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Research Paper

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Structural Characterization and Luminescence Properties of $Ca_2MgSi_2O_7$ (CMS) Phosphor

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Abstract— $Ca_2MgSi_2O_7$ (CMS) phosphor was successfully synthesized via traditional solid-state reaction technique. In order to find out the phase purity and crystal structure, characterization of the prepared powder samples was done by X-ray diffraction (XRD) technique. The results of the XRD studies obtained for this phosphor revealed its tetragonal, akermanite structure with a space group $P\bar{4}2_1m$. It is observed that the XRD pattern matched well with JCPDS file No. 77-1149. The average crystallite size (D) is calculated as 26.33nm and lattice strain as 0.29. SEM images reveal that the surface morphology of the particles was not uniform and they aggregated tightly with each other. The actual formation and identification of the functional group of phosphor was confirmed by Fourier transform infrared spectroscopy. The host CMS phosphor shows very poor PL emission intensity obtained in blue region at 462 nm wavelength and CIE chromaticity diagram displays the purple-blue light color emission of this phosphor. In this research paper, the XRD, SEM, FTIR and Photoluminescence (PL) properties as well as CIE chromaticity diagram of this phosphor are also reported.

Keywords— Photoluminescence (PL), CIE Chromaticity Diagram, XRD, FTIR, $Ca_2MgSi_2O_7$, Akermanite, Solid-State Reaction.

I. INTRODUCTION

Silicate phosphors have broadly revolutionized research in the remarkable area of material science. Especially, in the area of LEDs and lightening markets. Mellite are a large group of compounds characterized through the general structure formula $M_2T^1T^2_2O_7$, [where M= Barium (Ba), Strontium (Sr), Calcium (Ca); T^1= Magnesium (Mg), Zinc (Zn), Copper (Cu), Manganese (Mn), Cobalt (Co); T^2 = Germanium (Ge), Silicon (Si)], have been extensively investigated in the form of optical materials [1]. When compared with Alkaline earth silicates, sulfide phosphorescent phosphors and strontium aluminate phosphors. So, it was considered as appropriate hosts with high chemical stability and water-resistant property also discussed [2, 3]. Calcium magnesium silicate (CMS) has been broadly explored in the form of a host for long-lasting phosphor and plasma display panels [4]. Recently, in some literatures, long lasting alkaline earth silicate phosphors prepared via high temperature traditional solid reaction technique were reported [5, 6]. Akermanite, it belongs to the family of soro-silicates. The di calcium magnesium di silicate is also called as a host for long-lasting phosphors, generally activated with various RE ions, such as (Eu^{2+}, Nd^{3+}, Dy^{3+} or Mn^{2+}). This CMS phosphor has been extensively discussed in biological and medical areas of applications [7]. Silicate with akermanite structure may be a possible and attractive bio ceramics for tissue engineering applications [8]. Akermanite (CMS) has superior biocompatibility and bioactivity properties and is also considered as a probable bone material [9]. In addition, silicates have been broadly investigated because of their high thermal, excellent water resistance, chemical stabilities, cheaper and strong absorption in the near ultra-violet [UV] region [10]. In this study, $Ca_2MgSi_2O_7$ (CMS) phosphor was prepared via conventional solid-state reaction method. The structural characterization such as XRD, SEM, FTIR and optical properties like Photoluminescence spectra as well as CIE chromaticity diagram were discussed in detail.

II. EXPERIMENTAL PROCEDURE

2.1 MATERIAL PREPARATION

The phosphor was synthesized via traditional solid-state reaction method. All the starting materials employed in the experiment are of analytical grade. Initially, all raw materials $CaCO_3$ (99.99%), MgO (99.99%), SiO_2*H_2O (99.99%), and H_3BO_3 (99.99%) of Hi-media were purchased and weighed after calculation. Very little amount of H_3BO_3 (Boric acid) was added as a flux. It was mandatory that the all raw materials were mixed homogeneously by using acetone (CH_3COCH_3) and grinded thoroughly for 2 hours using the agate mortar and pestle in clock wise direction. The grinded sample was transferred in an alumina crucible. Sample was kept in

programmable muffle furnace and after subsequently fired at 1100°C for 3 hours. The heating as well as the cooling rate of the furnace were set at 5°C per minute. Final $Ca_2MgSi_2O_7$ phosphor (white powder) was obtained after additional grinding up to 1 hour. The resulting sample was restored in airtight bottle for characterization studies.

The chemical reaction process is given as follows:

$$8CaCO_3 + 4MgO + 8SiO_2{*}H_2O \rightarrow 4Ca_2MgSi_2O_7 + 8CO_2 (\uparrow) + 8H_2O (\uparrow) + 3O_2 (\uparrow) \quad (1)$$

Fig: 1 $Ca_2MgSi_2O_7$ Phosphor

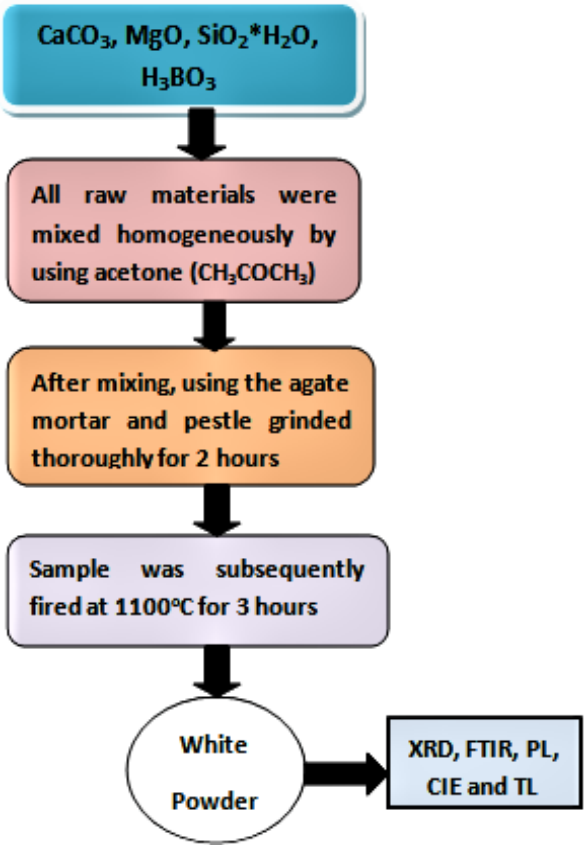

Fig: 2 Synthesization Process of phosphor

2.2 Material Characterization

XRD of the crystalline structure, size and phase composition of the synthesized phosphor were noted with the help of Bruker D8 advance X-ray diffractometer with Cu-K_α radiation having wavelength (λ = 1.5406 Å), at 40 kV, and 40 mA voltage and current values, respectively. Actual formation of this phosphor was obtained through FTIR. An FTIR spectrum was recorded with the help of Bruker Alpha Fourier transform infrared spectroscopy. For investigating the functional groups (4000 to 1400 cm-1) as well as the finger print area (1400 to 400 cm-1) of synthesized phosphor through mixing the sample with potassium bromide (KBr AR grade) with pallet preparation. In photoluminescence spectra (PL), emission spectra were recorded by a spectro-fluorophotometer (SHIMADZU, RF-5301 PC) using a xenon lamp of power 150 watt as excitation source. All experiments were performed in identical conditions and it was observed that the results were reproducible. All measurements carried out at the room temperature.

III. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction (XRD)

XRD patterns of $Ca_2MgSi_2O_7$ (CMS) phosphor as a host was synthesized via conventional solid-state reaction technique are shown in Fig. 3. It is recorded in the range (10^0\2θ\$80^0$). The position and intensity of diffraction peaks of this phosphor is well matched with the standard (Joint Committee on Powder Diffraction Standard) (JCPDS) No. 77-1149 [11]. The phase formation of the prepared phosphor was also confirmed by XRD characterization. The standard $Ca_2MgSi_2O_7$ structure cell volume and lattice parameters are observed from data base code AMCSD 0008032 [12]. All parameters of this phosphor are shown in Table no. 1. The prepared phosphor show akermanite-type structure which comes under the tetragonal type of crystallography with space group $P\bar{4}2_1m$.

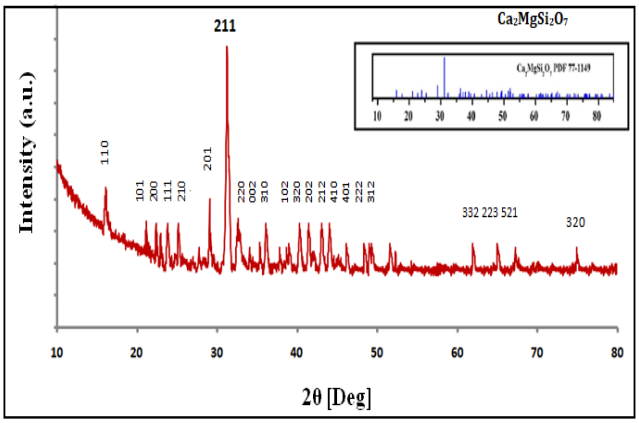

Fig: 3 XRD Pattern of $Ca_2MgSi_2O_7$ Phosphor

3.1.1 Debye–Scherrer Formula

For prominent peak (211), using Debye–Scherrer formula, the average crystallite size (D) of the $Ca_2MgSi_2O_7$ phosphor is calculated as (26.33) nm. Debye–Scherrer formula is represented as follows:

$$D = K\lambda/\beta Cos\theta \quad (2)$$

Where K is the Scherrer constant having value 0.94, λ is wavelength of incident X-ray (λ = 1.5406 Å), β =FWHM (Full Width Half Maximum) of the peaks and θ = corresponding Braggs diffraction angle.

3.1.2 Strain Determination By Uniform Deformation Model (UDM)

The strain induced broadening in the powder material was calculated via the following formula given as below

$$\varepsilon = \beta/4\tan\theta \quad (3)$$

Table: (1) According to prominent peak (211), position of the peak of the XRD patterns and the calculated value of following parameters.

No.	Parameters		
1.	Crystal Structure		Tetragonal
2.	Space Group		$P\bar{4}2_1m$
3.	Lattice Parameters	a=b=7.8071	α=β=γ=90°
		c=4.9821	
4.	Crystallite Size D (nm)		26.33
5.	2θ [Deg]		31.24
6.	Cell Volume		303.663 (Å)^3
7.	Crystal Plane Spacing d (Å)		2.8630 (Å)
8.	Strain		0.29

3.2 Scanning Electron Microscopy (SEM)

It is known that the luminescence characteristics of phosphor particles depend on the morphology of the particles, such as size, shape, size distribution, defects, and so on. The surface morphology of the $Ca_2MgSi_2O_7$ Phosphor is shown in fig. 4. at 10µm magnification. The surface morphology of the particles was not uniform and they aggregated tightly with each other. From the SEM image, it can be observed that the prepared sample consists of particles with different size distribution. In addition, there are some big aggregates is also present due to high temperature heat treatment. The surface morphology concludes that $Ca_2MgSi_2O_7$ phosphor is more amorphous.

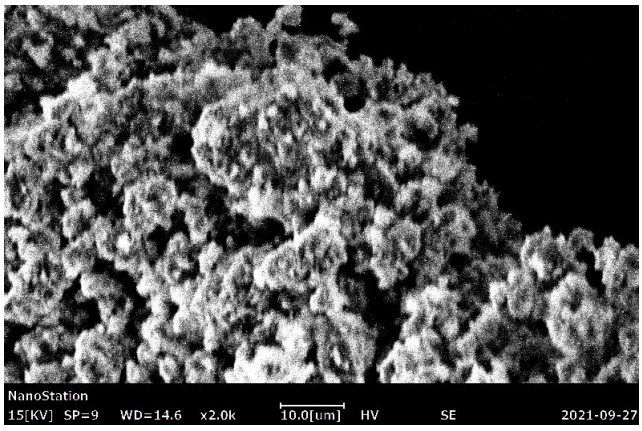

Fig: 4 SEM Images

3.3 FTIR SPECTRA

3.3.1 KBr Pallet Preparation

The KBr pallet is displayed in fig. 5(a) and 5(b). Before recording the FTIR spectra of a synthesized sample, it is very essential to mix the synthesized sample with KBr (IR Grade) powder and grind it. After applying with hydraulic pressure to form a thin pallet. It is important to be note that KBr powder and sample should be in very little quantities. In this way, FTIR spectra and reading are obtained very clearly.

Fig: 5(a) Hydraulic Pressure Equipment
Fig: 5(b) KBr-CMS Pallet

3.3.2 Functional Group Investigation

The FTIR (Fourier Transform Infra-Red Spectroscopy) spectrum of this CMS phosphor has been shown in fig: 5(c). FTIR spectra were recorded in the range of (4000 cm^{-1} to 400 cm^{-1}). An intense band centered at 975.19 cm^{-1} and 945.35 cm^{-1} are allocated as a result of (Si-O-Si) asymmetric stretch, the band at 848.65 cm^{-1} is allocated to the (Si-O) symmetric stretch. Bands at 587.73 cm^{-1}, 483.54 cm^{-1} are allotted to the (Si-O-Si) vibrational mode. Furthermore, the absorption bands at 686.81 cm^{-1}, 645.28 cm^{-1} and 1360.68 cm^{-1} can be allocated to the existence of (SiO_4) group. The band centered at 1860.62 cm^{-1} can be ascribed to the existence of small quantity of the calcite [2]. The asymmetry stretching of carbonate (CO_3^{2-}) can be recorded in the range of 1900-1700 cm^{-1} [13] and its spectrum band at 1860.62 cm^{-1} are allotted to the (CO_3^{2-}) and band at 1639.46 cm^{-1} is as a result of to the vibration in Mg^{2+} ions. Likewise bending of sharp peaks situated in the region of 848.65 cm^{-1}, 723.58 cm^{-1} are allocated to the vibration in the Ca^{2+} ions [14]. The band centered at 3428.40 cm^{-1} which displays stretching vibration of (O-H) group. The band at 483.54 cm^{-1} is allocated to the (Mg-O).

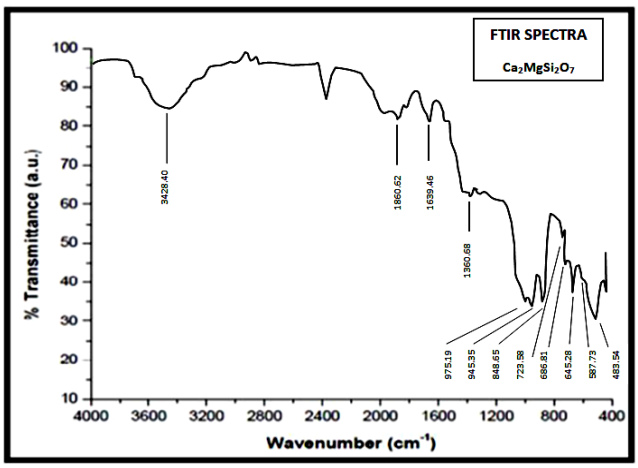

Fig: 5(c) FTIR Spectra of $Ca_2MgSi_2O_7$ (CMS) Phosphor

3.4 PHOTOLUMINESCENCE (PL) SPECTRA

Fig. 6(a) & 6(b) displayed the Emission spectra of host $Ca_2MgSi_2O_7$ phosphor. It is very clear that the excitation spectra in 337nm wavelength corresponding Emission spectra have been obtained in blue region at 462nm wavelength with very poor PL intensity. Another excitation peak situated in 357nm wavelength. Therefore, we have suggested that any phosphor as a host not displayed higher intensity in any wavelength, without any doping rare earth material.

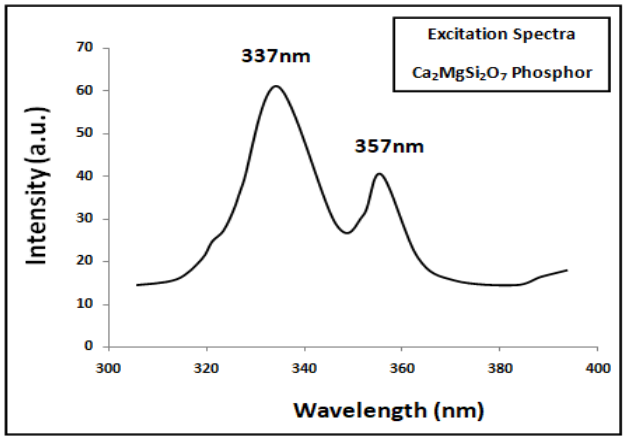

Fig: 6(a) Excitation Spectra of $Ca_2MgSi_2O_7$ Phosphor

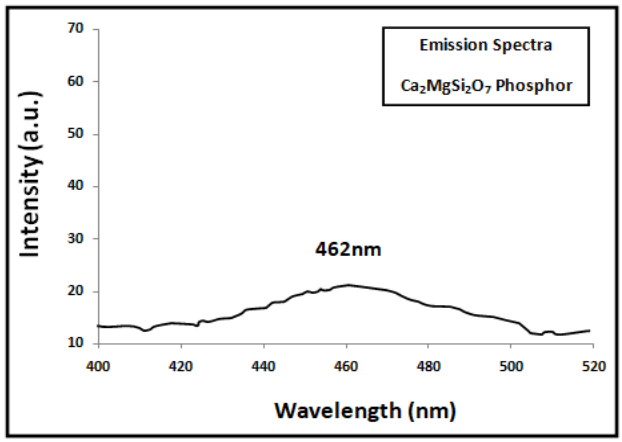

Fig: 6(b) Emission Spectra of $Ca_2MgSi_2O_7$ Phosphor

If we want to achieve higher intensity, then it will be mandatory to do rare earths doping with a host material with different concentrations. Due to which the PL intensity at various concentrations will be obtained in increasing order and at a certain concentration of rare earth, the PL intensity will be high.

3.4 CIE Chromaticity Diagram

CIE chromaticity diagram of this phosphor has been displayed in Fig. 7. In general case, color of any phosphor material is demonstrated through means of color coordinates. Color coordinates are one of the most significant factors for evaluating phosphors performance. The luminescence color of the samples excited under 462 nm has been characterized by the CIE chromaticity diagram. Luminescence colors of $Ca_2MgSi_2O_7$ phosphor were placed in the blue region (x = 0.2443, y = 0.2678), corners. The chromatic co-ordinates of the luminescence of this phosphor are measure and reached to purple- blue light color region [15].

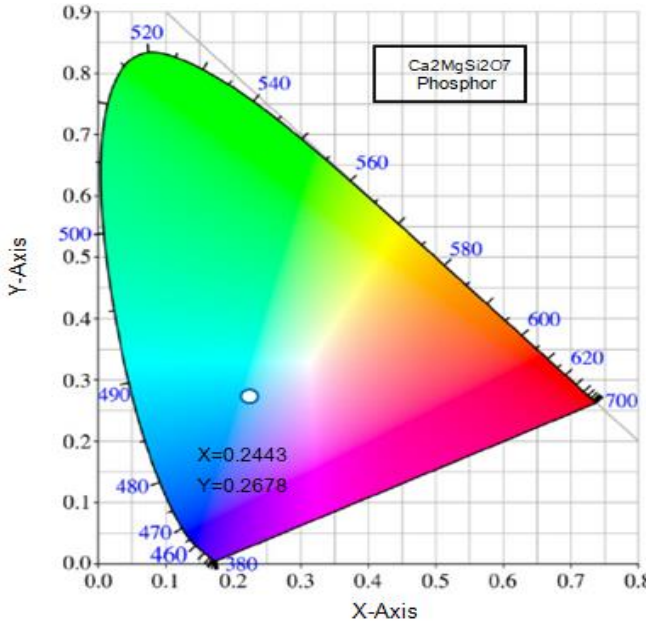

Fig: 7 CIE Chromaticity Diagram

IV. CONCLUSION

In brief, $Ca_2MgSi_2O_7$ phosphor was successfully synthesized via traditional high temperature solid-state reaction synthesis technique. The XRD spectra revealed that the standard pattern of the obtained phosphor was well matched through JCPDS file and lattice parameters were visualized with the data base code AMCSD 0008032. The grain size and homogeneity of the phosphor is much better in the micro range. The SEM images reveal that the surface morphology of the particles was not uniform and they aggregated tightly with each other. Actual formation of the phase structure and functional group identification of the CMS phosphor was determined via FTIR spectroscopy. The host CMS phosphor displays very poor PL emission intensity which obtained in blue region at 462 nm wavelength and CIE chromaticity diagram shows the purple- blue light color emission of this phosphor.

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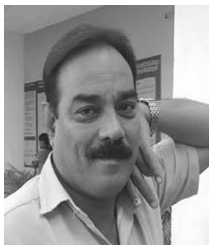

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