due to the presence of zinc oxide, copper oxide and cerium oxide respectively. The FTIR spectral results revealed the presence of Zn-O stretching, Ce-O stretching mode. From the XRD pattern the particle size was between 73-87 nm. Mixed oxide synthesized was in the nanometer range. The SEM images showed that the mixed oxide appeared spongy and lies in the range of 160-180 nm. The required composition of the mixed oxides was confirmed by EDAX. Hence, Zn,Cu,Ce-oxides were present in the mixed oxide.

REFERENCES

[1] Noguera, C., 1996, "Physics and Chemistry at Oxide Surfaces," Cambridge University Press: Cambridge, UK.

[2] Wyckoff R.W.G., 1964, Crystal Structures. Wiley: New York, 2nd ed.

[3] Wells, A.F., 1987, Structural Inorganic Chemistry. Oxford University Press: NewYork, 6th ed.

[4] Rodriguez, J.A., Liu, G., Jirsak, T., Hrbek, Chang, Z., Dvorak, J., and Maiti, A., 2002, J. Am. Chem. Soc., 124, 5247.

[5] Ram, S., and Mitra, C., 2001, "Formation of Stable Cu_2O Nanocrystals in a New Orthorhombic Crystal Structure," Mater. Sci. Eng., A 304-306.

[6] Musa, A.O., Akomolafe, T., and Carter, M.J., 1998, "Production of cuprous oxide, a solar cell material by thermal oxidation and a study of its physical and electrical properties," Sol. Energy Mater. Sol. Cells., 51: 305.

[7] Hirata, G.A, Mc Kittrick, J., Cheeks, T., . Siqueiros J.M., Diaz, J.A., Contreras, O., and Lopez O.A., 1996, "High Transmittance Low Resisitivity ZnO: Ga Films by Laser ablation," Thin Solid Films, 288(1-2): 29–31.

[8] Li, J.L, and Inui, T., 1996, "Characterization of precursors of methanol synthesis catalysts, copper/zinc/aluminum oxides, precipitated at different pHs and temperatures," Appl. Catal., 137(1): 105–117.

[9] Juraj Cernack., Jozef Chomic., Charles Kappenstein., Rachid Brahmi., and Daniel Duprez., 1996, "Copper-zinc oxide catalyst. Part II. Preparation, IR characterization and thermal properties of novel bimetallic precursors," Thermochim. Acta., 276: 209–220.

[10] Venkata Krishnan, R., and Nagarajan, K., 2006, "Heat capacity measurements on uranium- cerium mixed oxides by differential scanning calorimetry," Thermochim. Acta, 440 (2): 141–145.