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Ri-Ichi Murakami · Pankaj M. Koinkar · Tomoyuki Fujii · Tae-Gyu Kim · Hairus Abdullah *Editors*

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Preface

It is with great pleasure that we introduce this special issue of Springer Book containing 13 papers selected from about 100 papers presented at the 2nd International Conference on Nanomaterials and Advanced Composites (NAC 2019). The conference was held at National Taiwan University of Science and Technology in Taipei, Taiwan, from 9 to 11 August 2019, and the second in this series attracted a broad range of international researchers from various fields. The Conference of NAC is now in its second series of existence. It has been held on each year cycle starting in 2018—Busan (South Korea). This conference was initiated by a network of researchers and engineers from both academia and industry in the areas of new nanomaterials, nanotechnology and advanced composites. The key aim of NAC 2019 is to provide an opportunity for the delegates to meet, interact and exchange new ideas on nanomaterials and advanced composite development, performance and their applications. This conference covered a wide range of topics related to materials science and engineering. About 100 papers from 9 countries were submitted in this conference, and only a few manuscripts have been selected for publication in Springer Book. The papers selected for this issue of Springer Book are those represent innovative and leading work in the development field. Other papers from NAC 2019 are published in Modern Physics Letters B which are indexed by SCI. As guest editors, it is our hope that this issue will stimulate further discussion and additional research in nanomaterials and advanced composites so that in future, such a topical issue might include a range of empirical studies. We are grateful to all of the authors who contributed to the issue and the NAC 2019 participants and staff for their contributions.

Taipei, Taiwan — Prof. Ri-Ichi Murakami
Medan, Indonesia — Dr. Hairus Abdullah
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Chapter 2
Colour Tunable Photoluminescence from Samarium and Dysprosium Co-doped ZnO Nanofibers

Chaitali N. Pangul, Shyamkant W. Anwane and Subhash B. Kondawar

Abstract Electrospinning technique was used for the fabrication of samarium (Sm) dysprosium (Dy) co-doped ZnO nanofibers for colour tunable photoluminescence. The prepared nanofibers were characterized by SEM, EDX, XRD, UV-Vis and FTIR. Nanofibers diameter and morphology was studied through SEM and the diameter was found to be in the range of 100–180 nm while presence of elemental Zn, O, Dy and Sm was assured by EDX spectrum. XRD study reveals the crystalline structure while the presence of metal stretching bond of ZnO was observed around 450 cm^{-1} in FTIR studies. A tremendous enhancement in band gap was observed by UV-Vis spectrum. Photoluminescence spectra clearly depicts the energy transfer mechanism within the host ZnO and dopants Sm and Dy wherein CIE parameter confirms the colour tunability of co-doped ZnO nanofibers. Such materials can be a very good optimum candidate for colour tunable luminescent light emitting fabrics.

2.1 Introduction

One Dimensional Nanofibers had been seeking the attention in multiple research areas due to their exceptional physical and morphological properties. High aspect ratio and huge surface area to volume ratio has empowered to fetch the optimum results for the variety of applications [1–3]. Among the various methods of synthesizing nanofibers, Electrospinning had marked an unreplaceable position [4, 5]. Electrospun nanofibrous membranes are considered the chief promising and versatile filter media for fine particle filtration, possess several fascinating features such as remarkable specific surface area, high open porosity, and interconnected porous structure. More significantly, electrospun nanofiber-based filter media are expected to have extremely high filtration efficiency for fine particle and relatively low pressure drop due to the unique structure of electrospun nanofibers [6]. Functional garments

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with both robust waterproofness and breathability fabricated by Electrospinning technology had overcome the negative relationship between protective properties and comfort [7]. However, electro spun fibres generally exhibit a lower optical extinction capacity than the commonly used fibres because of their much smaller diameters [8]. Nanoparticles with excellent capabilities of refraction and absorption are generally incorporated into the polymer to improve the optical extinction capacity [9]. Zinc Oxide is one of the most eligible aspirants for enhancing the further properties of any material. Due to novel properties like high refractive index, binding energy, high thermal conductivity, antibacterial and UV protection of ZnO, it could be used in many materials and products [10]. Wurtzite zinc oxide is a typical native n-type semiconductor with a wide and direct band gap (3.37 eV) and a high exciton binding energy (60 meV) at room temperature and excellent chemical stability [11]. Various applications depend on ZnO properties that are influenced by different factors like deposition conditions, chemical composition, ZnO structural, defects and preferential orientation [12]. Photoluminescence properties for light emitting fibers had been studied as a fact of need for Smart and Technical Fabrics and Devices [13]. Zinc oxide is proved to enhance the light emitting properties when doped with optimum materials like Rare earth ions [14]. Martínez et al., had studied the influence of rare earth ion substitution properties of ZnO nanoparticles and found that magnetic and optical properties of Er^{3+} and Yb^{3+} ions play a significant role in the modification of electronic structure of ZnO resulting in remarkable magnetodielectric and photo dielectric effects which could be conjugated for different multifunctional photo-capacitors for operating microelectronics devices with light and magnetic field [15]. Fang et al., studied the effect of doping of La into ZnO nanostructure for efficient photocatalytic properties [16]. Wu et al., found Strong up-conversion luminescence of rare-earth doped oxide films enhanced by gap modes on ZnO nanowires due to the combined effect of the local confinement of the electric field in the spaces between the ZnO and reflections at the Ag layer [17]. Abdellah et al., hexagonal single-crystal perovskite-like nanofibers were tested as novel substrate materials for surface-enhanced Raman spectroscopy, showing exceptional performance [18]. Yang Ding et al., fabricated $CaMoO4{:}Ln^{3+}$ nanofibers based LEDs which present high efficiency and stability. These single-doped CaMoO4 nanofibers showed stronger emission, and the photoluminescence quantum efficiency [19]. Photoluminescent nanofibers were studied on a very wide aspect but still RE doped ZnO nanofibers needs to be investigated with reference to improved luminescence properties of ZnO via energy transfer process within dopant and host.

In this work, we report the fabrication and characterization of Samarium and Dysprosium co-doped ZnO nanofibers via Electrospinning process. The fabricated electro spun nanofibers were characterized by SEM, EDX, UV-Vis spectroscopy and FTIR strategies. Photoluminescence properties were also investigated in order to study the effect of dopant on the luminescence properties of ZnO. Energy transfer mechanism and CIE parameter were also studied. Efficient energy transfer mechanism was observed within the co-dopants and host ZnO which enables the ZnO to emit in visible range overcoming its defect state visible emission.

2.2 Experimental

Zinc Acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$); LobaChemie with 99.5% purity, RE(III) nitrate hydrate ($RE(NO_3)_3$); Sigma Aldrich with 99.9% purity, Poly Vinyl Alcohol (PVA); Sigma Aldrich mol. wt 89,000 were used as the starting materials. All the materials were used without further purification. The very first solution was prepared by dissolving 0.5 gm of zinc acetate dihydrate and x% of $(Dy(NO)_3)_3$ and 2% of $(Sm(NO)_3)_3$ in 1 mL of deionised water under magnetic stirring. Another solution was made by dissolving 0.35 gm of PVA in 5 mL of deionised water at 60 °C under continuous magnetic stirring for 2 h. Additional 0.059 mL of glacial acetic acid was added after mixing the first two solutions by continuous magnetic stirring for another 2 h. The resultant solution was filled in the syringe and electrospinning experiment was carried out. A constant high voltage potential of 20 kV was applied to the tip of the syringe and the ground was connected to the surface collector. The distance between the tip of the syringe and ground collector was maintained at 15 cm. The so formed electro spun fibers were first dried overnight in oven and then calcinations were performed at 300 °C for 2 h.

2.3 Results and Discussions

2.3.1 Scanning Electron Microscopy

Figure 2.1 represents the SEM images along with frequency distributions of x% Dy 2% Sm doped ZnO nanofibers. Figure 2.1a is the SEM image of 1% Dy 2% Sm doped ZnO doped ZnO nanofibers, Fig. 2.1b is the SEM image of 2% Dy 2% Sm doped ZnO nanofibers and Fig. 2.1c is the SEM image of 3% Dy 2% Sm doped ZnO nanofibers. From all the figures it is very evident that ultralong and continuous nanofibers are formed by the process of Electrospinning. The average diameter is found to be in the range of 100–160 nm for all the samples.

2.3.2 Energy Dispersive X-Ray Spectroscopy (EDX)

Figure 2.2 is the EDX spectra for x% Dy 2% Sm doped ZnO nanofibers. Figure 2.2a is the EDX spectrum for 1% Dy 2% Sm doped ZnO nanofibers, Fig. 2.2b is the EDX spectrum for 2% Dy 2% Sm doped ZnO nanofibers and Fig. 2.2c is the EDX spectrum for 3% Dy 2% Sm doped ZnO nanofibers. Presence of peaks for Zn, O, Sm and Dy confirms the presence of elemental Zinc, Oxygen, Samarium and Dysprosium. Additional peak of Carbon represents the presence of carbonaceous material due to calcination process.

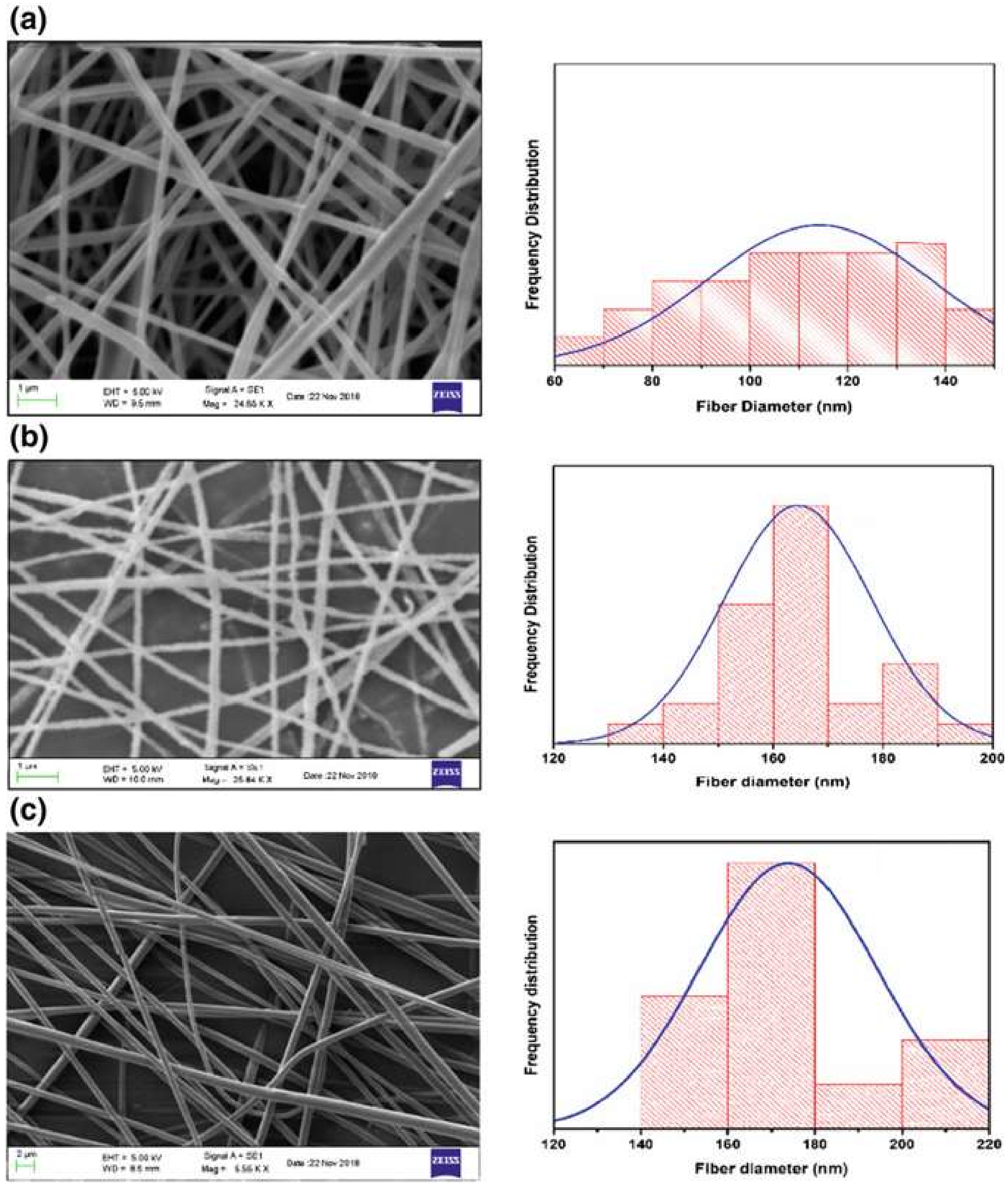

Fig. 2.1 **a** SEM image and frequency distribution of 1% Dy-2% Sm-doped ZnO nanofibers, **b** SEM image and frequency distribution of 2% Dy-2% Sm-doped ZnO nanofibers, **c** SEM image and frequency distribution of 3% Dy-2% Sm-doped ZnO nanofibers

2.3.3 X-Ray Diffraction

XRD patterns for x% Dy 2% Sm doped ZnO nanofibers are shown in Fig. 2.3. The samples clearly exhibit the diffraction peaks for wurtzite hexagonal structure of ZnO. Diffraction peaks at (100), (002) and (101) are the characteristic diffraction peak for crystalline ZnO. No other crystalline phase related to other impurity regarding Dy or Sm is observed indicating the successful incorporation of dopants into ZnO. A

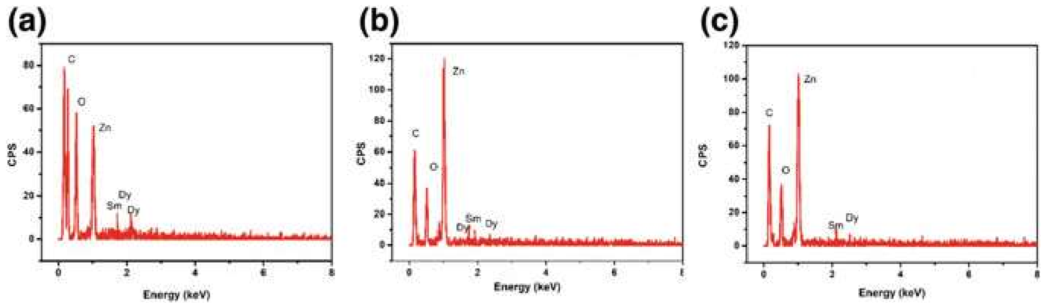

Fig. 2.2 EDX spectra of **a** 1% Dy 2% Sm doped ZnO, **b** 2% Dy 2% Sm doped ZnO, and **c** 3% Dy 2% Sm doped ZnO nanofibers

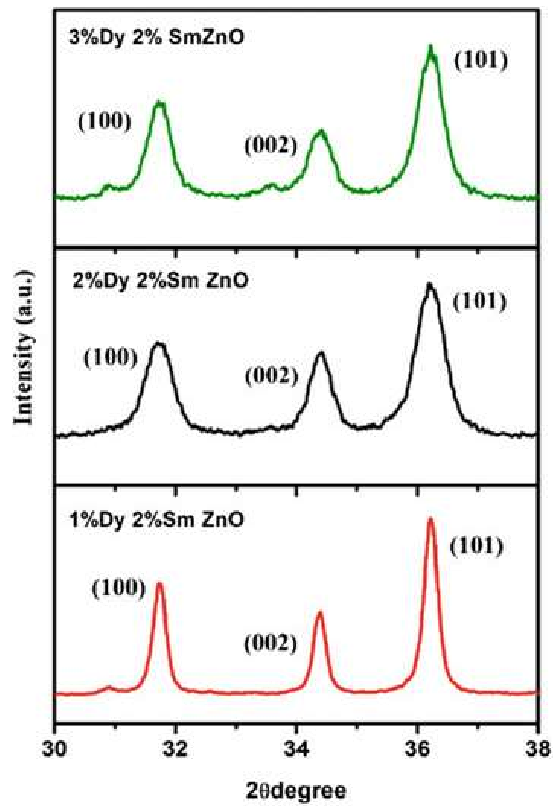

Fig. 2.3 XRD patterns of x% Dy-2% Sm-doped ZnO nanofibers

gradual shift has been observed in 2θ degree for all the diffraction peaks was observed which implies that the dopants had occupied Zn^{2+} sites [20].

2.3.4 UV-Vis Spectroscopy

Figure 2.4 is the absorbance spectra and Tauc's plot for Dy/Sm co-doped ZnO nanofibers. The absorption maxima is found to be blue shifted as compared to bulk ZnO. This could be explained with the help of Quantum confinement effect. Since the

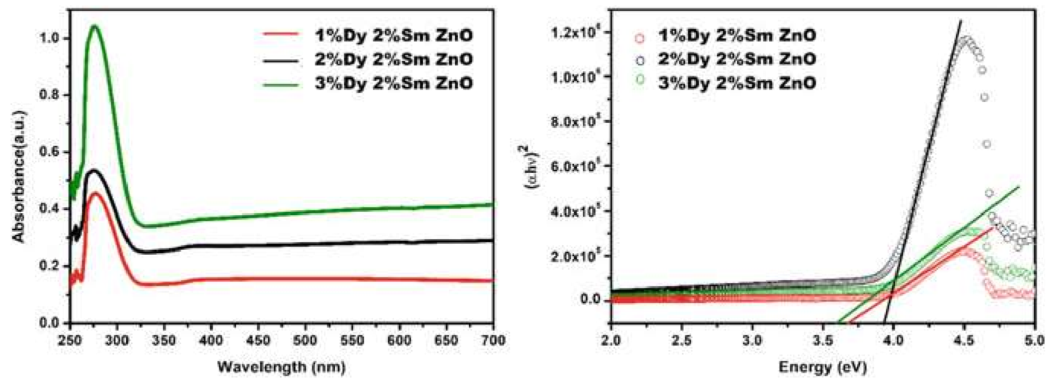

Fig. 2.4 UV-Vis absorbance spectra and Tauc's plot for Dy/Sm co-doped ZnO nanofibers

diameter of the nanofibers is in the range of 100 nm, the crystalline ZnO nanoparticles are assumed to be smaller than that and hence with the reduction of the diameter size the optical properties tends to enhance significantly [21]. Along with the blue shift in the absorbance maxima the band gap is enhanced tremendously with the reduction in the diameter size of nanofibers. With increasing dopant concentration, the band is found to be increased. For 2% Dy 2% Sm doped ZnO nanofibers the band gap is found to enhanced most and for further increasing dopant concentration the band gap starts reducing. This tailoring of band gap with dopant concentration could be explained by two terminologies, namely Burstein-Moss effect and other is Band gap narrowing. Due to increasing carrier concentration the Fermi level in the conduction band moves upwards, this is termed as Burstein-Moss effect whereas the latter occurs due to merger of the donor level and the conduction band above a critical carrier concentration termed as Mott density [22–24]. For optimal dopant concentration movement of Fermi level occurs, whereas when the concentration is increased further it results in concentration quenching.

2.3.5 Fourier Infrared Transform Spectroscopy (FTIR)

Figure 2.5 is the Fourier Transform Infrared Spectra for x% Dy 2% Sm doped ZnO nanofibers. All the samples confirms the presence of strong metal stretching bond Zn–O at around 450 cm^{-1} [25, 26]. This indicates the successful formation of ZnO in one dimensional nanofibers. Additional broad peak at 3500 cm^{-1} is observed. This resembles the presence of –OH which could be ascribed to the presence of ambient moisture retention [27].

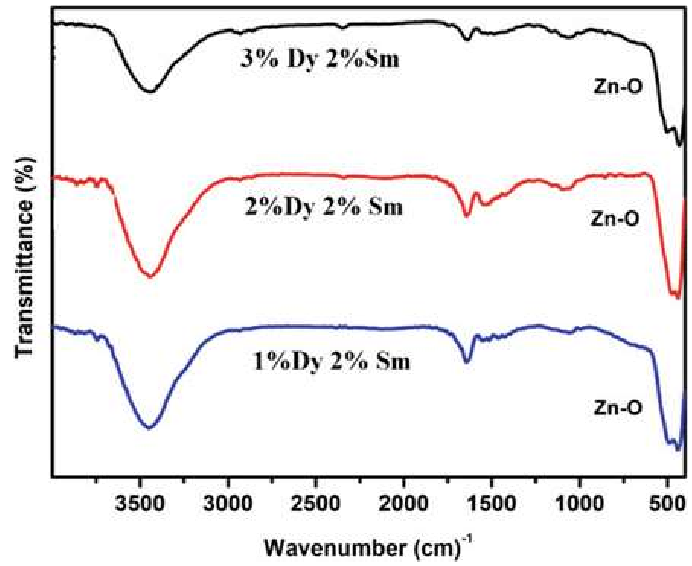

Fig. 2.5 FTIR spectra for x% Dy 2% Sm doped ZnO nanofibers

2.3.6 Photoluminescence

Figure 2.6 represents the photoluminescence spectra of dysprosium and samarium co-doped ZnO nanofibers. Figure 2.6a is the PL spectrum for 1% Dysproium and 2% Samarium doped ZnO nanofiber. It posses a NBE from host ZnO [28] and two characteristic emission from Samarium following $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition and $^4G_{5/2} \rightarrow {}^6H_{9/2}$ transition [29]. No peak related to Dysprosium emission is observed. Thus, the non-radiative energy transfer from Dy^{3+} ions to Sm^{3+} ion may had occurred. Figure 2.6b is the PL spectrum for 2% Dysprosium 2% Samarium doped ZnO nanofibers. It clearly possesses the NBE emission accompanied by the characteristic peaks for Dysprosium and Samarium [30]. Also, the intensity of the $^4G_{5/2} \rightarrow {}^6H_{9/2}$ transition is bit increased. Thus, dopant Dysprosium acts as the activator ion which sensitizes Samarium intensity. While Fig. 2.6c is the PL spectrum for 3% Dysprosium 2% Samarium doped ZnO nanofibers. Here no emission peak related to either of the dopant is observed. This led to the discussion that with the increase

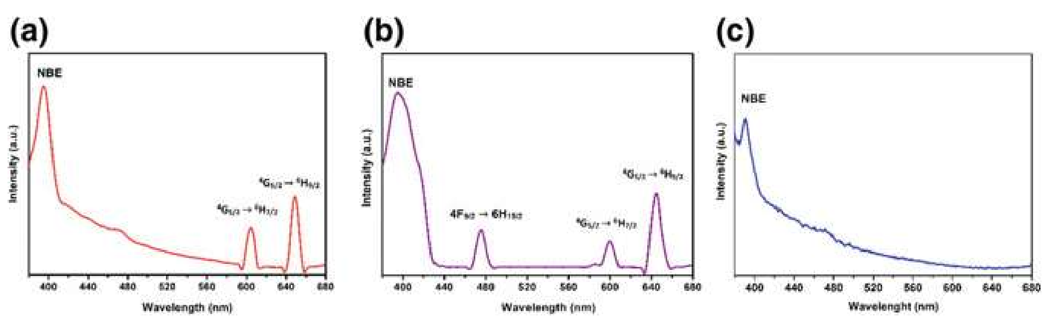

Fig. 2.6 Photoluminescence emission spectra of **a** 1% Dy 2% Sm doped ZnO, **b** 2% Dy 2% Sm doped ZnO, and **c** 3% Dy 2% Sm doped ZnO nanofibers

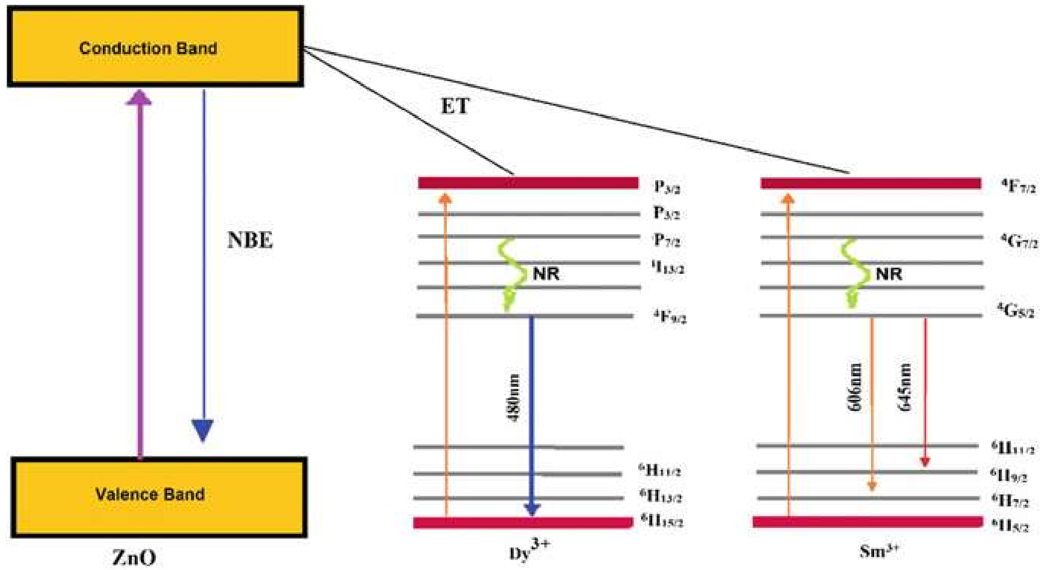

Fig. 2.7 Energy Transfer mechanism of host ZnO and dopant ions Dy^{3+} and Sm^{3+}

in doping concentration of Dysprosium characteristic peak for Dysprosium emerges but upon further increase the peak related to any of the dopants diminish. This could be explained as concentration quenching effect. Since a greater number of dopants when exists the energy is transferred from activator to activator and ultimately the energy is lost and does not result in effective emission from the dopant [31].

Figure 2.7 depicts the Energy transfer mechanism within the host ZnO and dopant ions Dy^{3+} and Sm^{3+}. Upon excitation the host ZnO gets excited to the conduction band. This excited ZnO then relaxes via two paths i.e. one is returning back to ground state and hence emitting into UV region and other is by transferring its energy to its dopants. This is referred and Energy transfer mechanism. The excited dopant ions then come back to ground state while emitting light in their characteristic region. Figure 2.8 illustrate the Commission Internationale de l'Elairage diagram (CIE) for x% Dy 2% Sm doped ZnO nanofibers. The CIE parameters such as colour coordinates, colour co-related temperature (CCT), luminescence efficacy of radiation (LER) and colour rendering Index (CRI) were calculated to know the photometric characteristics of the prepared nanofibers [32]. Colour coordinates (x, y) are one of the important parameter for evaluating luminescent materials performance. From chromaticity diagram, it is seen that the colour co-ordinates travel from warm orange white region to cool blue white region as the percentage of Dy increases from 1 to 3% while keeping 2% Sm fixed doping in ZnO. Thus, it could be clearly stated that the colour tunability can be achieved by varying the dopant concentrations and variety of dopants. It is evident from obtained values of CCT, CRI and LER as given in Table 2.1, ensures the use of these light emitting nanofibers in Light emitting devices, smart and technical textiles.

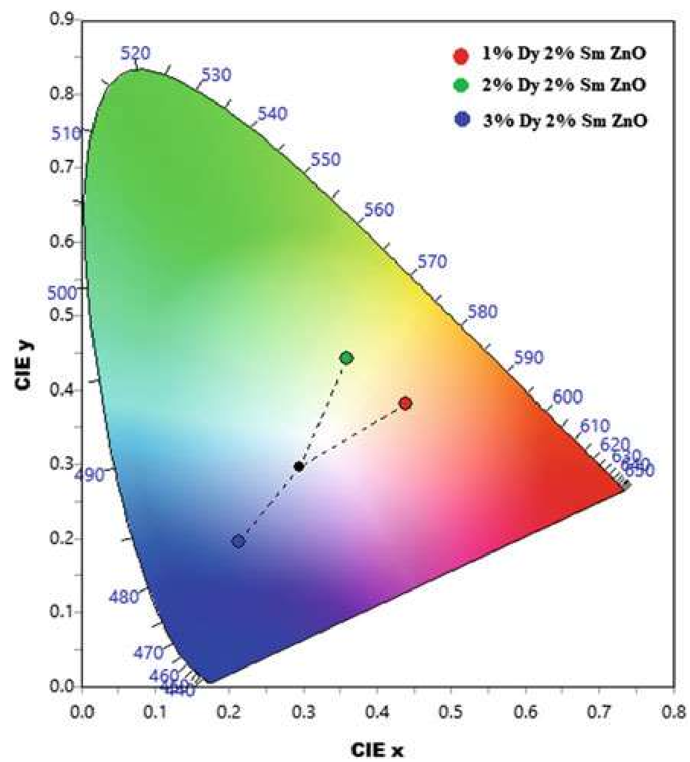

Fig. 2.8 CIE plot for Dy/Sm co-doped ZnO nanofibers

Table 2.1 CIE parameters for Samarium Dysprosium co-doped ZnO nanofibers

Samples	CIE x	CIE y	CRI	CCT	LER (lm/W)
1% Dy 2% Sm doped ZnO nanofibers	0.3592	0.4423	68	4857	289
2% Dy 2% Sm doped ZnO nanofibers	0.4389	0.3811	68	2771	241
3% Dy 2% Sm doped ZnO nanofibers	0.2135	0.1963	NA	NA	115

2.4 Conclusions

Dy–Sm co-doped ZnO nanofibers with diameters in the range 100–180 nm have been fabricated successfully by electrospinning. Photoluminescence spectra clearly depicts the energy transfer mechanism within the host ZnO and dopants Sm and Dy wherein CIE parameter confirms the colour tunabilty of co-doped ZnO nanofibers. Enhanced visible light luminescence transverse from warm orange white region to cool blue white region is observed in CIE diagram with increasing dopant Dy concentrations. Thus, it could be clearly stated that the colour tunability can be achieved by varying the dopant concentrations and variety of dopants. Such materials can be a very good optimum candidate for colour tunable luminescent light emitting fabrics.

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