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Microwave-Assisted Synthesis and Characterisation of Nanosized ZnO, CdO and ZnO-CdO Nanocomposite

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ABSTRACT

Science and technology of nanoscale materials will be enriched by adopting new synthetic route for nanomaterials and nanocomposites. Microwave-assisted technique is most energy efficient and less time consuming method for preparation of nanomaterial and nanocomposites. Nano sized single phase zinc oxide, cadmium oxide and its composite is prepared by microwave assisted method. Poly vinyl alcohol (PVA) is used as most efficient fuel for microwave combustion reaction. Metal oxides and its nanocomposites are prepared by microwave irradiation of metal hydroxides with PVA as fuel. As prepared metal oxides and its nanocomposites was well characterized for its structure by employing powder X-ray diffraction (XRD) tool. The morphology of as prepared samples was studied by Scanning Electron Micrograph (SEM) and Transmission Electron Micrograph tool. The presences of the metals are confirmed by EDAX analysis. Fourier Transform infrared (FT-IR) spectral study was undertaken to know the bonding nature of the samples.

1. Introduction

Microwave assisted method of metal oxide nanomaterials is gaining popularity because of its high rate of reaction, efficient heat transfer and environmental friendly nature. This heating shortens the reaction time compared to other usual heating methods. The recent advances reports the synthesis of nanomaterials through microwave-assisted route which enhances the synthetic nanotechnology [1]. Especially metal oxide nanomaterials are prepared with very simple experimentation by chemical route and irradiation with microwaves has been gaining significance in the synthesis of oxide nanomaterials [2-3]. In this process, materials is directly heated by radiation instead of indirect heating by thermal source leading to higher temperature homogeneity in the reaction. PVA is used as an efficient fuel for the combustion process which forms froths and ignite in efficient way. Initially metal salts are converted in to metal hydroxides and is burning with fuel controls the particle size of the materials.

The research on nanosized ZnO and CdO is catching fire fast due to its properties and wide range of applications especially for computing devices, sensor applications, electrical and optical properties [4-5]. Inorganic materials like metal oxides nanomaterials have attracted researchers due to its lower density, high surface area and distinct optical properties [6]. In addition to this these metal oxide nanomaterials found improved performance for applications like catalysis, sensing etc. [7]. The materialists prefer ZnO nanomaterials because of its versatile properties such as wide band gap, piezoelectric characteristics and applications in UV/blue emission devices [8]. nanocrystalline ZnO have superior compared to bulk crystals owing to quantum confinement effects. Similarly nanosized CdO is also one of the semiconducting materials having high band gap and shows low electrical resistivity [9]. This oxide is most preferred due to an ingredient for electroplating baths and pigments [10].

Multiple components of nanocrystalline materials my lead high degree of crystallinity and functionality. Nanocomposite materials like different phased nanomaterials may improve properties and applications [11]. Deepa et al studies the preparation of binary $CdO-Mn_3O_4$ nanocomposites and are well characterised [12].

Present work is an attempt to prepare nanosized ZnO, CdO and ZnO-CdO nano composite materials. These materials are prepared by microwave combustion process using PVA as a fuel. Initially metal hydroxide precursors are prepared and are microwave irradiated with PVA as fuel. As formed ZnO and CdO materials are used for its nanocomposite preparation. Solid state combustion method with PVA is adopted for ZnO-CdO nanocomposite materials. As prepared metal oxides and its nanocomposite materials are well characterised for its confirmation with various characterisation tools.

2. Experimental Methods

2.1 Materials

All chemicals are used in the present experimentation were of AR (Analytical Reagent) grade. Double distilled solvents are used in the present work. Microwave assisted method was used for combustion conversion process. Polyvinyl alcohol is used as a fuel for the combustion reaction and solid state combustion method is adopted for the synthesis of metal oxide nanocomposite materials.

2.2 Preparation of Metal Hydroxides

About 0.1 M zinc chloride and cadmium chloride dissolved in double distilled water and known volume of this salt solution was dissolved in 0.1 M NaOH solution slowly with 2 mL of polyethylene glycol in a separate beakers. Alcoholic media like methanol is maintained. The reaction mixture was stirred continuously on a magnetic stirrer for about two hours. The precipitate is filtered and washed with methanol. As formed metal hydroxide precipitates are dried hot air oven for complete evaporation of methanol. The possible reactions are given below.

$$ZnCl_2 + 2NaOH \rightarrow Zn(OH)_2 + 2NaCl$$
$$CdCl_2 + 2NaOH \rightarrow Cd(OH)_2 + 2NaCl$$

2.3 Preparation of ZnO and CdO Nanomaterials

Zinc hydroxide and cadmium hydroxide is grinded thoroughly with polyvinyl alcohol as a fuel in weight ratio 1:5 in a separate crucible. Initially it was burnt on electric oven for complete evaporation of fumes. Then it is transferred to microwave oven for complete calcination. The sample is calcined on microwave oven having 2.45 GHz frequency and

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power is 800 w for about 30 minutes. A white and brown colour product left behind is ZnO and CdO respectively. The possible reaction involved in the synthesis process is given below.

$Zn(OH)_2 \rightarrow ZnO + H_2O$
$Cd(OH)_2 \rightarrow CdO + H_2O$

2.4 Preparation of ZnO-CdO Nanocomposite

ZnO and CdO are not isomorphous systems, so it may not be possible for us to get lattice mixing sufficiently for the formation of single phased solid. Different phased CdO-ZnO nanocomposite is prepared by this assisted combustion system. Equimolar quantity of as prepared ZnO and CdO nanomaterials are grinded well with PVA thoroughly in a silica crucible. The reaction mixture was ignited on a microwave oven of 2.45 GHz for about ten minutes. A solid crystalline compound left behind is a ZnO-CdO nanocomposite materials (ZnO-CdO).

2.5 Characterizations

The structures of as prepared metal oxides were studied by X-ray diffraction using Phillips X-ray diffractometer (PW3710) with Cu Kα as source of radiation. The morphology was investigated using a field-emission scanning electron microscope (FE-SEM model: FEI-200NNL, Hillsboro, OR, USA), equipped with an energy-dispersive X-ray (EDX) spectrometer for elemental analysis and bonding by Perkin-Elmer 1600 spectrophotometer in KBr medium tools respectively. TEM was scanned by TEM model: JEM-2100 F JEOL, Akishima-shi, Tokyo, Japan.

3. Results and Discussion

3.1 X-Ray Diffraction

Figs. 1a and b show indexed XRD pattern of microwave derived ZnO and CdO samples. The both patterns shows the presence of some Bragg's reflections confirm the formation of crystalline product. Observed d-spacing values and literature d-spacing values are given in Table 1 and 2. The d-spacing values of the ZnO and CdO samples are match well with standard data of JCPDS file 36-1451 and 75-0594. Unit cell parameters ware obtained by least-square refinement of the powder XRD data. This study reveals that the samples are monophasic with cubic spinal structure having nanosized particles. Fig. 2 shows XRD pattern of as prepared ZnO-CdO nanocomposite. The pattern shows the presence of sharp and highly intenced peaks due to crystalline nature. Bragg's reflections of ZnO and CdO are identified as Zn and Cd in the composite pattern confirms the formation of different phased nanocomposite.

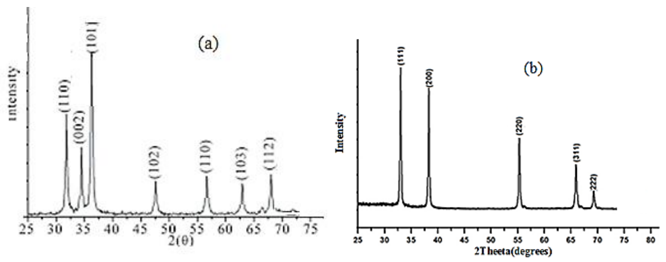

Fig. 1 XRD pattern of a) ZnO and b)CdO samples

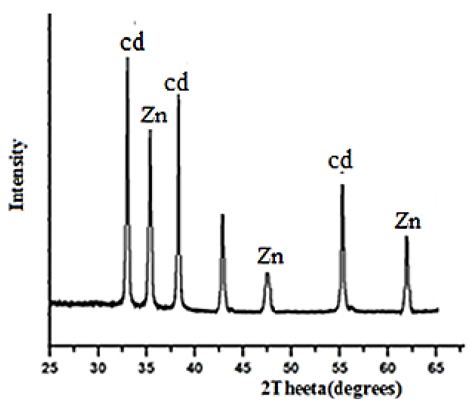

Fig. 2 XRD Pattern of ZnO-CdO nanocomposite

Table 1 XRD data of as prepared ZnO

S.No	d_{obs} (Å)	d_{Lit} (Å)	I_{obs}	(hkl)
1	2.61	2.60	43	002
2	2.47	2.48	100	101
3	1.93	1.91	20	102
4	1.68	1.62	25	110
5	1.52	1.48	18	103
6	1.41	1.38	28	112

Table 2 XRD data of as prepared CdO

S.No	d_{obs} (Å)	d_{Lit} (Å)	I_{obs}	(hkl)
1	2.74	2.70	100	(111)
2	2.33	2.35	80.5	(200)
3	1.59	1.66	58.3	(220)
4	1.46	1.42	48.4	(311)
5	1.38	1.36	21.22	(222)
6	2.74	2.70	100	(111)

3.2 Scanning Electron Microscopy (SEM)

Figs. 3a and b show SEM image of as prepared ZnO and CdO sample respectively. The SEM image of ZnO shows the spherical particles and are self-assembled compact structure. In addition to this some particles are irregular shape with a self-assembled arrangement due to crystalline behavior. SEM image of CdO shows irregular shaped particles out of which some are spherical globular arrangement. Fig. 4 shows SEM image of as prepared ZnO-CdO nanocomposite. The image shows crystalline nature with fine particle agglomeration. During measurement particle may attributed to its small size.

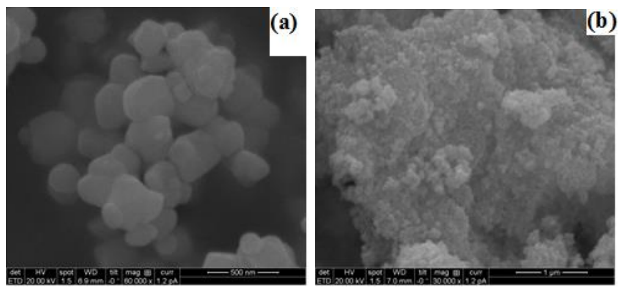

Fig. 3 SEM image of a) ZnO and b)CdO samples

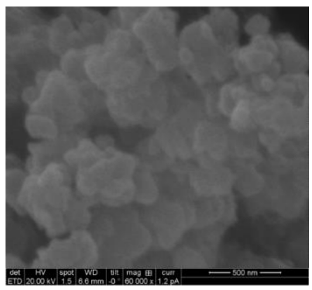

Fig. 4 SEM image of ZnO-CdO nanocomposite

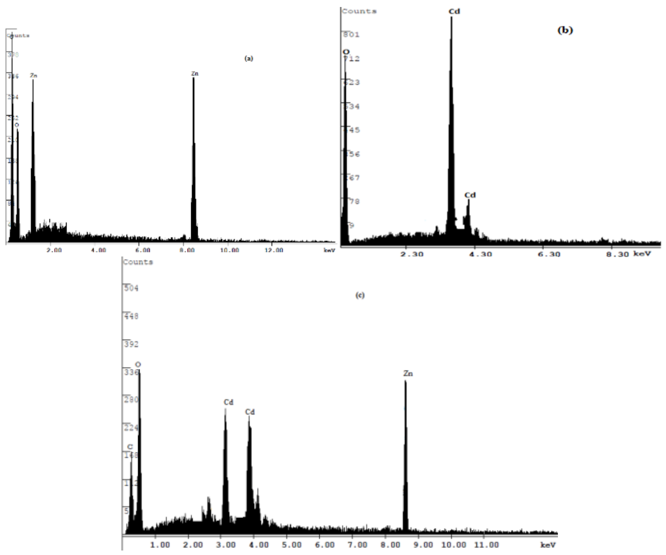

Fig. 5 EDX pattern of a) ZnO b) CdO c) ZnO-CdO sample

3.3 Energy Dispersive X-Ray Analysis Study (EDAX)

The presence elemental metal sample was confirmed by EDAX spectrum and is given Fig. 5a-c. EDX pattern of ZnO it indicates the presence Zn peak at emission energy 1.25 and 8.5 eV whereas CdO sample shows Cd peaks at 3.1 and 4.1 eV. However the EDX of composite shows the presence of both Zn and Cd peaks. The observation of these figures it clearly confirming the formation of metal oxides and its nanocomposite material.

3.4 Transmission Electron Micrograph (TEM)

Fig. 6a-c shows the TEM images of as prepared ZnO, CdO and ZnO-CdO nanocomposite respectively. The TEM image of ZnO shows most of the particles are spherical and are in nano range. Some compact structure is also observed in the image. TEM image of CdO is also indicating the almost particles are spherical in shape and are also in nano range. However the TEM image of ZnO-CdO nanocomposite shows the presence crystalline materials with randon arrangement of particles which are in high nano range.

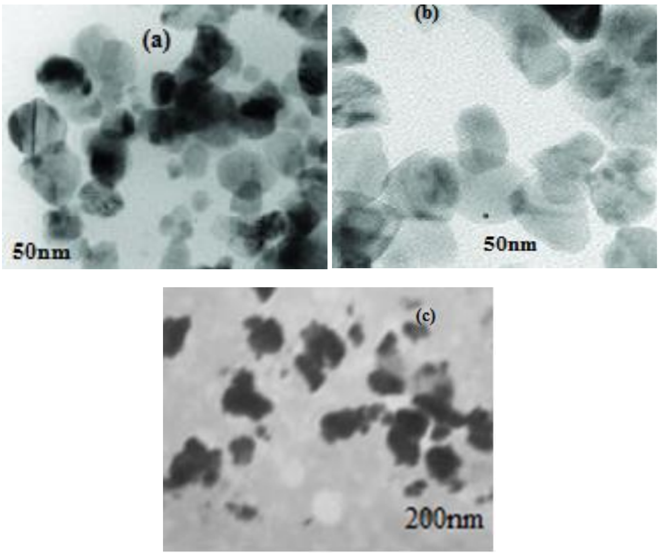

Fig. 6 TEM images of a) ZnO b) CdO c) ZnO-CdO nanocomposite

Table 3 FT-IR data of as prepared ZnO and CdO

S. No.	Frequency (cm^{-1})		
	ZnO	CdO	ZnO-CdO
1	3450	3560	3300
2	1630	1660	2900
3	1130	1130	1600
4	620	1050	995
5	600	650	680
6	580	610	620
7	-	560	605
8	-	-	520

3.5 Infrared Study

Table 3 shows obtained FT-IR data of as prepared ZnO and CdO samples. The metal-oxygen (M-O) bonding and nature of the synthesized samples was carried out by infrared study. Metal oxides generally appear absorption bands below 1000 cm^{-1} arising from inter-atomic vibrations [13]. The peak around 3400-3600 cm^{-1} corresponds to water of absorption due to vibration mode of OH group. Peak at 1700 cm^{-1} may be the presence of carbon dioxide. Vibrational frequency at 1100 cm^{-1} may due to the presence of some overtones. Peaks below 1000 cm^{-1} corresponds to Metal-oxygen (Zn-O, Cd-O) vibrational modes of the sample confirm the formation of ZnO and CdO samples. ZnO-CdO composite sample shows the similar behavior.

4. Conclusion

Metal oxides and its nanocomposites were prepared successfully by microwave-assisted method using polyvinyl alcohol. This method finds its importance because of simple experimentation. Various metal oxides nano materials and nanocomposites may be prepared by this method. As prepared nanomaterials shows the applicable morphology with the formation cubic arrangement, preparation of multi metallic oxides and its composites with polymer can be the future direction of this work.

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References

[1] A. Verma, R. Dwivedi, R. Prasad, K.S. Bartwal, Microwave assisted synthesis of metal oxide nanoparticles, J. Nanopart. 1155 (2013) 1-11.
[2] A. Lagashetty, V. Havanoor, S. Basavaraj, S.D. Balaji, A. Venkataraman, Microwave-assisted route for synthesis of nanosized metal oxides, Sci. Tech. Adv. Mater. 8 (2008) 484-493.
[3] V. Veeraputhiran, V. Gomathinayagam, A. Udhaya, K. Francy, B. Kathrunnisa, Microwave mediated synthesis and characterisation of CdO nanoparticles, J. Adv. Chem. Sci. 1 (2015) 17-19.
[4] D. Durgavijakarthic, M. Kirithika, N. Prithvikumaran, N. Jeyakumaran, Synthesis and characterisation of cadmium oxide nanoparticles for antimicrobial activity, Int. J. Nano. Simens. 5(6) (2014) 557-562.
[5] N.U. Sangari, Synthesis and characterisation of nanosized ZnO using conventional and microwave heating methods, Int. J. Chem. Tech. Res. 7(1) (2015) 181-184.
[6] J.H. Bang, K.S. Suslik, Sonochemical synthesis of nanosized hollow heamatite, J. Am. Chem. Soc. 129 (2007) 2242-2243.
[7] G. Ammar, H. Arnand, N. Jouini, F. Fievet, I. Rosemann, F. Villian, Magnetic properties of ultrafine cobalt ferrite particle synthesised by hydrolysis in a polol medium, J. Mater. Chem. 11 (2001) 186-192.
[8] S. Maensiri, P. Laoku, V. Promarak, Synthesis and optical properties of nanocrystalline ZnO powder by a simple method using acetate dehydrate and poly(vinyl pyrrolidine), J. Cryst. Gro. 289 (2006) 102-106.
[9] M. Herrera, P. Carrin, P. Baca, J. Liebana, A. Castillo, Invitro antibacterial activity of giass Ionomer cements, Microbios. 104 (2001) 141-148.
[10] Y. Matsumura, K. Yoshikata, S. Kunisaki, T. Tsuchido, Mode of bactericidal action of silver zeolite and its comparision with that of silver nitrate, Appl. Envron. Microbiol. 69 (2003) 4278-4281.
[11] Li. Jinghong, J.Z. Zhang, Optical properties and applications of hybrid semiconductor nanomaterials, Coord. Chem. Rev. 253(23-24) (2009) 3015-3041.
[12] G. Deepa, C.K. Mahadevan, A facile method to prepare $CdO-Mn_3O_4$ nanocomposite, IOSR J. Appl. Phy. 5(1) (2013) 15-18.
[13] C.N.R. Rao, Chemical applications of infrared spectoscopy, Academic Press, New York, 1963.