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SYNTHESIS AND CHARACTERISATIONS OF NANOCRYSTALLINE MAGNESIUM OXIDE PHOSPHOR DOPED WITH DYSPROSIUM

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ABSTRACT
In this work nanocrystalline MgO phosphors doped with Dy were prepared using combustion method. The obtained sample was characterized by XRD, SEM and EDX methods. The synthesized sample was observed by moving radiation detector with scan speed of 2˚/min at the range of 10˚-80˚ where monochromatic wavelength of 1.54 Å (Cu-kα) was used. The XRD pattern shows very broad peaks. The crystalline nature of sample is cubic and average size of particle is 20 nm .The maximum relative intensity was 100% for the peak (200).The instrument was operated at voltage of 10 Kv and the samples were scanned at a distance of 8.5 mm for SEM image. The spectrum obtained by EDX is 100%. From the data of EDX, it is observed that samples contain 55.71% Mg, 44.32% O and 0.87% Dy of atomic percentage respectively which agrees well the expected value.

KEYWORDS: Combustion synthesis, XRD, SEM, EDX, nanoparticles

INTRODUCTION

During the last few years, synthesis of nanostructures metal oxide materials has concerned the researchers due to its potential applications (F. Nastase et al. 2006). In current years, researchers have focused more on the synthesis of MgO nanoparticles due to its novel applications in advanced technologies (Mohammad Ali Karimi et al. 2011). Metal oxides are very significant technological materials to be used in electronic and photonic devices (M. C. Wu et al. 1991). The magnesium oxide (MgO) is a very suitable candidate for insulation applications due to its low heat capacity and high melting point (S. K. Shukla et al. 2004). Recently, it was reported that MgO has a good bactericidal show in aqueous environments due to the formation of super-oxide (J. Sawai et al. 2000). Various properties of MgO, such as catalytic behaviour, can be further enhanced if it is used as nanosized particles compared to micron-sized particles. Therefore, the formation of MgO nanostructures with a small crystallite size of less than 100 nm and homogeneous morphology has involved much attention due to their unique physicochemical properties including high surface area-to-volume ratio.

It is extensively accepted that the properties of MgO nanostructures depend strongly on the synthesis methods and the processing conditions. Magnesium oxide is a traditional raw material for use in a wide range of products, e.g. refractory, paints, paper, plastics, rubber, oil, pharmaceutical, fertilizer, animal feed, additive in superconductor products, waste treatment agent for neutralizing acids or cleaning water and as a catalyst material (Yuan YS et al. 1996 and Bhargava A et al. 1998). Some groups have devoted to the synthesis of fine MgO powders (Znaidi L et al. 1996 and Alvarado E et al. 2000). Recent progress in optical devices, such as lasers and optical amplifiers, based on electronic transitions of rare earth ions, has inspired a lot of working different materials doped with these ions (G. Blasse et al. 1994 and R. Scheps 1996). Dy3+ ions are well-known as activator dopants for many different inorganic lattices producing white light emission by suitably adjusting the yellow and blue emission (J. L. Sommerdijk, et al.

1975). The yellow color is due to the electric dipole transitions of electrons from the 4F9/2 level to the 6H13/2 level. However, the emission is not entirely controlled by the Dy3+ ions, since the host lattice also plays an important role. If a dysprosium site has no inversion symmetry the strong yellow emission is prominent, whereas, if the dysprosium site has inversion symmetry then the emission line would be expected to present blue (M. Yuet al. 2002).

MgO : Dy3+ has commonly being prepared using combustion syntheses method at temperature of 550˚ C. This method is one of the best methods because it is relatively simple, efficient, low cost and time consuming method. The scope of this work is to analyze crystalline nature, spectrum and atomic percentage of sample.

MATERIALS AND METHODS

MgO: Dy3+ has commonly being prepared using combustion synthesis. The starting raw materials are Mg(NO3)2.6H2O(99.99%),Dy (NO3)2.6H2O (99.9%) and NH2CONH2(99.5%).They were weighed with a stoichiometric ratio. The composite powders were ground in an agate mortar and then placed in an alumina crucible and were then introduced into muffle furnace at 550˚C for 20 min as the ignition occurs the reaction occurs vigorously for few seconds and the fluffy substance was obtained.

RESULTS AND DISCUSSION

XRD

XRD is used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. Xrays are high energy photons that are produced when electron make transitions from one atomic orbit to another. The crystalline structure of MgO: Dy3+ was analyzed by PAN analytical X-pert diffraction with Cu-kα radiation (λ=1.54060 A˚ or 0.154nm).The synthesized sample was observed by moving radiation descent speed of 2˚/min at the range of 10˚-80˚ where monochromatic wavelength of 1.54 A˚ (Cu-kα) was used. The XRD patterns show very broad peaks.

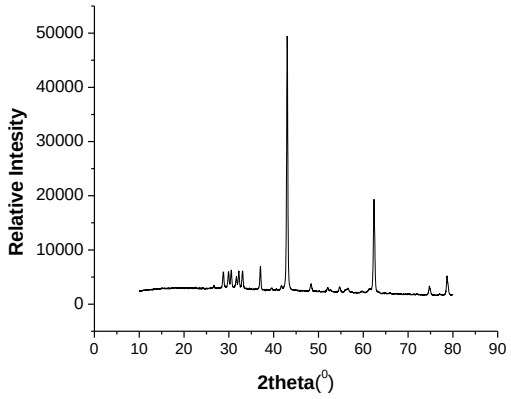

Fig. 1 Powder XRD Patterns of MgO and Dy^{3+}

Graph and d spacing

Indexing process of powder is done with the help of miller indices the value of hkl is being calculated. Following is the detail. There were number of braggs reflection can be seen with respect to (111), (200), (220) reflection. Peak value indexing from d spacing shown in table 1.

Table 1 shows graph and d spacing

2θ	d(A˚)	$1000/d^2$	$(1000/d^2)/C.F$	Hkl
36.9545	2.43052	169.280	2.980	111
42.9718	2.10308	226.090	3.980	200
62.3481	1.48827	451.497	7.497	220

Particle size

The average grain size of MgO: Dy3+ nanoparticles is determined using Debye Scherrer formula. The average particle size is calculated in the below table 2 i.e 20nm.

Table 2 shows particle size

2θ	FWHM B(rad)	d spacing (nm)	hkl	Size of particle D(nm)
36.9545	0.05761	0.243052	111	10nm
42.9718	0.02608	0.210308	200	23 nm
62.3481	0.05761	0.148827	220	28 nm

Intensity of XRD peaks

The maximum intensity of experimental for MgO: Dy3+ is 100% the peak (200).Peak Intensity are shown in the below table.

Table 3 shows relative intensity

Hkl	111	200	220
2θ of peak	36.9545	42.9718	62.3401
Height(cts)	2120.29	23904.14	10226.46
Relative intensity (%)	8.87	100	42.78

SEM

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition.

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The SEM image is carried out by using Zeiss. Evo 18 Special Edition in order to analyze the structure and morphology of doped samples. SEM was used for the morphological study of MgO: Dy3+. The instrument was accelerated at voltage of 10 Kv and the samples were scanned at working distance of 8.5 mm. The SEM images for the MgO: Dy3+ samples are shown in Fig. 2, Fig 3, Fig 4, respectively.

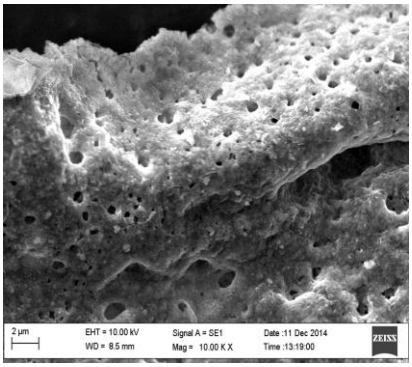

Fig. 2 The SEM image of MgO doped Dy for 2 µm

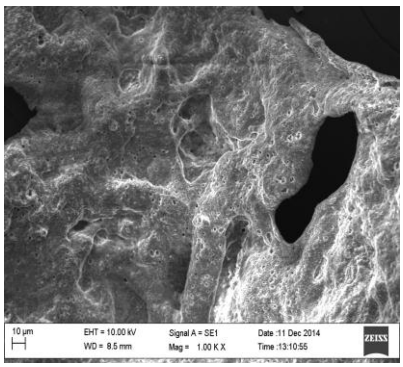

Fig. 3 The SEM image of MgO doped Dy for 10 µm

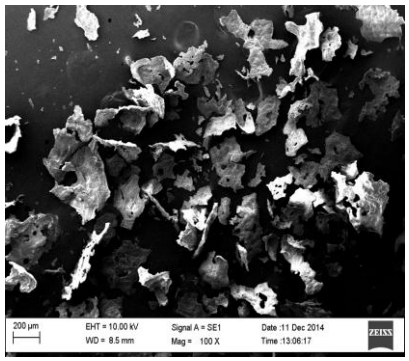

Fig. 4 The SEM image of MgO doped Dy for 100 µm

EDX analysis

Energy-dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. The spectrum obtained by EDX samples is shown in Fig. 5 (a). From the sample of MgO: Dy3+ , spectrum 100% of Mg metal was observed in the sample corresponding to peak shown in the Fig. 5 (b). In sample the inclusion of Dy 3+ is shown in the corresponding peaks. From the data it is observed that the synthesized sample contains about Mg, O and Dy with 55.71%, 44.32% and 0.87% of atomic percentage respectively which agrees with expected value. From the element count percentage, 55.71% of Mg and 0.87% of Dy have been observed.

Table 4 shows the elemental analysis

Element	Weight%	Atomic%
O	42.69	55.71
Mg	50.56	44.32
Dy	6.75	0.87

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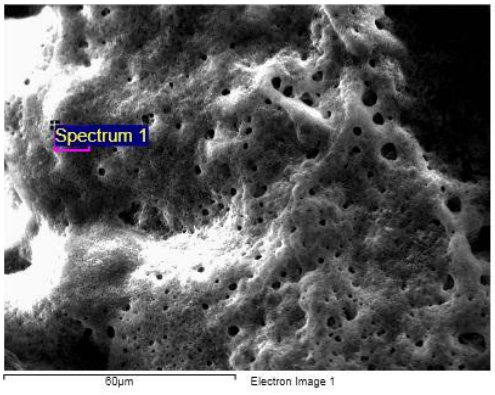

Fig. 5 (a) The EDX image of MgO doped Dy

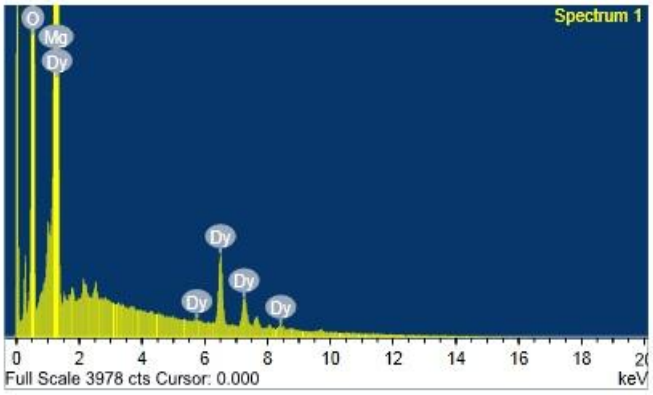

Fig. 5 (b) The EDX image of MgO doped Dy

CONCLUSION

The focus of this research is to study the properties of oxide based phosphors with rare earth doped elements have been discussed. A facile method to prepare high quality MgO and Dy doped magnesium nanocrystals, has been achieved. The structural perfection and the growth features of the synthesized crystals were studied .A morphology index based on FWHM of XRD data have been developed. The average grain size of magnesium oxide doped with dysprosium nanoparticles is determined 20nm. The quality of the nanocrystals was visualized by observing the surface morphology using SEM studies and control on size and size distribution were demonstrated by SEM results. The SEM image is carried out by using Zeiss. Evo 18 Special Edition in order to analyze the structure and morphology of doped samples. The spectrum obtained by EDX samples is 100% for Mg metals.

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