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New Investigation of Photoluminescence Properties of Red Dominated White Emitting $\text{YGd}_2\text{B}_2\text{O}_7:\text{Eu}^{3+}$ Phosphor

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Abstract: $\text{YGd}_2\text{B}_2\text{O}_7$ phosphors were synthesized using standard solid state reaction [SSR] method varying Eu molar concentration as 0.1, 1.0, 1.5 and 2.0 %. The mixture of reagents was ground together to obtain a homogeneous powder in acetone base. To prepare Yttrium Gadolinium Borate (YGB) doped with various concentrations of Eu, consists of heating stoichiometric amounts of reactants at 1000°C for 2 h in a muffle furnace. The received powder being ground thoroughly using an agate mortar, to ensure the best homogeneity and reactivity, powder was transferred to alumina crucible, and then reheated in a muffle furnace at 1200°C for 4 hours. The phosphor materials were cooled to room temperature naturally. All samples were found out to be white who are studied for photoluminescence [PL]. Photoluminescence spectra were recorded at room temperature using Shimadzu-5301 Spectrofluorophotometer. The prepared phosphors were characterized by using techniques such as powder X-ray diffraction (XRD), Photoluminescence (PL), Scanning electron microscopy (SEM), Particle size analysis and Commission International de l'Eclairage (CIE). The effect of doping of Eu in YGB on the PL emission/excitation was also studied. Eu shows all its primary allowed emissions hence it is concluded that this phosphor can be used in display devices.

Keywords: Photoluminescence, X-ray diffraction [XRD], Scanning Electron Microscopy [SEM], Solid State Reaction.

I. INTRODUCTION

Figures 1-2 are the emission & excitation of $\text{YGd}_2\text{B}_2\text{O}_7:\text{Eu}$ phosphor under different excitation wavelengths. From figure the excitation is a broad band from 240-320nm, absorption peaks at 254, 269 & 275 nm. As the Eu concentration increases in $\text{YGd}_2\text{B}_2\text{O}_7$ the excitation intensities gradually reduces and 275nm absorption dominates. As the excitation wavelength increases the emission intensities varies from low to high energy. All the four phosphor samples are excited with 254, 269 and 275nm. The excitation spectra are monitored at 594nm. The 254nm excitation is considered due to application potential of this phosphor in devices CFL, Fluorescent Lamps etc. When excited with 254, 269 & 275 nm the emission peaks are dissolved at 584, 594, 613, 618 and 624nm. These phosphors find applications in fluorescent lamps, color TV screen, cathode ray tubes, Plasma Display Panels, discharge lamps etc. These phosphors are not commercially synthesized in India, and are imported. However, phosphors with delayed emission are also extremely useful as sources of light energy (lamps) at the time of sudden power failure or intentional blackouts. These have great potential for luminous paints, watches, display industries, exit signage and emergency escape route guidance systems. The interest in these phosphors has grown due to increasing number of applications in radiation detectors, sensors for structural damage and temperature detection. Considering its innumerable applications, the present work was concentrated on the synthesis, characterization and studies on luminescence properties of multi colour emitting phosphors.

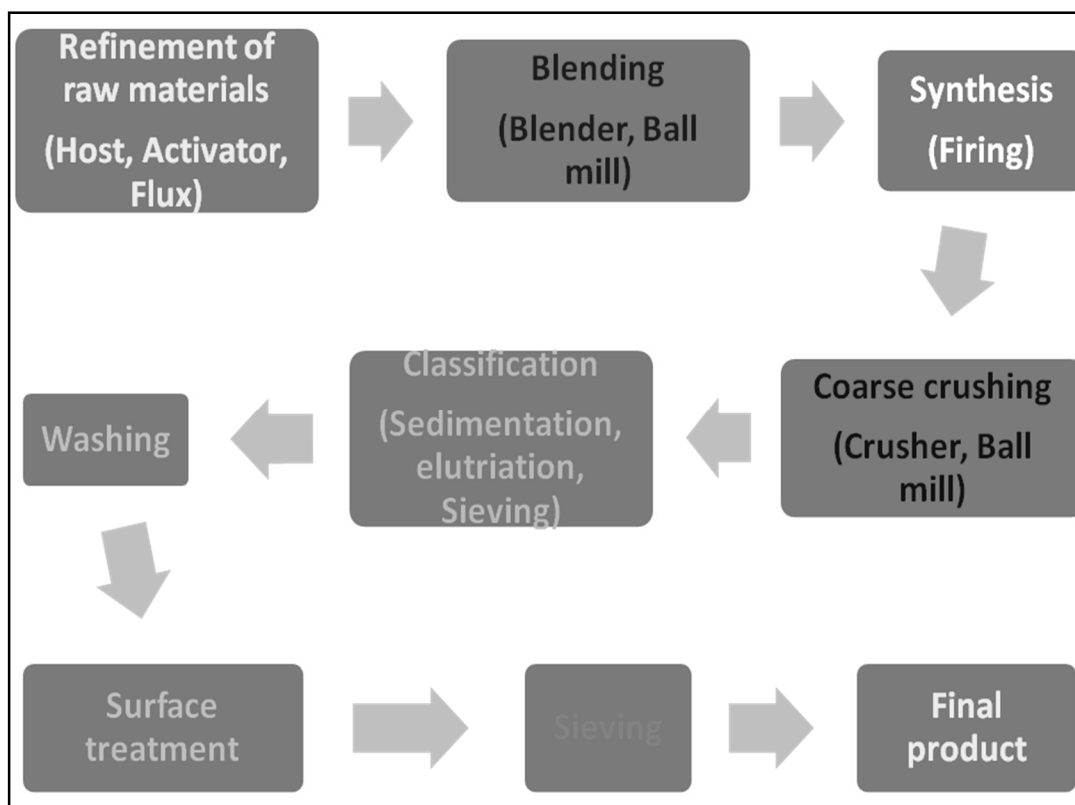
II. PHOSPHOR PREPARATION TECHNIQUES

Almost all phosphor materials are synthesized or manufactured by a high temperature solid-state reaction or by a sol-gel process. During synthesis, a host matrix is formed from high-purity starting chemicals and the impurities, also known as activators and coactivators, are diffused into the crystal lattice in the required quantities. The activators are mostly responsible for the luminescence. In some cases, coactivators play an important role in diffusing the activators effectively into the crystal lattice and

sometimes participate in keeping crystal neutrality through charge compensation. The synthesis sequence varies depending on the type of phosphor being prepared.

A. Solid-State Diffusion Reaction (SSR)

The formation of a phosphor host and doping process by solid solution is critical and is highly dependent on the reaction on the reaction temperature and conditions. Since the purity of starting chemicals is very important to the synthesis of phosphors, the starting chemicals are typically 99.9-99.999% in purity. It is important to minimize the concentration of specific contaminants such as Fe, Co, and Ni, which can seriously degrade phosphor performance. Required amounts of starting ingredients are mixed in the presence of an appropriate flux (if necessary) and fired at high temperatures (900-1500°C) in air or in a controlled atmosphere (N₂, C, CO, or N₂ with 2-5% of H₂). The calcinations conditions such as firing temperatures, duration of firing, firing atmosphere, and rate of heating and cooling for a particular phosphor are optimized empirically. The phosphor particle size and shape are also related to the morphology of starting chemicals and flux. The presence of flux materials of low melting compounds such as alkali halides helps to complete the doping process at lower temperatures. In the past decade many publications on the subject of PDP phosphors have become available; most of the phosphors are prepared by a solid-state reaction. The general preparation procedure of solid-state reaction is well described in the literature.

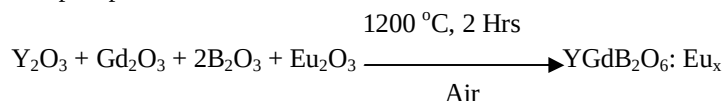


B. Preparation of RE³⁺ Doped YGdB₂O₆ Phosphors

The general formula of the prepared phosphors:

YGdB₂O₆: Eu_x (x=0.1, 1.0, 1.5 and 2.0 mol%)

Basic Reaction of the YGdB₂O₆: Eu_x phosphors:



C. Introduction to Characterization Techniques

The tools used for characterization in this study are as follows:

- 1) Spectrofluorophotometer (SPF) using Shimadzu – RF 5301 PC
- 2) X-ray diffraction technique (XRD) using Scintag.inc, USA

- 3) Scanning Electron Microscopy (SEM) using JEOL 5600LV SEM
- 4) Particle size analysis
- 5) Commission Internationale de l'Eclairage (CIE) using Radiant Imaging software.

III. RESULTS AND ANALYSIS

Fig.1 is the Excitation & Emission studies of Eu doped $\text{YGd}_2\text{B}_2\text{O}_6$ phosphor. The excitation spectra is monitored at 594 nm. It is interesting to note that the excitation is broad are from 240-320nm peaking at 269, 275 & 314nm. As it is known fact the emission below 600nm are due to magnetic dipole of host where Eu occupies in $3+$ state. The hyper five interactions in the host lattice when Eu occupies in inverse site symmetry are called Electric dipole and the emissions are generally found about 600nm. When Eu is 0.1% the excitation intensity is around 180, when 1.0% it is 170; when 1.5% it is 150 and when 2.0% it is 80 units. It is interesting to note that as Eu concentration in $\text{YGd}_2\text{B}_2\text{O}_6$ increases the excitation intensity gradually decreases and the 275nm absorption dominates when compared to 269nm.

Fig.1 Excitation & Emission spectra of $\text{YGd}_2\text{B}_2\text{O}_6:\text{Eu}(1.0\%)$ phosphor

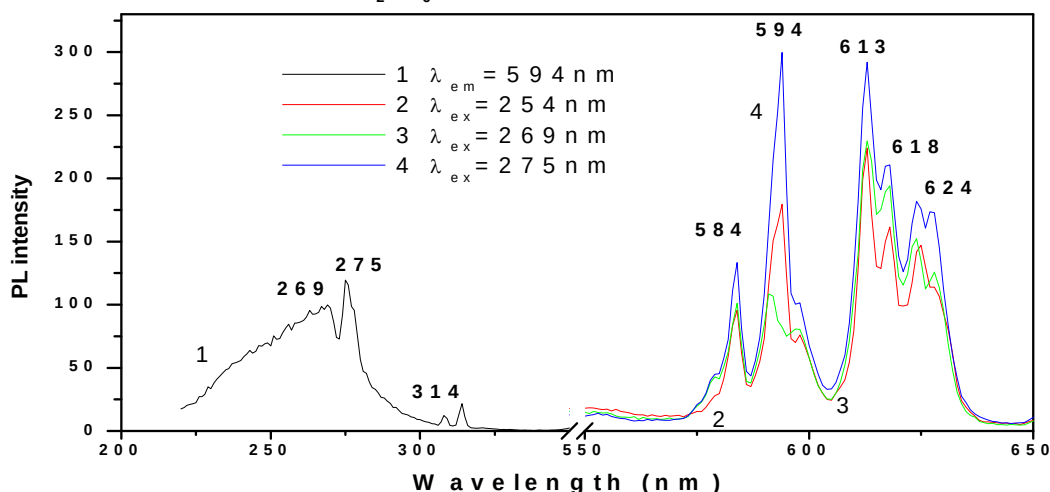


Fig.2: Variation of different emission peaks with respect to Eu concentrations under 275nm Excitation

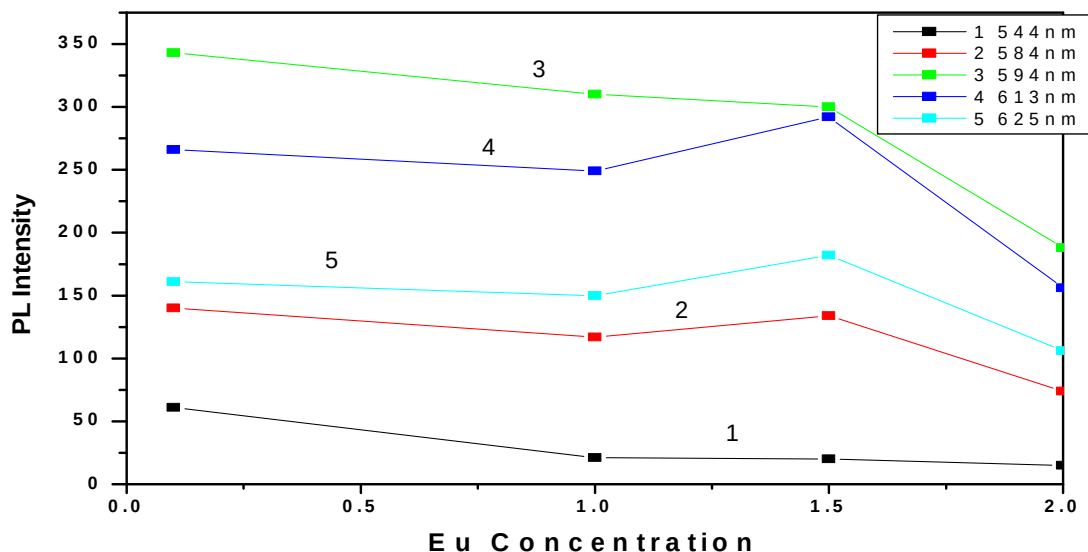


Table-1 Intensity of various emission peaks of $\text{YGd}_2\text{B}_2\text{O}_6:\text{Eu}^{3+}$ (0.1, 1.0, 1.5 & 2.0 mol%) phosphor under 275nm excitation

Sr. No.	Excitation Wavelength	Eu Concentration Mol(%)	Different Emission peaks under 275nm Excitation				
			584	594	613	618	624
1	275nm	0.1	117	310	249	150	150
2		1.0	140	343	266	161	130
3		1.5	134	300	292	182	170
4		2.0	74	188	156	106	90

Fig.2 is the variation of different emission peaks with respect to Eu concentrations under 275nm Excitation. Table-1 Intensity of various emission peaks of $\text{YGd}_2\text{B}_2\text{O}_6:\text{Eu}^{3+}$ (0.1, 1.0, 1.5 & 2.0 mol%) phosphor under 275nm excitation for better understanding. The following emissions with different are found at 584,594,613,618 and 624nm under 275nm excitation. Among the PL emissions the 594nm peak intensity dominates when compared to other PL emissions when excited with 275nm. On comparison where the excitation is varied from 254, 269 & 275nm, the emissions are not affected. However the variations in emission intensities are found.

A. XRD Study

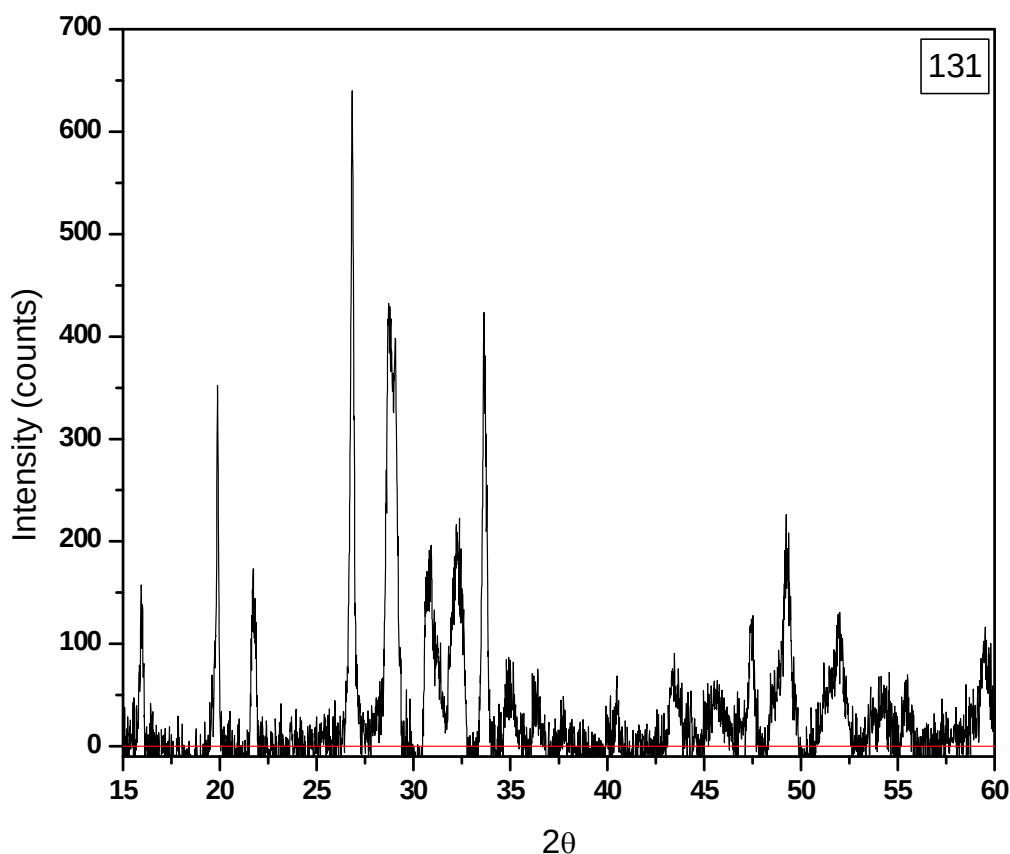


Fig.3 XRD pattern of $\text{YGd}_2\text{B}_2\text{O}_6:\text{Eu}(1.5\%)$ phosphor

Fig.3 is the XRD patterns of $\text{YGd}_2\text{B}_2\text{O}_6$ doped with Eu phosphors crystallite size is calculated using Scherer's formula for all the samples using the formula, $D = K\lambda/\beta\cos\theta$. The calculated crystallite sizes are 48.06nm. More the diameter of the ions, lesser is the crystallite size.

B. SEM Study



Fig. 4 SEM images of 1.5% Eu doped YGdB₂O₆ phosphor

Fig.4 is the SEM micrographs of YGdB₂O₆: Eu (1.5%) phosphor of different magnifications. The particles are irregular in shape and agglomerated and the size is < 5 microns.

C. Particle Size Analysis

This property is determined mainly by particle size distribution and surface treatment. To fine particle size, luminescence efficiency tends to become lower. Phosphors having a small particle size and high efficiency would be most useful. The particle size of the YGdB₂O₆: Eu (1.5%) phosphor was found to be 0.52 and 3.9 microns and specific surface area is 2.8810 m²/gram.

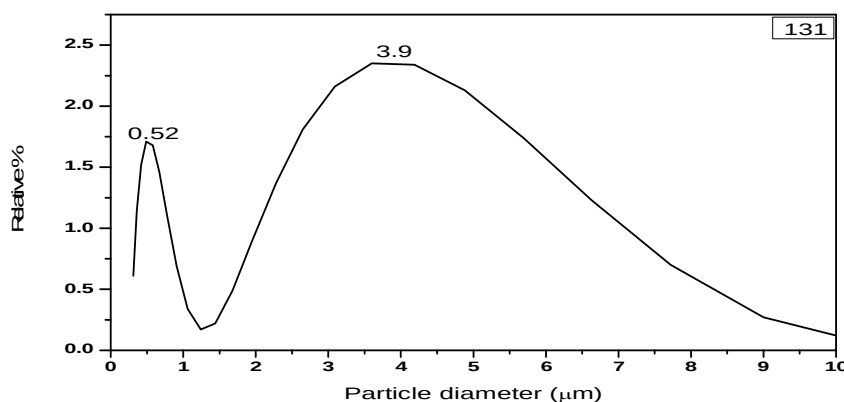


Fig. 5 Particle size histogram of 1.5% Eu doped YGdB₂O₆ phosphor

D. CIE colour co-ordinates study

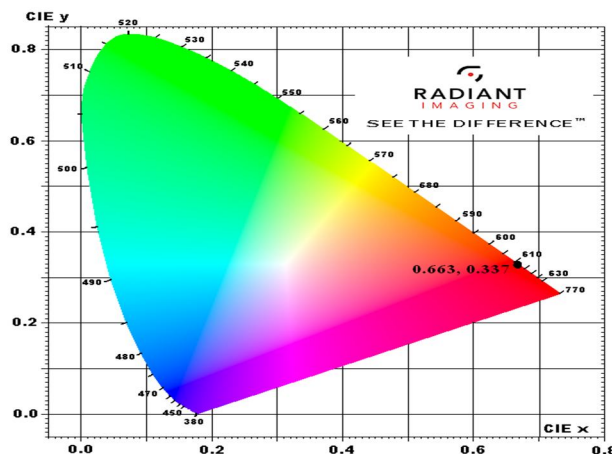


Fig.6 CIE colour co-ordinates of YGdB₂O₆:Eu(1.5%) phosphor under 275nm ex.

Calculated colour coordinates [CIE] for the phosphors under characterization. X and Y values are calculated using Radiant Imaging software. Other phosphors emit either red dominated white emissions YGdB₂O₆. The colour co-ordinates of YGdB₂O₆:Eu(1.5%) phosphor are X=0.663, Y=0.337.

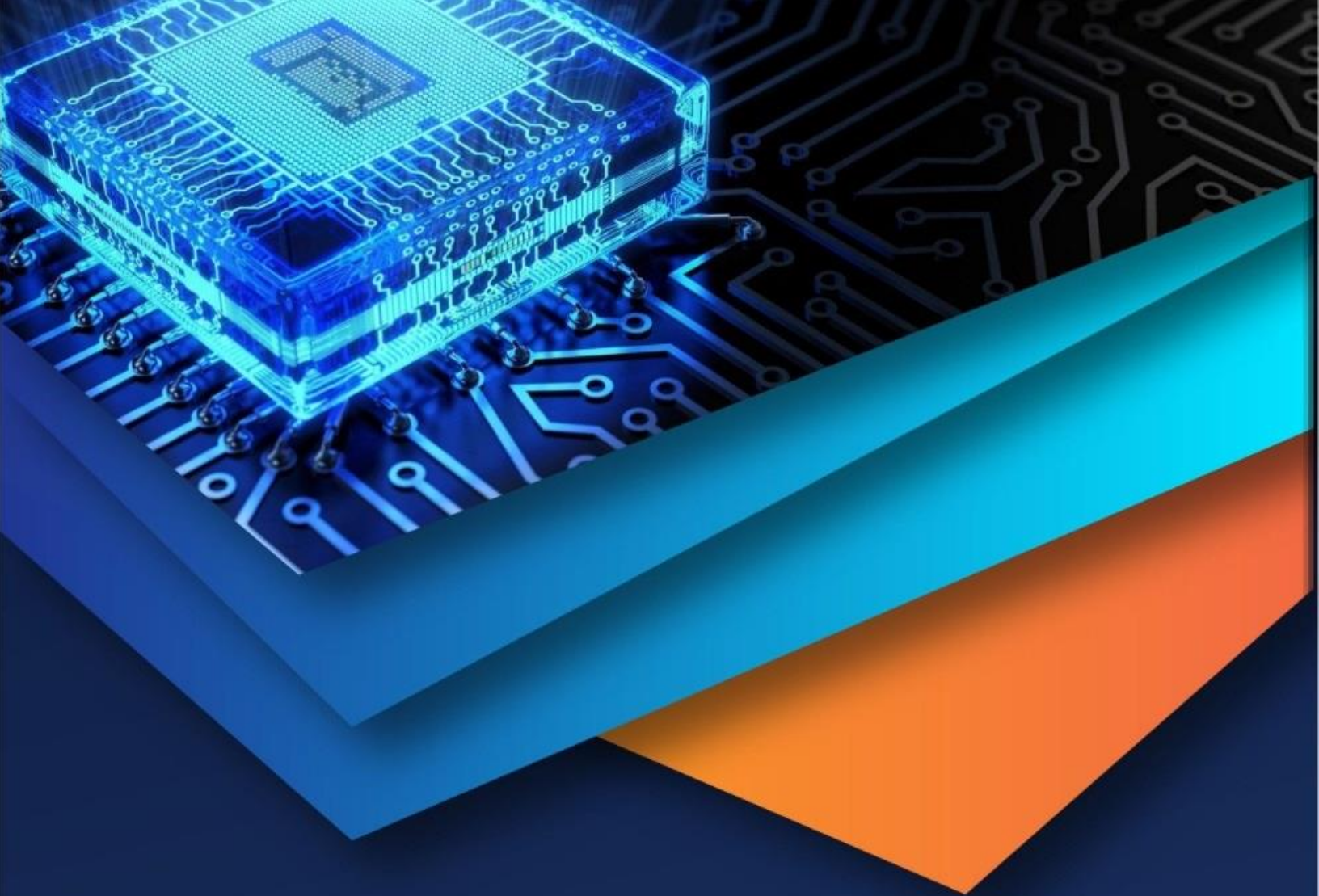
IV. CONCLUSIONS

Therefore the following conclusions are drawn from the above studies.

- A. When the phosphor is excited with 254 & 269nm the emissions from Eu doped YGdB₂O₆ phosphor are dominates in Electric Dipole region.
- B. When the phosphor is excited with 275nm the 594 peak dominates among the emission which is attributed to Magnetic Dipole of the host when doped Eu in 3+ state.
- C. It is interesting to note that as the Eu concentration increases in YGdB₂O₆ the excitation intensities gradually reduces and 275nm absorption dominates.
- D. It is rare case in the same Eu doped host (YGdB₂O₆) phosphor as the excitation wavelength increases (high to low energy) the emission peak intensities varies from low to high energy. This may be due to the resonance energy transfer between the Gd³⁺ state Eu³⁺.
- E. From the XRD pattern the crystallite sizes is found to be 48.06nm.
- F. From the SEM observation the particles are irregular in shape and agglomerated and the size is < 5 microns.
- G. The particle size observed for Eu(1.5%) doped YGdB₂O₆ phosphor is 0.52 and 3.9 microns and the surface area is 2.8810 m²/gram.
- H. From the CIE study the phosphor is red dominated micrographs of YGdB₂O₆: Eu (1.5%) phosphor of different magnifications.

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