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SYNTHESIS AND CHARACTERIZATION OF BIS (2 METHYL-8-HYDROXYQUINOLINE) LEAD NANOPARTICLES FOR ORGANIC LIGHT EMITTING DIODE APPLICATIONS

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In this work, Bis (2-methyl 8-hydroxyquinolate) Lead nanoparticles were synthesized by a simple chemical precipitation method. The crystalline nature of the Pb(mq_2) nanoparticles was characterized by powder X-ray diffraction (PXRD) analysis. The morphology and the elemental compositions were analyzed by High-resolution scanning electron microscopy (HRSEM) and Energy dispersive X-ray analysis (EDAX). The various functional groups of the synthesized samples were confirmed by Fourier transform infrared spectroscopy (FTIR) analysis. The thermal stability of the Pb(mq_2) nanoparticles was studied using TGA and DSC. The optical properties of the Pb(mq_2) nanoparticles were studied by UV–Vis absorption spectroscopy. The luminescence property of the synthesized particles was observed from the Photoluminescence spectrum confirm the possible application in Organic Light Emitting Diode.

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Keywords: Pb (mq_2) nanoparticles, HRSEM, EDAX, TGA &DSC, and Photoluminescence spectrum

1. Introduction

The organic light emitting diodes (OLEDs) are tending to cause to approach much significance on account of their possible used for solid state lighting and flat panel displays [1]. The optoelectronic engineering is to revamp in accordance with lighting sources such as blazing and fluorescent lighting with ample power efficient semiconducting light sources [2]. OLED includes many advantages such as easy manufacturing, low cost, brightness, low power usage etc. OLED covers a lot of vital pliabilities and capabilities for the color tune. OLEDs become an ultimate modern light source because of these properties [3]. Very few attempts have been made on organic functional nanomaterials to investigate the organic and inorganic materials. The synthesis of organic materials, therefore, draws attention to opening up a simple and prompt method with the same sizes and novel properties. Metal 2 methyl 8 hydroxyquinoline chelates affect a large area have been studied due to their application in photoluminescence, electroluminescence and so on [4-10]. In this paper, a simple chemical precipitation method is used to report the synthesis of Pb(mq_2) nanoparticles. PXRD, HRSEM, EDX, UV-Vis absorbance and PL analysis confirmed the prepared Pb(mq_2) nanoparticles.

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2. Materials and methods

2.1. Materials

Lead (ii) acetate, 2 methyl 8hydroxy quinoline, and ethanol were used as starting materials. All these chemicals were of high purity and no further purification was done.

2.2. Synthesis of Pb(mq_2) Nanoparticles

The Pb(mq_2) nanoparticles were synthesized by simple precipitation method using Lead acetate, 2-methyl 8-hydroxyquinolate and ethanol in the stoichiometric ratio 1:2:1. A solution of 2-methyl 8-hydroxyquinolate (2.9 g) was prepared using the solvent of ethanol (40 ml). The solution was stirred using a magnetic stirrer, for 1 hour, at room temperature to attain homogenization reaction. An aqueous solution of lead acetate (2.095 g) in 50 ml of water added dropwise to the reaction mixture, which yields a yellowish precipitate. The yellow precipitate is separated from the reaction mixture and dried at 60°C in a vacuum oven for 6 hours.

2.3. Characterization techniques

The powder X-ray diffraction pattern (PXRD) of the synthesized sample was performed with CuK_{α} radiation on the Rich Siefert 3000 diffractometer (λ = 1.5406 Å). The FTIR spectrum is recorded in the range of 4000-500 cm^{-1} using a BRUKER 66 V FTIR spectrophotometer with the KBr pellet. The TG-DSC thermograms of the sample were recorded using NETZSCHSTA 449F3 under a nitrogen atmosphere at a heating rate of 10°C/min. The optical absorption spectrum of the sample was taken in the range between 200-800 nm using double beams CARY 5E UV-Vis-NIR spectrometer. The photoluminescence study was done using a JOBIN YVON FLUROLOG-3-11 spectrofluorometer with an excitation wavelength of 300 nm.

3. Results and discussion

3.1. Powder X-Ray diffraction analysis

The XRD analysis of crystalline compounds provides a diffraction pattern consisting of a well-defined, narrow, sharp, and significant peak while amorphous materials do not give clear peaks, rather the pattern has noise signals, smeared peaks, or some short-order bumps may have. Powder XRD can be used to determine crystallinity by comparing the background pattern's integrated intensity with that of the sharp peaks. Hence, the XRD pattern of Pb(mq_2) nanoparticles is shown in Fig.1. The strong peaks are observed and its depicted in Fig.1. The sharp peaks corresponding ascertaining the phase of the lead complex. In addition, the sharp peaks show that the obtained product has a high crystalline nature.

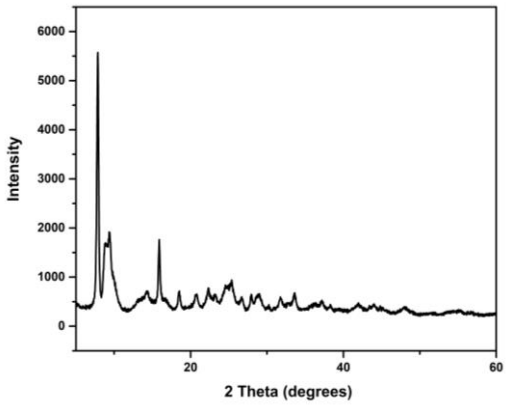

Fig. 1. Powder XRD pattern of Pb(mq_2) nanoparticles

3.2. HRSEM and EDAX analysis

The surface morphology of the Pb(mq_2) nanoparticles was analyzed using HRSEM. Fig.2(a)shows the High-Resolution scanning electron microscope images of Pb(mq_2) nanoparticles. It is clearly showed that the as-prepared products were mainly composed of spherical and rod-shaped nanoparticles. One of the spherical nanoparticles is about 421.4 nm breadth and 555.1 nm length as determined. Energy Dispersive X-ray (EDAX) study was carried out for looking at the elemental composition of the synthesized samples and for the confirmation. Fig.2(b) shows the EDAX spectrum of Pb(mq_2) nanoparticles which confirmed the presence of the elements of Lead, carbon, oxygen, and nitrogen.

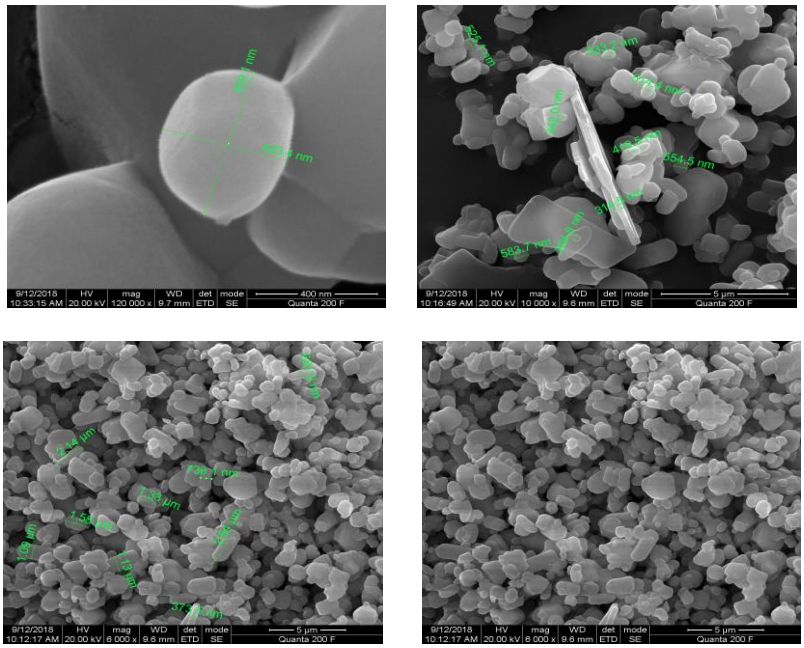

Fig. 2. (a) SEM Images of Pb(mq_2) nanoparticles

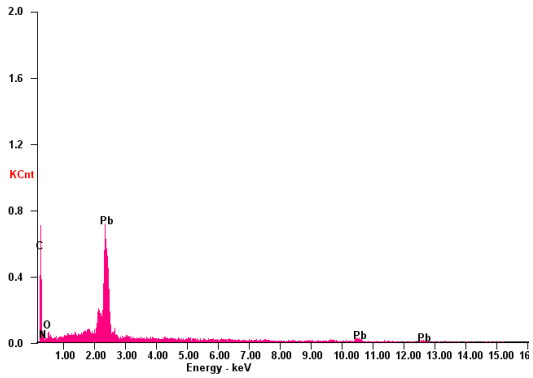

Fig.2. (b) EDAX pattern of Pb(mq_2) nanoparticles

3.3. FTIR analysis

The FTIR spectrum of Pb(mq_2) nanoparticles is shown in Fig.3. The vibrations at 3045 cm^{-1} and 2925 cm^{-1} illustrate the presences of O-H stretching vibration of water. The vibration at 1569 cm^{-1}, 1420 cm^{-1}, and 1340 cm^{-1} are assigned to the quinoline group of Pb(mq_2) [11]. The band at 1420 cm^{-1} corresponding to the presences of phenyl group [12]. The peaks at 842$cm^{-1}$and 713cm^{-1} are associated with C-H deformation vibrations [13]. The peaks between the ranges 1000-

400 showed the bending of phenyl group [14]. These results confirm the execution of quinoline ring formation and manifestation of quinoline structure in the metal complex.

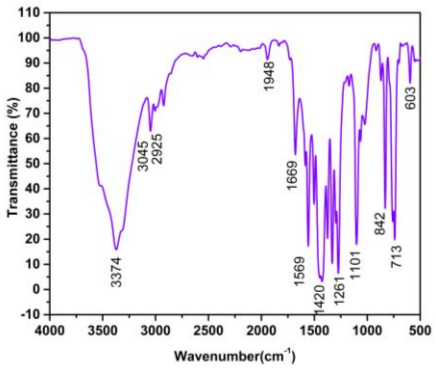

Fig. 3. FTIR spectrum of Pb(mq_2) nanoparticles

3.3. Thermal analysis

TGA analysis of the prepared material was done by the temperature range from 100 °C to 1000 °C shown in Fig. 4 (a). The TG thermogram shows two stages of weight loss of Pb(mq_2). The first stage occurs at to 85.9°C and ends at 112°C with the weight loss of 3.57 % due to moisture or impurities present in the material Pb(mq_2) nanoparticles. The second stage occurs from to 229.8 °C to 288.9°C with the weight loss of 41.82%. From the graph, it is clear that the sample is stable up to 229. 8°C.In the corresponding DTG curve of the Pb(mq_2) nanoparticles the exothermic peak which was observed at the 100.8, 241 and 280°C. Temperature range can be assigned to the dehydration of the correlated water molecules of the intercalated complex [15]. The first exothermic peak observed in DSC thermogram at 100.8°C coincides with the first stage of weight loss of Pb(mq_2) and the second exothermic peak in DSC at 241°C coincide with the second stage of weight loss of Pb(mq_2) and decomposition of the sample at 288.9 °C was observed [16].

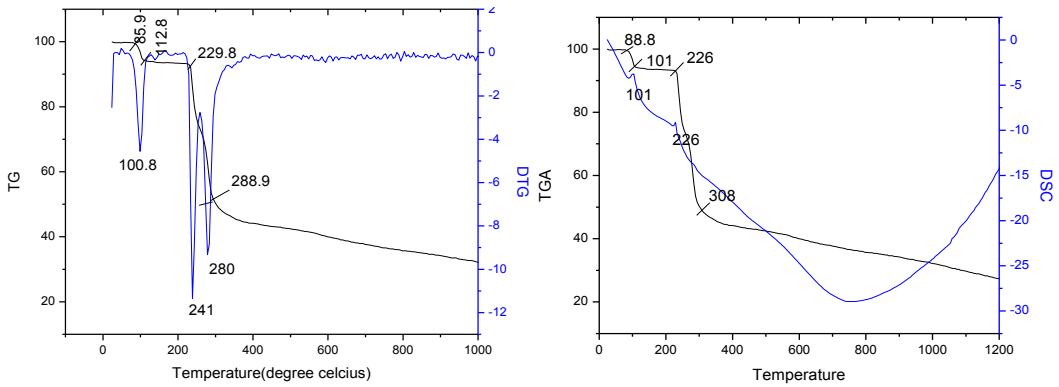

Fig. 4. Analysis of Pb(mq_2) nanoparticles
(a) TGA-DTG, (b) TGA-DSC

DSC analysis of the material was done by the temperature range from 100°C to 1200°C shown in Fig.4 (b). The first exothermic peak observed in DSC thermogram at 101°C coincides with the first stage of weight loss of Pb(mq_2) and the second exothermic peak in DSC at 226°C coincide with the second stage of weight loss of Pb(mq_2) and decomposition of the sample at 308°C was observed [17].

3.4. UV-Vis spectral analysis

One of the best methods for determining impurities in organic molecules is UV absorption spectroscopy. Impurities can also be detected by measuring the absorbance at a specific wavelength. UV spectroscopy is useful in the elucidation of the structure of organic molecules, the presence or absence of unsaturation, the presence of heteroatoms and aromatic compounds. Therefore, the UV-Vis absorption spectrum of Pb(mq_2) nanoparticles is shown in Fig.5. From this absorption spectrum, the maximum of the UV-Vis absorption peak was observed at 373 nm, which is due to the π-π* transition of the aromatic ring and the band gap value was found to be 3.32 eV.

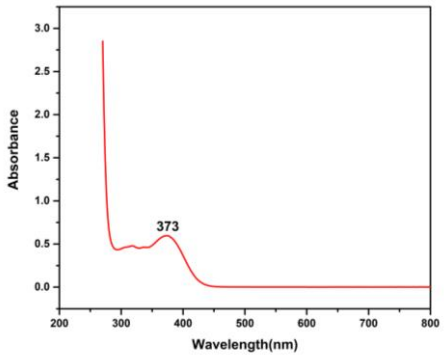

Fig. 5. UV-Vis absorption spectrum of Pb(mq_2) nanoparticles

3.5. Photoluminescence analysis

The photoluminescence (PL) emission spectrum of Pb(mq_2) nanoparticles is shown in Fig.6 at the excitation wavelength of 380 nm. Two emission peaks are observed at 410 nm and 433 nm in the bluish-violet region of the spectrum. The emission peak at 433 nm corresponds to the band-edge emission. The emission wavelengths of the Lead complexes are dependent on their size. When the particle size decreases, the effective band gap increases, therefore the emitted photon has comparatively higher energy giving photoluminescence peak at a shorter wavelength.

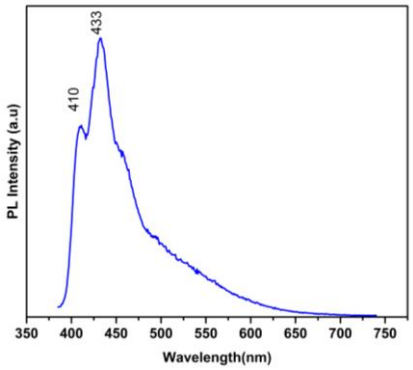

Fig. 6. Photoluminescence spectrum of Pb(mq_2) nanoparticles

4. Conclusions

The Pb(mq_2) nanoparticles were synthesized by chemical precipitation method. The crystal structure, morphology, absorption, functional groups and emissive properties of Pb(mq_2) nanoparticles were studied by PXRD, HRSEM, UV-Vis, FTIR, and PL analyses. The XRD analysis confirms that the crystalline nature of the Pb(mq_2) nanoparticles. The relatively sharp

absorption indicates the narrow size distribution of nanoparticles. HRSEM confirms the spherical and rod-like the structure of $Pb(mq_2)$ nanoparticles. The measurement of EDAX shows the presence of elements Pb, C, O and N. The different functional group is identified using FTIR analysis. The TGA and DSC analysis of the powder sample suggests that the sample is stable up to 288.9°C. The photoluminescent spectrum showed a prominent peak around 433 nm which indicated a strong PL emission in the bluish-violet region. The observed optical properties of the $Pb(mq_2)$ nanoparticles suggest that this compound is a potential candidate for application in organic light emitting devices (OLEDs).

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