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Evaluation of Synthesized S, N-CQDS/$ZNCo_2O_4$ Heterojunction Nanocomposite as an Efficient Photocatalytic Decolourisation of Organic Pollutants under Visible Light Irradiation

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Abstract

New synthesized materials exhibit promising applications as photocatalysts for ecological rehab and better usage of non-conventional energy source i.e. Sun. Semiconductor-based photocatalysts have been massively utilized for the overall elimination or fading of organic pollutants from wastewater in the presence of solar light irradiation. The heterojunction photocatalyst was synthesized by decorating S, N-CQDs onto the surface of $ZnCo_2O_4$ nanomaterials. The structural and morphological features of the developed material were analysed using powder X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and energy–dispersive X-ray spectroscopy (EDX) analyses. It was found that the morphology of the prepared sample was spherical in shape. The EDX-Spectrum of the nanocomposite revealed the existence of Zn, Co and O elements. The photocatalytic activity of the prepared catalyst was evaluated for the degradation calmagite dye an organic dye was found to be 85% after 90 min of solar light irradiation.

Keywords: Nanocomposite, Organic contaminant, Photocatalyst, Semiconductor.

Introduction

The massive production and global use of organic dyes in a wide range of industries, especially the textile one, contributes significantly substantial to environmental pollution[1]. Dyes are mostly toxic and non-biodegradable and thus, cannot be eliminated from the environment by conventional means[2]. Advanced oxidation processes are recognized as the most promising technologies for degrading polluted water through the destruction of organic compounds while maintaining high efficiency and processability[3].

Many methods for the degradation of organic wastes have been developed in recent years and photocatalytic oxidation is one of the best techniques currently under study. The method is based on the decomposition of organic molecules under the action of light by a semiconductor photocatalyst[4]. Thus, the possibility of using this method makes it possible to achieve the complete mineralization of organic dyes into non-toxic products, CO_2 and H_2O_2, which are produced as a result of the degradation[5]. Thus, the technology provides an economically viable and environmentally safe way of wastewater treatment. In recent years, studies devoted to metal oxide nanomaterials have received much attention in the literature since they allow one to study their properties and perform comparative analysis of synthesis methods. These materials have found their applications almost in all areas of science and technology[6]. $ZnCo_2O_4$, which has a wide absorption edge, and therefore, the development of methods for its preparation and investigation of its properties are of great interest. A mixed metal oxide with a spinal structure, has been widely studied. In recent decades, this type of metal oxide has been the subject of many studies. $ZnCo_2O_4$ is the most suitable option for decolourisation of dyes with the help of photocatalytic applications by photodegradation under solar irradiation[7].

To increase the surface area of $ZnCo_2O_4$ and thus its adsorption capacity, S and N-doped carbon quantum dots (S, N-CQDs) were introduced on its surface. The composite material was prepared by growing S,N-CQDs heterostructures on the surface of $ZnCo_2O_4$ nanomaterial. As a result, the surface area of the nanocomposite was increased, which made it possible to remove a larger amount of dye from the solution in comparison with the initial $ZnCo_2O_4$. This nanocomposite showed good adsorption ability and could be used for water purification[8].

Materials and Methods

We used all the chemicals without purifying them. We bought all the chemicals, including $Zn(NO_3)_26H_2O$, $Co(NO_3)_26H_2O$, NH_4OH, Calmagite, Thiourea, and Ascorbic acid, from Bangalore, India.

We used $Zn(NO_3)_26H_2O$, $Co(NO_3)_26H_2O$, NH_4OH using a method called Precipitation. We made S, N-CQDs using a machine that uses high pressure and temperature called a Teflon-lined autoclave and a method called hydrothermal method. We obtained $ZnCo_2O_4$ through coprecipitation method.

To put S, N-CQD on the surface of $ZnCo_2O_4$, we mixed S, N-CQD and $ZnCo_2O_4$ together in a ratio stirred them for two hours then washed and dried them at 70 degrees Celsius.

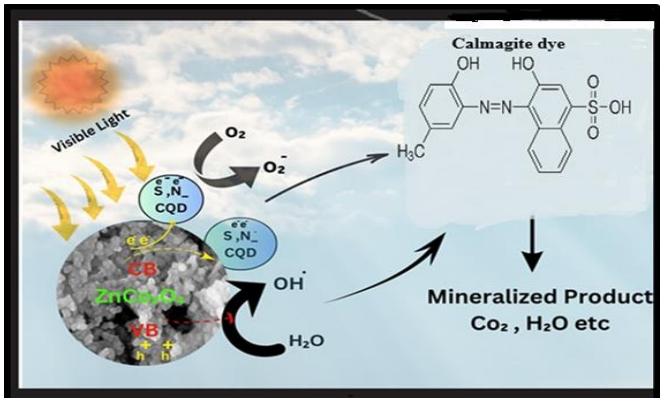


Figure-1: Photocatalytic activity.

Characterisation Method: X-ray diffraction (XRD) analysis was employed to investigate the structural properties of the prepared materials using a Bruker AXS D8 diffractometer with Cu Kα radiation (wavelength = 0.15406 nm). The prepared samples were further characterized using field-emission scanning electron microscopy (FE-SEM) and energy-dispersive X-ray spectroscopy (EDX) (Hitachi, PU SAIF, Chandigarh) to examine the surface morphology and elemental composition of the nanocomposites. Furthermore, the internal morphology and structural features of the nanocomposites were investigated using high-resolution transmission electron microscopy (HRTEM) (Hitachi, PU SAIF, Chandigarh).

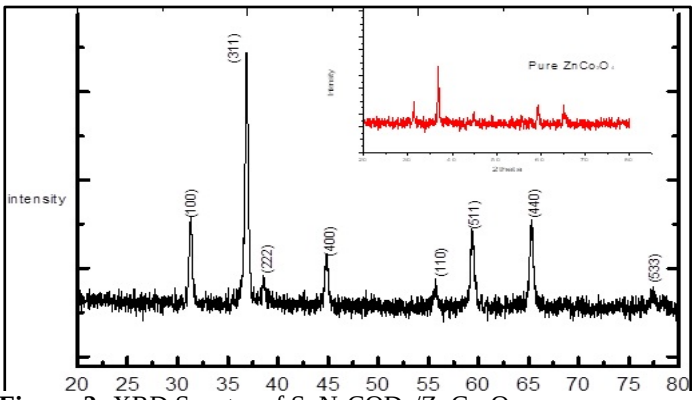


Figure-2: XRD Spectra of S, N-CQDs/$ZnCo_2O_4$.

Photocatalytic Activity Measurement: The photocatalytic activity of the S, N-CQDs/$ZnCo_2O_4$ composite was characterized based on the dissoluteness of the calmagite dye under illumination from visible light. In the study, 40 mg of the composite sample was mixed with 100 ml of calmagite dye solution and a Xe lamp (300W) was considered an alternate light source in place of sunlight. From time to time, 3 ml of the reaction mixture was extracted and centrifuged at a speed of 3500 rpm for two minutes to separate out the composite particles[9]. The amount of dye solution absorbed was measured at 520nm through a spectrophotometer (Model 166, Systronics). The decolourization percentage of the dye was calculated through the following formula –

$$\%\, degration = \frac{C_0 - C}{C} \times 100 \qquad (1)$$

Where: C_0 is the incipient dye concentration and C is the dye concentration later the dye adsorption at time t.

Figure-1 shows that the photocatalytic activity of S, N-CQDs/$ZnCo_2O_4$ nanocomposites is responsible for the disintegration of Calmagite dye under visible light irradiation. The efficiency of decolorization of Calmagite dye is observed to be 85% in 90 minutes in the presence of visible light. The Langmuir-Hinshelwood kinetic expression was simulated for the heterogeneous pseudo- first order kinetic reaction for dye decolourisation (Figure-2)[11].

$$ln\frac{C_0}{C} = kt \qquad (2)$$

Where: k is constant of Langmuir adsorption and the rate constant of calmagite dye decolourisation was showed 0.0214 min^{-1} (Figure-2).

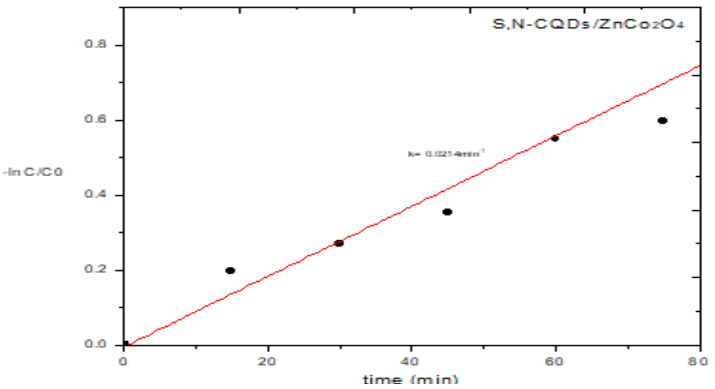


Figure-3a: Photocatalytic kinetic curves using S, N-CQDs/$ZnCo_2O_4$ nanocomposite.

Results and Discussion

The diffraction pattern obtained by XRD techniques for the S, N-CQDs/$ZnCo_2O_4$ nanocomposite photocatalyst is presented in Figure-3a. All the peaks observed with respect diffraction angle corresponding to 100, 311, 222, 400, 110, 511, 440, 531, 533 are crystal planes, respectively, well indexed to the cubic structure (JCPDS- 00-001-1149)[12,13]. No other peaks of S, N-CQDs have been seen in the XRD spectrum. The average size of particles is analysed using the Scherrer equation and found to be 16.85nm. Figure-3b shows the FE. spherical morphologies were observed. Figure-3c the HRTEM image showing the presence of S, N-CQDs are uniform distribution in on the surface of $ZnCo_2O_4$ nanoparticles. EDX image In Figure-3d for the elements such as Zn (1.11%), Co (13.14%), O (30.70%), N (3.93%), C (50.77%), and S (0.34) can be the element composition are available in this nanocomposite photocatalyst.

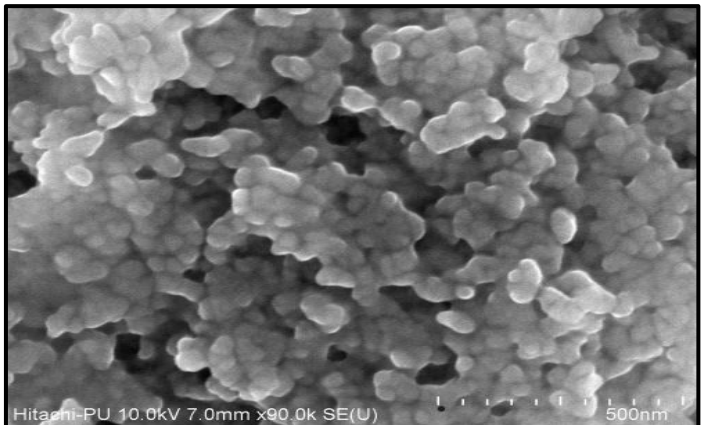


Figure-3b: FESEM image of S, N-CQDs/$ZnCo_2O_4$.

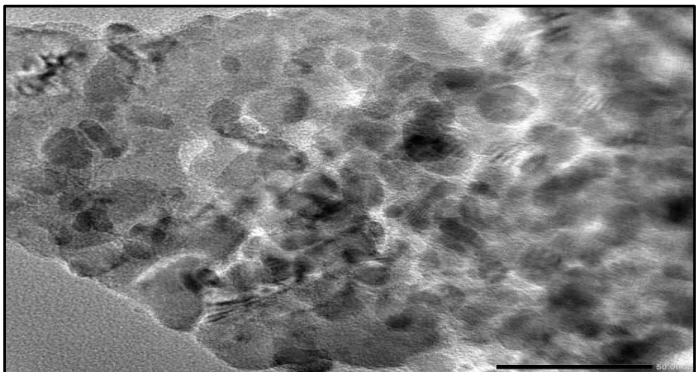


Figure-3c: HRTEM image of S, N-CQDs/$ZnCo_2O_4$.

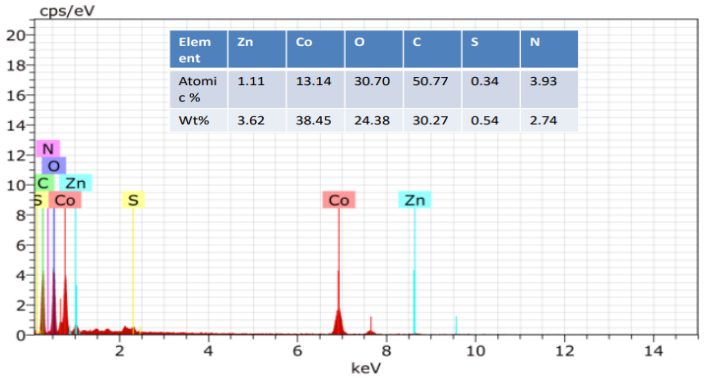


Figure-3d: EDX spectrum of S, N-CQDs/$ZnCo_2O_4$ nanocomposite.

Photocatalytic activity: Impact of pH on decolourisation efficiency: The pH of the sample is an important parameterwhich impacting the photochemical decolourisation efficiency of dye molecules[14], since it affects the surface charge characteristics of S, N CQD/$ZnCo_2O_4$ nanocomposite. The role of impact of pH on the photo induced decolourisation of Calmagite dye using S, N CQD/$ZnCo_2O_4$ nanocomposite catalyst under Visible light was illustrated at some pH (pH Span 4 to 10) of the solution. The optimum photo decolourisation efficiency was obtained at 5.82×10^{-4} at 8 pH. After the maximum pH, further increment in the pH value from 9 to 10 it is observed that the decolourisation rate was declined from 5.10×10^{-4} to 4.73×10^{-4} shown in Figure-4(a).

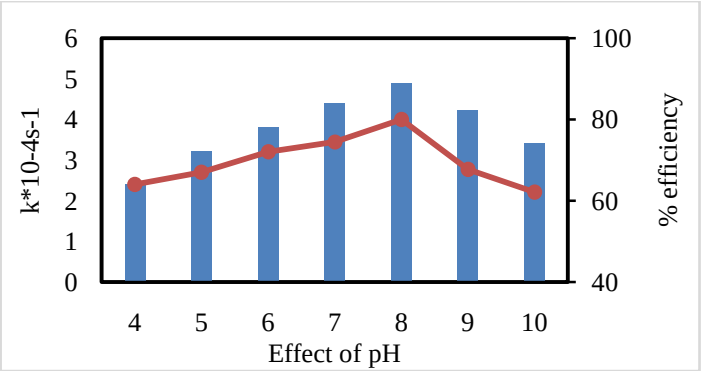


Figure-4a: Impact of pH on decolourisation efficiency.

Effect of catalyst loading: This is well known fact that excess use of catalyst is harmful. This causes reduction in the rate of decolourisation of organic compounds. In the current experiment the loading of S, N-CQDs/$ZnCo_2O_4$ Nanocomposite has been varied from 20mg to 80 mg/100 ml in dye suspension under visible light irradiation by keeping all factor constant. According to Figure-4b achieved the best photo decolourisation efficiency was at the concentration 50mg/100ml with a rate constant $4.87\times10^{-4}s^{-1}$. Increasing the nanocomposite concentration, the decrease in decolourisation rate constant result due to screening[15].

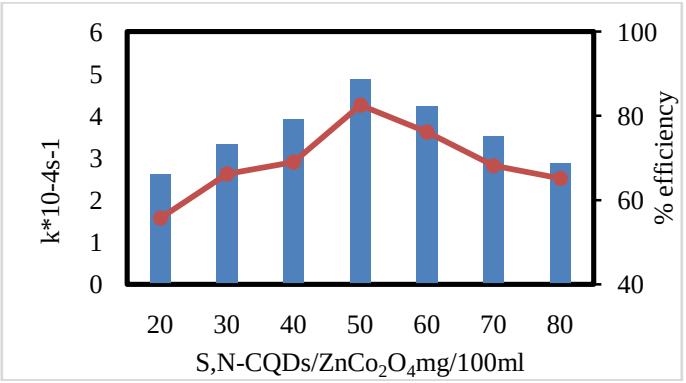


Figure-4b: Effect of catalyst loading.

Impact of dye concentration: To calculate the effect of photocatalytic decolourisation of organic pollutants[16]. In the recent research, this dye concentration ranges from 2×10^{-6} to 10×10^{-6} mol/dm^3. When the dye concentration increased, the rate constant also increased from 3.45×10^{-4} to 4.87×10^{-4} s^{-1}. This is due to the increase in dye concentration, increases the availability of dye molecules by transferring energy through excitation process[17]. But the decrease in rate constant when the dye concentration is increased from 6×10^{-6} to 10×10^{-6} mol/dm^3 is due to high concentration of dye solution, the intensity of light will be reduced. The obtained outcome as in Figure-4c.

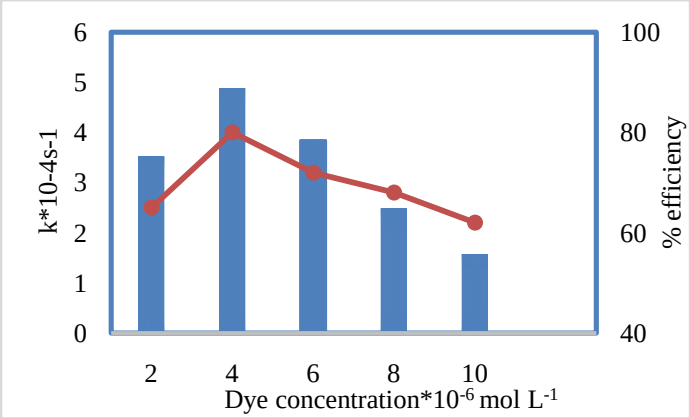


Figure-4c: Effect of dye concentration.

Reusability: Reusability is another important factor considered while assessing the sustainability and practicality of a photocatalyst[18]. In order to determine this factor, the experiment was analyzed through multiple cycles of photocatalytic decolourisation under similar experimental conditions[19,20]. Photocatalyst was used four times in a row and without any loss in its activity and sustainability. In each cycle, the photocatalysts were collected, washed and centrifuged and then dried at 70◦C in the furnace. In the fourth cycle, the efficiency of the photocatalytic decolourisation was measured at 91% of the initial efficiency of decolourisation.

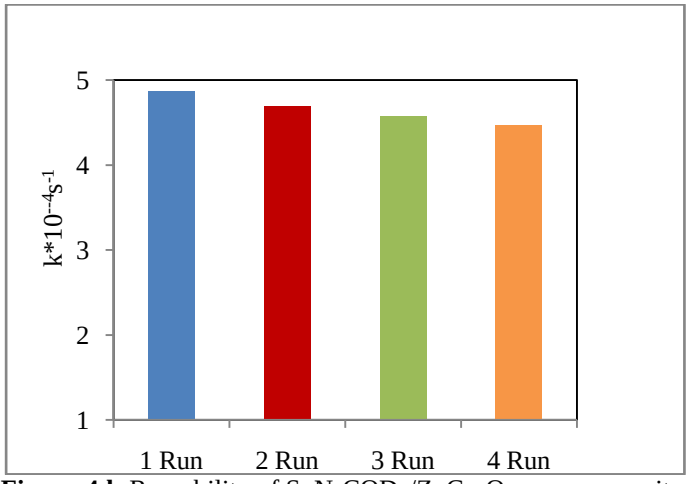


Figure-4d: Reusability of S, N-CQDs/$ZnCo_2O_4$ nanocomposite.

Chemical Oxygen Demond (COD): Chemical Oxygen Demand (COD) for the calmagite dye solution was determined both before and after the photo decolourisation. After completing 3 hours experiment, 90% COD was observed, which is illustrated in Figure-4e.

Though full decolourization took place in one hour, but the efficiency of COD reduction was much slower as compared to decolourization process. Reduced COD values show that the molecules of dye were greatly mineralized, besides their decolourization.

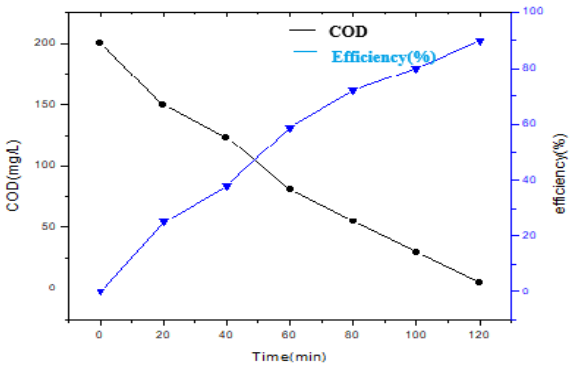


Figure-4e: COD reduction and percentage efficiency during photocatalytic decolourisation of calmagite dye using S, N-CQDs/$ZnCo_2O_4$.

Conclusion

In this work, S, N-CQDs/$ZnCo_2O_4$ nanocomposites have been synthesized via the impregnation technique. The phase structure, morphology and composition were determined using XRD, FE-SEM, EDX and HRTEM techniques. The outcome of this work shows that the S, N-CQDs/$ZnCo_2O_4$ composite can degrade 87.93% of the calmagite dye in 80 minutes in the presence of visible light. S, N-CQDs/$ZnCo_2O_4$ composite exhibits good reusability.

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