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Led Preparation of Cdsio₃ nanopowder Prepared by Different Silicates

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ABSTRACT

Pure CdSiO₃ nanopowderwas prepared by solution combustion (*SC*) technique at low temperature of at 800 °C for 2 h by using different silicates like meso-porous silica, tetraethyl ortho silicate (*TEOS*), fumed silica (*SiO*₂) and oxyledehydrazide(*ODH*) used as a fuel. The PXRD showed the pure monoclinic phase and the crystallite sizes were in the range of 23 - 43 nm. The SEM picture reveal that the pure CdSiO₃nanopowder were porous and agglomerated with irregular in size. The FTIR analysis is identify the chemical functional groups in the sample. The absorption peaks for pure CdSiO₃nanopowderprepared by different silicates were found in the range of 250 - 266 nm as observed in UV-Visible spectra. The structural defects present in the material band gap (E_g) value of all the prepare samples were in the range of 5.55 - 5.71 eV. ThePhotoluminescence(*PL*) emission spectra of pure CdSiO₃nanopowder prepared by meso-porous silica, TEOS and SiO₂ consist of peaks at 574, 580, and 583 nm respectively when excited at 353 nm. Emission spectra of pure CdSiO₃ nanopowder prepared by meso-porous silica, it is useful for the fabrication of yellow component of WLEDs and display applications.

Keywords: - Solution combustion; Meso-porous silica; Tetraethyl ortho silicate; Fumed silica; Photoluminescence;

1. INTRODUCTION

Phosphors with highly stable, good morphology and better yield are in great demand for energy saving applications like display, lasers, scintillators, safety indicators etc. In this regard, a silicate hosts found to be multi-color phosphorescence and shows inactive with alkali, oxygen and acid environment [1, 2]. The various silicates $CdSiO_3$ host exhibit a remarkable optical and luminescent property. The combined nature of ionic and covalent due to the presence of Cd^{2+} ions and strong interaction between Si-O present in the SiO₃ group. The crystal structure of $CdSiO_3$ shows one dimensional chain of edge-sharing SiO₄ tetrahedron as a result dopants can be easily embedded into the host by replacing the Cd site. In order to maintain the charge neutrality, the charge compensation of Cd^{2+} and O^{2-} were tuned by the dopants. These dopants are responsible for the creation of deep traps at appropriate depths, which stores the excitation energy and emit the light in the visible range [3].Therefore, to improve the properties of the luminescent materials, the exothermic reaction based solution combustion technique was developed. The reported work in this section is the first time synthesis of CdSiO₃ ananoparticles using ODH as a fuel and different silicates like meso-porous silica, tetraethyl orthosilicate (TEOS) and fumed silica (SiO₂) by CS technique. The prepared samples were characterized by Powder X-ray diffractometer (PXRD), scanning electron microscope (SEM), Fourier Infra-red Spectroscopy (FTIR), and UV-Vis spectroscopy. The Photoluminescence (PL) studies were discussed in detail.

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II. PREPARATION OF CdSiO3 NANOPARTICLES USING DIFFERENT SILICATES

The synthesis of pure CdSiO₃ nanoparticles were cadmium nitrate (Cd(NO₃)₂·4H₂O) and freshly prepared meso-porous silica, TEOS and SiO₂was used [4] as the source of Cd and Si respectively. PXRD analysis was performed using Shimadzu X-ray diffractometer with CuK_{α} radiation (λ =1.5405Å) along with a nickel filter. The data was collected in 2 θ ranges from 10-60 degree. Morphology of the sample was analyzed by using Hitachi table top SEM. FTIR spectrum was recorded along with KBr pellets. UV-Vis spectrum of the sample was recorded using Visible Spectrophotometer.PL spectra were carried out using Horiba, USA made model Fluorolog-3, spectrofluorimeter.

III. RESULTS AND DISCUSSION

A. Powder X-ray diffractometer (PXRD)

Fig. 1 shows the PXRD of pure $CdSiO_3$ nanopowder (a) As-formed, calcined samples for 2 h. at (b) 600 (c) 700 (d) 800 (e) 900 0 C prepared by (A) meso-porous silicate (B) TEOS and (C) SiO₂. Thepeaks of pure CdSiO₃were well matched with the JCPDS card No. 35-0810 confirms the formation of CdSiO₃. Among all the prepared silica, the sample calcined at 800 $^{\circ}$ C for 2 h shows better crystallanity and monoclinic phase. The average particle size 'D' was calculated by employing the Debye-Scherrer's formula and was found to be ~ 23 - 43 nm for the samples calcined at 800 $^{\circ}$ C for 2 h. Further, strain [5] present in the CdSiO₃ nanoparticles synthesisedwas estimated using the W-H equation and shown in Fig.2. It is known that the FWHM can be expressed as a linear combination of the contribution from the lattice strain and crystalline size. The particle size was calculated from Scherrer's method and Williamson–Hall plots ~ 23-32 nm were in good agreement also shows nano in size. Also, observed that the crystallite size is 23 nm least in TEOS as a silica source.

B. Morphological analysis

SEM pictures of pure $CdSiO_3$ nanoparticles prepared by different silicates shown in Fig.3 reveal the morphologies of $CdSiO_3$ nanoparticles are highly porous and agglomerated with poly crystalline nature. The pores and voids can be produced due to the large amount of gases escaping out of the reaction during combustion [7].

C. FT-IR spectral analysis

Fig. 4 showsFTIR spectra were recorded in the range 4000-400 cm⁻¹ using Perkin Elmer Spectrum 65 with KBr pellets. The spectra showed the band in the range 875–1114 cm⁻¹ was owing to unequal stretching vibration of Si–O–Si bond and stretching vibrations of edge Si–O bonds. The peak at 468 cm⁻¹ was the characteristic stretching vibrations of Si–O–Si bridges. A weak absorption peak at 2397 cm⁻¹ specifies the existence of C=O bond, may be due to adsorbed CO₂ in the sample [8]. The sharp peak to ~ 680 cm⁻¹ can be ascribed to Si–O bond, which exists in the SiO₃form. The absorption at 3490 cm⁻¹ indicates the presence of hydroxyl groups.

D. Optical Absorption Studies

The inset picture Fig. 5 shows the pure CdSiO₃nanoparticlesprepared by different silicates calcined at 800 0 C for 2 h UV-Vis absorption spectrum in the range of 200-2100 nm. The optical E_g (Fig.5) of pure CdSiO₃nanoparticles prepared by different silicates calcined at 800 0 C for 2 h was estimated by Wood and Tauc relation and E_g values.(Inset Fig.5) [9].

F.Photoluminescence (PL) Studies

Pure $CdSiO_3$ nanoparticles were best hosts for PL and radiate luminescence according to luminous theory of rare earth atoms [10, 11]. The excitation spectra of pure $CdSiO_3$ nanoparticles prepared by different silicates the broad band at 353 nm and weak band appeared at 343 nm. The emission spectra of pure $CdSiO_3$ nanoparticles prepared by meso-porous silica, TEOS and SiO_2 consist of peaks at 574, 580, and 583 nm respectively shown in Fig. 6. The yellow emission was frequently attained in the emission spectrum.

Pure CdSiO₃ nanoparticles emission at 480 and 583 nm was of magnetic dipole and 574 nm an electric dipole. The 575 nm was less intensity compared to others. Other two emission peaks were observed at 580 and 583 nm respectively, which were mainly attributed to ${}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2}$ and ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ respectively. Wang et al studied PL of Dy₂O₃nanorods prepared by hydrothermal method at an excitation wavelength of 300 nm. Twin emission peaks recorded at 490 and 575 nm. The 490 and 575 nm peaks were attributed to electron transition from ${}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2}$ and ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ respectively. Further, PL emission was recorded for bulk CdSiO₃nanopowder and observed 490 nm peaks along with less intense peak at 575 nm.

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A large variation in PL intensity of pure $CdSiO_3$ nanoparticles prepared by TEOS to other silica precursor and was observed due to higher activity in nanoparticles. Further, we concluded that TEOS were suitable for the preparation of $CdSiO_3$ nanoparticles. CIE chromaticity coordinates (x, y) for prepared products excited at 353 nm. The co-ordinate values were given in table inset of the Fig.7. The CCT values were well located in the yellow region shown in the star mark (Fig. 7).

CCT can be caluculated by Planckian locus. This defines the color temperature of a light source. Calculated values of CCT by using transforming equations 1 and 2 shows the temperature of the closest point of the Planckian locus to the light source on the (U', V') uniform chromaticity diagram (Fig. 7). Hence, this shows that pure CdSiO₃ nanopowder prepared by TEOS produced more intensity at 580 nmwere potential materials for the fabrication of WLEDs and solid state display applications.

IV. Conclusion

Pure $CdSiO_3$ nanopowderwas successfully synthesized by self-sustainable propellant wet chemical technique using different silicate precursor.PXRD analysis confirms the monoclinic phase was formed at low calcined temperature of 800 °C for 2 h compared to high temperature conventional methods. SEM picture show that the powder was highly porous, foamy and agglomeration. From the UV-Vis spectra the average energy band gap of 5.6 eV was calculated. PL emission spectrum shows strong high intensity at 580 nm excited at 353 nm. It will be used for yellow component of WLEDs andsolid state display applications.



Fig.1. PXRD of pure CdSiO₃nanopowders (a) As Formed, Calcined for 2 h at (b) 600 ⁰C (c) 700 ⁰C (d) 800 ⁰C (e) 900 ⁰C prepared by (A) meso-porous silicate (B) TEOS and (C) SiO₂



Fig.3. SEM micrographs of pure CdSiO₃nanopowders(a) as formed, calcined at (b) 600 ⁰C (c) 700 ⁰C (d) 800 ⁰C and 900 ⁰C for 2 h prepared by (A) meso-porous silicate (B)

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Fig.6. Emission spectrum of pure CdSiO₃nanopowderscalcined at 800 C for 2 h prepare by different silicates(Inset: Figure shows excitation spectrum of pure CdSiO₃ nanopowders prepare by different silicates)



Fig.7.(a) CIE chromaticity coordinates of pure CdSiO₃nanopowderscalcined at 800 ⁰C for 2 h prepare by different silicates. (Inset: table shows x, y values) (b) CCT diagrams of pure CdSiO₃nanopowderscalcined at 800 ⁰C for 2 h prepare by different silicates (Inset: table shows CCT values)

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