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Synthesis and Characterization of CuO-Ag_2S Composite: A Novel Photocatalyst Efficient Degradation of Eosin B and Methylene Blue Dyes

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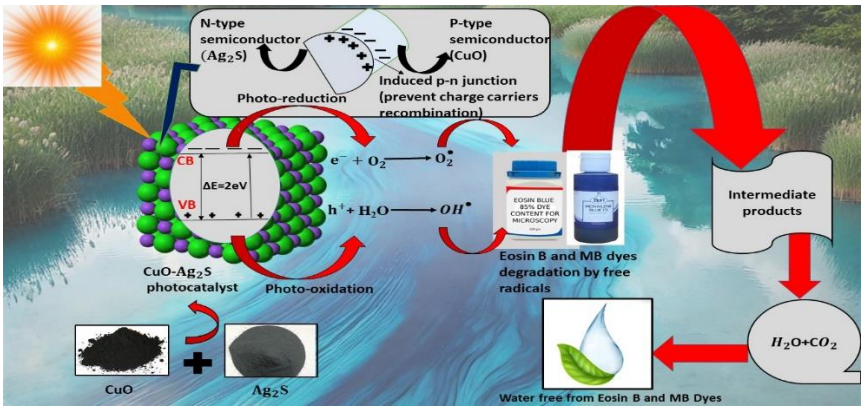
ABSTRACT

Presently, the contamination of water by synthetic dyes such as Eosin B and Methylene Blue (MB) is a matter of concern. Being toxic and non-biodegradable, these dyes cause harm to nature, human health and aquatic life. Amongst the numerous remediation methods, photo catalytic degradation has emerged as an effective approach for the degradation of these toxic pollutants into harmless byproducts like H_2O and CO_2. The present study investigates the use of CuO-Ag_2S as a photocatalyst for the breakdown of Eosin B and MB in the presence of light. CuO-Ag_2S composite was synthesized by hydrothermal method and characterized for its crystal structure, surface morphology, elemental analysis, functional groups and optical features using XRD, SEM, EDX, FTIR and UV-Vis spectroscopy respectively. The degradation of Eosin B and MB was carried out under different conditions in order to figure out its efficacy. This study was conducted on 60ppm Eosin B solution having dosage of 0.015g, where a maximum degradation of 97% was attained after 60min of irradiation time at pH 5. On the other hand, the optimized conditions for MB were 20ppm, 0.015g dose, and pH 11 at which the highest degradation was attain i.e. 68%. The degradation kinetics were analyzed, revealing a pseudo second order kinetics for both processes. The study highlights the effective degradation of Eosin B and MB dyes using CuO-Ag_2S, providing valuable insights into the impact of various operational parameters and reaction kinetics. The findings contribute to the development of efficient and sustainable dye removal techniques, paving the way for future advancements in waste water treatment.



INTRODUCTION

Water is essential for life, so every decision and action about it is very important [1]. Food, textile, plastics, leather goods, industrial paintings, and cosmetic industries are using different varieties of organic synthetic dyes over the years. These industries release a lot of tinted substances into water [2] and thus affect the water quality parameters like pH, chemical oxygen demand (COD), turbidity, gas solubility, aesthetic merit and water transparency etc. [3]. The most commonly used dyes that contaminate water are Eosin blue (Eosin B) and Methylene Blue (MB) [4-6].

These dyes are widely used in biological staining, ink manufacturing and textile dyeing due to their bright color and strong fluorescence properties [1]. Water pollution

from Eosin B and MB dyes has become a serious environmental problem. Even in small amounts, these dyes are toxic and can build up in the food chain, causing harm to nature and human health. The problem is getting worse in many countries due to industrial growth and a rising population [7]. In addition, the presence of Eosin B and MB in water bodies can lead to the destruction of aquatic life. Because of this, removing and neutralizing these dyes has become more important for protecting the environment [8-11].

Various strategies have been put forward to remove Eosin B and MB from water including coagulation, precipitation, solvent extraction, reverse osmosis, adsorption, and filtration. Numerous phtocatalysts such as TiO_2, Zr doped TiO_2/reduced Graphene Oxide (rGO) nanocomposite, tridoped Titania decorated on SWCNTs and MWCNTs, AgBr-ZnO nanocomposite, UV-ZnO NPs, ZnO, Fe_2O_3@TiO_2, Bi_2O_3@$MoSe_2$ mixed nanostructures, SnO_2, Cerium Oxide Nanoparticles, Graphene Oxide-doped Polyacrylamide and $MnTiO_3$, have been tested to improve the effectiveness of photocatalysis for Eosin B MB degradation. However, even though they have benefits, they also have some problems like low visible light activity, rapid recombination of electron-hole pairs, stability, reusability and reduced effectiveness of Photodegradation [1, 3, 7, 12-16].

Photocatalysis has emerged as a promising technology for the degradation of synthetic dyes. In this process the material absorbs light energy and generates reactive oxygen species that can degrade organic pollutants by converting them to carbon dioxide, inorganic compounds and water. It is considered as a cost effective alternate technology for the purification of environmental pollutants [17, 18]. High surface area, small particle size, stability, suitable bandgap energy, high absorption coefficient, suitable energy levels, non-toxicity and high electron mobility are the important requirements through which a photocatalyst can efficiently facilitate photocatalytic degradation reactions.

Semiconductor materials such as copper oxide (CuO) and silver sulphide (Ag_2S) have been explored for their photocatalytic properties. However, the individual use of these materials often results in limited efficacy and stability. To address these limitations, this study investigates the photocatalytic degradation of Eosin B and MB dyes using a novel CuO-Ag_2S composite. To achieve high Photodegradation efficiency, photocatalyst semiconductors need a smaller energy gap to absorb more light. This was done by combining two nano-sized semiconductors, one in oxide form and the other in sulphide form to improve their performance [19]. CuO is a P-type semiconductor material with a narrow bandgap energy [20], while Ag_2S is an N-type semiconductor material with a high absorption coefficient [18]. The combination of these materials can form a heterojunction, which can enhance the photocatalytic activity and stability. The key advantage of mixing copper oxide and silver sulphide is that their individual properties can be modified and optimized to create a more efficient nanocomposite i.e. CuO-Ag_2S. By carefully adjusting these properties, the combined material can achieve improved performance compared to using either copper oxide or silver sulphide alone. This synergy enhances the photocatalytic activity, making the nanocomposite more effective in applications like pollutants degradation [21]. To the best of our knowledge such composite material has not been reported in the literature for the photocatalytic degradation of Eosin blue and MB dyes, and this manuscript focuses on the synthesis, characterization and photocatalytic activity of CuO-Ag_2S composite material for Eosin B and MB under various conditions.

Experimental Part

Instrumentation: In this work X-ray diffraction (XRD), scanning electron microscope (SEM), Energy dispersive X-ray spectroscopy (EDX), fourier-tramsmission infrared (FTIR) spectroscopy and UV-Vis spectroscopy were used to determine the crystallinity and phase structure, morphology, elemental analysis, to identify functional groups and to monitor the concentration of dye over time by measuring the absorption spectra respectively.

Chemical reagents: Before starting the experiment distilled water was used for cleaning of all apparatus. After cleaning they were placed in oven for drying. Copper sulphate ($CuSO_4.5H_2O$), Sucrose ($C_{12}H_{22}O_{11}$), Silver Nitrate ($AgNO_3$) and Thiourea (CH_4N_2S) were used as sources of Copper, Oxygen, Silver and Sulphur respectively for the synthesis of CuO-Ag_2S composite. All the solutions were prepared in distilled water. Sodium hydroxide (NaOH) was used for adjusting the pH of solution and distilled water and ethanol were used for washing the desired material.

Synthesis of CuO: CuO was synthesized by hydrothermal method. 1g copper sulphate and 5g sucrose dissolved in 20mL and 30mL distilled water respectively. Both solutions were then mixed in a beaker and placed on a magnetic stirrer for 30min to make a homogeneous solution. The solution was then transported to Teflon-lined autoclave and placed in oven at 180°C for 24hrs. Once the reaction was finished, the final product was washed (4-6 times) with distilled water and ethanol. After washing it was placed in oven for drying at 60°C for 8hrs. The dried material was then converted to powder form using a mortar and pestle and then placed in muffle furnace for calcination at 550°C for 4hrs and after that the attained product was stored in a vial for later use [20].

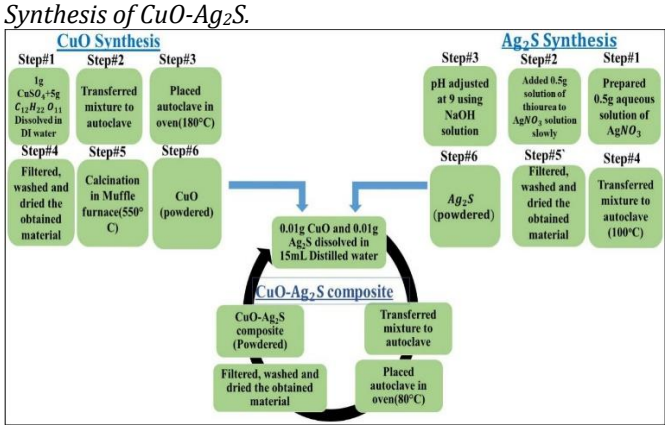
Synthesis of Ag_2S: Ag_2S was prepared by using hydrothermal method. 0.5g silver nitrate and 0.5g thiourea were dissolved in 15mL of distilled water separately. The solution of Silver nitrate was then placed on a magnetic stirrer for 30min to acquire a uniform solution. The thiourea solution was slowly added to the above solution. NaOH was used to adjust the solution pH at 9. The above reaction solution was then transported to Teflon-lined autoclave and placed in oven at 100°C for 8hrs. After finishing the reaction time, the autoclave was left out to cool down and the final product was washed with distilled water and ethanol several times and then placed in an oven for drying at 70°C for 2hrs. After drying the obtained product was converted to powder form by using mortar and pestle and kept for further use [18].

Synthesis of CuO-Ag_2S composite: CuO-Ag_2S composite was also synthesized by hydrothermal method. 0.01g CuO and Ag_2S were dissolved in 15mL distilled water separately. Bothe solutions were then mixed together and placed on magnetic stirrer at 80rpm for 15min and then

transferred to Teflon-lined autoclave and kept in an oven at 80°C for 2hrs. After the intended time, the autoclave was left to cool slowly. The desired product was filtered and placed in an oven for drying at 80°C, then converted to powder form and kept for further use. The schematic diagram for the synthesis of CuO-Ag_2S is given in fig 1.

Figure 1

Synthesis of CuO-Ag_2S.



Mechanism of photocatalytic degradation of Eosin B and MB

The photocatalytic degradation of various organic pollutants has been studied in a lot of literature. When light hits the surface of photocatalyst (CuO-Ag_2S), it absorbs enough energy equal to the difference between the two energy levels i.e. valence and conduction band. The absorbed energy by the electron present in the lower energy level (valence band) causes the electron to jump to the higher energy level (conduction band) leaving behind a positive charge called "hole". Electron-hole pairs (e^-/h^+) created by this process act as the starting species for the photocatalytic reaction on the surface of photocatalyst. The electron (e^-) gets captured by O_2 and the hole (h^+) reacts with H_2O forming superoxide free radicals ($O_2^{\cdot}$) and hydroxyl free radicals ($OH^{\cdot}$) respectively which are very reactive species and degrade the organic pollutants like Eosin B and MB as shown in fig 2. The induced p-n junction in the photocatalyst prevents the recombination of these charge carriers which helps in the sustainability of the reaction.

CuO-Ag_2S + hν_____h^+ (VB) + e^- (CB)

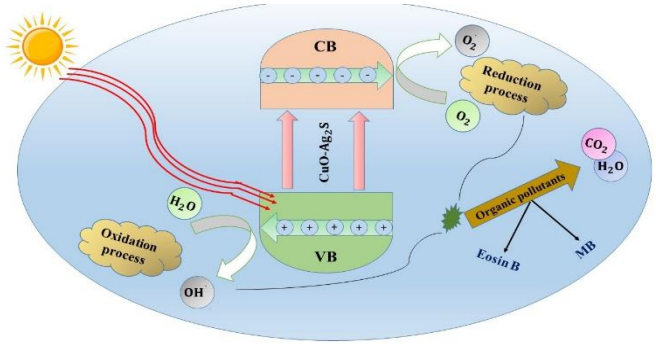
$h^+ + H_2O$_____$OH^{\cdot}$

$e^- + O_2$_____$O_2^{\cdot}$

Pollutants(EosinB+MB)+$OH^{\cdot}/O_2^{\cdot}$____Degradation/discoloration

Figure 2

Mechanism of photocatalytic degradation of Eosin B and MB.



Characterization techniques

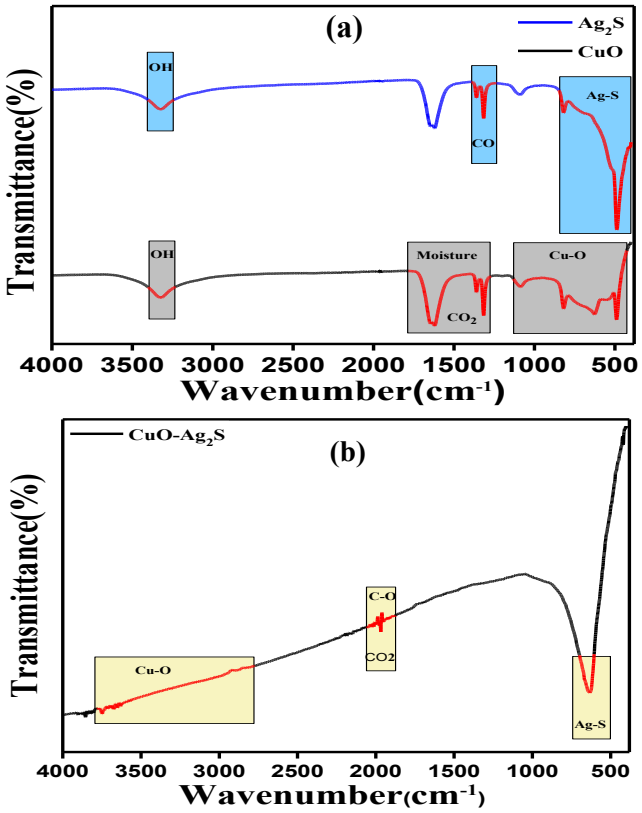
FTIR

The FTIR spectra of CuO and Ag_2S synthesized by hydrothermal method are shown in fig 3(a). For CuO synthesis the peak at 3321 attributed to the presence of O-H bond, the peaks in the region 1300-1700cm^{-1} show the presence of moisture and CO_2. Many peaks around 500-1100cm^1 confirms the presence of Cu-O bond. On the other hand for the synthesis of Ag_2S, the band appears at 3326cm^{-1} shows O-H stretching, the peak at 1310cm^{-1}attributed to the stretching of C-O and the characteristic peaks between 812-475cm^{-1} confirm the formation of Ag-S bond.

The FTIR spectra for composite CuO-Ag_2S is shown in fig 3(b). The characteristic peaks in the region 500-600cm^{-1} attributed to the stretching vibrations of Ag-S and 2800-3700cm^{-1} confirm the presence of hydrated Cu-O bond. Band around 1900-2000cm^{-1} can be assigned to C-O or to surface adsorbed CO_2 [20, 22-24].

Figure 3

(a) FTIR of CuO and Ag_2S (b) FTIR of CuO-Ag_2S composite.

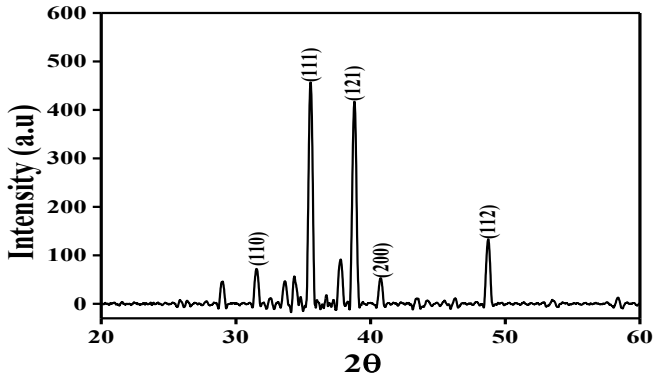


XRD study

The investigation of the crystalline structure of the synthesized composite CuO-Ag_2S was done by X-ray diffraction (XRD). The pattern of diffraction displayed characteristic peaks with a monoclinic structure, given space group C 1 2/c 1 with a space group number 15. Prominent 2Θ peaks at 32.5°, 35.5°, 38.8°, 340.8° and 48.7° are indexed to the 110, 111, 121, 200 and 112 crystal planes respectively as illustrated in fig 4. These results showed the crystallinity, purity and interaction of CuO and Ag_2S with the pattern aligning with reference code 96-721-2243. The average crystallite size i.e. () was determined by Scherrer equation which is given below.

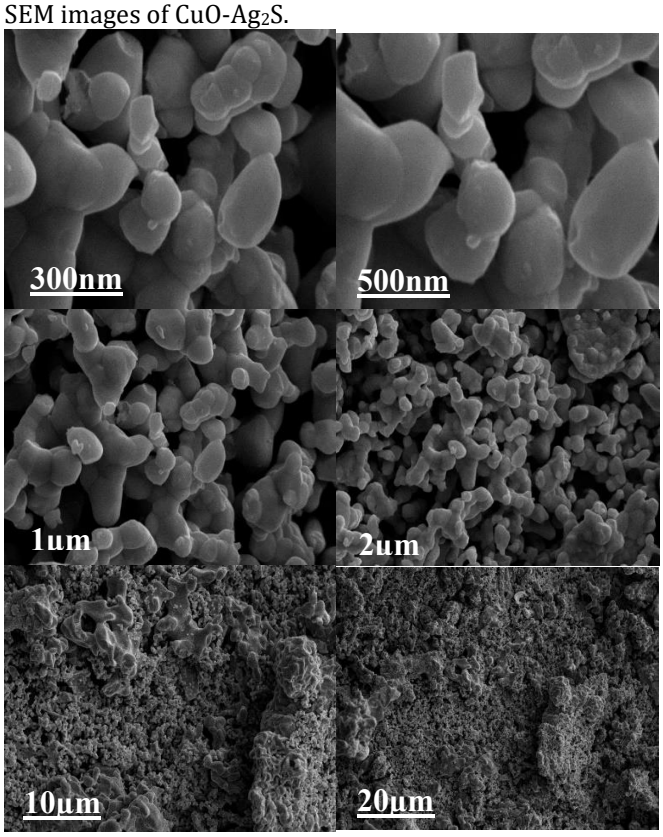
$D=K\lambda/\beta\ (\cos\theta)$

Figure 4
XRD study of CuO-Ag2S.



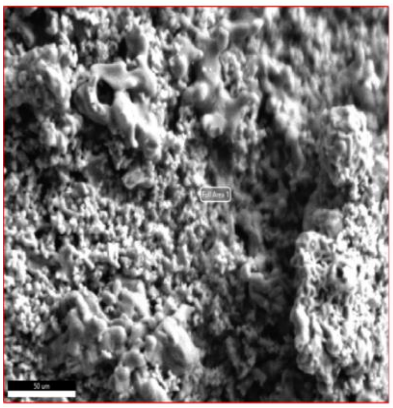
SEM study
The surface morphology of the synthesized composite CuO-Ag_2S is examined by scanning electron microscope (SEM). The images of SEM revealed the morphology of the composite having particle size in the range of 300nm, 500nm, 1µm, 2 µm, 10 µm and 20 µm as shown in fig 5. The images reveal a highly porous and agglomerated structure and the intimate interaction between the two phases offers a synergistic effect which enhances the chemical and electrical characteristics of the synthesized material. This interconnected and compact structure increases the surface area and provides large number of active sites which can be beneficial for the processes like photocatalytic degradation of organic pollutants.

Figure 5
SEM images of CuO-Ag_2S.



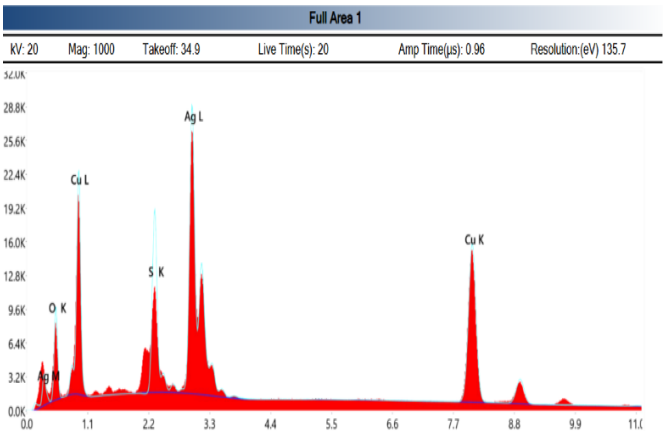
EDX and EDX mapping
EDX, an advanced technique used for the investigation of chemical characterization and analysis of elements present in the material. It gives information about the appropriateness of the synthesized material for the intended use. The characteristic bands in the EDX analysis represent the presence of central elements of the composite which are O, S, Cu and Ag, having weight percent composition 14.83%, 9.01%, 43% and 33.16% respectively as depicted in fig 6. It proved the effective synthesis of CuO-Ag_2S composite. The uniform and homogenous dispersion of CuO and Ag_2S proved the purity of the synthesized composite. The properties of composite like electrical, conductivity and photocatalytic activity can also be determined by the uniform composition of the composite.

Figure 6
EDX and EDX mapping of CuO-Ag_2S.



Smart Quant Results

Element	Weight %	Atomic %
O K	14.83	42.28
S K	9.01	12.82
CuK	43	30.87
AgL	33.16	14.02



UV-Vis analysis
After passing the amount of light through the sample of CuO/Ag_2S in UV-Vis spectrophotometer, the amount of light absorbed at each wavelength is recorded and the maximum absorption was observed at 551nm as shown in fig 7(a) recommending it as an effective photocatalyst for the degradation of organic pollutant Eosin B and MB. The band gap was also concluded from this spectrum following Tauc's equation.

Band gap
Band gap is one of the most important parameter which influences the ability of a material to generate charge carriers by absorbing light and also plays a crucial role in the determination of photocatalytic activity. The following Tauc's equation was used to calculate the band gap (2eV) as shown in fig 7(b) of the synthesized photocatalyst i.e. CuO-Ag_2S.

$(\alpha h\nu)^{\gamma} = A\ (h\nu - Eg)$ (1)

Where "α" is absorption co-efficient, "h" is Plank's constant (6.6262×10^{-34}), "ν" is photon's frequency, "γ" is

nature of electronic transition, "A" is proportionality constant and "E_g" is band gap.
At this band gap a large number of free radicals formation takes place because the electrons move from ground state to excited state easily and degraded the Eosin B dye effectively.

PZC

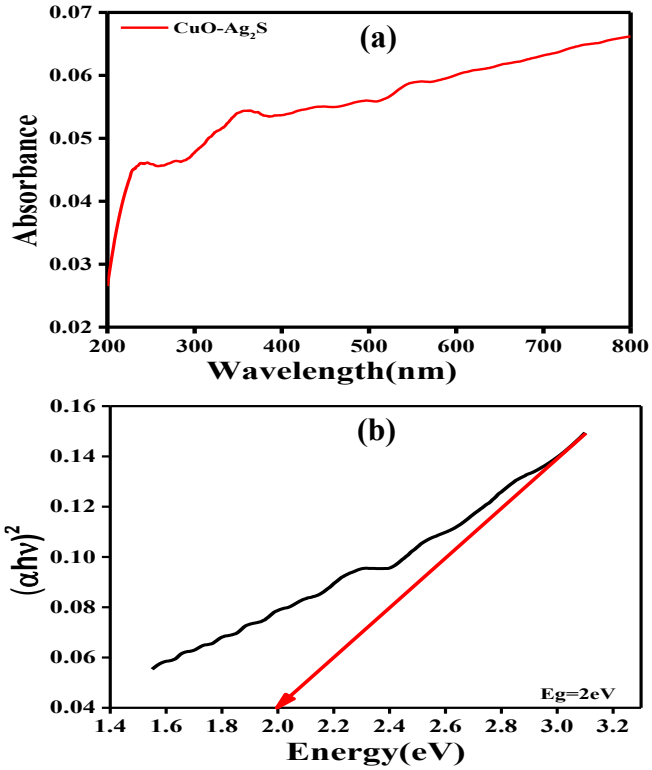
The point of zero charge (PZC) was determined by means of pH drift method. PZC is the pH value at which the net surface charge of the composite is zero, influencing its adsorption and photocatalytic activity. Five different solutions of 0.1M of KNO3 each having volume of 50mL were prepared and 0.1M of sodium hydroxide and hydrochloric acid were added to the above solutions of potassium nitrate for adjusting their pH at 3,5,7,9 and 11. A 0.01g of CuO-Ag_2S was added to all these solutions and their pH were determined after the completion of 24hrs of agitation. The PZC 7.0 as shown in fig 7(c) was figured out by drawing the graph of ΔpH as a function of pHi. It was concluded that the pH values below the PZC, the composite surface is positively charged, and facilitating the adsorption of negatively charged Eosin B dye molecules and enhanced its photocatalytic degradation efficiency.

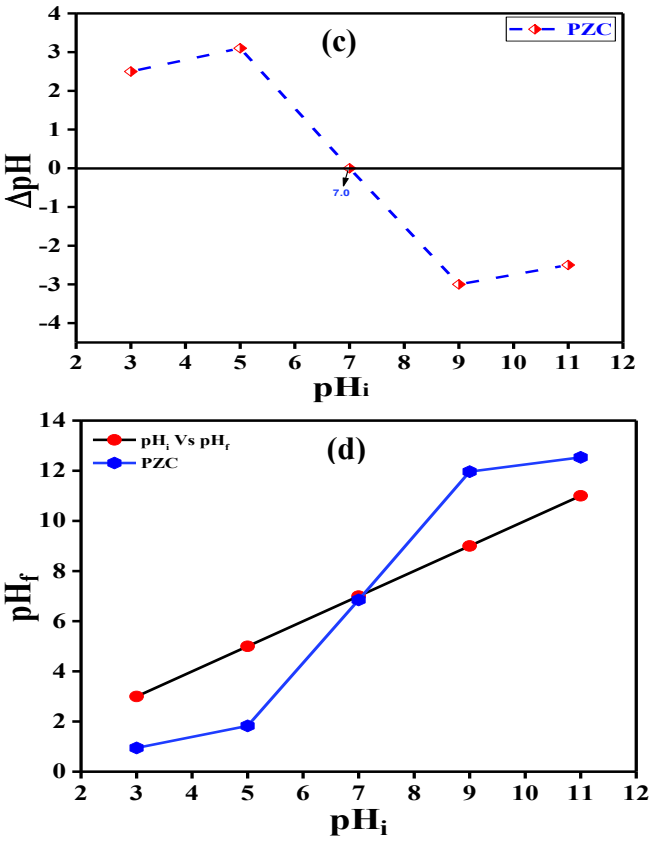
Effect of pH on PZC

pH has a great effect on the value of PZC as shown in fig 7(d). This study indicated that the material has high positive charge at pH 5 and as Eosin B is an anionic dye having negative charges on the surface so a strong interaction created between them. As a result, a large number of dye molecules adsorbed on the surface of the catalyst leading to higher rate of degradation. When the pH increased above 7, the rate of degradation decreased due to repulsion between the catalyst and the dye molecules.

Figure 7

(a) UV spectra of CuO-Ag_2S (b) Band gap (c) PZC (d) Effect of pH on PZC





Photocatalytic activity

0.015g of the photocatalyst CuO-Ag_2S was added to 60ppm and 20ppm solutions of the Eosin B and MB having pH 5 and 11 respectively and placed on a magnetic stirrer under light in order to evaluate the photocatalytic activity of the synthesized photocatalyst for the degradation of these toxic dyes. UV-Vis spectroscopy was used to examine the samples (taken every 10min) in the form of spectra as a function of irradiation time. The reduction in the absorption peaks showed that the catalyst has degraded the dyes up to a large extent and the percent degradation i.e. 97% for Eosin B and 68% for MB was calculated by using the following equation

$$\% \text{ Photodegradation} = [(A_0 - A_t)/A0] \times 100 \quad (2)$$

Where A_0 and A_t are the initial absorbance and absorbance at time't'

Effect of time

The time of illumination has a significant impact on the photocatalytic degradation process of Eosin B and MB by CuO-Ag_2S composite. The efficacy of degradation improved with increase in reaction time due to the strong interaction of the photocatalyst CuO-Ag_2S with these dyes for a longer period of time, as there is a direct relation between the reaction time and Photodegradation process but up to a certain point. In the beginning, the rate of degradation was rapid because of the availability of large number of active sites but after a specific point of time it slowed down and remained constant. The decrease in the light absorption by Eosin B and MB from 0.57au to 0.029au as depicted in fig 8(a) and from 2.9a.u to 0.9a.u as shown in fig 8(b) respectively over a time period of 10 to 60 minutes showed that the composite worked effectively and broke down the dyes considerably. As a result, a change in color was observed.

Effect of dye concentration

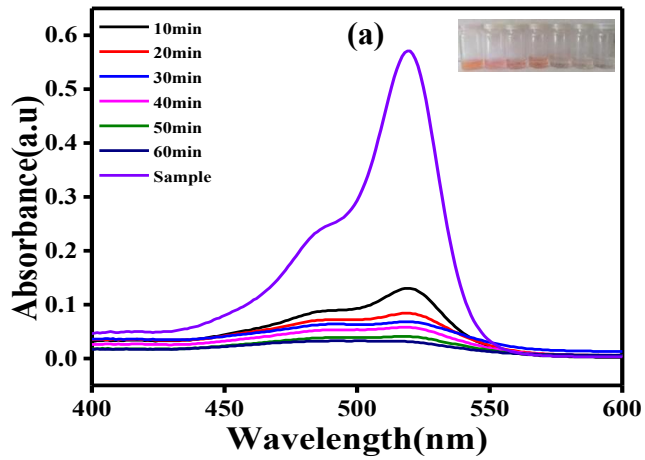
Different amounts of Eosin B and MB dyes i.e.20ppm, 40ppm, 60ppm and 80ppm were prepared to perform experiments using photocatalyst CuO-Ag_2S having dose of 0.015g. It was shown that the rate of percent degradation decreased above 60ppm for Eosin B and above 20ppm for MB as shown in fig 8(c). This happened because, as the concentration of dye increased, more organic pollutants adsorbed on the surface of catalyst and blocked the active sites reducing its capability to degrade the Eosin B and MB or preventing the UV light from reaching the photo catalyst's surface and reducing the production of free radicals which are the essential species for the photocatalytic degradation process.

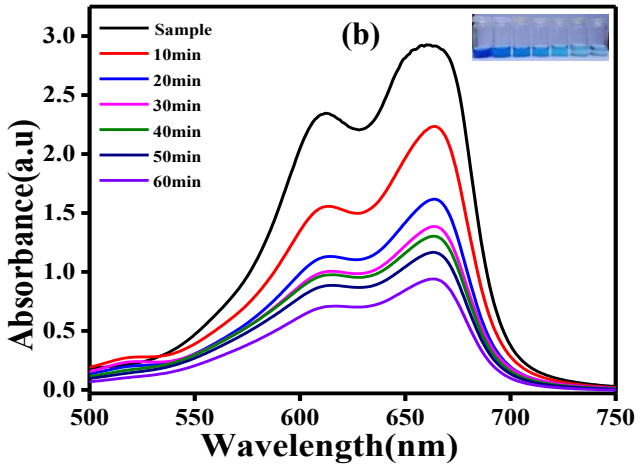
Effect of catalyst concentration

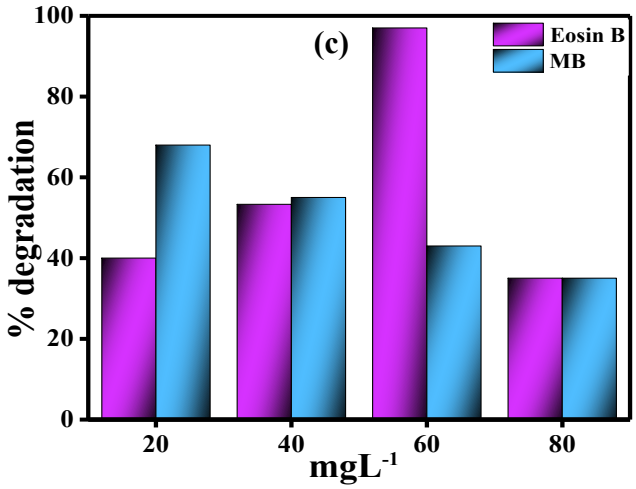
The amount of catalyst is important and should be optimized to avoid using too much. To study its effect, different amounts of the photocatalyst CuO-Ag_2S ranges from 0.01g- 0.25g were tested for the photodegradation of Eosin B and MB dyes. This study revealed the 0.015g concentration of the catalyst as an optimized dose as shown in figure 8(d). At this concentration the degradation efficiency was highest after 60 min of irradiation time. Further increase in catalyst concentration decreased the percent degradation due to decrease in the absorption of light as a consequence of its scattering and screening. Therefore, 0.015g dose was used for further experiments to achieve the highest degradation for both dyes.

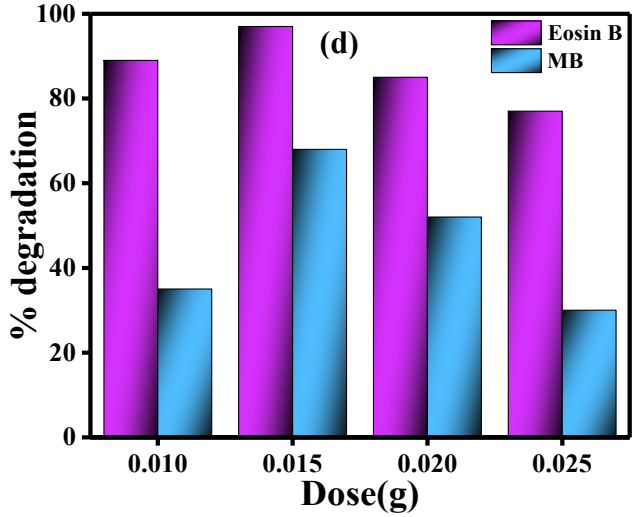
Figure 8

(a) Time study of Eosin B (b) Time study of MB (c) ppm study (d) Dose study









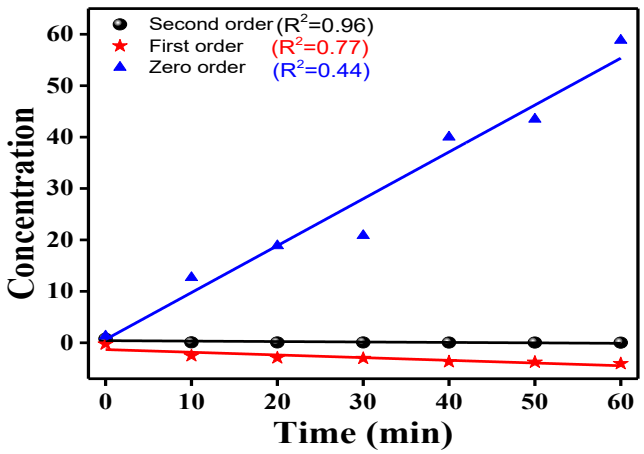
Kinetics

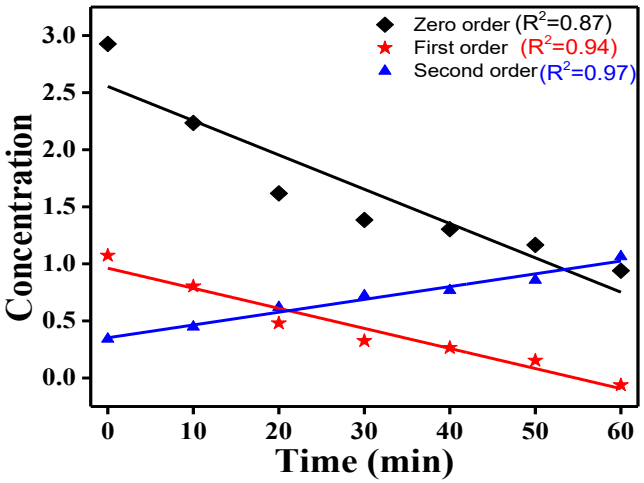
To investigate the kinetics of photodegradation of Eosin B and MB by CuO-Ag_2S, zero, first, and second models were applied. The following equations were used for applying these models. From the results it was concluded that both the processes follow pseudo-second order which best fit the model having correlation coefficient (R^2) values 0.96 for Eosin B dye degradation process as shown in fig 9(a) and 0.97 for Methylene B dye degradation process as shown in fig 9(b).

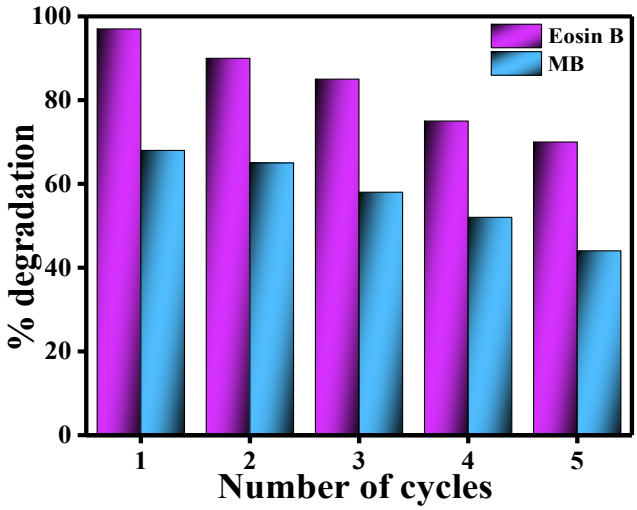
$$\frac{C0}{Ct} = kt \qquad \ln\frac{C0}{Ct} = kt \qquad \frac{1}{Ct} - \frac{1}{C0} = kt$$

Figure 9

(a) Kinetics of Eosin B dye (b) Kinetics of Methylene B dye







The pH effect

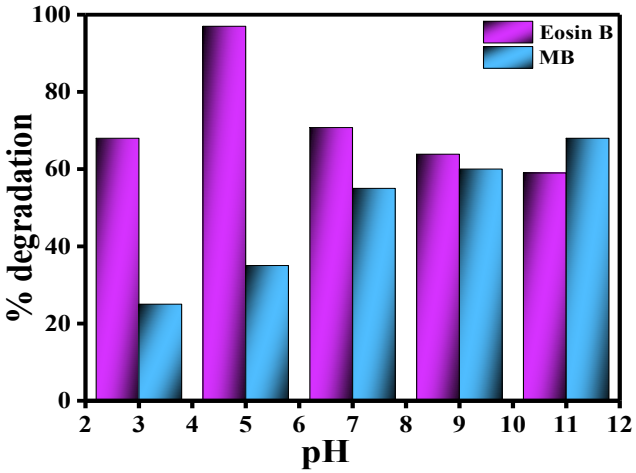
The pH of the solution highly influenced the photocatalytic degradation of Eosin B and MB dyes by CuO-Ag_2S. The pH was adjusted based on the nature of the dyes to improve the adsorption of these toxic dyes on the surface of the photocatalyst. The degradation efficiency was examined in the pH range of 3, 5, 7, 9 and 11. The highest degradation was observed at pH 5 for Eosin B as depicted in fig 10(a) and at pH 11 for MB. At these pHs a strong electrostatic interaction was created between the catalyst and Eosin B and MB. At basic pH the degradation activity of Eosin B decreased due to increased competition between $OH^{\bullet}$ and the dye molecules for the active sites on the photocatalyst surface. While, the degradation activity of MB in acidic medium decreased due to the repulsive forces between the photocatalyst and the dye.

Recyclability

The catalyst was reused five times for the photocatalytic degradation of Eosin B and MB dyes to evaluate the recyclability of the synthesized photocatalyst or to check its capability that either it performs another photocatalytic degradation cycle for Eosin B and MB or not. Distilled water and ethanol were used for the washing of the catalyst after its recovery from these dye solutions. After drying and reusing the CuO-Ag_2S photocatalyst for 5X in different solutions of Eosin B and MB, it still showed 70% efficacy as shown in fig 10(b) and degraded the dye effectively.

Figure 10

(a) pH study (b) Recyclability



CONCLUSION

In this research, we investigated the degradation of Eosin B and MB dyes (commonly used synthetic dyes in industries) using a composite photocatalyst CuO-Ag_2S. Their presence in water cause significant environmental and health issues. Our study aimed to explore an effective photocatalyst for their degradation under specific conditions. The composite CuO-Ag_2S was synthesized by hydrothermal method and its synthesis was confirmed by XRD, EDX, SEM and FTIR. The results showed that the prepared photocatalyst displayed the capacity to degrade the dyes up to 97% and 68% under optimum conditions i.e. 60ppm, 0.015g dose, 60min and pH 5 for Eosin B and 20ppm, 0.015g dose, 60min and pH 11 for MB in the presence of light, which highlights the versatility of the material and its potential application as environmental clean-up tool for dyes pollution. Most of the dye molecules were fully decomposed into simple, harmless substances i.e. CO_2 and H_2O. This indicates that the degradation process was highly effective. Additionally, the photocatalyst remained stable throughout the process. It could be used repeatedly up to 5 times without losing its ability to break down the dyes efficiently.

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Contribution statement

Shahzaib Ahmad: Writing – review & editing. **Ayesha khan:** Data curation. **Nimra Saeed:** Investigation. **Shagufta:** Formal analysis. **Muhammad Idrees:** Supervision. **Muhammad Adnan:** Revision and Final check.

Data availability

No datasets were generated or analyzed during the current study.

Declaration

Ethics approval and consent to participate

Not applicable.

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