
Silicon dioxide ultraviolet to visible phosphors material

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Abstract: The present work include preparation of phosphors materials from rare earth (Eu) ion doping Silicon Oxide (SiO₂), and study the characteristics of phosphors for ultraviolet to visible conversion. The phosphor materials have been synthesized by two steps, the first was preparing the powder by solid state method using SiO₂, and Eu₂O₃ with doping concentration of 5% and different calcination temperature (1000, 1200 and 1400 °C), the second step is the preparing of the colloid by dispersing the produced powder in a polyvinyl alcohol solution (4%). Powder preparation achieved by mixing the powder according to weight percentage, milling then calcining the mixture at the above temperatures. The produced powder was characterized by X-Ray diffraction .The X-Ray resultsshow a mismatch with the standard peaks. The difference may be due to the variation in purity of the materials and the phases before calcination. Colloid preparation achieved by dissolving the PVA in water (4%) then dispersing the powder into the solution by using the hot plate magnetic stirrer and ultrasonic bath. The produced powder was characterized by using Fourier transform infrared (FTIR) and Photoluminescence spectra (PL). The results of photoluminescence spectra show that samples doped with Europium ions emit red color with wave length 612nm. The intensity of emission was increased with increasing calcination temperature.

Keywords: Nano Smart Material, Chemical Doping Processor, Fourier Transform Infrared, Photoluminescence Spectra

1. Introduction

Luminescence is an optical phenomenon, whereby a material is excited with high energy radiation (typically ultraviolet light, but other forms of energy such as beta rays can also be used). The phenomenon of luminescence has been known to mankind for over a thousand years. Descriptions have been found of ancient Chinese paintings that remained visible during the night, by mixing the colors with a special kind of pearl shell [1].

Until the end of the 20th century, very little research was done on the phenomenon of luminescence. For many decades, zinc sulfide (ZnS) doped with copper (and later codoped with cobalt) was the most famous and widely used persistent phosphor. It was used in many commercial products including watch dials, luminous paints and glow-in-the-dark toys[2,3]. In recent years, considerable research has been done on the synthesis and characterization of large band gap oxide materials such as Y₂O₃ and SiO₂ doped with rare-earth elements using different chemical methods such as sol-gel precipitation and combustion[4].

There are many advantages of luminescent materials, Precise material deposition on substrate at well-defined positions, Small material consumption , Less material losses, Less material losses and It has excellent chemical stability.

There are many applications of luminescent materials which can be used in many fields, Security ink[5], Widely used in Cathode ray tube displays[5], Plasma display panel[6], High radiation energy detection (such as to make films for X-ray detection) applications[7] and white light emitting diode (LED)[6] The aim of this work is to prepare luminescent ink from rare earth materials by solid state method.

2. Experimental

The experimental part is formed from two steps the first is the preparation of the powder and the second step is preparation of the colloid.

Raw materials used in the present work were: Nano

SiO₂ and Eu₂O₃ Their properties and sources were listed in table (1) below:

Table (1). Raw Materials and the properties

Item	Purity	Source	characterization
SiO ₂	99.5%	Sigma Aldrich	Nano powder <50nm
PVA	99%	Sigma Aldrich	n=89000-98000
Eu ₂ O ₃	99.5%	Sigma Aldrich	Nano powder <150nm

2.1. Methods of Preparation

2.1.1. Powder Preparation

The powder is prepared by using solid state reaction. The starting materials and weights were listed in table (2) below:

Table (2). The percentages of starting materials.

Batch No.	Item	Weight Percentage %	Weight (gm.)
1	SiO ₂	95	3.819
	Eu ₂ O ₃	5	1.179

Raw materials were weighed according to the above table in an electronic balance (five digits) inside a Nano-filtration system. The powders feeds in a poly vinyl chloride bottles with yttrium stabilized zirconia (YSZ) balls as milling media, then the mixture was milled by using a ball mill for 24 hrs with 64.5 rpm, this process achieved with the addition of propanol to achieve homogenization.

After 24hr take off the mixture and poured inside stainless steel pan then dry it in dryer for 4hr in 80 °C to evaporate propanol. The dried sample calcined in an electrical furnace using an alumina crucible as a boat at a temperature 1000, 1200 and 1400 °C for 9 hrs.

The powder after calcination was secondly milled in the same previous way for 24 hrs with 64.5 rpm in order to prevent agglomeration.

X- Ray diffraction was used to characterize the powder after preparation by solid state method.

2.1.2. Colloid Preparation

To prepare the security ink, the powder was produced as colloid, this can be achieved by dispersing the powder in a poly vinyl alcohol [CH₂CHOH]_n, (n=89000-98000) inside a Nano-filtration system. First a solution of polyvinyl alcohol in water (4%) was prepared with the aid of magnetic stirrer, homogenizer and heater.

The powder added to the PVA solution 0.2 gm. for 20 ml of the solution. The suspend also mixed and heated in a magnetic stirrer and homogenizer with heating up to 50°C for 1 hr. Then mixing without heating for 2 hrs with the addition of 10 ml ethanol, ultrasonic bath was used for third stage mixing and homogenization, then the fourth stage of mixing and homogenization was achieved by magnetic stirrer and homogenizer for 2 hrs, the final product is a white solution which then tested by photoluminescence spectra and Fourier transform infrared.

2.2. Characterization

The characterization of powder achieved by X-Ray

diffraction.

2.3. X-Ray Diffraction

The X-ray powder diffraction patterns presented in this work were measured using a theta to theta diffractometer (Philips), equipped with a Cu K α source (generator: 40 kV and 40mA) with wave length 1.5418Å, a scintillation detector with pulse-height analysis, and a variable knife-edge collimator for high resolution X-ray diffractometry. The best achievable instrumental resolution was 0.001° in 2 θ .

2.4. Colloid Characterization

The prepared colloid characterization achieved by measuring its photoluminescence spectra, in order to predict its capability to emit color (luminescence).

2.5. Photoluminescence Spectra

Photoluminescence spectra is measured by using Fluoro Max-4 spectrofluorometer. The excitation wave length for all samples were 254 nm (at the ultraviolet region) produced by Xenon flash lamp then the emitted wave detected by signal detector (photomultiplier tube and housing).

2.6. Fourier Transform Infrared

Fourier transform infrared is measured by NEXUS FTIR.

The variables of the instrument was

Number of scan: 64

Resolution: 4

Data spacing: 1.928cm⁻¹

3. Result and Discussion

3.1. X-Ray Diffraction

The analysis of X-ray diffractions for all synthesized samples were shown below according to their calcination temperature:

SiO₂: Eu 5% Calcined at 1000°C:

Figures (1), (2) and (3) shows the XRD patterns for the system SiO₂: Eu 5% Calcined at 1000, 1200 and 1400°C respectively. There was mismatch with the standard peaks. The difference may be due to the variation in purity of the materials and the phases before calcination.

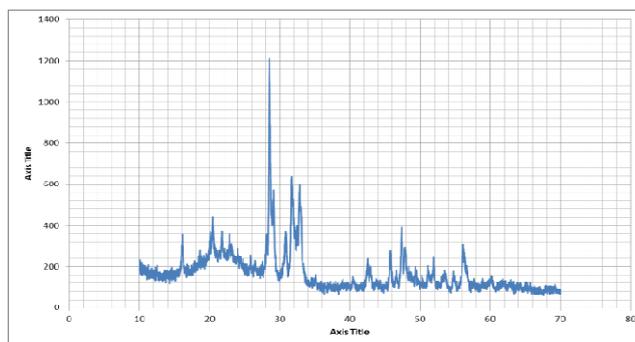


Figure (1). XRD patterns of SiO₂: Eu 5% Calcined at 1000°C

SiO₂: Eu 5% Calcined at 1200°C:

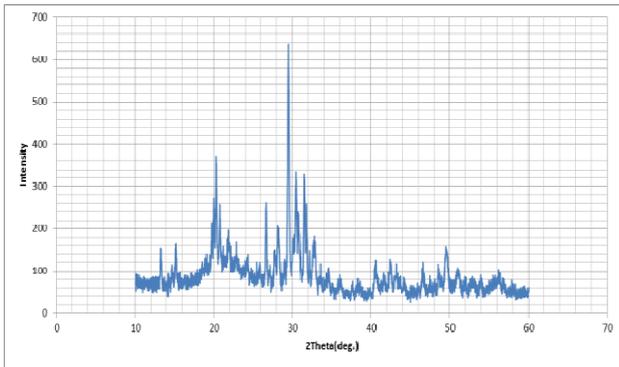


Figure (2). XRD patterns of *SiO₂: Eu 5% Calcined at 1200°C*

SiO₂: Eu 5% Calcined at 1400°C:

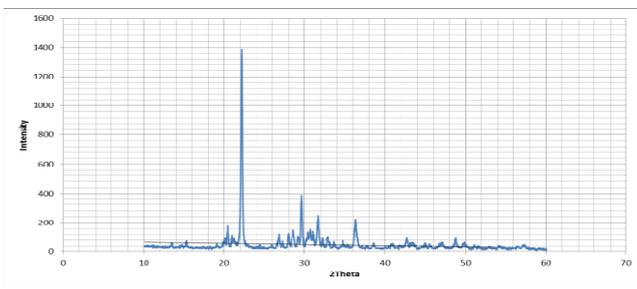


Figure (3). XRD patterns of *SiO₂: Eu 5% Calcined at 1400°C*

3.2. Fourier Transform Infrared

The analysis of Fourier transform infrared for all synthesized colloid was shown in figure (4) can be discussed by comparing it with the data of infrared absorption of pure materials according to the wave number. The broad peak at around 3340 cm^{-1} is originated from O-H stretching in hydroxyl groups. Both PVA and alcohol contain hydroxyl group. C-H stretching was observed at around 2980 cm^{-1} . The peak at around 2300-2400 cm^{-1} it is coming from $\text{C}\equiv\text{C}$ group.

The peak at around 1600-1800 cm^{-1} was coming from $\text{C}=\text{O}$ group. Absorption peak at around 1213-1420 cm^{-1} might originated from C-H bend and C-C stretching. $\text{C}-\text{O}$ stretching was observed at around 1020-1100 cm^{-1} .

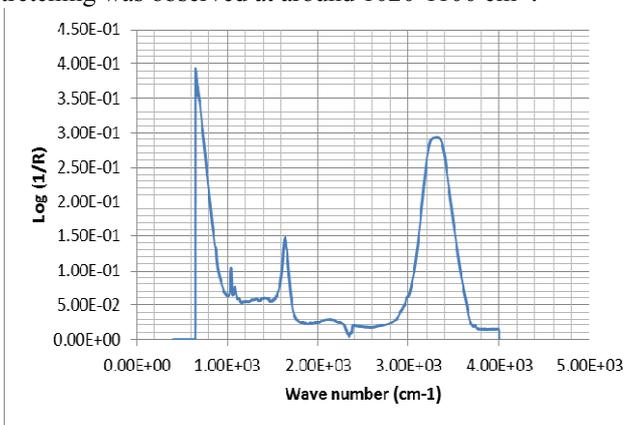


Figure (4-a). The FTIR of *SiO₂: Eu 5% calcined at 1000°C*

The absorption peak at around 890-1000 cm^{-1} was originated from interaction between PVA chain and the surface of $\text{Y}_2\text{O}_3:\text{Eu}_2\text{O}_3$. Figures for all samples are shown below according to the calcination temperature :

SiO₂: Eu 5% calcined at 1000°C

SiO₂: Eu 5% 1200°C:

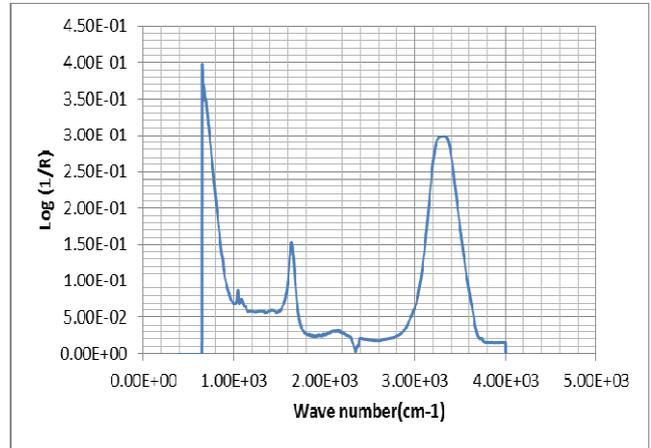


Figure (4-b). The FTIR of *SiO₂: Eu 5% 1200°C*

SiO₂: Eu 5% calcined at 1400°C

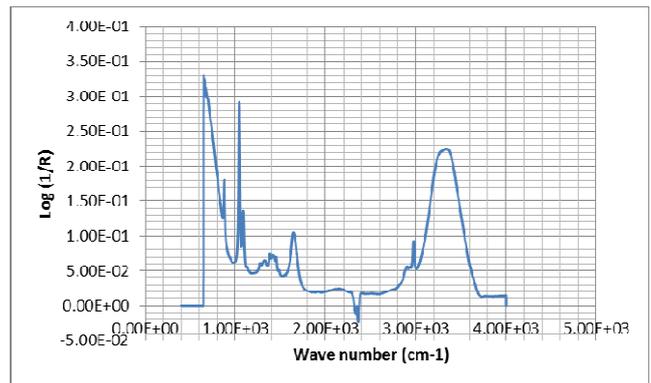


Figure (4-c). The FTIR of *SiO₂: Eu 5% calcined at 1400°C*

3.3. Photoluminescence Spectroscopy

The analysis of Photoluminescence spectroscopy for all synthesized colloid are shown below according to the calcination temperature as shown in figure (5):

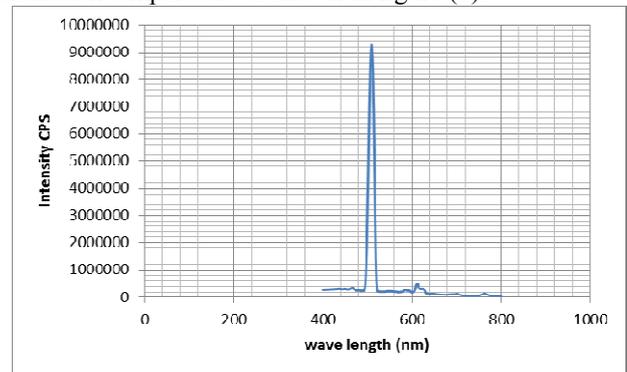


Figure (5-a). Photoluminescence spectra of *SiO₂:Eu calcined at 1000°C*

SiO₂: Eu 5% calcined at 1000°C

From figure (5-a) below there are three peaks, the first one at wave length 508 nm it comes from 2λ where the applied λ was 254 nm. The second tiny peak at wave length 612 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$. The third peak at wave length 760 nm it comes from 3λ .

SiO₂: Eu 5% calcined at 1200°C

From figure (5-b) below there are four peaks, the first one at wave length 585 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$. The second peak at wave length 612 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$. It is clear that photoluminescence increased with increasing calcination temperature. The third peak at wave length 700 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_3$. The fourth peak at wave length 762 nm comes from 3λ where the applied wave length was 254 nm.

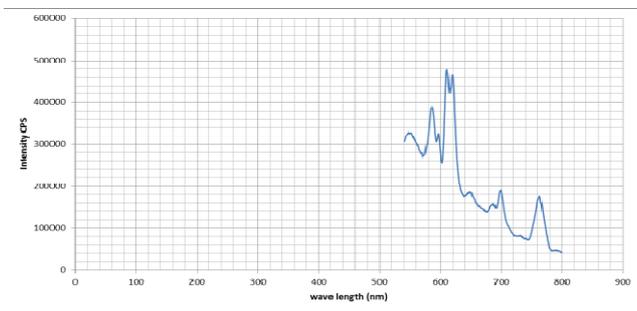


Figure (5-b). Photoluminescence spectra of $\text{SiO}_2:\text{Eu}$ calcined at 1200°C

SiO₂: Eu 5% calcined at 1400°C

From figure(5-c) below there are four peaks, the first one at wave length 585 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$. The second peak at wave length 612 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$. It is clear that photoluminescence increased with increasing calcination temperature. The third peak at wave length 700 nm comes from electron transition in Eu^{+3} ion from ${}^5\text{D}_0 \rightarrow {}^7\text{F}_3$. The fourth peak at wave length 762 nm comes from 3λ where the applied wave length was 254 nm.

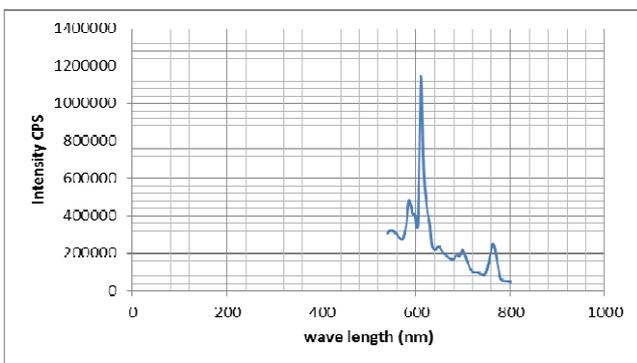


Figure (5-c). Photoluminescence spectra of $\text{SiO}_2:\text{Eu}$ calcined at 1000°C

4. Conclusions

Stable luminescent colloid was produced from powder of $\text{SiO}_2:\text{Eu}$ powder dispersed in poly vinyl alcohol solution. $\text{SiO}_2:\text{Eu}$ played a major role as luminescent centers in the colloid which emitted red luminescence. This colloid can meet several applications in optoelectronics such as for production of light emitting devices, light sensor, and luminescent displays in a very definitive sizes and shapes. Increasing calcination temperature lead to increase luminescent intensity.

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