



Morphology convenient flower like nanostructures of CdO-SiO₂ nanomaterial and its photocatalytic application

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ABSTRACT

Highly efficient CdO-SiO₂ nanomaterial was synthesized by Sol-gel method and sonication technique. The nanomaterial was characterized by Field-emission Scanning electron microscopy (FE-SEM) with elementary dispersive X-ray (EDX), High-resolution transmission electron microscopy (HR-TEM), photoluminescence spectroscopy (PL) and Ultraviolet-Visible diffuse reflectance spectra (DRS). The photocatalytic activity of CdO-SiO₂ nanomaterial was studied from the photodegradation of Rh B as it has maximum efficiency at pH = 7 under UV-light irradiation at 365 nm. The photodegradation of dye various Catalyst loading and dye concentration is parameters have been studied. The hydroxyl radical formation in the mechanism was confirmed by fluorescence quenching technique. The mineralization of dye is confirmed by chemical oxygen demand measurements. An achievable mechanism is proposed for higher activity of CdO-SiO₂ under UV-light at 365 nm. This catalyst was found to be stable and reusable.

Keywords: FE-SEM; HR-TEM; Photocatalytic activity; ·OH analysis

1. INTRODUCTION

This CdO-SiO₂ is large interest for many applications from catalysis, medicine and .sensing [1-4]. The silica used can be tailored to act as water, thus pretty the ability to maintain the biological activity of the entrapped antibodies, enzymes and cell [5-7]. Cadmium oxide nanomaterial is a exceptional photocatalyst for optoelectronic applications [8,9]. Rhodamine B dye is useful in various industries such as food, textiles, paper and cosmetics. Advanced Oxidation Process (AOP) is a photocatalysis in water treatment technique (10, 11). In this present work, CdO-SiO₂ nanomaterial was synthesised by sol-gel method and sonication technique.

The synthesized nanomaterial was characterized by Field-emission Scanning electron microscopy (FE-SEM) with elementary dispersive X-ray (EDX), High-resolution transmission electron microscopy (HR-TEM), Fourier transform-Raman spectra (FT-Raman), photoluminescence spectroscopy (PL) and Ultraviolet-Visible diffuse reflectance spectra (DRS).

The photocatalytic activity of CdO-SiO₂ nanomaterial was studied from the photodegradation of Rh B as it has maximum efficiency at pH = 7 under UV-light irradiation at 365 nm. The photodegradation of dye various Catalyst loading and dye concentration is parameters have been studied. The hydroxyl radical formation in the mechanism was confirmed by fluorescence quenching technique. The mineralization of dye is confirmed by chemical oxygen demand measurements. A achievable mechanism is proposed for higher photocatalytic activity. This catalyst was found to be stable and reusable

2. EXPERIMENTAL

2. 1. Reagents

Tetraethoxysilane (TEOS), Cadmium acetate dehydrate, Hydrochloric acid and ethanol (C₂H₅OH) were the guaranteed reagents of Sigma Aldrich and aqueous solutions were prepared by using deionized water.

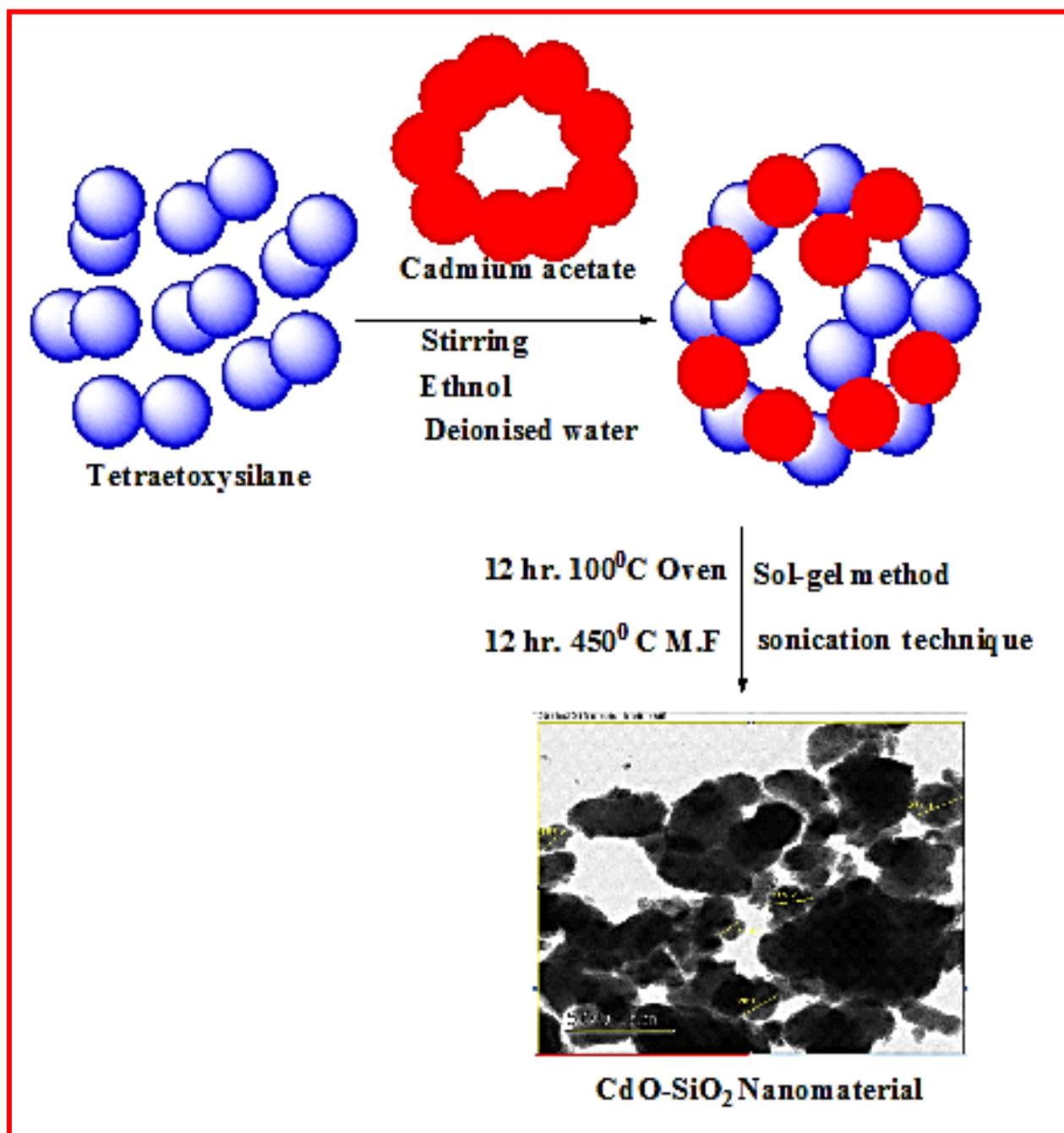
2. 2. Synthesis of the CdO-SiO₂ nanomaterial

The CdO-SiO₂ nanomaterial has been obtained synthesized by the sol-gel method. In the synthesis of SiO₂ nanomaterial, tetraethoxysilane (TEOS) (1M) prepared by dissolving in solvent ethanol (15 ml) and deionized water (35 ml) mixture was stirred for 30 min, and then added cadmium acetate dihydrate (0.2 M) with catalyst HCl (1M) drop wise into the above mixture and stirred magnetically 50 min at temperature 60 °C and to become a white transparent homogeneous solution than sonication 40 min. Now the prepared sample is kept for aging about 3 hr for gel preparation at room temperature. The suspensions obtained were dried in an oven for 12 hr at 100 °C; CdO-SiO₂ white powder. The obtained powder sample was calcined in muffle furnace for 12 hr at temperature 450 °C for further characterization.

2. 3. Photocatalysis

The photocatalytic activities of the photocatalysts (SiO₂ and CdO-SiO₂) were evaluated by the photodegradation of dye. The light source was UV lamp at 365 nm. The reaction was carriedout at ambient temperature (303 K). In a typical experiment, aqueous suspensions of

dye (40 mL, 1×10^{-4} M) and 0.150 g of photocatalyst were loaded in reaction tube of 50 mL capacity. Prior to the irradiation, the suspension was magnetically stirred in dark to ensure the establishment of an adsorption/desorption equilibrium. The suspension was kept under constant air-equilibrated condition. At the intervals of given irradiation time. The suspension was measured spectrophotometrically Rh B dye within the Beer–Lambert law limit.



Scheme 1. Preparation of CdO-SiO₂ nanomaterial

2. 4. Analytical Methods

The Field-emission Scanning electron microscopy (FE-SEM) with elementary dispersive X-ray analysis (EDX) was carried out on a FEI Quanta FEG 200 instrument with EDX analyzer facility at 25 °C.

The sample was prepared by placing a small quantity of prepared material on a carbon coated copper grid and allowing the solvent to evaporate. The morphology of the sample was examined using a JEOL 3010 high-resolution transmission electron microscope (HR-TEM). FT-RAMAN spectra were recorded with an integral microscope Raman system RFS27 spectrometer equipped with 1024 - 256 pixels liquefied nitrogen-cooled germanium detector.

The 1064 nm line of the Nd:YAG laser (red laser) was used to excite. To avoid intensive heating at the sample, the laser power at the sample was kept not higher than 15 mW. Each spectrum was recorded with an acquisition time of 18s. Photoluminescence (PL) spectra at room temperature were recorded using a Perkin-Elmer LS 55 fluorescence spectrometer.

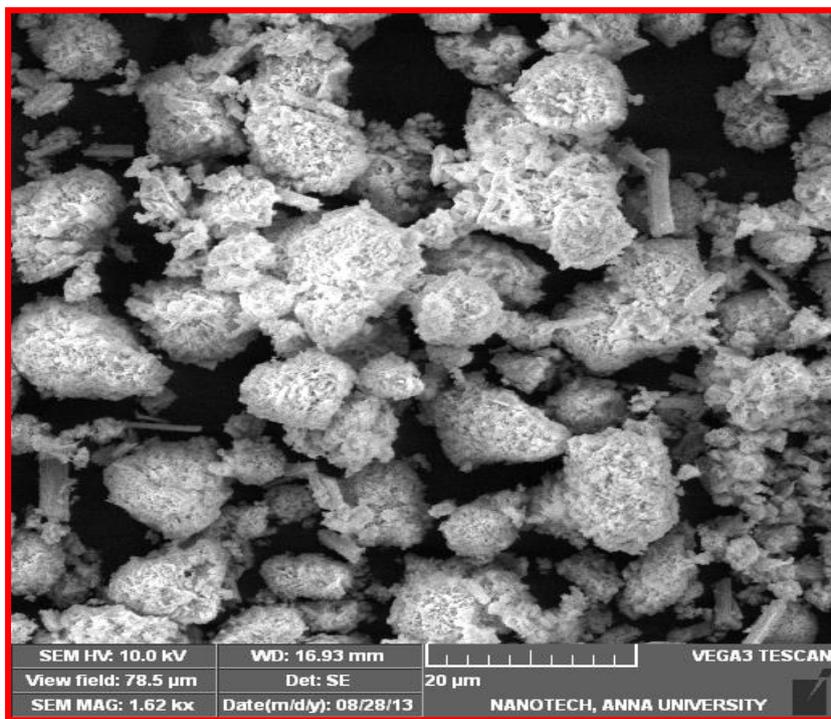
The crystallinity was characterized by an UV-vis DRS the direct band gap energy, was analyzed UV-visible (Shimadzu UV-1650 PC) spectrophotometer. UV spectral measurements were done using a Hitachi-U-2001 spectrometer. Ultraviolet and visible (UV-vis) absorbance spectra were measured over a range of 800-200 nm with a Shimadzu UV-1650PC recording spectrometer using a quartz cell with 10 mm of optical path length. Fluorescence technique with coumarin (1 mM of 4-hydroxycoumarin) were measured on a Hitachi F-7000 fluorescence spectrophotometer

3. RESULTS AND DISCUSSION

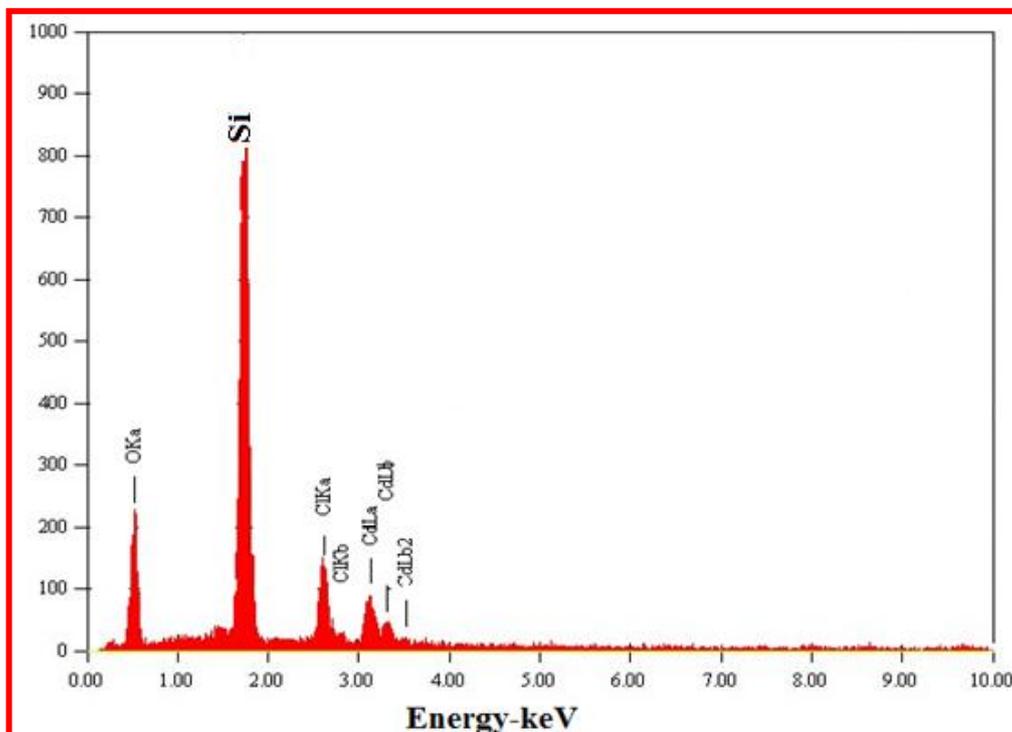
3. 1. FE-SEM with EDX analysis

The sample of SiO₂ and CdO-SiO₂ were prepared by sol-gel method. Scanning electron microscope image was done to study the morphology as show in (Fig. 1a). SEM micrographs (Fig. 1a) reveal that the particles are nano flower in shape. The SiO₂ small particles have been agglomerated and the particles were slightly better in size. The average particle size of SiO₂ sample is 76 nm and CdO-SiO₂ is 89 nm. CdO-SiO₂ sample observed that this sample has smaller particle size than that of SiO₂.

Hence CdO-SiO₂ is effective due to less agglomeration which produces more surface area so high photocatalytic activity. EDX analysis is shown in (Fig. 1b). The presence of Cd, Si and O is confirmed from the peaks at 3.2 KeV, 1.8 KeV and 1.5 KeV various respectively.



(a)



(b)

Fig. 1. FE-SEM images of (a) CdO-SiO₂ nanomaterial and (b) CdO-SiO₂ EDX analysis

3. 2. HR-TEM analysis

The High-resolution transmission electron microscope (HR-TEM) measurements of CdO-SiO₂ are shown in Fig. 2a it is established that the presence of particles are depicted from the HR-TEM micrographs of the mixed nanomaterial at 500 nm as well crystalline and spherical shaped. Fig. 2b represents an image surface plot, and Fig. 2c particle size distribution 195.6 nm by selected particle area highlighted show in Fig. 2a.

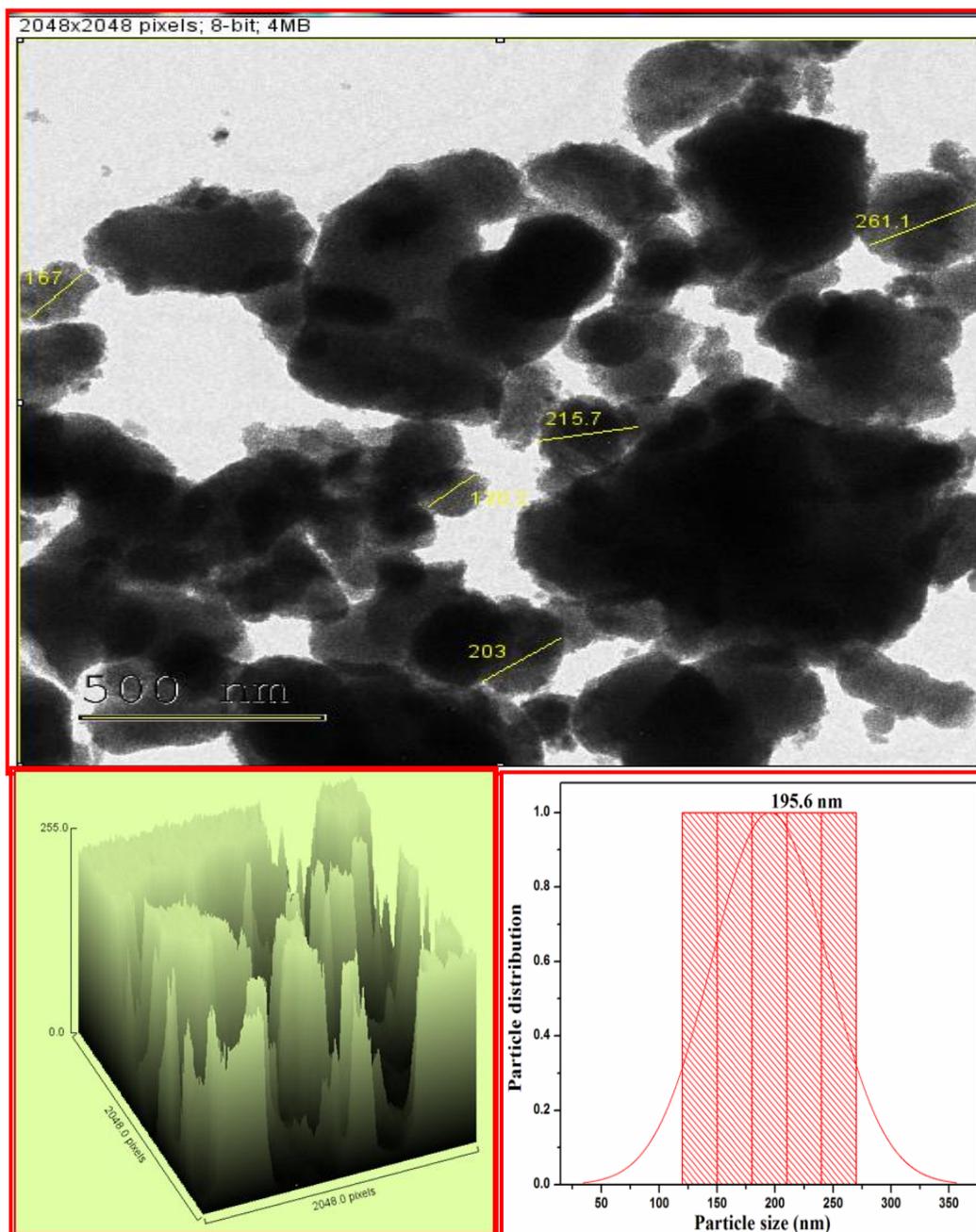


Fig. 2. TEM analysis of (a) Image of CdO-SiO₂ nanomaterial (b) surface plot and (c) particle size in selected area highlighted fig (a)

3. 3. PL analysis

PL analysis of SiO_2 and CdO-SiO_2 nanomaterial are shown in Fig. 3 (a and b) respectively. As photoluminescence occurs due to electron/hole pairs under equivalent light irradiation important to highly developed photocatalytic activity, the intensity is directly proportional to the rate of electron-hole recombination. [12,13] The emission spectra of synthesis CdO-SiO_2 show that both section exhibited characteristic green emission in the range 350-600 nm. It is well-known that the PL of a nanomaterial is due to radioactive recombination process of generated electron/hole pairs. Therefore, the decreased PL intensity of CdO-SiO_2 as a low recombination rate of electron/hole pairs, through a explanation for the enhancement in photocatalytic activity

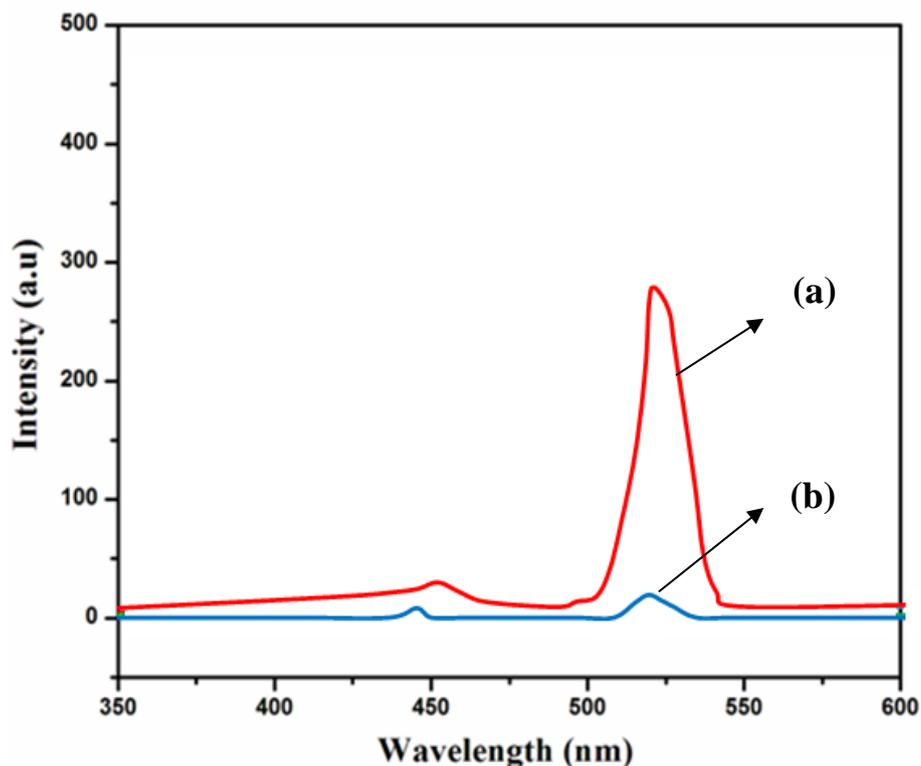


Fig. 3. Photoluminescence spectra of (a) SiO_2 (b) CdO-SiO_2 nanomaterial

3. 4. UV-Vis-DRS analysis

The UV-Vis-DRS spectra of SiO_2 and CdO-SiO_2 are shown in Fig. 4. The direct band gap of the synthesized material has been resolute from the tauc plots. The plots of $[F(R) h\nu]^2$ Vs the photon energy ($h\nu$) give the direct band gap of the synthesized SiO_2 and SiO_2 as 5 eV and 4.6 eV, correspondingly. The UV-Vis-DRS consequences confirm a decrease in the direct band gap of CdO-SiO_2 that of SiO_2 . The band gap energy obtained from the plot of the modified Kubelka-Munk function $(F(R) E)^{1/2}$ versus the energy of the absorbed light E by the equation -- (1)

$$F(R) E^{1/2} = \left[\frac{(1-R)^2}{2R} X h \nu \right]^{1/2} \text{----- (1)}$$

UV-Vis-DRS results established a decrease in the direct band gap of CdO-SiO₂ compared to SiO₂. The results recognized CdO-SiO₂ having high photocatalytic and UV-Vis activity show in Fig. 4. (a and b).

3. 5. Photocatalytic activity

3. 5. 1. Effect of solution pH

Effect of solution pH may also effect the degradation of Rh B. The effect of pH on the degradation Rh B was inverstigated in the pH range of 3-9 and the results are shown in Fig. 5. It was observed that the degradation increases with an increase in pH up to 7 and then decreases. After 60 min of irradiation the Rh B degradation values were 35, 73, 95 and 53 % at pH 3, 5, 7 and 9 respectively. The optimum pH was found to be 7 for Rh B degradation. The low removal efficiency in the acidic pH range was due to the dissolution of the SiO₂ in CdO-SiO₂. CdO-SiO₂ is more effective than SiO₂ in the degradation of Rh B because it has maximum efficiency at pH = 7.

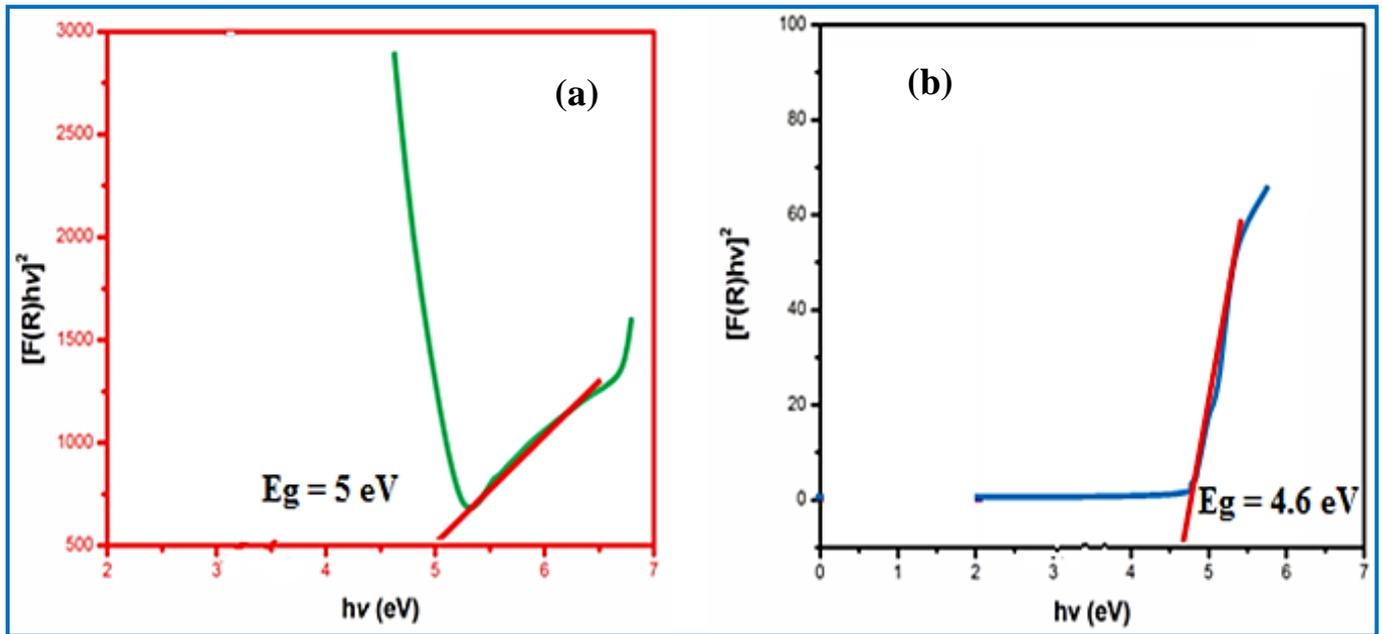


Fig. 4. Plot of transferred Kubelka–Munk versus energy of the light absorbed of the (a) bare SiO₂, (b) CdO-SiO₂ nanomaterial

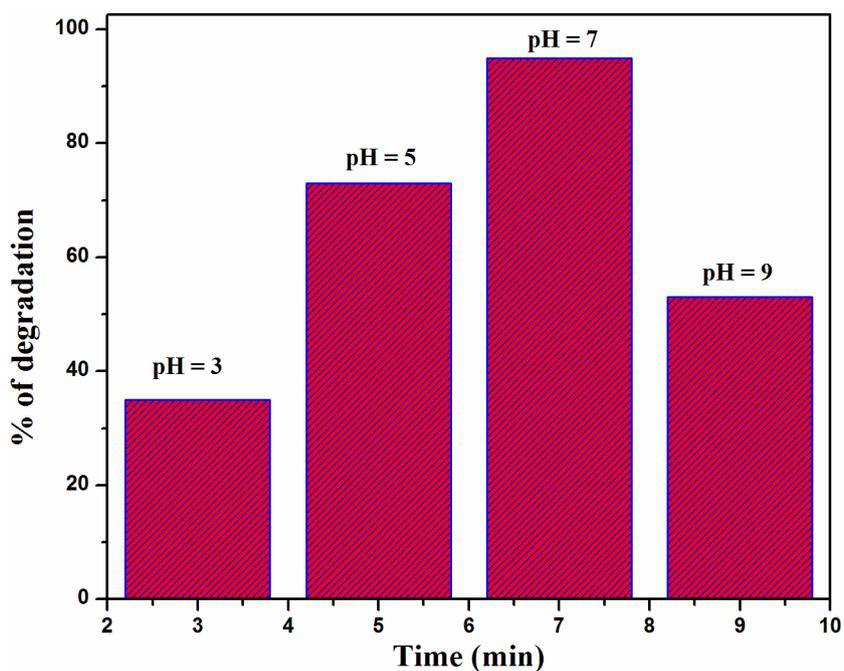


Fig. 5. pH analysis of CdO-SiO₂ nanomaterial

3. 5. 2. Degradation of Rh B dye

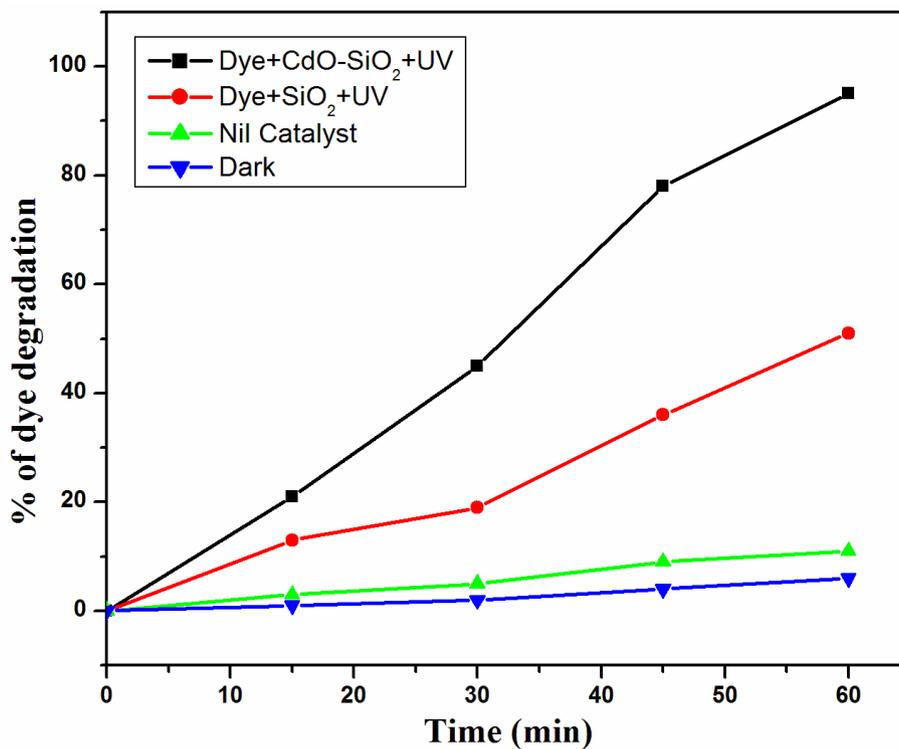


Fig. 6. Primary analysis: photodegradation of Rh B under UV-light (365 nm) irradiation by CdO-SiO₂ nanomaterial

CdO-SiO₂ nanomaterial can degrade the Rh B in aqueous solution up to (0, 21, 45, 78 and 95 %) shows superior photocatalytic activity when compare to that of SiO₂ (0, 13, 19, 36 and 51 %), Nil catalyst (0, 3, 5, 9 and 11) and Dark (0, 1, 2, 4 and 6) The reaction under UV-light at 365 nm. The photocatalytic degradation is in the order of the catalyst material used CdO-SiO₂ > SiO₂ > Nil catalyst > Dark are shown in Fig. 6.

3. 5. 3. The effect of catalyst loading

The effect of catalyst loading affect the degradation of Rh B and hence different amounts of CdO-SiO₂ were used. The catalyst loading in photocatalytic processes is an significant feature that can strongly influence dye degradation. different of the amount of CdO-SiO₂ suspended in the reaction medium leads to increase of the degradation at catalyst loading amounts of 0.100 g, 0.150 g and 0.200 g, and the total volume of solution used in each 40 mL are shown in Fig. 7. This study can be explained in terms of the accessibility of active sites on the catalyst surface and access of UV-light at 365 nm into the suspension. The total active surface area increases with increasing catalyst loading. However, with excess catalyst dosage there is a decrease in UV-light penetration as a result of increased light scattering by photocatalyst particles. As a result, the photo activated volume of the suspension decreases. Additionally, it is important to keep the treatment expenses low for industrial use. Thus, we used 0.150 g as the optimal catalyst amount in high activity in our work. (CdO-SiO₂ on Rh B) catalyst was faster than SiO₂. The degradation increases with increases 0.150 g (0, 21, 45, 78 and 95%) then decreases 0.200 g (0, 3, 7, 12 and 16.5 %) on Rh B dye.

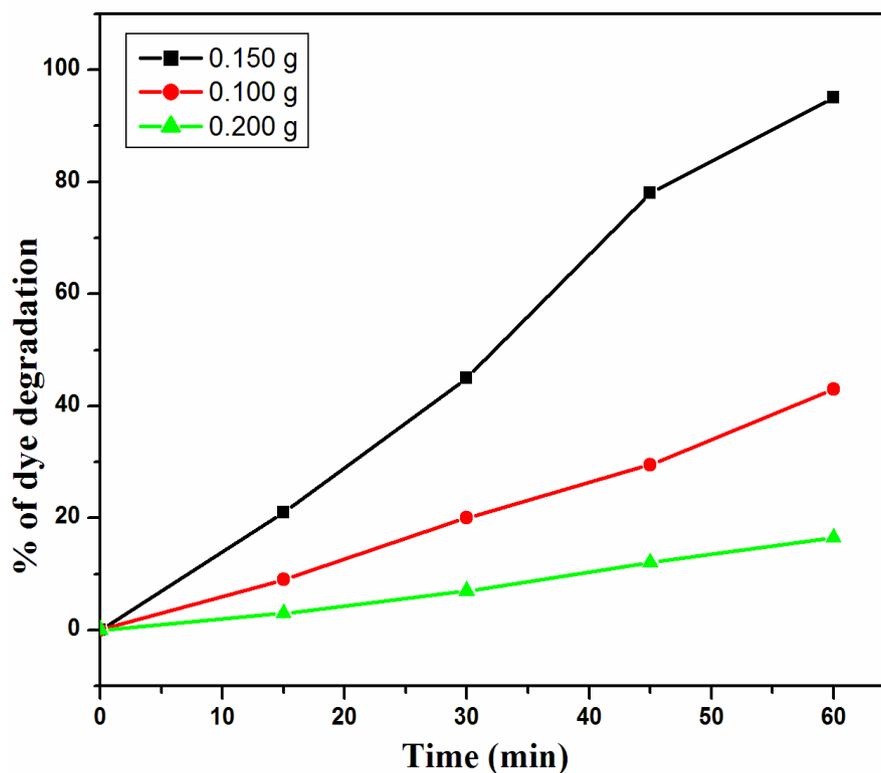


Fig. 7. The effect of Catalyst loading CdO-SiO₂ nanomaterial

3. 5. 4. The effect of dye concentration

The effect of dyes concentration of Rh B, and hence different amounts of dye concentration with CdO-SiO₂ material were used. The dyes concentration in photocatalytic processes is an significant feature that can strongly influence dye degradation. Different dye concentration of CdO-SiO₂ suspended in the reaction medium leads to increase of the degradation at 1x10⁻⁴ than decrees 2x10⁻⁴ the total volume of solution used in each 40 mL are shown in Fig. 8. This study can be explained in terms of the accessibility of active sites on the catalyst and access of UV-light at 365 nm into the suspension. The effect of dye concentration is explained on the basis that on increasing the concentration of Rh B, more molecules of Rh B are available for degradation. However on increasing of degradation concentration 1x10⁻⁴ M then 2x10⁻⁴ decrease in CdO-SiO₂ nanomaterial.

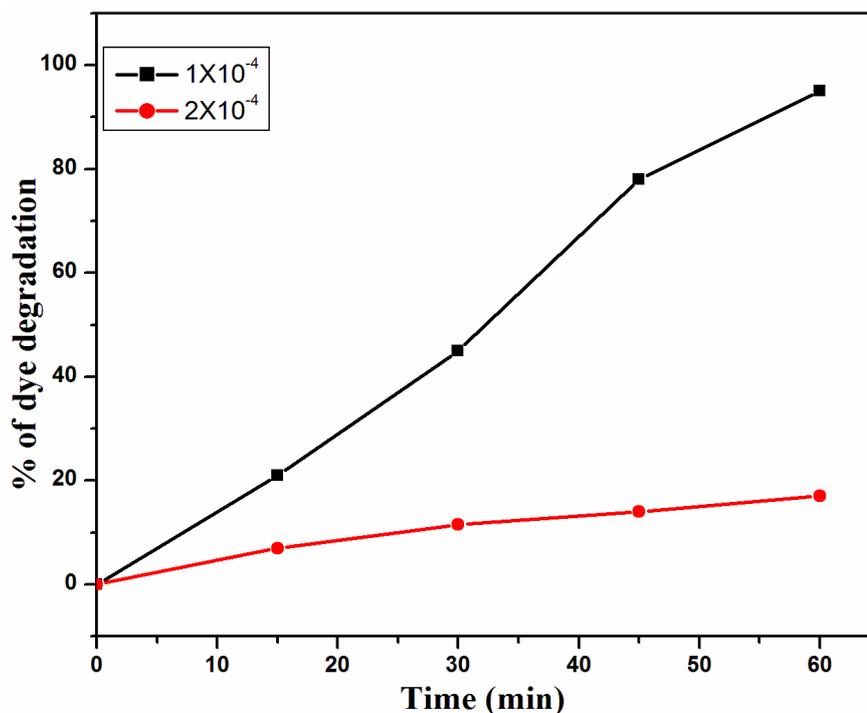
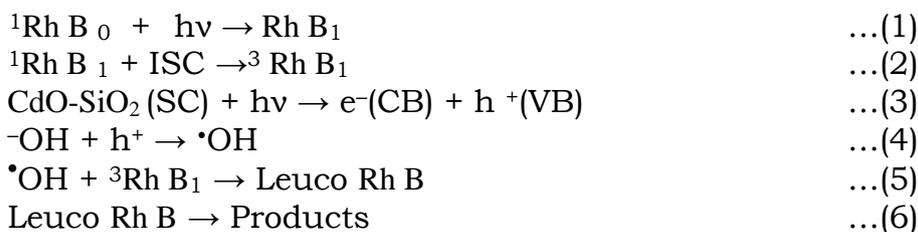


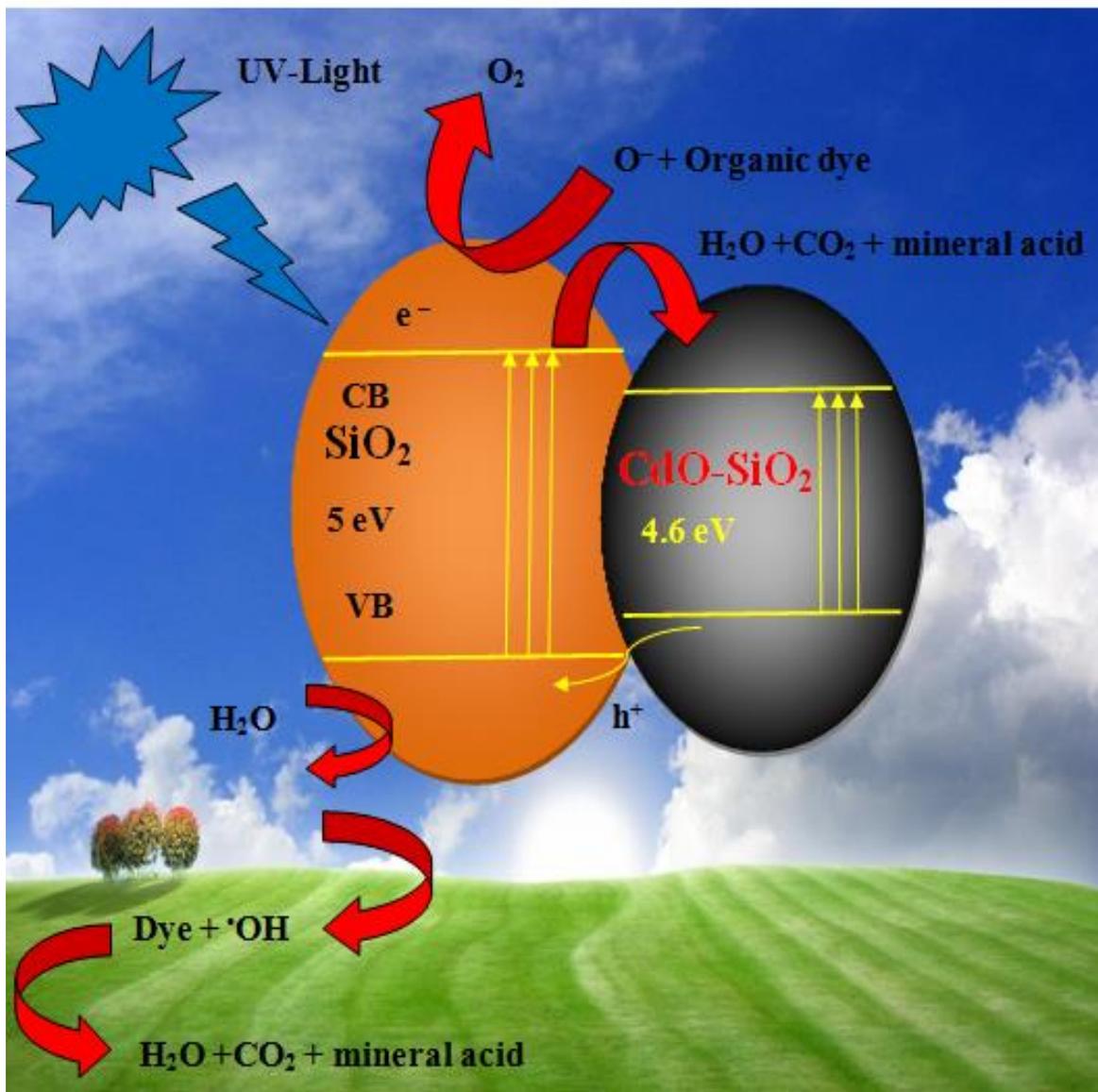
Fig. 8. The effect of dye concentration of CdO-SiO₂ nanomaterial (1x10⁻⁴ and 2 x10⁻⁴)

Mechanism

Tentative mechanism by CdO-SiO₂ under UV-light irradiation on Rh B dye is proposed as follows:



Rh B dye absorbs radiation of preferred wavelength and it forms excited singlet state. Promote, it undergoes intersystem crossing (ISC) to give its more stable triplet state. Along with this, the semiconducting CdO-SiO₂ (SC) also utilizes this energy to excite its electron from valence band to the conduction band. An electron can be abstracted from hydroxyl ion (⁻OH) by hole (h⁺) present in the valence band of semiconductor generating [•]OH. This [•]OH will oxidize Rh B to its leuco form, which may ultimately degrade to products. It was confirmed that the [•]OH radical participates as an active oxidizing species in the degradation of Rh B as the rate of degradation was appreciably reduced in presence of hydroxyl radical scavenger (2-propanol) Shown in Scheme 2 [14].



Scheme 2. Schematic diagram of photodegradation of Rh B on CdO-SiO₂ nnomaterial under UV- light for successive mineralization.

3. 5. 5. Hydroxyl radical analysis

The photocatalytic activity of the prepared SiO₂ and CdO-SiO₂ nanomaterial was further confirmed by the detection of [•]OH from the change in the fluorescence spectra with coumarin solution at UV-light irradiation in 45 min. It is suggested that the fluorescence intensity arises [13,15,16] from the chemical reaction between coumarin solution and [•]OH occurrence of the emission peaks in the UV-visible region is due to the electronic transitions mediated in the defect levels such like oxygen vacancies in the band gap. [17,18] The hydroxyl radical analysis confirms the radicals are active group in photocatalytic reactions. On the other hand, the formation rate of [•]OH is directly related to the photocatalytic activity of CdO-SiO₂ nanomaterial. Formed on the illuminated CdO-SiO₂ nonmaterial show in Fig. 9.

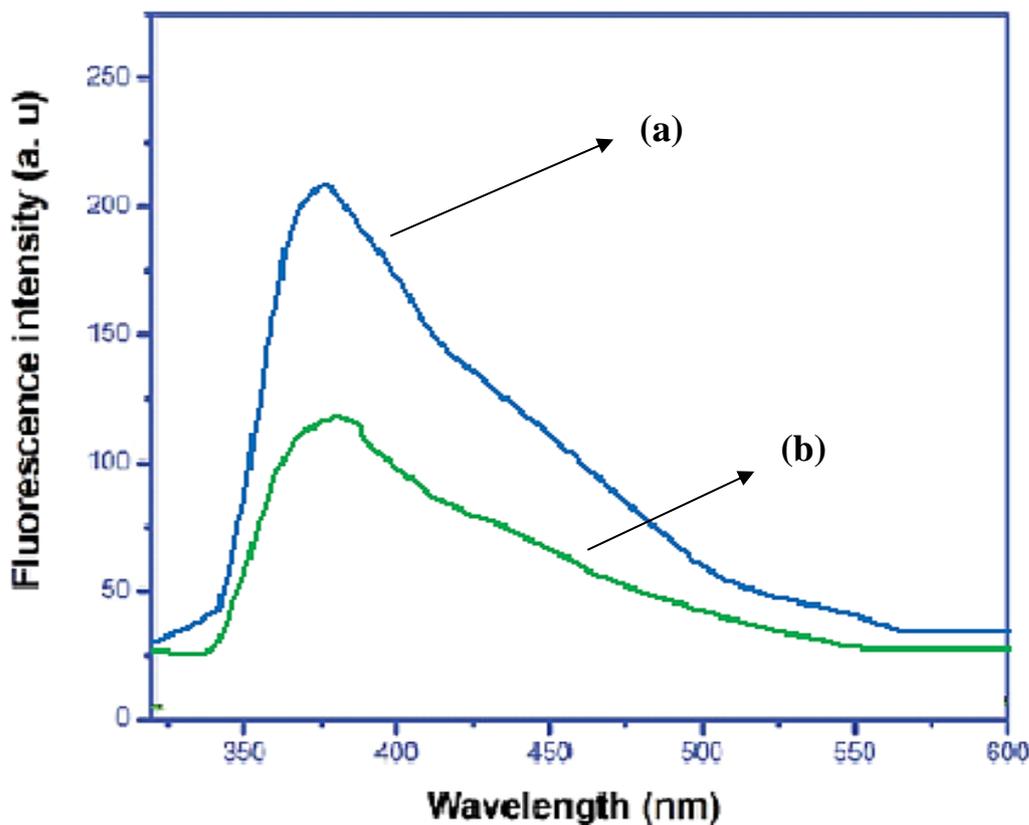


Fig. 9. Fluorescence spectra measured at $\lambda_{\text{max}} = 320 \text{ nm}$ for the (a) CdO-SiO₂ (b) SiO₂ samples obtained using optimum time (60 min) in coumarin solution (sample was illuminated for 60 min of UV light irradiation)

3. 5. 6. Stability and reusability

The stability and reusability of the CdO-SiO₂ nanomaterial are achieved by repeating Rh B degradation experiments for fifth more times. After each cycle the CdO-SiO₂ catalysts were washed thoroughly with water, and a fresh solution of Rh B dye was made before every run in the photoreactor. The complete degradation occurs in the Rh B [1st cycle (100 %), 2nd cycle

(98 %), 3rd cycle (97 %) and 4th cycle (97 %)] the photocatalytic efficiency of decreased gradually with reuse. The lost cycle a 3% loss in material was observed show in Fig. 10. The result indicates that the prepared catalysts are stable and reusable. After the completion of the degradation process, the solution was tested for Cd³⁺ by leaching with Na₂S. There is no precipitation of Cd³⁺ S (black color) was found. As there is no further leaching of Cd³⁺. So this responded material is non-hazardous for waste water treatment [19].

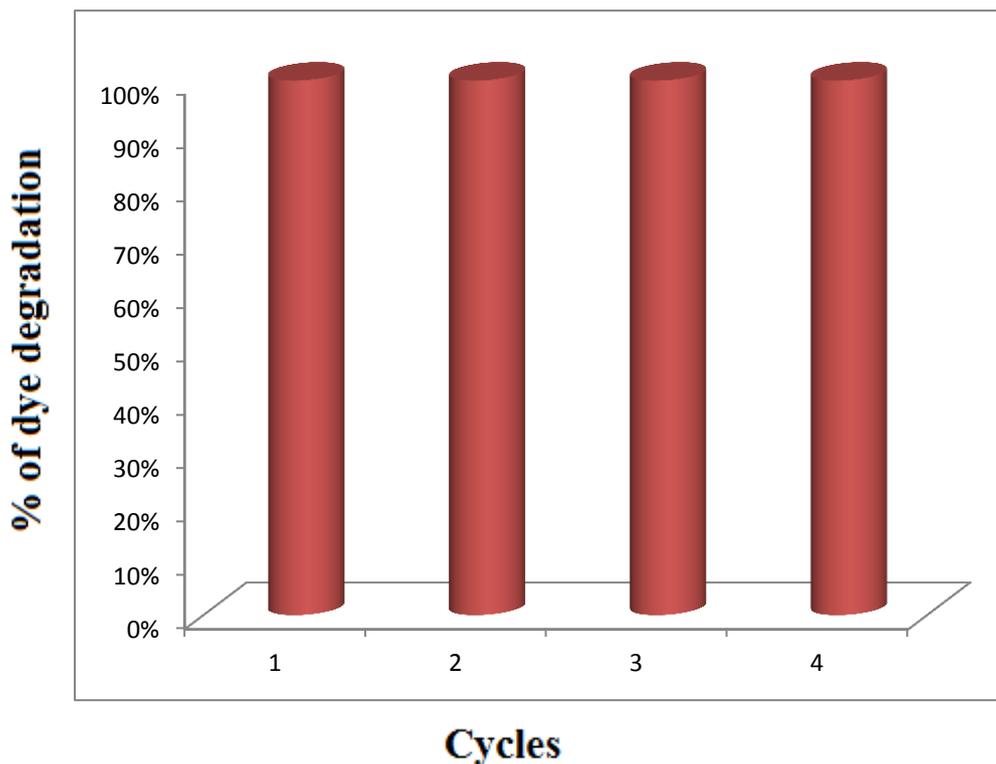


Fig. 10. Stability and reusability of CdO-SiO₂ on dye concentration = 1X10⁻⁴ and irradiation time = 60 min

3. 5. 7. Chemical oxygen demand analysis (COD)

COD analysis of Rh B on mineralization of CdO-SiO₂ photocatalyst loading amount of 0.150 g on dye concentration (1x10⁻⁴) suspension for 40 mL pH = 7 solution and air passing with UV- light at 365 nm.

The CdO-SiO₂ high % of Chemical oxygen demand analysis reduction of Rh B [0 (0%), 15 (20 %), 30 (42 %), 45 (76 %)] and 60 (94%) min than [0 (0%), 15 (13 %), 30 (18 %), 45 (35%) and 60 (50 %)] of Chemical oxygen demand analysis measurements reduction is obtained.

The mineralization is also specific by formation of calcium carbonate when the evolved gas (carbandedioxide) through photodegradation is accepted and calcium hydroxide is obtained show in Fig. 11.

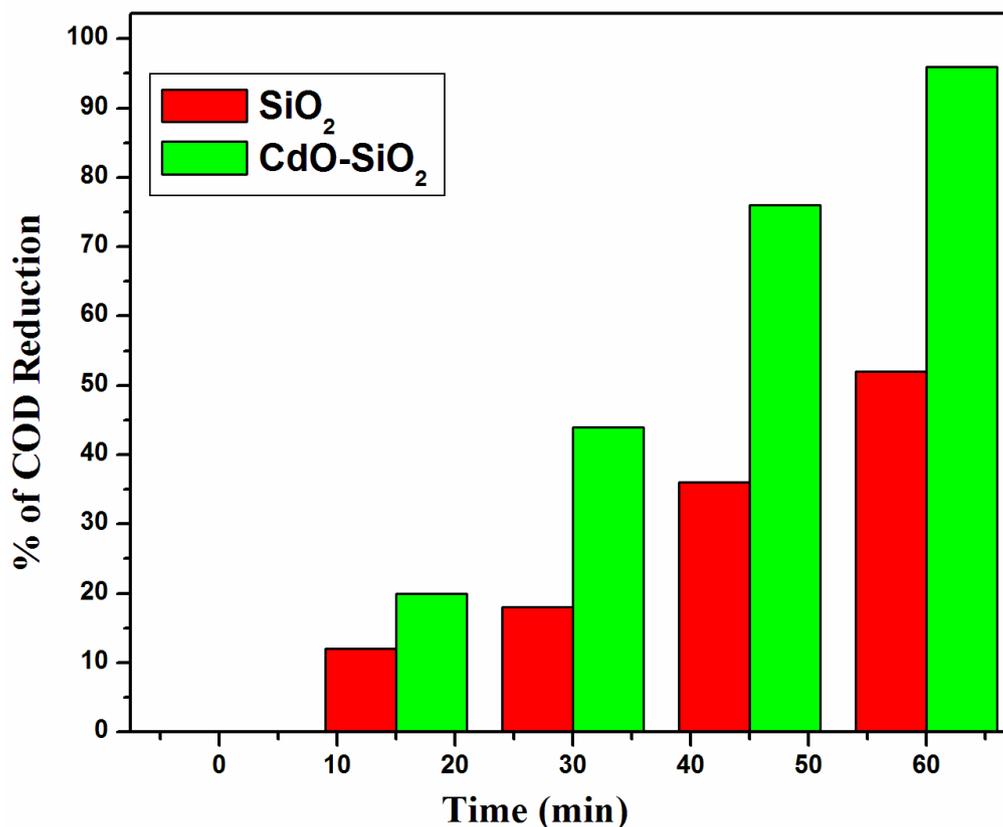


Fig. 11. Chemical Oxygen Demand (COD) analysis of SiO₂ and CdO-SiO₂ nnomaterial UV-light irradiation of dye

4. CONCLUSIONS

In this present work, CdO-SiO₂ nanomaterial was synthesised by sol-gel method and characterized by suitable analytical methods. These results confirmed FE-SEM analysis showed CdO-SiO₂ had a nano-flower like structure. EDX analysis conform revealed presence of Cd, Si and O in catalyst. HR-TEM showed CdO-SiO₂ had a nano-spherical like structure. PL spectra analysis showed repression of recombination of photogenerated electron-hole pairs by CdO-SiO₂. The UV-DRS spectra indicated a reduction in the band gab of the CdO-SiO₂ when compared to SiO₂.

The photocatalytic activity of CdO-SiO₂ nanomaterial was studied from the photodegradation of Rh B as it has maximum efficiency at pH = 7 under UV-light irradiation at 365 nm. The photodegradation of dye various Catalyst loading and dye concentration is parameters have been studied. The formation of [•]OH is directly related to the photocatalytic activity of the CdO-SiO₂ nanomaterial, which was confirmed by fluorescence analysis. The mineralization of dye is confirmed by chemical oxygen demand measurements. A achievable mechanism is proposed for higher photocatalytic activity. This catalyst was found to be stable and reusable such as mineralization of pollutants and water purification technology.

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CAPTIONS FOR SCHEME AND FIGURES

Fig. 1. FE-SEM images of (a) CdO-SiO₂ nanomaterial and (b) CdO-SiO₂ EDX analysis

Fig. 2. TEM analysis of (a) Image of CdO-SiO₂ nanomaterial (b) surface plot, (c) particle size in selected area highlighted fig (a)

Fig. 3. Photoluminescence spectra of (a) SiO₂ (b) CdO-SiO₂ nanomaterial

Fig. 4. Plot of transferred Kubelka–Munk versus energy of the light absorbed of the (a) bare SiO₂, (b) CdO-SiO₂ nanomaterial

Fig. 5. pH analysis of CdO-SiO₂ nanomaterial

Fig. 6. Primary analysis of photocatalytic activity for the photodegradation of Rh B under UV-light (365 nm) irradiation by CdO-SiO₂ nanomaterial

Fig. 7. The effect of Catalyst loading CdO-SiO₂ nanomaterial

Fig. 8. The effect of dye concentration of CdO-SiO₂ nanomaterial (1×10^{-4} and 2×10^{-4})

Fig. 9. Fluorescence spectra measured at $\lambda_{\max} = 320$ nm for the (a) SiO₂ (b) CdO-SiO₂ samples obtained using optimum time (60 min) in coumarin solution (sample was illuminated for 60 min of UV light irradiation),

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Scheme 1. Preparation of CdO-SiO₂ nanomaterial

Scheme 2. Schematic diagram of photodegradation of Rh B on CdO-SiO₂ nanomaterial under UV-light for successive mineralization