RESEARCH ARTICLE

Comparison of Photocatalytic Efficiency of TiO_2 and In_2S_3 Thin Films under UV and Visible Light Irradiance

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We use simple, cost effective deposition techniques of doctor blading and chemical bath deposition to synthesize uniformly dispersed nanoparticle thin films of Titanium dioxide (TiO_2) and mesh fractal thin films Indium sulphide (In_2S_3) respectively. The resulting microstructures and phase composition of these films are characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and the optical studies are carried out using UV-Visible-NIR spectroscopy. Further, we report these thin films are photocatalytically active in different spectral regions and a comparative study is detailed in this work. The photocatalytic studies of both the films are carried out by using methylene blue (MB) dye as pollutant and their efficiencies are compared under UV and visible light irradiation. The results obtained shows that the photocatalytic efficiency of TiO_2 film was conspicuous in the UV region where as In_2S_3 films showed significant activity in the visible region. Present study also confirms that photocatalysts in thin film forms are ideal for practical applications as they are efficient and also can be retrieved easily from the solution. These features favour their extensive use in eco-friendly environmental applications such as degradation of environmental pollutants, waste water treatment in textile industries and water purification.

Introduction

Nowadays, water sources are often polluted by the industrial effluents and dyes from textile industry. The discharge of the toxic dyes in water bodies affects the flora and fauna and therefore, is a matter of serious concern to the ecosystem [**1,2**]. Photocatalysis is an effective method of degradation of dyes that does not require further secondary purification techniques. Recently, the role of semiconductor photocatalysts in enhancing the degradation of various dyes, organic pollutants and pharmaceuticals has attracted much attention [**3-5**]. Great significance is given to works that explore new catalysts or that compares with the existing catalysts, for better and efficient performance in various applications [**6**].

Titanium dioxide (TiO_2) is a conventional photocatalyst having exceptional optical and electronic properties, strong oxidizing power, non-toxicity, chemical stability, high photosensitivity and biocompatibility [**7, 8**]. TiO_2 exists in three crystalline structures namely anatase, rutile (both having tetragonal structure) and brookite (orthorhombic structure). Among these crystalline structures, anatase phase offers better photocatalytic activity and rutile phase is the thermodynamically most stable state. In all these phases, each titanium atom is surrounded octahedrally by six oxygen atoms. Each of the crystal structures differ with respect to spacing distortion of each octahedron or by the assembly pattern of the octahedra chains [**9**]. The photocatalytic performance of TiO_2 has been widely studied in degradation of various dyes [**10**]. In the present work, we aim to compare the photocatalytic efficiency of TiO_2 with a newer photocatalyst, Indium Sulphide (In_2S_3). In_2S_3 is considered as a promising material with a variety of applications due to its chemical stability, non-toxicity and transmittance in visible region. In_2S_3 is a III-VI compound material which exists in three different forms α, β and γ depending upon the synthesis temperature. Above 693 K, the cubic α-form exists and γ-In_2S_3 with trigonal symmetry exists at temperature above 1047 K. β-In_2S_3 with tetragonal structure is the stable room temperature phase [**11,12**].

Here, the materials for photocatalytic studies are deposited and used in thin film form. The advantage of coating photocatalysts in thin film form is that they can be easily introduced in continuous flow systems for cleaning applications, requires no difficult filtration steps and prevents the clustering of particles [**13**]. Much of the works that reports on high efficiency photocatalytic degradation is mostly in the powder form [**14-17**]. Usually, the deposition techniques used for deposition of these thin films are sputtering, successive ionic layer adsorption and reaction etc. [**18,19**]. The uniqueness of the present work is

that the comparison of photocatalytic efficiency study is carried out between TiO_2 and In_2S_3 deposited in thin film forms which alleviate the need for complex filtration processes facilitating the suitability in practical applications. To the best of our knowledge, no studies have been reported yet on the comparison of photocatalytic efficiencies of Titania (TiO_2) thin film with mesh fractal structured Indium sulphide (In_2S_3) thin film. Also, the comparison and contrasting has been carried out in two distinct spectral regions (UV and visible). Moreover, the methods used for deposition of the photocatalysts as thin film in the present work are simple, cost effective and scalable, thereby making it ideal for practical applications.

Experimental techniques

Materials and methods

The present work focuses on simple and cost-effective deposition techniques of films that do not require vacuum technology and complex equipments. The glass substrates used for coating the thin films were of the dimension (25 mm x 26 mm x 1.1 mm, Labtech) respectively. These glass substrates were initially cleaned with soap solution, washed with distilled water, ultrasonically cleaned and dried. TiO_2 thin films were coated on the substrate using doctor blading technique and the In_2S_3 thin films by chemical bath deposition (CBD). For the preparation of TiO_2 thin films, we used 1M Titanium (IV) oxide, (TiO_2, 99.8%, Sigma Aldrich) and triton X-100 (Sigma Aldrich) as the binding agent. The mixture was then ultrasonicated for about 1 hour. This viscous paste was then coated on the glass substrate by doctor blading using a glass rod. The film was then heated at 100°C for 1 hour followed by sintering at 450°C for 30 min [**20**]. The thickness of the TiO_2 film is about 17 μm. The deposition of pristine In_2S_3 films used in the present work were carried out by CBD process whose details are already been reported elsewhere [**21**]. The thickness of In_2S_3 film was found to be 2 μm.

Characterization methods

The structural studies were investigated by XRD using Rigaku Mini Flex 600 X-ray diffractometer with Cu-K_α irradiation (λ = 1.5406 A^o) and morphological characterizations was done by field emission scanning electron microscopy (FE-SEM, Carl Zeiss Sigma). The absorption spectra were recorded using JASCO V-670 UV-Vis-NIR spectrophotometer.

Photocatalytic test

In order to determine the photocatalytic efficiency of TiO_2 and In_2S_3 films, the degradation of methylene blue (MB) dye was measured under both UV and visible light sources. For carrying out the photocatalytic studies, we used a 9 watt UV light source (KACOOL UV flashlight) and a 9 watt LED for visible light source (Aelius LB LED Lamp 9W).

An amount of 0.1 mg MB dye (Merck) is diluted to 300 ml of distilled water and then stirred. For studying the photocatalytic efficiency, 15 ml of dye solution is taken in a beaker and exposed to the light source. In order to maintain a constant temperature, the system is kept water cooled. The beaker containing the solution was placed in a water bath, into which running water flows without disturbing the system. There were outlets in the bath that allows water to flow out, thereby keeping the water level constant. The light source was then placed in a holder in such a way that the beaker containing the dye solution is uniformly illuminated. The distance between the light source and MB solution was maintained at about 10cm.

The degradation of MB dye solution in the presence of TiO_2 and In_2S_3 films were carried out under UV and visible light sources respectively [**22,23**]. For this, the degradation of MB dye was noted at an interval of 1 hour continuously for 4 hours, by taking 3 ml of the sample using a dropper for measuring the absorbance. The absorbance spectrum of MB dye has the characteristic peak at 663nm [**24**]. Therefore, by measuring the variation in absorption intensity of this peak, the degradation of dye in the presence of the two catalysts (TiO_2 and In_2S_3) was studied under both UV and visible illumination. The studies were systematically carried out for both these catalysts and the photocatalytic performance was compared and evaluated. The percentage of degradation of MB can be obtained from the equation given below; since absorbance and concentration are being proportional as per the Beer Lamberts Law [**14,25**].

$$\eta = \frac{(A0 - A)}{A0} \times 100\% = \frac{(C0 - C)}{C0} \times 100\%$$

Here C0 and A0 are the initial concentration and absorbance of MB, C and A are the respective values after t hours.

The apparent rate constant for the degradation of MB dye by both the catalysts is estimated by plotting the ln (A_0/A) versus the irradiation time.

Results and discussion

Structural analysis

Fig. 1(a) and **1(b)** shows the XRD of TiO_2 films and In_2S_3 films respectively. XRD pattern determines the crystal structure of the as-synthesized thin films. In the XRD of TiO_2, the most dominant peak is at 2θ=25.17^o which corresponds to (1 0 1) plane and the other less intense peaks are along (1 0 3), (0 0 4), (1 1 2), (2 0 0), (1 0 5), (2 1 1), (1 1 0), (2 2 0) and (2 1 5) orientations. These peaks correspond to the anatase phase of TiO_2 (ICDD card no: 00-002-0387). The peaks at 2θ = 27.2^o, 62.6^o, 76^o corresponding to (1 1 0), (0 0 2) and (2 0 2) planes confirm the presence of rutile phase (ICDD card no: 00-001-1292). Therefore, the prepared TiO_2 film has a combination of anatase and rutile phases and it shows tetragonal structure [**26,27**]. The multiple peaks, which are sharp, reveal the poly crystalline nature of the TiO_2 films.

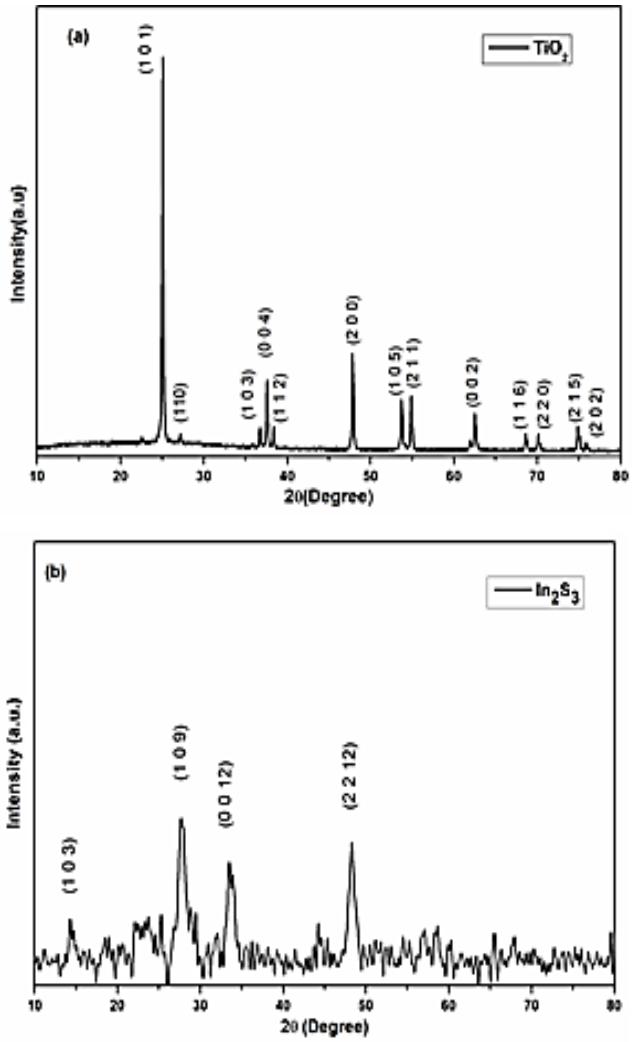

Fig. 1. XRD patterns of (a) TiO_2 and (b) In_2S_3 thin films

The XRD pattern of In_2S_3 shows its intense peaks at 2θ = 14.24°, 27.46°, 33.25° and 48.1° corresponding to (1 0 3), (1 0 9), (0 0 12) and (2 2 12) planes. This matches with ICDD card no: 00-025-0390 and thus confirms the tetragonal structure of In_2S_3 film [**28**]. The crystallite size was calculated from the most intense peak using Debye Scherrer formula, which is found to be 48.5 nm for TiO_2 film and 17.3 nm for In_2S_3 film respectively.

FESEM analysis

Fig. 2(a) shows the surface morphology of the as prepared TiO_2 films. From the figure, it is evident that TiO_2 particles are uniformly distributed throughout the film. Grain size is often considered as an important parameter in determining the photocatalytic efficiency [**29**]. The average grain size was obtained to be 156 nm. It is seen that the particles are about spherical in shape with irregular size distribution [**30**]. The FESEM image of In_2S_3 film is shown in **Fig. 2(b)**. Here, the particles have mesh fractal structure and they are evenly dispersed throughout the film [**31, 32**].

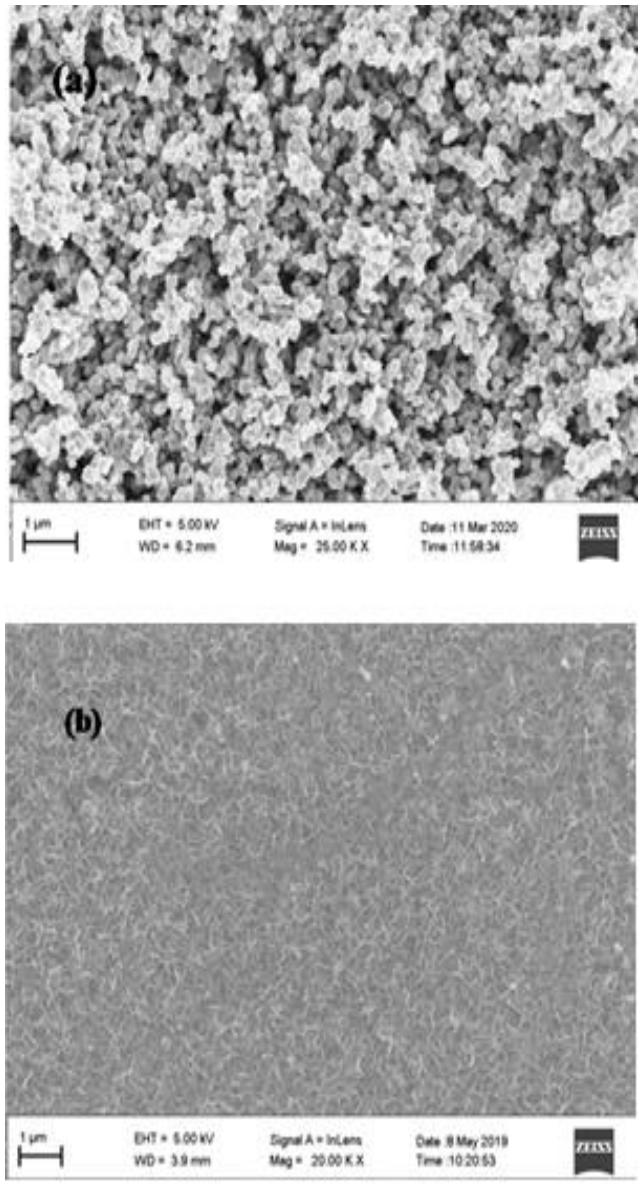

Fig. 2. FESEM images of the thin films of (a) TiO_2 and (b) In_2S_3

Optical studies

UV-Visible spectroscopy is a powerful tool in determining the optical properties and also the band gap of the sample. **Fig. 3** shows the tauc's plot of TiO_2 films and In_2S_3 films. The optical absorption spectrum of TiO_2 films was recorded using Diffuse Reflectance Spectroscopy (DRS) measurements. **Fig. 3(a)** shows the plot of $(F(R)\ h\nu)^{1/2}$ versus hν, where F(R) represents the Kubelka-Munk function. The band gap energy of the film is calculated by extrapolating the linear portion of $(F(R)\ h\nu)^{1/2}$ on the *x*-axis [**11**]. Thus, the obtained optical band gap energy is 3.75 eV.

Fig. 3(b) gives $(\alpha h\nu)^2$ versus hν plot of In_2S_3 film and the band gap energy is obtained by extrapolating the straight-line portion of the plot on x-axis. Here, the obtained band gap is 2.57 eV [**19**].

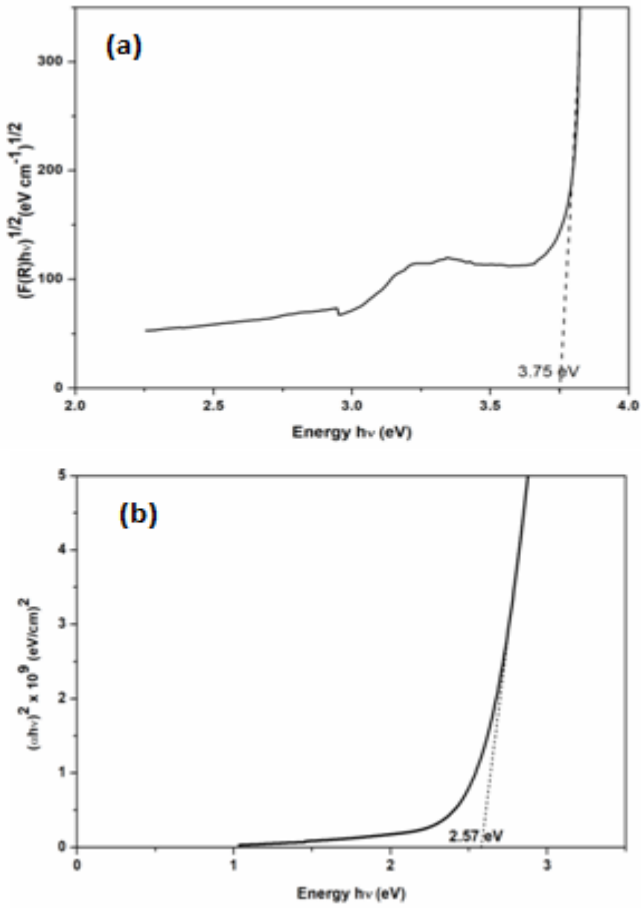

Fig. 3. The optical band gap energy of a) TiO_2 thin film and b) In_2S_3 thin film.

Photocatalytic studies

The photocatalytic property of TiO_2 and In_2S_3 films were studied by carrying out the photocatalytic degradation of MB in the presence of these films under both UV and visible light sources [**33-36**]. The decrease in the absorption intensity of the characteristic peak of MB dye measures the amount of dye degradation and UV-Vis-NIR spectroscopy was used to monitor this.

Fig. 4(a) and **4(b)** show the absorbance spectra of MB dye in the presence of TiO_2 film under UV and visible light respectively. It is evident from the figure that MB dye degrades more in the UV light source than in the visible light with TiO_2 thin films as the photocatalyst. On noting the degradation of MB dye after 4 hours in the presence of TiO_2 photocatalyst, it is found that there is 31% more degradation, when irradiated by UV light source. This is because TiO_2 has strong absorption in the UV range because of its wide band gap [**37**].

In the process of photocatalytic degradation, initially the MB dye molecules get adsorbed on the surface of the catalysts which exist in the form of thin films. When these catalysts adsorbed with the dye molecules are illuminated under appropriate light with energy equal or greater than bandgap, separation of charge carriers take place i.e electrons from valance band (VB) moves to conduction band (CB) leaving holes in the VB. The dye molecules too get excited under the irradiation which results in movement of electrons to the CB of the catalyst [**38**]. In general, the excited electrons have strong reduction capacity while holes possess strong oxidation capacity and therefore, they would act as reductant and oxidant respectively [**39**]. The excited electron in the conduction band reacts with air to generate super oxide radical anions (O_2^-). The photo generated holes in VB react with adsorbed water molecules forming hydroxyl radicals (OH). These super oxide and hydroxyl radicals would further undergo secondary reactions and finally decompose the pollutants. This photo induced generation of electron-hole pair, the formation of reactive radicals and oxidation of pollutants by these radicals represents the overall mechanism of photocatalytic reaction [**40,41**].

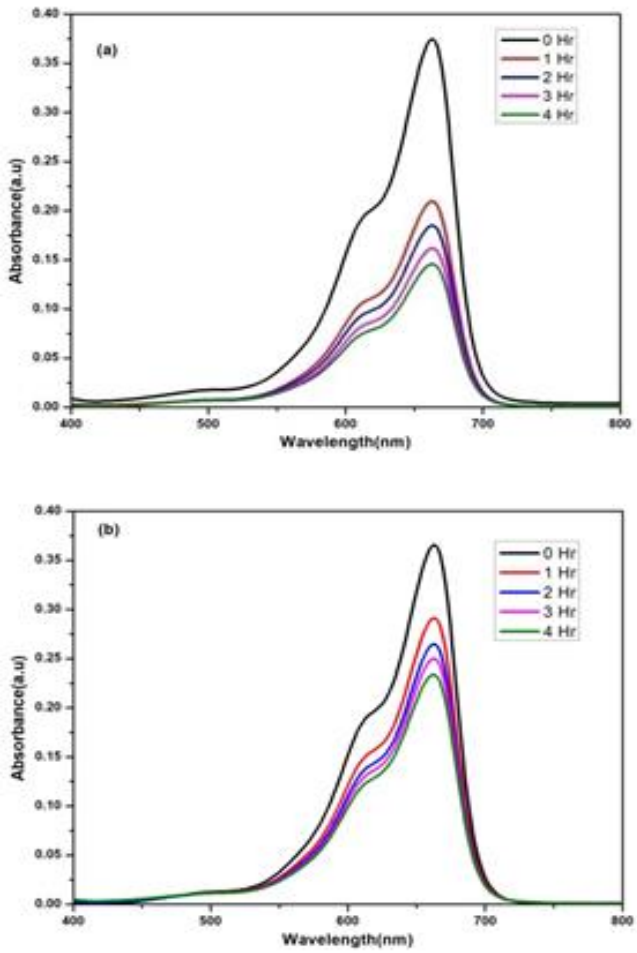

Fig. 4. The absorbance spectra of MB in the presence of TiO_2 as photocatalyst under a) UV light b) visible light.

Fig. 4(b) shows that there is degradation in MB dye under visible light. The photocatalytic degradation is clearly lower compared to that of UV light irradiation. Thus, it is confirmed that the TiO_2 photocatalyst is more activated under UV exposure. In order to improve the absorption of TiO_2 in visible region, proper dopants could be incorporated and thus the photocatalytic property can be

enhanced [**42**]. The **Table 1** shows the percentage of dye degradation sampled at regular interval for a period of 4 hours.

Table 1. Percentage of degradation of MB dye.

Catalyst	Irradiation time (In Hours)	Photocatalytic degradation (%)	
		UV	Visible
TiO_2	1	44	20
	2	51	27
	3	57	31
	4	67	36

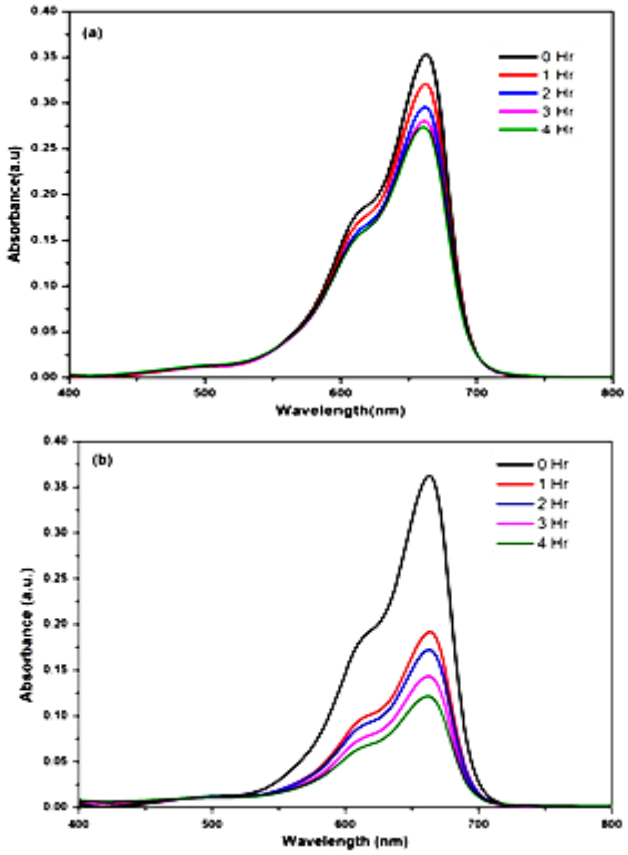

Fig. 5. The absorbance spectra of MB in the presence of In_2S_3 as photocatalyst under (a) UV light and (b) visible light.

Fig. 5(a) and **Fig. 5(b)** show the absorbance spectra of MB in the presence of In_2S_3 thin films under UV and Visible light exposure. It is noted that, with In_2S_3 films as photocatalysts, the MB dye undergoes substantial degradation in visible light when compared to UV light. After 4 hours of exposure, it is found that MB has degraded only 23% under the UV light irradiation whereas degradation is 69% under visible light. The reaction rate decreases with irradiation time as a competition for degradation may occur between the reactant and the intermediate products. Initially there is availability of larger area of unreacted catalytic surface. As time progresses, there is a reduction in unreacted catalyst surface which may be the cause of reduction in photocatalysis [**43**]. The work has been repeated many times to ensure the accuracy in measurements.

Present study demonstrates that In_2S_3 is a good photocatalyst which has more activation in the visible range than in UV. In case of In_2S_3, visible light has a major role in degradation of the MB dye, since it gives sufficient energy to excite the electron and generates more electron-hole pairs, thereby improving the photocatalytic efficiency. Hence, it can be a suitable replacement to various conventional photocatalysts which works in UV region and thus can be used for real time applications [**16**].

Table 2 shows the percentage of dye degradation under UV and visible light irradiation in the presence of In_2S_3 thin film as the photocatalyst, sampled at an interval of 1 hour.

Table 2. Percentage of degradation of MB dye.

Catalyst	Irradiation time (In Hours)	Photocatalytic degradation (%)	
		UV	Visible
In_2S_3	1	9	50
	2	16	55
	3	21	63
	4	23	69

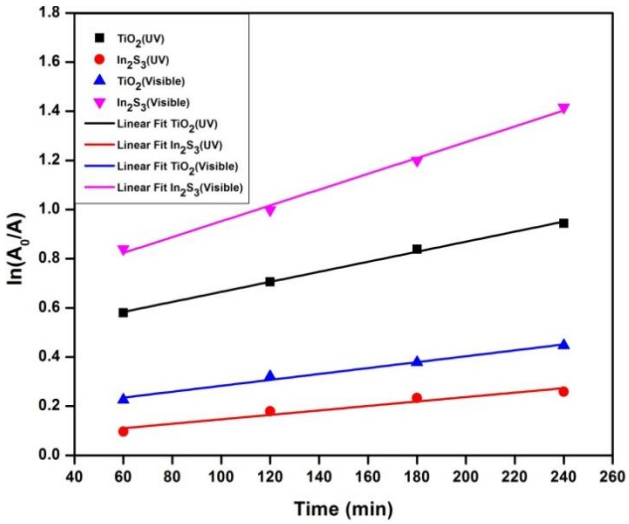

Fig. 6. Plot of $\ln(A_0/A)$ versus irradiation time of TiO_2 and In_2S_3 films under UV and visible light.

The photocatalytic degradation kinetics of MB dye can be obtained using a pseudo first-order kinetic model by plotting $\ln(A_0/A)$ versus irradiation time, where A_0 is the absorbance at initial time and A is the absorbance after time t. **Fig. 6** shows the $\ln(A_0/A)$ versus time curve of the MB dye degradation using TiO_2 and In_2S_3 thin films under both UV and visible light radiation. The apparent rate constant of dye degradation using these photocatalysts was estimated from the slope of the linear fitting curves [**44-46**]. The percentage of degradation of MB dye using TiO_2 thin film after 4 hours of irradiation under UV and visible light was 67% and 36% respectively. Similarly, the percentage of degradation of MB dye using In_2S_3 thin film under UV light was found to be 23%, which has increased to about 69% under visible light. It is found that the apparent rate constant (K_{app}) for the TiO_2 photocatalyst

under UV irradiation is 0.002 min^{-1} and decreases to 0.001 min^{-1} under visible light irradiation. The corresponding K_{app} values for In_2S_3 photocatalyst on UV irradiation is 0.0009 min^{-1} and increases to 0.003 min^{-1} under visible irradiation.

Conclusion

This work reports the comparison of photocatalytic efficiencies of TiO_2 and In_2S_3 thin films prepared by simple and cost-effective deposition techniques. The photocatalytic efficiency of conventional photocatalyst TiO_2, is limited to the UV region. Present study demonstrated that MB dye degradation using In_2S_3 films as the photocatalyst is equally promising because of its substantial activity in the visible region. The deposition of photocatalyst in thin film form facilitated its easy recovery from solution, thereby eliminating cumbersome filtration processes. The advantage of present study is that it deals with the comparison and contrasting of photocatalysts focusing on its applicability in cost effective water treatment process. This work also opens up prospects for tailoring absorption of In_2S_3 thin films using suitable dopants so as to improve absorption over wider visible spectral range thereby enhancing its suitability as an effective photocatalyst.

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Conflicts of interest
There are no conflicts to declare.

Keywords

Titanium dioxide thin film, Indium sulphide thin film, doctor blading, chemical bath deposition, photocatalytic degradation.

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Authors biography

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Presently, Dr. Saji Augustine serves as HOD, Dept. of Physics, Deva Matha College, Kuravilangad, Kerala, India. He was CSIR postdoctoral fellow at IISc Bangalore, India and KAIST, South Korea. He has been actively involved in Materials synthesis and characterization especially in single crystal growth, synthesis of new perovskite structures, and bulk carbon nanotubes for hydrogen storage. He has got fifteen peer reviewed international publications, two US patents and two Korean patents to his credit.

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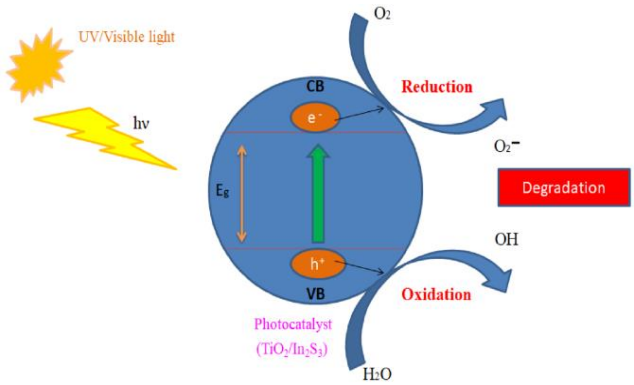
Graphical abstract

TiO_2 and In_2S_3 thin films are compared and contrasted for photocatalytic degradation of methylene blue in water, which acts as pollutant. When light with energy equal or greater than the band gap of semiconductor photocatalysts are used for irradiation, electrons from valence band get excited to conduction band and these photogenerated carriers on reaction with water and oxygen molecule generates superoxide anion and hydroxyl radicals. These would further undergo secondary reactions and finally decompose pollutant.



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