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Structural, Optical and Photocatalytic Activity of Polymer Capped Zinc Selenide Nanoparticles

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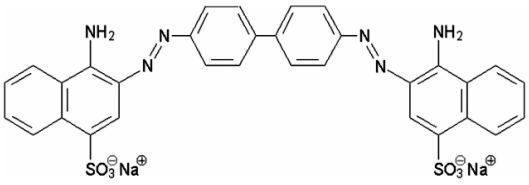
Abstract - *In the present paper study, ZnSe nanoparticles have been successfully synthesized by hydrothermal method using polyvinyl alcohol (PVA) as the capping agent. The photocatalytic degradation reaction of azo dye was studied using polymer capped nanoparticles. The nanoparticles were characterized by XRD, SEM with EDAX, AFM, HRTEM and UV-vis absorption spectroscopy. The XRD results revealed that the hexagonal wurtzite structure of the zinc selenide nanoparticles. Furthermore, the morphology of the nanoparticles was obtained from SEM micrographs and elemental composition was also obtained from EDAX spectrum. The band gap energy of the material was calculated from UV-visible absorption spectroscopy. The effects of various parameters such as contact time, amount of adsorbent, initial concentration of dye and pH of the dye on degradation have been determined. The percentage removal of dye was calculated by using UV-visible spectrophotometer.*

Key Words: Polymer, Hydrothermal, Azo dye, Photocatalyst, Nanoparticles, Capping agent.

1.INTRODUCTION

Colour is considered as the first pollutant to be identified in wastewater and it is mostly caused by the effluents discharged from dyeing industries such as paper, rubber, leather, cosmetics, textile, pharmaceuticals, plastics and food industries [1]. The Azo dyes one of the important classes of synthetic organic pigments utilize in manufactured coloured productions Worldwide for this reason the Organic dyes appear in many industrial effluents [2]. When these coloured effluents enter rivers or any surface water system they upset biological activity. Ground water systems are also affected by these pollutants because of leaching from the soil. In addition, many dyes are difficult to degrade due to their complex aromatic structure and they tend to persist in the environment and creating serious water quality and public health problems such as allergic dermatitis, skin irritation, cancer and mutation [3] and teratogenic effects on aquatic life and humans [4]. Hence, the removal of dyes from wastewater is essential to prevent continuous environmental pollution.

Congo red (CR) is an azo dye; it is toxic to many organisms and is a suscepted carcinogen and mutagen. It is a benzidine – based anionic disazo dye. Benzidine and congo red are, however, banned in many countries because of health concerns. But, it is still widely used in several countries. It also represents a significant effluent problem along with related dyes from textile, printing and dyeing, paper, rubber and plastic industries. Its structural stability makes it highly resistant to biodegradation and obviously its bright color and toxicity are entirely undesirable in the environment [5]. Structure of Congo red (CR) dye is shown below:



Congo red

Due to the high concentration of organics in the effluents and the higher stability of modern synthetic dyes, the conventional biological methods are ineffective for the complete colour removal and degradation of organics and dyes [6]. Therefore, many physicochemical methods such as adsorption, coagulation, precipitation, filtration and oxidation have been attempted for dye removal. The adsorption process has been found to be the most effective [7]. In order to decrease damage caused by organic dye pollution to environment and humans, the use of photocatalyst to degrade organic compounds in contaminated air or water or to convert them into harmless chemicals has been extensively studied [8].

Semiconductor nanoparticles are very important materials due to their tunable electrical and optical properties [9–12]. Their physical properties depend on their crystallite sizes [13-15]. ZnSe is an important material in shorter wavelength applications. It has wide range of applications in blue laser diodes, optical instruments [16-17], high speed optical devices, photovoltaic and laser screens [18] due to wide band gap (2.7 eV), wide transmittance range (0.5–2.2 μm) [19–21], high luminescence efficiency [22-23], low absorption coefficient and excellent transparency to infrared radiations.

Recently, semiconductor – assisted photo-catalysis has been extensively investigated mainly due to its capacity to degrade a large number of recalcitrant chemicals in aqueous system. In this work, we have prepared polymer

capped zinc selenide nanoparticle for the degradation of congo red dye under UV and visible light irradiation. In our previous work, we have reported the synthesis and characterization of polymer capped zinc selenide nanopartilce [24].

2. Experimental method:

2.1. Preparation of polymer solutions:

The PVA solution was prepared by adding 1 g of PVA into 50 ml of deionised water and heated at 90°C for 1 hour under constant stirring to obtain a viscous transparent solution.

2.2. Synthesis of different polymer capped Zinc selenide nanoparticles:

In order to synthesis different polymer capping ZnSe nanostructure materials by hydrothermal route, Zinc chloride [0.2M $ZnCl_2$], Aqueous ammonia [25% NH_3], Selenium dioxide [0.1N SeO_2], Polyvinyl alcohol [PVA]. The PVA act as polymer capping agent. A 20 ml amount of 0.2M $ZnCl_2$ aqueous solution was mixed with 20 ml of ammonia solution(25% NH_3) under continuous stirring then 20 ml of PVA solution and SeO_2 (0.1N) were added into the mixed solution. The pH is 9 using sodium hydroxide solution. After one hour of stirring, the resulting reaction mixture was transferred into a Teflon-lined stainless autoclave. The autoclave was sealed and maintained in an electric oven at 180°C for two hours and then cooled to room temperature. The precipitates were carefully collected and washed with deionised water and absolute ethanol several times and then dried.

2.3. Photocatalytic Activity:

The photocatalytic activity of the polymer capped zinc selenide nanoparticles was evaluated by photodegradation of an aqueous CR (Congo red) textile dye under UV and Visible light irradiation. The experiment was carried out in a cylindrical double walled hollow photo reactor with water circulation facility. A 20W UV lamp [wave length of 365 nm] and visible lamp was placed inside the reactor. The catalytic experiments were carried out with 100 mL solution of dye [5×10^{-5} M] and 20 mg of the catalyst under constant stirring. About 3 mL of the aliquot solution was withdrawn at predetermined time intervals from the reaction mixture, centrifuged and the decrease in absorbance values was measured. A control experiment was carried out under identical experimental condition using commercial CR and without catalyst.

$$\%\ Decolourization = [(C_o - C) \div C_o] \times 100$$

Where,
C_o is the initial concentration of dye solution and
C is the concentration of dye solution after photocatalytic degradation.

3. Results and Discussion:

3.1. XRD Analysis:

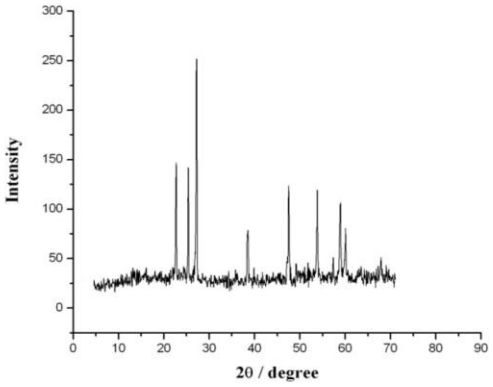


Figure. 1. XRD pattern of PVA capped ZnSe nanoparticles

The X-ray diffraction pattern of the as prepared PVA capped ZnSe sample is shown in Fig. 1. All the diffraction peaks in this pattern can be indexed to Wurtzite (hexagonal) structure. Which is in very good accordance with the JCPDS card no.80 - 0008 for ZnSe (a = b = 3.996 Å, c = 6.55Å). From this figure, the characteristics peaks located at 2θ= 25.56°, 27.23°, 29.44°, 38.03°, 49.87° and 54.6° are corresponding to (100), (002), (101), (102), (103) and (112) planes respectively. No other impurity peaks can be detected, indicating that the ZnSe samples are of high purity. The strong and sharp diffraction peaks show that the as-obtained products have high crystallinity. This result suggests that the growth of PVA capped ZnSe is good. The information on the particle size (A) of PVA capped ZnSenanoparticles have been obtained from the following Scherrer relations [25].

$$D = \frac{0.9\lambda}{(\beta cos\theta)} \text{---------- (1)}$$

Where D is the average crystallite size, λ is the wavelength of incident X-ray (1.541 8Å), β is the full width half maximum (FWHM) of X-ray diffraction expressed in radian and θ is the position of the diffraction peak in the diffractograms. The particle size was obtained in the above there XRD pattern as 34.8nm. Xiong et al[26] and Archana et al[27] have reported hexagonal wurtzite ZnSe having the lattice planes (100), (002), (101), (102), (103) and (112) for the zinc selenide nanoparticles. The XRD patterns reported by various authors are found to be highly consistent with our report.

3.2. SEM with EDAX:

The surface morphology of the synthesized PVA capped nanoparticles was identified on nanoscale by Scanning Electron Microscope. SEM image showed the shape and distribution of the zinc selenide nanoparticles

synthesized by polymer capping. The inset figure show high magnification image of the sample. The PVA capped zinc selenide shows a large agglomeration of undistinguishable morphology. The nanoparticles (Figure.2.a) showed needle type morphology. The nanoparticle confirms the enhancement of well-defined morphological crystallite in polymer capped zinc selenide nanoparticles.The present report is well matched with the report of celine et al [28].

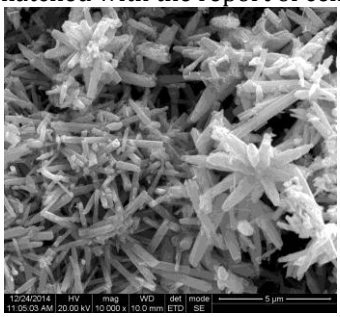


Figure.2.a. SEM image of PVA capped Zinc selenide nanoparticles

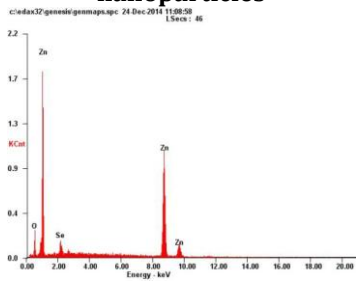


Figure.2.b. EDAX spectrum of PVA capped Zinc selenide nanoparticles

The PVA was used as a capping agent, which prevents the nanoparticles to aggregate. It is proved that the prevention of aggregation of nanoparticles in the presence of the surfactant is more effective. This capping agent's molecules bind to the surface of the particle by stabilizing the nuclei and larger nanoparticles against aggregation, hence controlling the growth of nanoparticles [29]. The chemical composition of this nanoparticle was further investigated by energy dispersive X-ray spectrum (Fig. 2.b). In the spectrum, besides the O signals come from the substrate, only Zn and Se are detected. The similar result was reported by Qingzi Zeng et al [30].The EDAX results corresponded to the XRD results demonstrate that sample is hexagonal wurtzite ZnSe.

3.3. AFM Analysis:

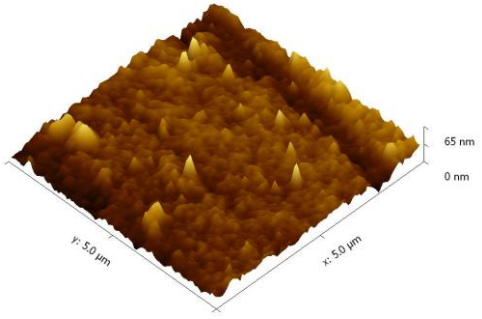


Figure.3. AFM 3D image of PVA capped Zinc selenide nanoparticles

Atomic force microscopy (AFM) is also known as scanning force microscopy (SFM). AFM is a basic technique and inevitable for all nanoscopic research. The AFM image of PVA capped zinc selenide nanoparticles is shown in figure.3. Surface topological features of polymer capped ZnSe samples as-observed under AFM 3D pattern. The micrograph at 5 µm × 5 µm exhibit an uniform surface with cone like grains covering the ZnSe surface can be seen for this sample.

The surface roughness, RMS average value and heights were determined by AFM analysis. The surface roughness was found to be in the range of 1.97 µm to 2.32 µm. The surface roughness of the samples was increased in the presence of polymer capping agent due to the presence of functional groups. The average crystallite size of the sample was 34.7 nm, which has good agreement with the XRD results. AFM observation showed uniform distribution of ZnSe particle in polymer matrix. Murali et al [31] have given the similar report. The grains size is observed to increase from 54-76 nm as the deposition temperature increases and Mazher et al [32] has investigated the zinc selenide nanoparticle show the surface is rows of pyramidal nanostructures. Unlike in our case, there is a cone like grains is observed.

3.4. HRTEM Analysis:

Figure 4 show that the HRTEM image and particle size distribution of the PVA capped ZnSe nanoparticles. This HRTEM image indicating the monodispersed spherical crystallite. The nanoparticles are clearly well identified and no effective aggregation of bulk particle is formed. The particle range is 30 nm to 50 nm.

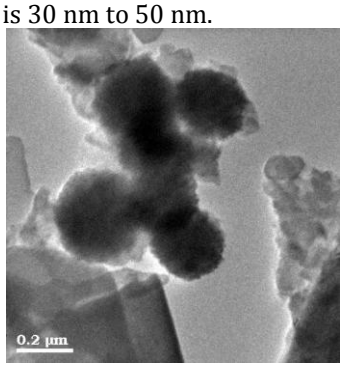


Figure. 4. HRTEM image of PVA capped Zinc selenide nanoparticles

While the HRTEM photograph shows the aggregates of the particles, which are formed because of the high surface energy of the nanometer-sized crystals. The similar report was observed by mamta Sharma et. al [33]. This is in accordance with the XRD results. From the HRTEM image, which is observed that the polymer plays an important role in enhancing the disperse property of ZnSe nanoparticles. This is reported on the capping mechanism of polymers in growth process of ZnSe nanoparticle and also the influence polymers dosage in tailoring the shape and size of the ZnSe nanostructures.

3.5. Optical Analysis:

The optical property of the sample was characterized by UV–Vis spectroscopy. The optical absorption spectrum of the ZnSe sample was recorded at room temperature in the wavelength range between 200 and 800 nm and is shown in Fig. 5.a. The absorption data were analyzed using the classical relation for near edge optical absorption of semiconductors.

$$\alpha = \frac{A(h\upsilon - E_g)^n}{h\upsilon} \text{ --------(2)}$$

Where hν is the photon energy. A and n are constants. Where n is 2 for a direct energy gap and ½ for an indirect energy gap. The plot of $(\alpha h\nu)^2$ vs. hν is shown in Fig. 5.b. Extrapolating the straight line of this plot for zero absorption coefficients it gives the direct band gap of nanostructure materials which is shown in Fig. 5.b.

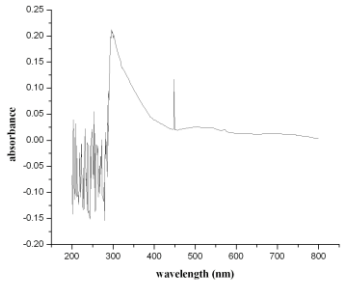


Figure. 5.a. UV-Vis absorption spectrum of PVA capped ZnSe nanoparticles

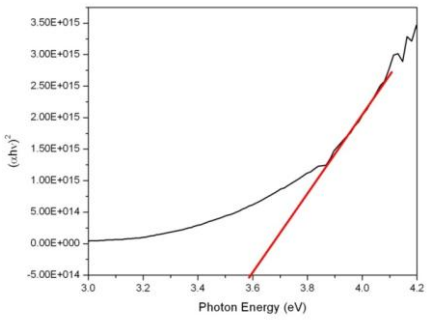


Figure. 5.b. Band gap energy of PVA capped ZnSe nanoparticles

The absorption band gap edge was shifted to shorter wavelength and the corresponding band gap is 3.6 eV for PVA capped ZnSe nanomaterial was observed. It is well known that the absorption edge is related to the size of the nanoparticles. The absorption edge of the ZnSe nanoparticles shows a blue-shift as compared with the value for bulk ZnSe. This blue shift could be attributed to the size reduction effect of PVA capped zinc selenide nanoparticles[33] and structural defect of nanomaterials. The capping agent is used to arrest the growth of nanoparticles and to stabilize them from aggregation. The capping agent also modifies the structural, morphological and optical properties of nanoparticles [34].

3.6. Photo catalytic activity of Congo red dye using PVA capped ZnSe Nanoparticle in presence of UV and Visible light irradiation:

3.6.1. Effect of contact time:

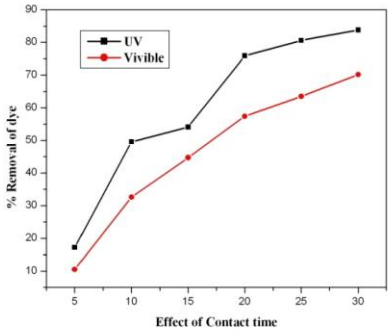


Figure.6. a. Effect of contact time

The rate of removal of congo red dye is shown in figure.6.a. The extent of removal of CR dye by polymer capped zinc selenide nanoparticles was found to increase, reach a maximum value with increase in contact time. The equilibrium time was found to be 30 minutes for UV and visible light irradiation. This is may be due to with increase in irradiation time dye molecules and catalysts have enough time to take part in photocatalytic degradation process and hence percentage of degradation increases [35]. As in shown figure.6.a. desorption occurred after saturation. Therefore, considering technical and economic aspects, a contact time of 30 min was chosen for dye removal from aqueous solution by the adsorbent.

3.6.2. Effect of the amount of adsorbent:

Adsorbent dose is an important parameters because it determines the capacity of an adsorbent for a given initial concentration of dye. The effect of adsorbent dose was studied on dye removal keeping all the experimental conditions constants. The removal of congo red by nanoparticles at different adsorbent doses from 10 mg to 20 mg of dye concentration was studied.

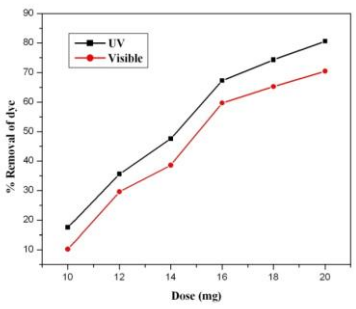


Figure.6.b. Effect of dosage

The results show that as the adsorbent mass increases from 10 mg to 20 mg, the percent congo red dye removal increases from 18 to 81 % for UV and 10 to 70 for Visible light irradaiation. The percent congo red dye removal increases as the adsorbent dose increase due to increase in total number of surface active sites, increase in the extent of dye adsorption molecules on the catalyst surface and enhanced generation of hydroxyl radicals due to increase in the concentration of charge carriers [36-40].

3.6.3. Effect of initial concentration of congo red dye:

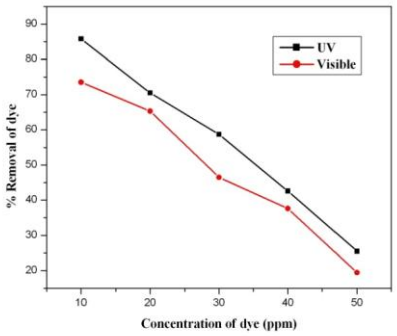


Figure.6.c. Effect of initial concentration of dyes

The effect of initial concentration of congo red dye on photocatalytic activity was studied by varying the initial dye concentration from 10 ppm to 50 ppm. The samples were taken in 100 ml of dye solution and percentage decolourization was calculated after experiment. The degradation efficiency is significantly dependent on the initial dye concentration. The results are shown in figure.6.c. The dye degradation efficiency decreases as the concentration of congo red dye increased. This can attributed to the number of hydroxyl radicals which remains constant as the catalyst dose was kept constant. The maximum dye degradation was observed at 10 ppm (Fig.6.c.). As the concentration of the dye increased the degradation efficiency decreased.

3.6.4. Effect of pH:

Congo red is a dipolar molecular. It exists as anionic form at basic pH and as cationic form at acidic pH. It has also been found that as the pH decreases, the colour of Congo red solution changes from red to dark blue.

These variations of colour with pH suggest that the extent and nature of ionic character of congo red molecule depends on the pH of the medium. The pH value increases from 2 to 10 for congo red dye. The zero point charge for different polymer capped ZnSe is 3.1. In CR dye the pH values 2, 4, 6, 8 and 10 higher percentage removal was occur became at this basic condition. The surface of the catalyst will become positively charged and hence the anionic dye (CR) easily attracted by the catalyst.

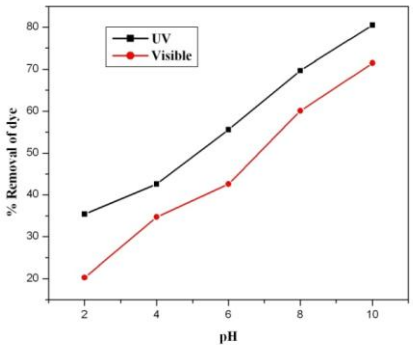


Figure.6.d. Effect of pH

4. Conclusion:

The present study outlines the synthesis of nanaostructured zinc selenide by hydrothermal method using poly vinylalcohol (PVA) as capping agent. The physico-chemical characterization involved XRD, SEM with EDAX, AFM, HRTEM and UV-Visible absorption spectroscopy. The XRD studies showed that the polymer ZnSe nanoparticles exihibit hexagonal crystal structure. Band gap energy of ZnSe nanoparticles was around 3.6eV for polymer capped nanoparticles, which is higher than that of bulk ZnSe nanoparticles indicate the capping agent modifies the band gap energy of the nanoparticles. The nanoparticles could find use in solar cells, in modern electronics and electrooptical devices. The polymer capped zinc selenide nanoparticle was done to enhance its degradation efficiency. The maximum efficiency was observed in the UV light irradiation. This nanoparticles exhibits excellent photocatalytic activity and can be considered as a promising photocatalyst for treatment of dye effluents.

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