RJC

RASĀYAN *J. Chem.*
Vol. 10 | No. 1 |298 -304 | January - March | 2017
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

STRUCTURAL AND OPTICAL CHARACTERIZATION OF ORGANIC LIGHT EMITTING DIODES

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ABSTRACT

A new type of organic light-emitting diodes (LEDs) has been fabricated, by extracting the Ruthenium (III) chloride and complexed with polyvinyl alcohol (PVA) by sol-gel technique. Various Spectroscopic techniques like X-ray Diffraction (XRD) studies, photoluminescence (PL) and Ultra Violet Visible (UV) spectroscopy were carried-out for the obtained LED samples. The structural analysis of an organic LED showed polycrystalline nature. Photoluminescence studies have been performed through Spectroscopy in the wavelength range 430-1000 cm^{-1}. The optical properties of ligands were analyzed by UV-visible spectroscopy in the wavelength region 200-800 cm^{-1}. These results find potential applications as organic light-emitting diodes (OLEDs), light-emitting electrochemical cells and solid-state organic lighting devices.

Keywords: Ruthenium (III) chloride, sol-gel, PVA, XRD, PL and UV-Visible.

INTRODUCTION

In present scenario, an attractive attention has been made towards organic light-emitting diodes (OLED) devices using organic materials, which are widely used in many applications[1]. In fact, organic material based devices have great potential rather than inorganic material based devices. In present aspects a tremendous effort has been made for developing a new kind of materials in order to improve the efficiency of phosphorescent dyes in OLEDs[2]. Organic materials are used in many applications such as display devices, solar cells, chemical sensors, solid state OLEDs etc[3]. The OLEDs are prepared by inter-relating the nanoparticles in the polymer matrixes[4]. The nanoparticles are incorporated in the polymer nanocomposites in such a way an interaction takes place with polymer matrix directly there by enhancement of the luminescence properties.

The inherent properties of organic materials make them well suited for fabrication on a flexible substrate. Recently, step growth interest has been made to investigate OLEDs based thin films of polymers. By stacking of these materials properly, one can design a device with high efficiency and long lifetime[5]. The core for display devices has a matrix combination of red, green and blue, which facilitate mass scale production and large internal area with low cost[6]. The charge carrier barrier is continuously reduced by increasing the doping level of materials. The prompting usage of doping levels allows scientists to quickly screen for the optimum electronic devices[7].

The display phenomenon involve in OLED devices is the hole or electron ejection layer of electroluminescence, the efficiency of the light output increase while reducing the operating voltage. Owing to the technical advantages, OLED has been considered as the promising material for flat panel display technology due to its excellent behavior characteristics such as self-emission, full color capability and flexibility[8]. Polyvinyl alcohol (PVA) is used for the preparation of these LEDs due to its excellent polymeric behavior with high tensile strength and good in mechanical, thermal and electrical properties, low permittivity. PVA is having good chemical, potential resistance and has very high dielectric strength, thus it can be used in good charge storage capacity and dopant-dependent electrical and optical properties.

Rasayan J. Chem., 10(1), 298 -304(2017)
http://dx.doi.org/10.7324/RJC.2017.1011621

PVA is freely water soluble poly hydroxyl polymer which is dependent on the degree of polymerization and alcoholysis. Due to its physical, chemical and mechanical properties it is widely used in many industrial and many practical applications such as adhesives, textiles, pharmaceutical and biomedical industries due to non-toxic and biogradable properties[9]. Rao et al. published their results on different materials in the earlier studies[10-68]. In the present study reduced ruthenium nanoparticles are incorporated in polymer matrix which results as an intermediate medium in OLEDs.

EXPERIMENTAL

Chemicals Required

$RuCl_3\cdot 3H_2O$,2,2'-dipyridyl,NaH_2PO_2, $NaBF_4$, was obtained from Sigma Aldrich India Ltd., PVA polymer was purchased from Sigma Aldrich chemicals India Ltd.(with average MW 124,000-186,000) and H_3PO_2 (50%),NaOH pellets, with 98 % purity were obtained from Loba chemicals India are used in the present investigation without any further purification.

Synthesis of $[Ru(bpy)_3](BF_4)_2$

Initially 10 ml of 50 % H_3PO_2 (Hypo phosphorous acid) is added to 6 ml of water and stirred slowly, then NaOH pellets (3.9g) is added until it is neutralized.0.083 g $RuCl_3\cdot 3H_2O$ is added to 8 ml of water. Add 0.188 g 2, 2'-dipyridyl to the solution and then 0.44 ml of NaH_2PO_2.Cover the beaker with a watch glass and reflux for half an hour. 0.333 g of $NaBF_4$ is added to the solution which was dissolved in 1.5 ml of water. Let the complexed solution is placed in a cool ice tub for about 2°C, until the crystals were formed. Later the obtained crystals were centrifuged to get the $[Ru(bpy)_3](BF_4)_2$ which was rinsed with ethanol.

Preparation of OLED

Add 0.30 g PVA is taken in 10 ml of water and stirred well until the transparent solution is obtained. Dissolve approximately 0.035 g $[Ru(bpy)_3](BF_4)_2$ in 3 ml of PVA solution, such that the rubidium is incorporated in the polymer matrix.

In the final step, the obtained solution of $[Ru(bpy)_3](BF_4)_2$with PVA is placed as a layer on ITO glass and dried the layer with the help of a hair drier, trying to keep some uncoated regions at the edges. Indium or gallium alloy is spotted on a layer which is acting as an active metal electrode. Apply positive lead to 4.5 volt power supply around the uncoated ITO glass slide and negative lead to the gallium-indium until the OLED will emit light. The samples were fabricated with different wt% concentrations referred as (a) $0.035[Ru(bpy)_3](BF_4)_2$+0.30g PVA (S1), (b) $0.035[Ru(bpy)_3](BF_4)_2$ +0.35 g PVA (S2) and (c) $0.035[Ru(bpy)_3](BF_4)_2$ +0.40 g PVA (S3).

RESULTS AND DISCUSSION

XRD Analysis

X-ray diffraction studies were performed by using a Philips analytical X'pert diffractometer, with the wavelength of 1.5403 Å and the diffraction peaks were recorded in between 10° and 90° diffraction angles. The crystallinity of the polyquinolines show a wide diffraction peak observed at 38°, as shown in Figure-1. This diffraction reveals the polycrystalline nature of the polymer. A broad peak is obtained due to the amorphous component, while the diffracted sharp peaks obtained are due to the crystallinity region. Our results are similar to the earlier reported XRD results of $Ir(ppy)_3$ and $Ir(btp)_3$ powder samples[69].

Photoluminescence Studies

Photoluminescence studies were carried-out for Ruthenium based OLED with the help of Spectra Aco at room temperature as shown in Figure-2. The intensity of the peak in the wavelength region 900–930 nm is due to the interlinking of C=C bond formation. We believe that the wavelength bands on the long wavelength side are associated with spin–orbit coupling enhanced π–π^* transitions. It is noted that with the increase in wt% of PVA in the sample the intensity of the peak is increasing and found better for the 0.035 $[Ru(bpy)_3]$ $(BF_4)_2$ +0.40 g PVA (S3) sample. The highly intense PL emission peak of 0.035 [Ru (bpy)$_3$] (BF$_4$)$_2$ +0.40 g PVA (S3) was located in the red region with the λ_{max} value at 925 nm. However the emission peaks of the complex [Ru(bpy)$_3$] (BF$_4$)$_2$+ PVA show clear differences. The peak shift can be attributed due to the excitation of molecules.

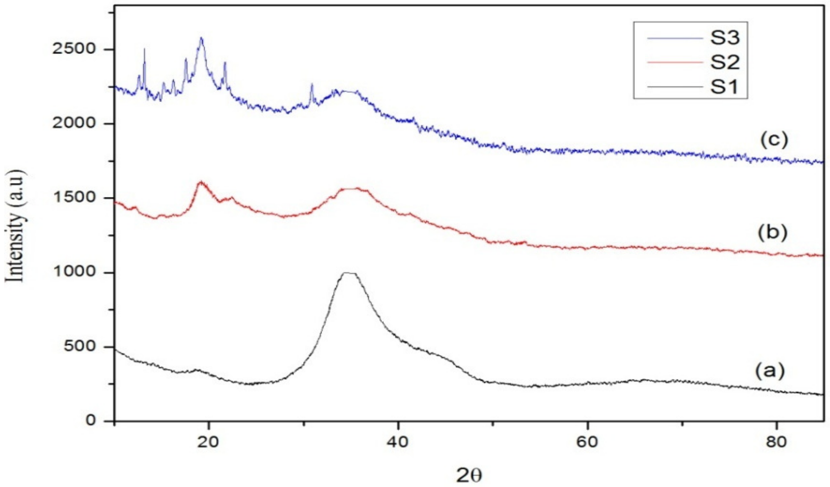

Fig.-1: XRD spectra of (a) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.30 g PVA (S1); (b) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.35 g PVA (S2) (c) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.40 g PVA (S3)

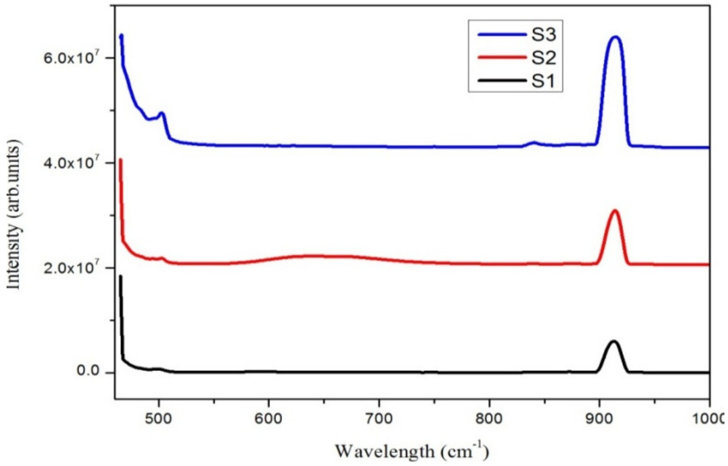

Fig.-2: PL spectra of (a) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.30 g PVA (S1) (b) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.35 g PVA (S2) (c) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.40 g PVA (S3)

UV-visible Spectroscopy

UV-vis spectroscopy is an important tool which is used to identify the temperature dependence vibrational bonds and characteristic vibrational modes in an energy level. Figure-3 shows the UV-visible absorbance spectra of Ruthenium (Ru). This spectra shows an absorption peak at 550 nm which corresponds to $\pi - \pi^*$ transition of the C = C bond of Ru which is observed in Figure-4. These results attributed to surface plason bond where the electrons are exited from energy level.

Optical Properties

Optical analysis is used to identify the optical band gap of the materials in the transmitting radiation. In an energy level a photon is absorbed, when an electron jumps from lower energy level to higher energy level. Transition takes place in band gap energy as it rises in the absorption process called absorption edge from where the optical band gap energies are determined. Absorption coefficient (α) was calculated from the absorbance (A) value by using the following equitation:

$$I=I_o \exp(-\alpha x) \quad (1)$$

Hence,

$$\ln(I_o/I) = \alpha x, \qquad \alpha=2.303(A/x) \quad (2)$$

Insulators and semiconductors are classified in to two types (i) direct band gap and (ii) indirect band gap. If the top of the valence and bottom of the conduction band are same then direct band gap exists, where as in indirect band gap exists if the top of the valence and bottom of the conduction band are not same. The indirect band gap transitions takes place from valence band to conduction band which is accumulated with phonon of magnitude of crystal momentum[70]. The direct, indirect band gaps and absorption edge values are shown in Table-1.

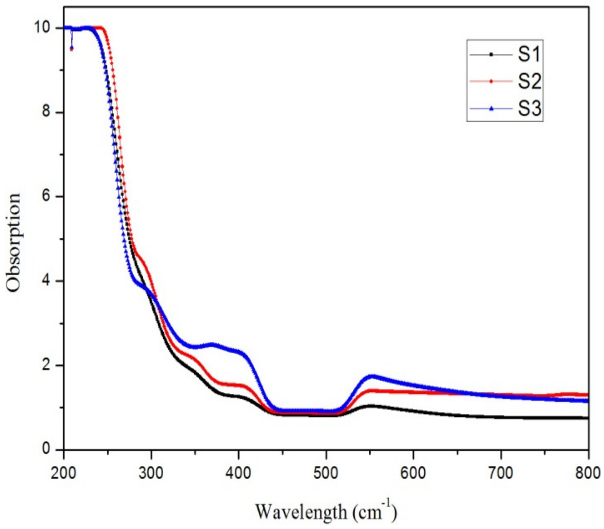

Fig.-3: UV-visible spectra of
(a) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.30 g PVA (S1)
(b) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.35 g PVA (S2)
(c) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.40 g PVA (S3)

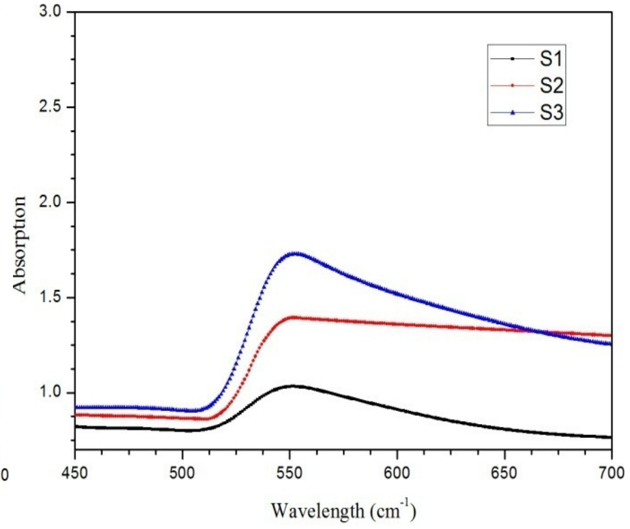

Fig.-4: Absorption peak of
(a) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.30 g PVA (S1)
(b) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.35 g PVA (S2)
(c) 0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.40 g PVA (S3)

Table-1: Energy band gap values for different compositions of polymer films

Polymer electrolyte	Optical band gap		Absorption edge
	Direct (eV)	Indirect (eV)	
0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.30 g PVA (S1)	4.9	2.8	2.7
0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.35 g PVA (S2)	4.5	2.9	3.1
0.035[Ru(bpy)$_3$](BF$_4$)$_2$ +0.40 g PVA (S3)	4.2	3.1	3.4

To calculate band gap energy values, graphs were plotted between α, $(\alpha h\upsilon)^2$ and $(\alpha h\upsilon)^{1/2}$ as a function of *hυ*. The linear portion of α versus *hυ* curves to zero absorption value. Absorption value for 0%, 5 %, 10 %, weight ratios of [Ru(bpy)$_3$](BF$_4$)$_2$ doped PVA lies at 2.7, 3.1 and 3.4 eV, which is shown in Figure-5. In the case of direct bandgap transitions, the absorption coefficient and energy of incident photon can be determined as follows[70,71]

$$\alpha h\upsilon= C\,(h\upsilon-E_g)^{1/2} \quad (3)$$

Where, E_g is the band gap, C is a constant dependent on the specimen structure, α is the absorption coefficient, υ is the frequency of incident light and h is Planck's constant. From the graph $(\alpha h\upsilon)^2$ versus $h\upsilon$, direct band gap values are obtained. Energy gap values are 4.9, 4.5 and 4.2 eV as shown in Figure-6. Whereas indirect band gap values are obtained by plotting $(\alpha h\upsilon)^{1/2}$ versus $h\upsilon$ as shown in Figure-7 and the values obtained from the graph are 2.8, 2.9 and 3.1 eV respectively.

In the case of indirect transitions, phonon assistance is required for transition where the absorption coefficient has the following dependence on photon energy[64,72].

$$\alpha h\upsilon = A\,(h\upsilon - E_g - E_p)^2 + B\,(h\upsilon - E_g - E_p)^2 \tag{4}$$

Where, E_p is the energy of the photon associated with the transition, A and B are constants depending on the band structure.

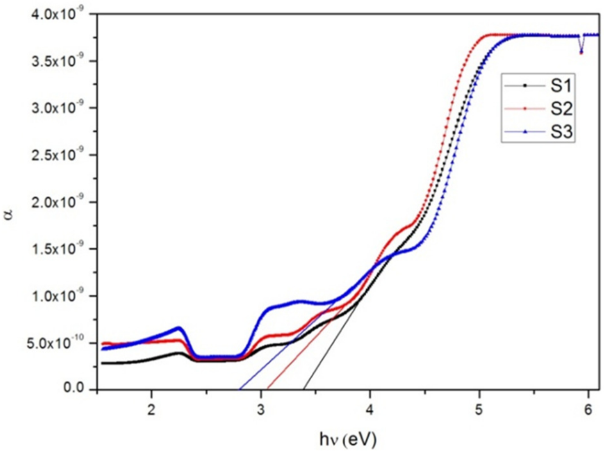

Fig.-5: α vs $h\upsilon$ plots of
(a) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.30 g PVA (S1)
(b) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.35 g PVA (S2)
(c) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.40 g PVA (S3)

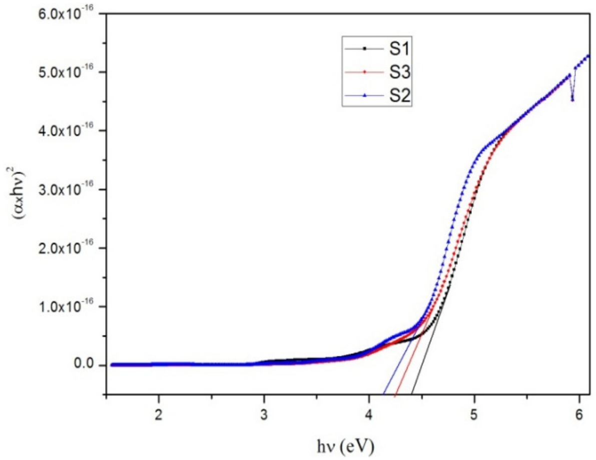

Fig.-6 α vs $(\alpha h\upsilon)^2$ plots of
(a) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.30 g PVA (S1)
(b) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.35 g PVA (S2)
(c) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.40 g PVA (S3)

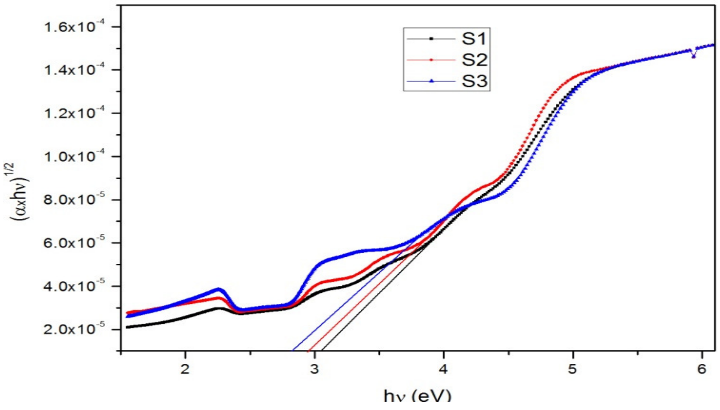

Fig.-7: α vs$(\alpha h\upsilon)^{1/2}$ plots of (a) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.30 g PVA (S1)
(b) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.35 g PVA (S2) (c) 0.035$[Ru(bpy)_3](BF_4)_2$ +0.40 g PVA (S3)

From the obtained values it is clear that due to incorporation of small amount of dopant in a host lattice results in the decrease of activation energy in polymer chain. The direct and indirect band- gap values are shifted to lower energiesdue to the doping of Ruthenium in PVA polymer.

CONCLUSION

OLEDs were prepared with the combination of $[Ru(bpy)_3](BF_4)_2$ + PVA by sol-gel method. The structural analysis of sharp diffraction peak is observed at 38^o, which reveal the polycrystalline character of the synthesized polymeric complexes. Photoluminescence studies revealed the intensity of peak in the wavelength region 900–930 nm is due to the interlinking of bond formation between π–π^* transition. Optical absorption data concludes the lower band gap values are formed due to the strong interaction of vibrational molecules between C=C bonds.

ACKNOWLEDGEMENT

One of the authors (G. Sunita Sundari) is thankful to Dr. K. Satyanarayana, President, Dr. M. Rama moorthy (Chancellor), Dr. L.S.S Reddy (Vice chancellor), Prof. K.L.Narayana (Academic Dean) and Prof. B. Jay Kumar Sing (Associate Dean, Sponsored and Internal Funded Research) of K. L. University for providing the Internal fund and given their constant encouragement for completion of Internal project. The author sincerely thanks Dr.B. Mahendran, Department of Biotechnology K.L.University for providing Lab facility.

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[RJC-1621/2017]