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Research Article

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Synthesis and Characterization of Copper Selenide Quantum Dots for Photocatalytic Application

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Abstract: Copper Selenide quantum dots were prepared by a simple wet chemical method known as the reflux condensation method from which uniform-sized crystalline nanocrystals were obtained. The resultant nanocrystals strong confinement, phase shift, morphology, and purity has been found with the use of characterization technique such as by X-ray diffraction (XRD), UV–Visible spectroscopy (UV–Vis), Photo-luminescent spectroscopy (PL), Raman Shift (RS) and Scanning Electron Microscopy (SEM). The Experimental investigation of XRD, SEM reveals that the CuSe obtained are with high crystalline crystals which exhibits hexagonal structure and the diameter ranges from 10-15nm. The UV-Vis and PL studies revealed that there is a large "blue shift". Hence by this large shift, the material can be used for Photo-degradation and Photocatalytic activity. The photocatalytic activity was done with the Rhodamine B Dye (RhB). The result shows an effective degradation of 83%. Future nanocrystals can be used for developing potential electronic devices such as solar cells.

Keywords: CuSe, Quantum Dots, Reflux Condensations, Rhodamine B, Photocatalytic Activity

1. Introduction

Quantum dots are nanoparticles in the form of crystals that exhibit semiconducting properties with a diameter in the range of 2-10 nanometers [1]. It has distinctive electronic properties, which is an intermediate between those of bulk semiconductors and discrete molecules, these results in a high surface-to-volume ratio for these particles. [2-4]. Quantum dot exhibits Fluorescence which determines the size of the particles. Because of their bright and pure color, it exhibits a band of colors together with their high efficiencies, high excitation co-efficient, and longer lifetime they are much employed in the optical applications [5-7].

Currently, Metal-selenide QDs have found to be broadly studied which exhibit excellent electrical, optical, and optoelectrical properties that absorb light in the visible region and transfer electrons in the larger bandgap semiconductors. [8–11] Among these, the copper selenide (CuSe) QDs are found to be the most interesting metal which can be used as an alternative for optoelectronic devices [12]. CuSe appears in different phases and forms: such as CuSe, Cu_2Se, and Cu_3Se_2, etc, and also non-stoichiometric form ($Cu_{2-x}Se$) and multiple crystal structures like orthorhombic, hexagonal, tetragonal, cubic, etc. [13]. Though a small amount of selenide in nature is toxic, it counters effects with the other metal toxins to remove the toxicity and which can be used for a wide range of application such as solar cells, superionic conductors, photodetectors, photothermal conversion, electroconductive electrodes, microwave shielding, coating, thermoelectric cooling, optical filter, photo-catalysis as an optical recording material [14-21]. Among these Photocatalytic activity found to important because of its potential application in the depollution technology to remove hazardous materials from water. Also, nanostructured semiconductors are very much in use to find a solution to environmental issues. [22]. consequently, to exhibit significant photocatalytic activity for photodegradation of organic contaminants, the nanostructures semiconductors can be employed. [23].

Therefore the present work involves the development of CuSe nanocrystal quantum dots by a novel method of preparation along with the control of their size and shape which is used for photocatalysis activity to remove the impurities in water. In this article, the reflux condensation method is used because of its low cost and highly sufficient material. The Reflux procedure involves the condensation of vapors and the return to the original system. The common precursors used here is a simple metal material such as copper and selenium. To prepare the Cuse nanocrystalline quantum dot usually hydrazine hydrate has been employed which acts as a reducing agent [23]. Here in this study hydrazine hydrate or any other reducing agents are not employed, instead, the Sodium hydroxide is used which helps in maintaining the pH. The experiment is carried out at various temperatures and time duration, to obtain the nanocrystal less than 10nm.

2. Experimental Setup

2.1. Materials Used

Copper chloride, Selenium metal powder, Sodium hydroxide was purchased from Sigma Merck. All chemical were in analytical grade and used as such without any further refinement.

2.2. Synthesis Route

The reaction solution was prepared by dissolving 0.4g of cuprous chloride ($CuCl_2.2H_2O$) and 0.545g of selenium metal powder in 40ml of distilled water under mild stirring at room temperature. After 5mins of stirring, 3.2g of sodium hydroxide (NaOH) is dissolved in distilled water and added dropwise until it reaches a pH of 12. The reaction mixture is transferred into a round bottom flask and fitted to the reflux condenser and heated to a specific temperature under vigorous stirring. The reaction mixture was maintained at a different temperature like 50ºC, and 70ºC with a time duration of 4hrs, 8hrs, and 12hrs which is given in Table 1. The flow diagram of the synthesis of CuSe Quantum Dots is shown in Figure 1.

Table 1. CuSe quantum dot formation with different temperature and time duration with NaOH as the base

S.No	Precursor	Base	pH	Temperature ºC	Reaction Time (hrs)		
1	Copper Chloride and selenium metal powder	NaOH	12	50	4	8	12
2	Copper Chloride and selenium metal powder	NaOH	12	70	4	8	12

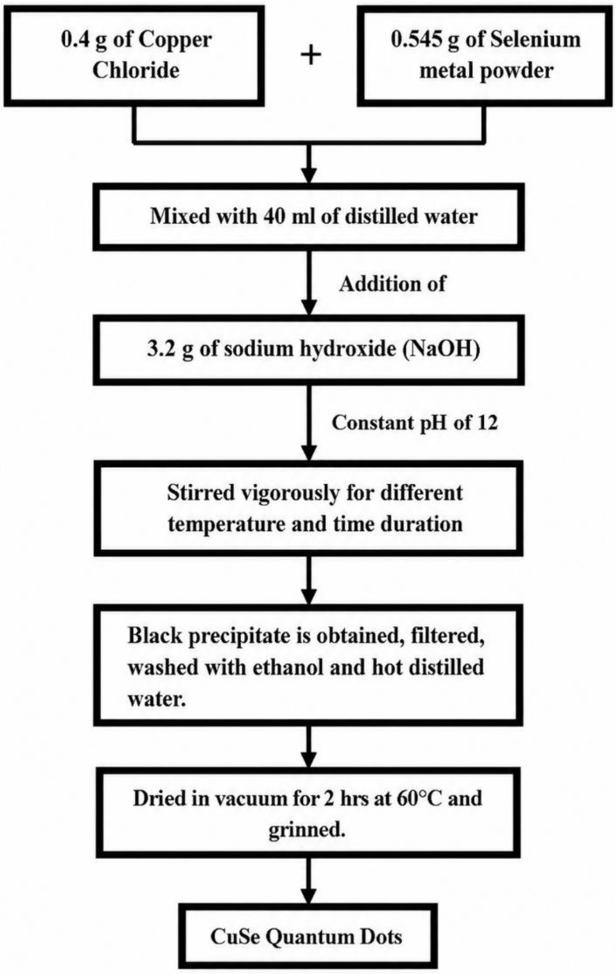

Figure 1. Flow Diagram of Synthesis of Cuse Quantum Dot

3. Results and Discussion

3.1. X-Ray Diffraction (Xrd)

Powder XRD patterns of the as-prepared copper selenide quantum dots for various reaction times and the temperature is shown in Figure 2a, b The XRD pattern of the copper selenide quantum dots with different time and the temperature has the characteristics feature corresponding to (101), (102), (006), (110), (108), and (116) planes which are in very good accordance with the JCPDS Card no 89-7391 of CuSe which possess a hexagonal structure with a=3.94 and c= 17.25. It was noted that the maximum intensity peak was obtained at the (102) plane in all cases and also the peak obtained was broadened compared to the bulk due to the size confinement.

The average grain size was estimated by using the Scherrer formula, $D=0.94\lambda/\beta\cos\theta$, where D is the average grain size, $\lambda=1.5406$ Å is the wavelength of the X-ray radiation, β is the full width half maximum (FWHM) of the diffraction peak and θ is the angle of diffraction. From the different θ values the average grain size at $50^{o}C$ for 12 hrs was around 10nm and at $70^{o}C$ for 12hrs was around 15nm which is in good accordance with the SEM results.

Also, the average grain size at $50^{o}C$ and $70^{o}C$ for 12hrs was confirmed by the W-H plot as shown in Figures 3a and b W-H plot is the between the $\beta\cos\theta$ Vs $4\sin\theta$, the inverse of they value pointing on the y-axis gives the grain size. It was noted interestingly that when the reaction time increases the average grain size was reduced, but conversely when the temperature increases the grain size increases since the reaction was completed at $50^{o}C$ and when the reaction extended to $70^{o}C$ the particles get aggregated and so the volume ratio is reduced.

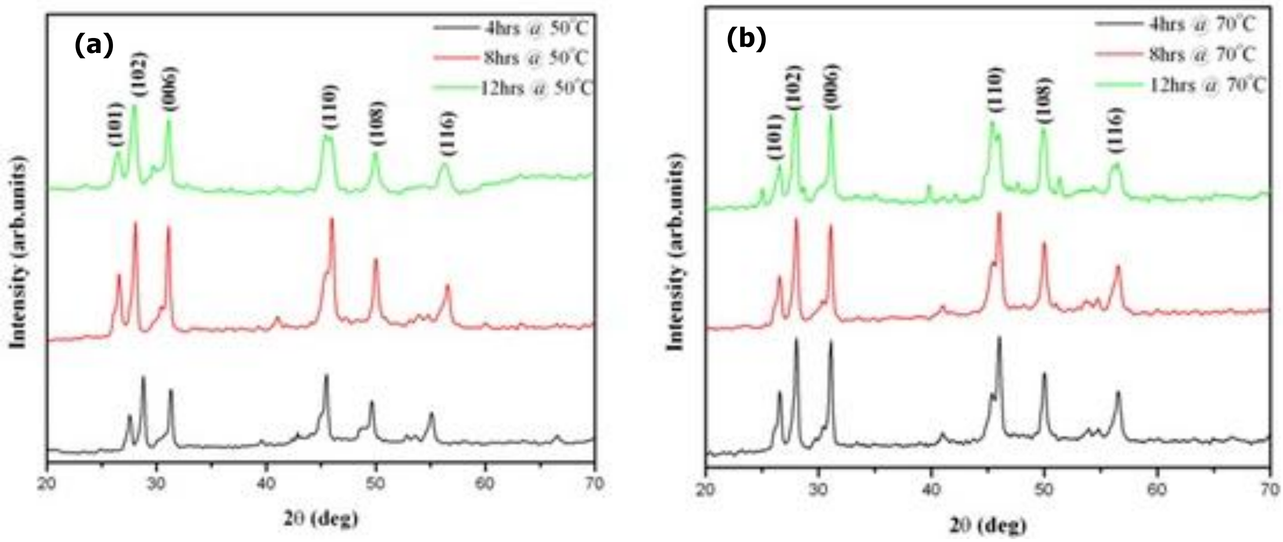

Figure 2. XRD patterns of the copper selenide quantum dots at $50^{o}C$ & $70^{o}C$ for various reaction times

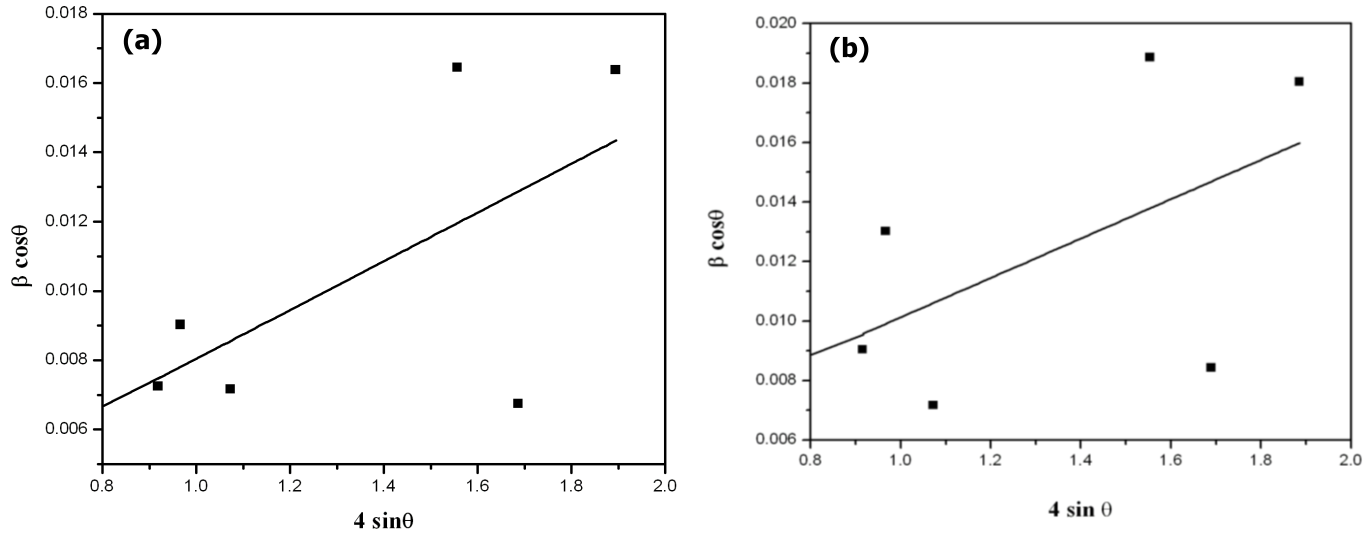

Figure 3. **(a)** W-H plot for 12hrs at $50^{o}C$, **(b)** W-H plot for 12hrs at $70^{o}C$

Table 2. Various parameters value obtained for 12hrs at 50ºC

Θ	d_{ob}	d_{ex}	hkl	d_{ob} - d_{ex} / d_{ob} = €	Lattice parameter		Volume		No. of the unit cell	Grain size (D)	Dislocation density
					A	C	V_{ob}	V_{ex}			
13.2785	3.3537	3.3472	101	0.0019			233.611	231.900	15.726	19.14	0.0027
13.9893	3.1864	3.1728	102	0.0042					8.125	15.36	0.0042
15.5708	2.8696	2.8750	006	-0.0018		17.2179			16.218	19.34	0.0026
22.9054	1.9791	1.9700	110	0.0046	3.95825				1.337	8.42	0.0141
24.9355	1.8270	1.824	108	0.0016					19.439	20.55	0.0023
28.2674	1.6265	1.6250	116	0.0055					1.3582	8.464	0.0139

Table 3. Various parameters value obtained for 12hrs at 70ºC

Θ	d_{ob}	d_{ex}	Hkl	d_{ob} - d_{ex} / d_{ob} = €	Lattice parameter		Volume		No. of unit cells	Grain size (D)	Dislocation density
					A	C	V_{ob}	V_{ex}			
13.2341	3.3647	3.3472	101	0.005			234.710	231.900	8.029	15.33	0.0042
13.9893	3.1864	3.1728	102	0.0042					2.6922	10.65	0.0088
15.5619	2.87126	2.8750	006	-0.0013		17.2275			16.122	19.34	0.0026
22.8554	1.9322	1.9700	110	-0.0195	3.9664				0.8864	7.354	0.0184
24.9888	1.8234	1.824	108	-0.0030					9.903	16.44	0.0036
28.1252	1.63406	1.6250	116	0.00554					1.0111	7.684	0.0169

Table 2 and 3 gives the various parameters value determining the strain, disl^{oc}ation density, volume, lattice parameter, and No. of the unit cell for 12 hrs at 50°C and 70°C. It was observed that the lattice parameter a and c are in good accordance with the standard value. The volume of the CuSe quantum dots grown at 70°C 12hrs was found to decreasing when compared to CuSe quantum dots grown at 50°C for 12 hrs but with that of the bulk of the standard value is was found to be increasing because of the large surface to volume ratio. It can be predicted that as the reaction time increases the surface to volume ratio increases. Thus from the XRD results can conclude that the reaction product formed is CuSe quantum dots which having increased surface.

3.2 Scanning Electron Microscopy (SEM)

The surface morphology of the as-synthesized copper selenide quantum dots was studied by Scanning electron microscopy (SEM). Figure 4 shows the SEM image of prepared CuSe for 12hrs at 50°C and 70°C respectively. It is clear from the SEM image that the particles are spherical in shape and the average size of the particles obtained to be around 10nm for 12hrs at 50°C and 15nm for 12hrs at 70°C which is in correct accordance with the XRD results. Also at 50°C for 12 hrs shows the uniform distribution of particles than 70°C. From these results, it is confirmed that the prepared CuSe nanoparticles are the quantum dots since the size of the particles is ≈10nm. Thus prepared CuSe quantum dots to possess a hexagonal structure and a spherical shape and as the temperature increases, the size increases, and structure gets destroyed.

3.3 Uv-Vis Measurement

The optical absorption measurements were carried out in the spectral range of 200-500 nm for prepared CuSe quantum dots for various reaction times and temperatures as shown in Figure 5. It is clear that there is no sharp absorption at the longer wavelength, but a sharp absorption peak was found at the shorter wavelength due to the exciton absorption which formed at the low temperature, and confinement was formed at the higher temperature. And also it reveals that the absorption intensity increases as the temperature increases, this is due to the increase in the grain size and decrease in the inter-distance between CuSe nanocrystals in the solvent causing the increased absorption with increasing temperature.

At low temperatures, the coulomb attraction between an electron and hole becomes prominent so that bound excitonic states were formed; as a result, sharp absorption peaks have appeared in the spectra. The optical bandgap of the CuSe quantum dots was determined with the transmission data's and the grain size obtained from the XRD calculation. From an established method, a plot is obtained between the $(\alpha h\upsilon)^{2}$ and hυ for direct, $(\alpha h\upsilon)^{1/2}$ and hυ for indirect as shown in Figure 6. where the absorption coefficient(α)and photon energy(hυ).

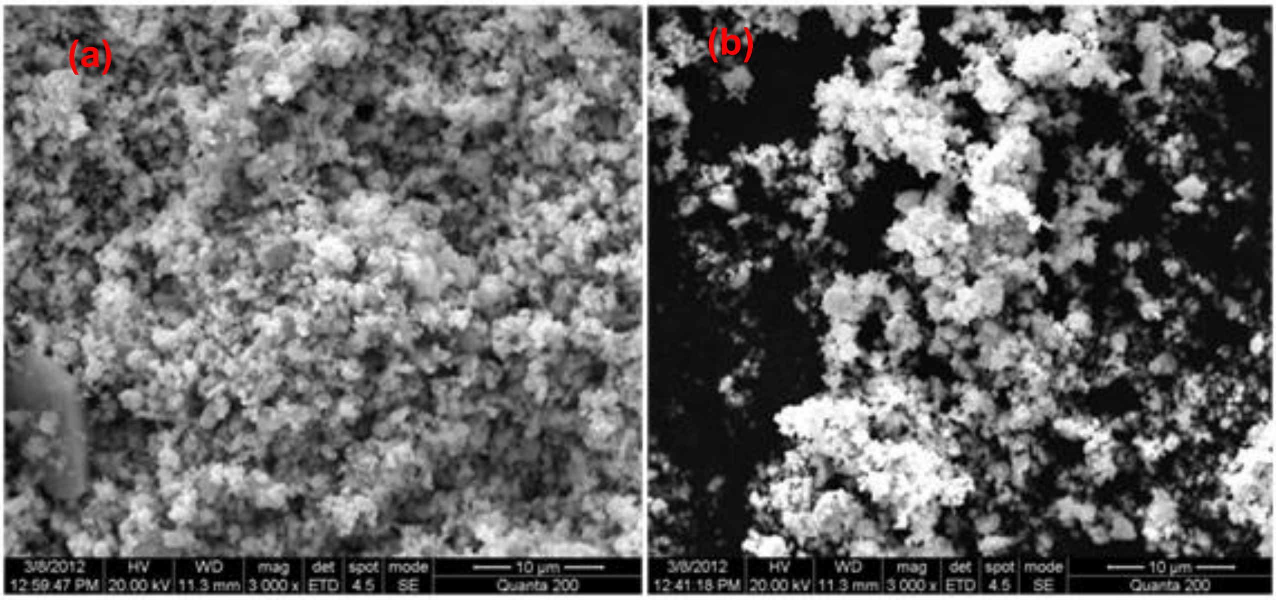

Figure 4. SEM Results for Prepared CuSe Quantum Dots for 12hrs at 50°C & 70°C

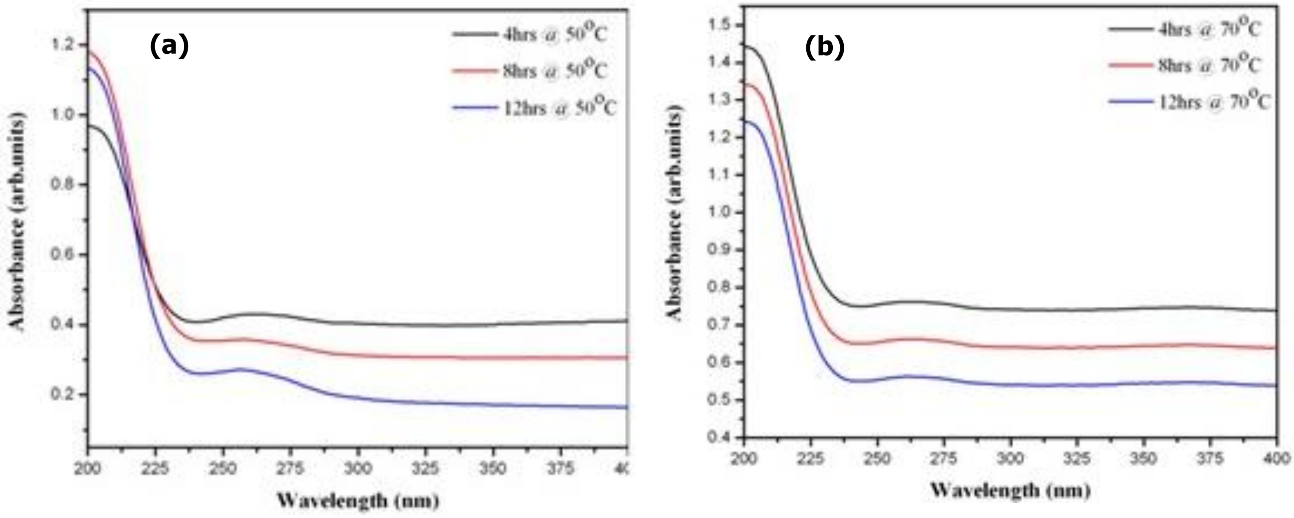

Figure 5. Absorption Spectra of CuSe Quantum Dots for 12hrs at 50°C & 70°C

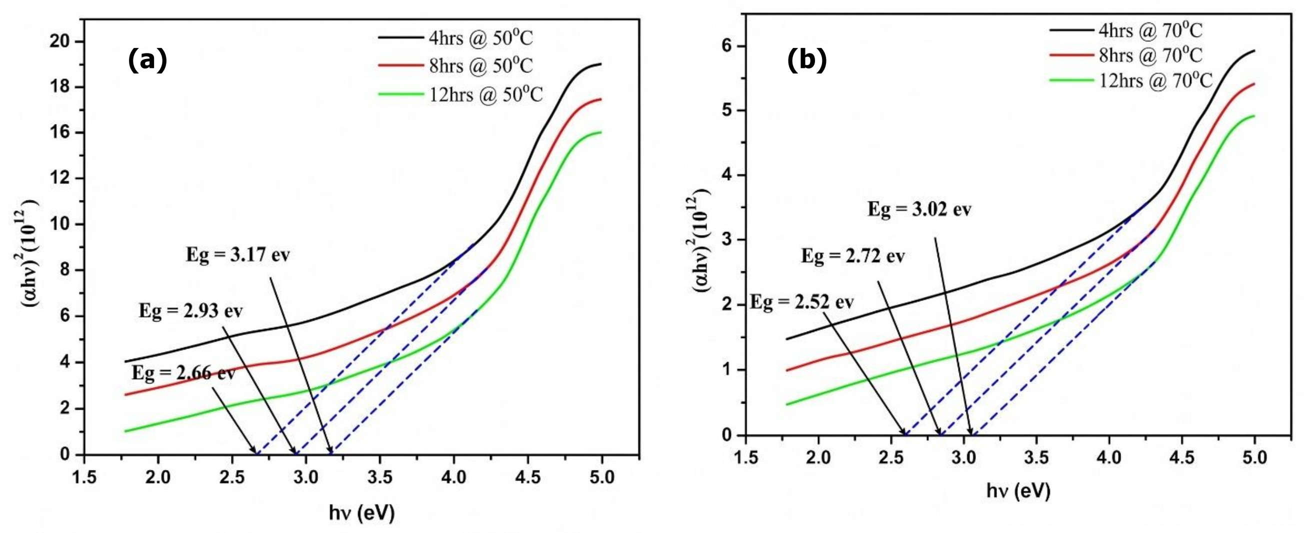

Figure 6. Bandgap plot for direct transitions for 12 hrs at 50°C & 70°C

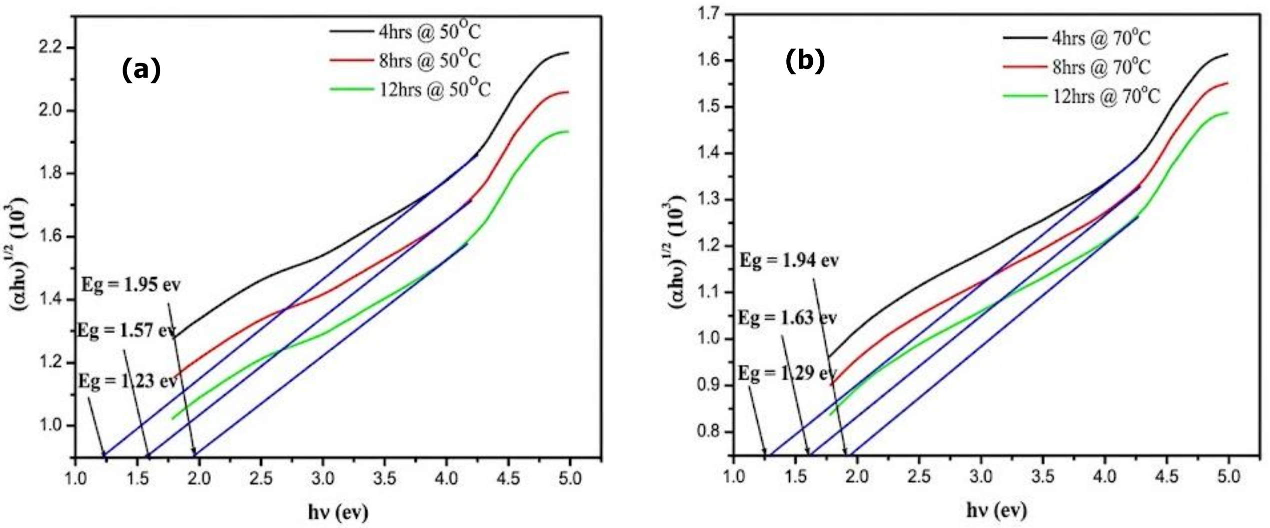

Figure 7. Bandgap plot for indirect transitions for 12 hrs 50°C & 70°C.

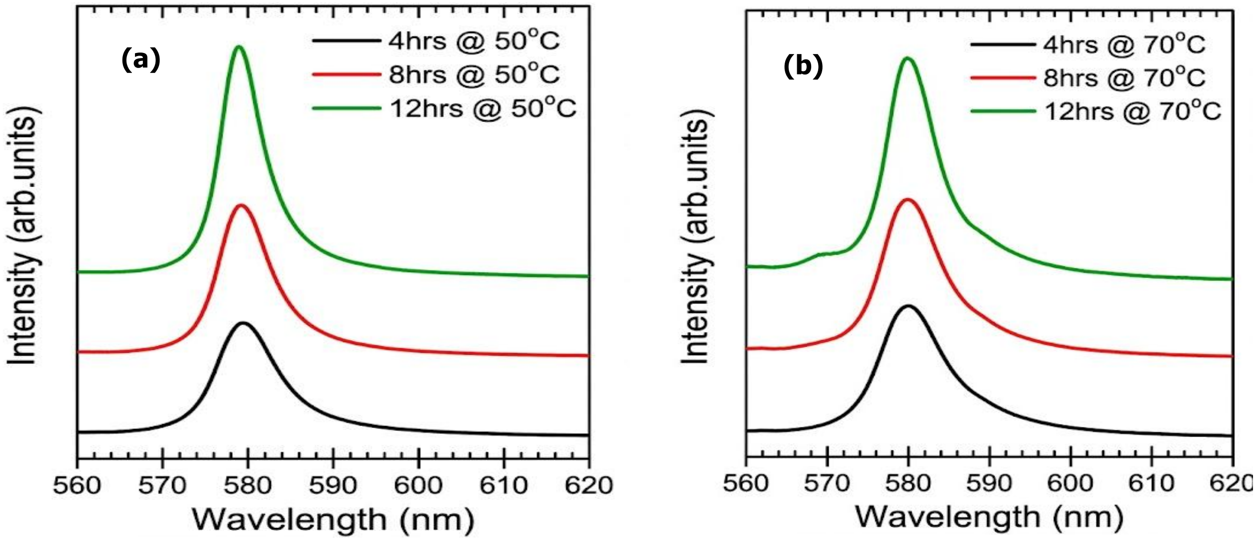

Figure 8. Photoluminescence spectra of CuSe quantum dots for 12hrs at 50°C &70°C

Table 4. Comparison chart between the reaction time and temperature concerning the intensity

Temperature °C	Reaction time		
	4hrs	8hrs	12hrs
50	580.17nm	579.6nm	577.998nm
70	577.83nm	577.032nm	576.712nm

Absorption coefficient (α) at different wavelength was calculated using the Transmittance (%) values by α = (2.303*log (1/T))/a. Figure 7 shows the direct bandgap and indirect bandgap plot. The direct bandgap value obtained for various reaction times at 50°C of 4hrs, 8hrs, and 12hrs is 2.66, 2.93, 3.17, and similarly at 70°C are 2.52, 2.72, and 3.02. The indirect bandgap value obtained for various reaction times at 50°C of 4hrs, 8hrs, and 12hrs is 1.23, 1.57, 1.97 ev, and similarly at 70°C are 1.29, 1.63, and 1.94 ev. Form the figures it is clear that the bandgap values increases as the reaction time increases, which means that as the grain size decreases band gap value increases. Thus can conclude the band gap values and grain size are inversely proportional.

3.4. Photoluminescence Spectroscopy

The photoluminescence spectra of copper selenide quantum dots at different temperatures and various reaction time durations are shown in Figure 8. Mainly the photoluminescence is used to investigate the photon generated electrons and holes. The recombination of the electrons and holes results in the photoluminescence emission. The PL spectra for different temperatures were found in the visible range from 550 to 600 nm. The broad emission PL spectra at 577 and 580nm were excited at 520nm which attributed to the blue emission. It was found that with the increase in temperature the PL intensity decreases and when the reaction time increases the PL intensity increases as shown in figure 8. Thus intensity concerning reaction time is the inverse to that of the temperature.

The wavelength of peak intensity concerning the time duration and the temperature shows a very slight blue shift as shown in Table 4. This reveals that as the reaction time increases there is a decrease in the wavelength which results in very small strokes of blue shifts. These broadbands are mostly attributed to the recombination of the carriers trapped in the surface states of CuSe quantum dots these surface defects are mainly due to the vacancies of Cu^{2+} and Se^{2-}, resulting in the degradation of luminescence properties. This conclusion is incorrect following the photocatalytic activity, where the degradation rate was 83%, hence we can conclude that the product formed is a CuSe quantum dot.

3.5 Raman Spectroscopy

Raman spectroscopy of CuSe quantum dots was analyzed in the range100-500cm^{-1}.

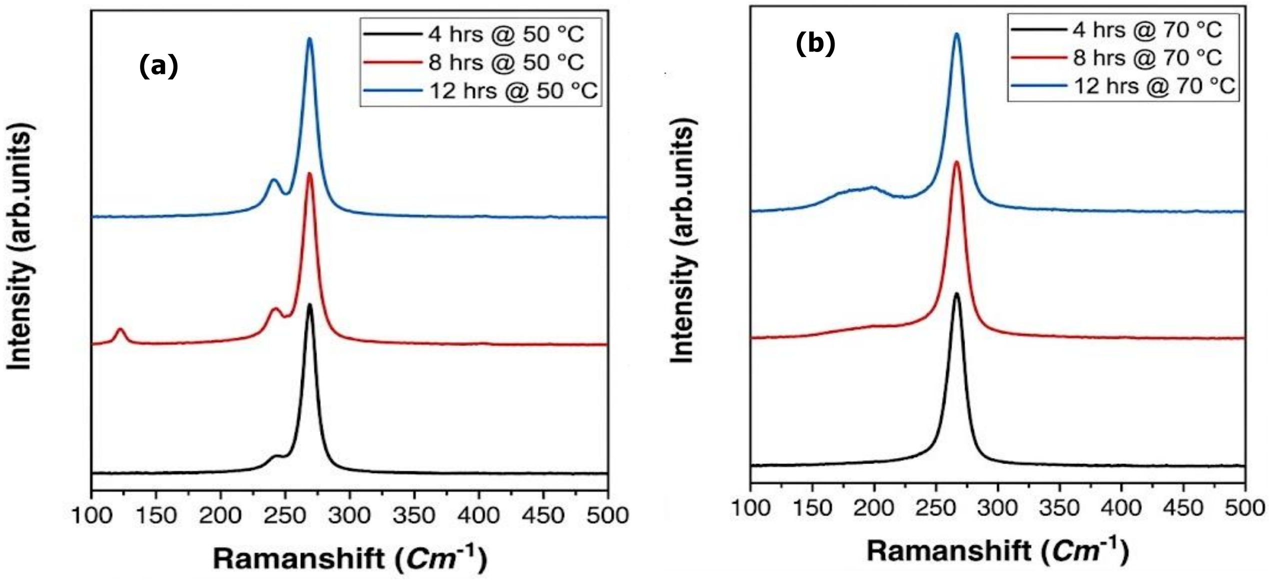

Figure 9. Raman spectra of as-prepared CuSe Quantum for 12 hrs at 50ºC & 70ºC

Comparing the Raman spectra of prepared CuSe at 50ºC (Figure 9) with those of prepared CuSe at 70ºC, the characteristic peak of Cu–Se bonds at 253 cm^{-1} was found, which is consistent with the standard Raman spectra of copper selenide (CuSe). The most intense peak observed corresponds to Cu–Se vibration and is in good agreement with the literature. The Raman shift intensity of 12 hrs was found to be strong enough then of 4hrs and 8hrs and similarly, the intensity at 50ºC was stronger than at 70ºC. The above facts highly suggested that the CuSe quantum dots were successfully synthesized in the NaOH solution.

3.6 Photocatalytic Activity

The photocatalytic activity of prepared samples was evaluated by the decoloration of the Rhodamine B(RhB) solution on the irradiation of UV light. The experimental procedure involves as 0.002g of as-prepared copper selenide(CuSe) and 1ml of aqueous Rhodamine B was taken in a quartz cell. Then the cell was put in the ultrasonic bath for 5mins for the homogeneous dispersion of CuSe and RhB.

The prepared CuSe and RhB solution were kept in the dark for about 1hour to reach an adsorption/desorption equilibrium between the Photocatalyst and the RhB molecules. After 1 hour of dark, the solution was irradiated with UV-vis light to obtain the absorption intensity and also irradiation with light was obtained every 1 hour to determine the absorption change of RhB in solution along with CuSe. This was repeated until the RhB color degrades. The decoloration percentage is evaluated by,

$$\text{Percentage of degradation (\%)} = \frac{A_0 - A}{A_0} * 100$$

Where A_0 and A are the absorption intensity before and after irradiation of UV light.

3.7 Measurement of Photocatalytic Activity

By measuring the decoloration of RhB under UV light the Photocatalytic activities of the samples are identified. Figure 10 shows the degradation of the RhB aqueous solution at different intervals after UV visible light irradiation in the presence of CuSe quantum dots.

The absorption spectra of RhB peaking at 554nm decreased gradually with the irradiation, which determines the Photocatalytic decoloration of RhB. In the qualitative analysis, the initial red-orange color of the RhB gradually degrades and then completely bleached, which indicates the chromophoric structure of the dye was destroyed. Also by quantitatively the percentage of degradation was around 83% as shown in Figure 11. Under the UV light irradiation of the RhB aqueous solution without the CuSe quantum dots, the absorption intensity did not decrease.

Rate constant value of the photocatalytic activity is determined by, $A=\ln (A_0/A_t)$Where A_0 is the absorption of the Rhodamine B and At is the absorption of Rhodamine B with CuSe for different time duration which is shown in Figure 10. The slope of any two points gives the rate of constant value.

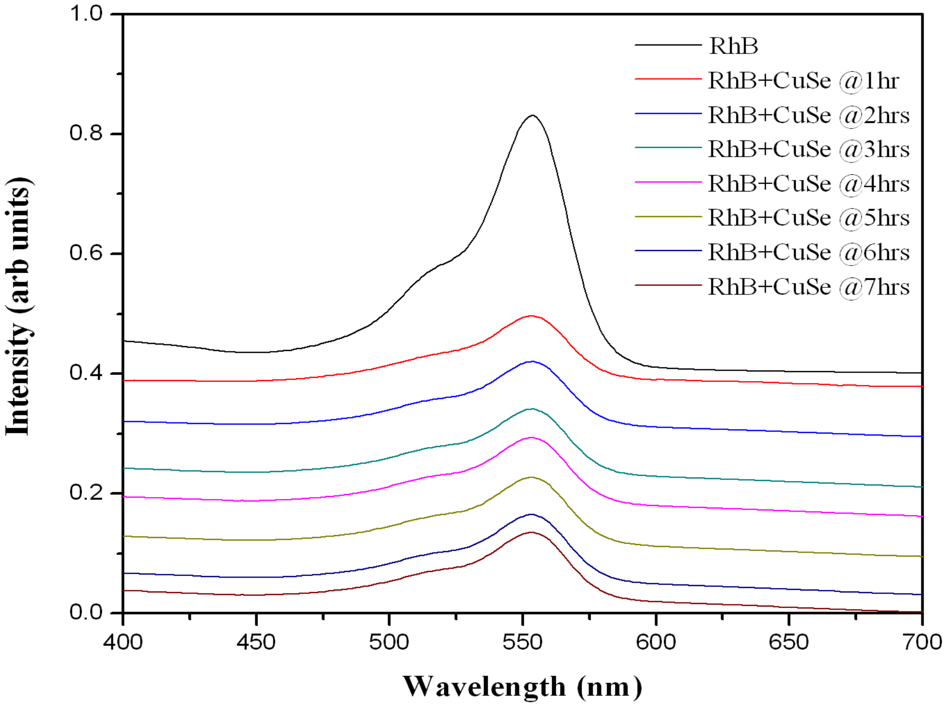

Figure 10. UV absorption spectra of RhB aqueous solution in the presence of CuSe quantum dots under UV-visible light irradiation

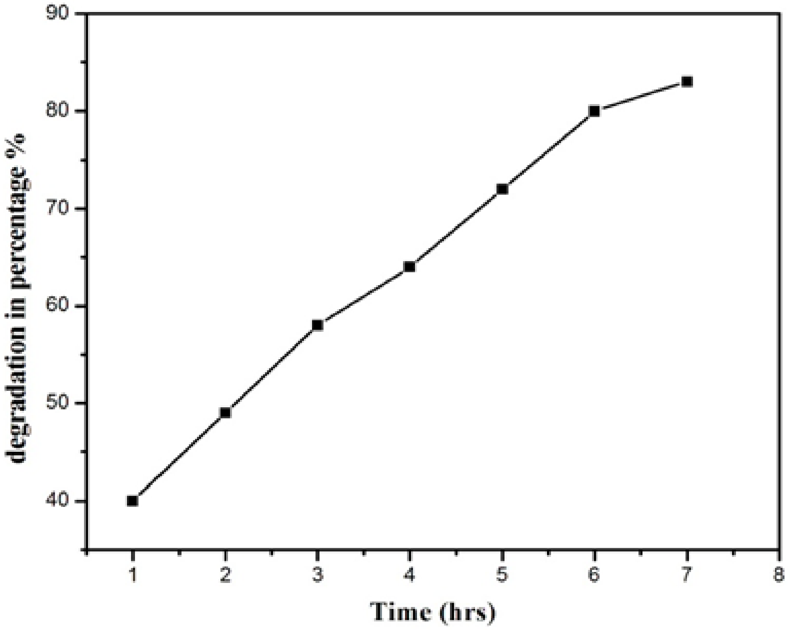

Figure 11. The plot of Degradation in percentage vs time

The rate constant found to be 0.2571. Thus these results both qualitative and quantitative analysis determine that the degradation of RhB aqueous solution caused by the photocatalytic reactions with the CuSe surface under UV light irradiation.

4. Conclusion

Copper Selenide Quantum dots have been prepared using a low cost (reflux condensation) method. A series of copper selenide Quantum dots can be obtained by varying the reaction time and temperature. Using structural studies like XRD, SEM confirmed the size and shape of the copper selenide quantum dots. Strong blue shifts were observed from the optical studies like UV and PL and also from these studies band gap energy was calculated. Thus quantum confinement effect results from the strong blue shifts. Photocatalytic activity determined with CuSe along with RhB which results in the complete degradation of the color. Finally from these results obtained can conclude that copper selenide obtained were in the size of 10-15 nm which confirms as a quantum dot and also it exhibits excellent photocatalytic properties to degrade the dye and hence it can be used for removing the impurities from the water.

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Does this article screened for similarity?

Yes

Author Contribution Statement

K. Chamundeswari: Conceptualization, Methodology, Writing - Original Draft, Investigation, Validation. R. Saranya: Methodology, Writing - Original Draft. A. Leggins: Conceptualization, Writing - Review & Editing, Supervision. All the authors read and approved the final version of the manuscript.

Conflict of interest

The Author's declares that there is no conflict of interest anywhere.

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