



PHOTOCATALYTIC PROPERTIES OF CuBi_2O_4 PREPARED BY THE CO-PRECIPIATION METHOD: DEGRADATION OF ROSE BENGAL AND POSSIBLE REACTION MECHANISM UNDER VISIBLE LIGHT IRRADIATION

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ARTICLE INFO

Article History:

Received 17th September, 2017

Received in revised form 21th October, 2017

Accepted 28th November, 2017

Published online 28th December, 2017

Key words:

Photocatalysis, Rose Bengal (RB), CuBi_2O_4 , Picric acid.

ABSTRACT

Monoclinic CuBi_2O_4 was synthesized by Co-precipitation method at 300 °C. Crystallographic and morphological characterizations of prepared sample has been carried out using X-ray diffraction and scanning electron microscope. The results indicate that the sample prepared at 300 °C has the structure of monoclinic CuBi_2O_4 with average crystallite size less than hundred nanometer. The UV-Vis diffuse reflectance spectra show that the sample has good absorption ability in the wavelength range of 300–800 nm. The results of photocatalytic experiments show that the photocatalyst exhibit excellent photocatalytic activity for Rose Bengal (RB) dye solution under visible light irradiation. Rose Bengal(RB) dye was successfully photodegraded using visible light active monoclinic CuBi_2O_4 as photocatalyst. 10 ppm of Rose Bengal(RB) dye was photodegraded using 0.1 g of CuBi_2O_4 under irradiation for 2 hours. The prepared CuBi_2O_4 photocatalyst was characterized by XRD,UV-DRS,SEM-EDX and FT-IR. Photoluminescence studies confirmed the formation of ·OH free radicals due to irradiation.

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INTRODUCTION

Heterogeneous photocatalysis has been widely investigated for the abatement of several toxic organic pollutants because of its ability to completely mineralize hazardous organic contaminants into harmless products at ambient temperature (Ahmed *et al.* 2011). TiO_2 is used extensively as a photocatalyst because it is inexpensive, easy to synthesize, non-toxic, chemically inert, and highly photostable. But, the wide band gap of TiO_2 restricts its absorption to U.V region with $\lambda < 380\text{nm}$ which require substantial electrical input. Terrestrial solar radiation is composed of 4% U.V radiation, 52% visible light and 44% near infrared light. In order to exploit the large component of solar radiation, it is essential that the photocatalyst needs to be visible light responsive. Different strategies have been demonstrated (Suresh 2016) to extend photo response of TiO_2 into visible light through (i) doping of metal atoms/anions and/or cations, (ii) surface sensitization with dyes, phthalocyanins, porphyrins and (iii) fabrication of nano composites with higher surface to volume ratio. Simultaneous research on ternary metal oxides as potential photocatalysts led to the development of ZnWO_4 (Montini *et al.* 2010), CaBi_2O_4 (Tang *et al.* 2004),

BaBiO_3 (Tang *et al.* 2007), BiVO_4 (Martinez-de la *et al.* 2010), Bi_2WO_6 (Liao *et al.* 2011), Bi_2MoO_6 (Martinez-de la Cruz and Obregon Alfaro 2010), $\text{Bi}_2\text{Mo}_2\text{O}_9$ (Li, Li, and Wang 2009), $\text{Bi}_2\text{Mo}_3\text{O}_{12}$ (Martinez-de la Cruz and Perez 2010), $\text{Fe}_2\text{Mo}_3\text{O}_{12}$ (Photo *et al.* 2014), NaBiO_3 (Chang *et al.* 2010), FeV_3O_8 (Zhang, Zhou, and Zhang 2014) etc. as potential photocatalysts for degradation of dyes and other aromatic pollutants. Majority of these visible light active photocatalysts contain Bi as one of the components. Binary metal oxide systems $\text{Bi}_2\text{O}_3\text{-V}_2\text{O}_5$, $\text{Bi}_2\text{O}_3\text{-MoO}_3$ and $\text{Bi}_2\text{O}_3\text{-WO}_3$ form a large group of visible light responsive photocatalysts, probably due to the valance band formed from Bi (6s) and O (2p) orbitals resulting in a smaller band gap (M. Oshikiri 2006). Remediation of several dyes using $\text{Bi}_2\text{Mo}_3\text{O}_{12}$, $x\text{MoO}_3$ (Suresh and Prasada Rao 2015; Suresh, Umabala, and Rao 2016) and aromatic pollutants such as nitrobenzene (A.M. Umabala 2015), 2, and 4- nitrophenols (A M Umabala 2015), acetophenone (A.M. Umabala, P. Suresh 2016b), and Brilliant green (Umabala, Suresh, and Rao 2016) using BiVO_4 have been recently reported from this laboratory.

Among different types of dyes, xanthene dyes are most widely used and these are characterized by presence of xanthenes nucleus with aromatic groups as chromophore (Kaur J 2014). Rose bengal is an important xanthene dye widely used in textile and photochemical industries whose molecular structure as shown in Figure 1. It has severe toxic effects on the human health especially on corneal epithelium (HM 1998). This dye is

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very dangerous when it comes to contact with skin and causes itchiness, irritation, reddening and blistering. It also affects to eyes like inflammation, eye redness, itching etc. (Vinoda BM 2014). Present paper describes visible light heterogeneous photocatalytic degradation of Rose Bengal (RB) using monoclinic CuBi₂O₄.

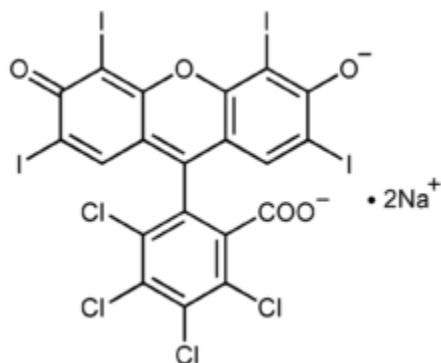


Fig 1 Rose Bengal

MATERIALS AND METHODS

Synthesis of Photocatalyst

CuBi₂O₄ was prepared by co-precipitation synthesis at 300 °C. Stoichiometric amounts of Cu(NO₃)₂·3H₂O (LOBA CHEMIE PVT. Ltd) and Bi(NO₃)₃·5H₂O (98% HIMEDIA) were dissolved in Ethylene Glycol separately by using magnetic stirrer. After completion of solubility two solutions were mixed with each other and heated at 50 °C. By maintaining this temperature, 5N NaOH was added to maintain the P^H at 8.5. At this P^H the mixed precursors start react with each other to produce a precipitate. This precipitate was washed several times with distilled water to completely remove the excess NaOH and the precipitate was filtered and dried at 80 °C in an air oven. The dried powder was calcined at 300 °C. The resultant catalyst was grounded to several hours to get fine powder and subjected to phase identification, microstructural investigation and photocatalytic studies.

Characterization Techniques

Phase purity of the resultant powder was investigated with X-ray diffractometer (PANalytical- X^{PERT} PRO, Japan) at room temperature, using Nickel filtered Cu-K_α radiation ($\lambda = 1.54059 \text{ \AA}$), with a scan rate of 2° min⁻¹. Microstructural investigation of the sample was performed on the powdered sample using SEM (JEOL-JSM-6610LV, Tokyo, Japan). UV-visible diffuse reflectance spectrum (UVDRS) of the sample was obtained with dry pressed disk samples using Shimadzu UV-visible spectrophotometer (UV-3600) between 200 to 800 nm range. Spectral grade BaSO₄ was taken as reference in the UV-DRS.

Photocatalytic activity measurements

Photo catalytic activity of CuBi₂O₄ was evaluated in terms of degradation of Rose Bengal dye under visible light. 50 mg of the catalyst was dispersed in 100ml RB aqueous solution (10 mg/L) and the suspension was magnetically stirred for half an hour in dark to ensure adsorption/desorption equilibrium between photo catalyst powder and dye. The suspension was then exposed to 400 wt metal halide lamp; 5ml aliquots were pipetted at periodic time intervals and filtered through 0.45 micron Millipore filters to remove the suspended powder. The

spectra as a function of irradiation time were recorded using UV-Visible spectrophotometer (Schimadzu). The extent of photodegradation was calculated using the following equation

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

where A₀ and A_t correspond to the initial absorbance and absorbance at time 't' respectively.

Photoluminescence studies

50 mg CuBi₂O₄ catalyst is added to the beaker containing 100 ml of terphthalic acid (TPA) solution (0.25 mmol L⁻¹ in 1mmol L⁻¹ NaOH solution). The solution is stirred for 15 min in dark followed by irradiation by 400 w metal halide lamp for 45 min. The reacted solution was centrifuged and the clear solution is used for photoluminescence measurements in a fluorescence spectro fluorometer (Flouromax 4) with the excitation wavelength of 315 nm.

RESULTS AND DISCUSSION

X-ray diffraction studies

The crystal structure and particle size of the obtained CuBi₂O₄ was analyzed by X-ray diffraction. Fig. 2 displays the XRD pattern of the sample. The XRD pattern of the CuBi₂O₄ can be indexed with six CuBi₂O₄ diffraction peaks at 27.8° (211), 29.6° (220), 33.1° (310), 44.9° (302), 46.5°(411), 52.71°(213) (JCPDS 72-0493), which suggests the presence of pure monoclinic phase of crystalline CuBi₂O₄ in the sample. The main diffraction peaks were selected to calculate the average grain size of CuBi₂O₄ by using the Scherrer's formula. The calculated average size of CuBi₂O₄ nanoparticles by using Scherrer's formula (Crystallite size D_p = K λ / (β cos Θ)) observed is 68.29 nm.

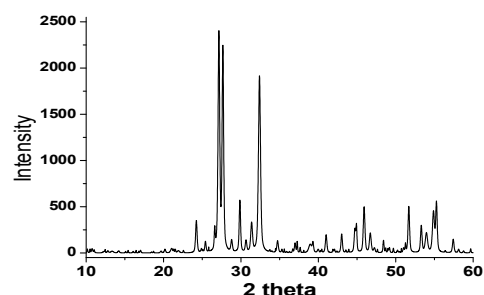


Fig 2 X-ray diffraction patterns of CuBi₂O₄.

UV-Vis diffuse reflectance studies

The UV-visible diffuse reflectance spectrum of monoclinic CuBi₂O₄ prepared at 300 °C was measured as shown in Fig. 3. It can be seen that the absorption wavelength edge of CuBi₂O₄ is extended greatly toward visible light and its absorption intensity is also greatly increased. CuBi₂O₄ has greater absorption in the wavelength range of 300–800 nm. This indicates that the prepared sample absorbs both UV and visible light quite well, which is better than that of TiO₂. So the formation rate of electron-hole pairs on the photocatalyst surface also increases greatly in visible light irradiation. As can be seen in Fig. 2, the as-synthesized CuBi₂O₄ exhibits an absorption onset at 489.502333 nm, which corresponds to band gap energy of 2.61 eV. This value is much lower than that of TiO₂ (3.2 eV) and thus the as synthesized CuBi₂O₄ sample can be predicted to be a satisfying visible light active photocatalyst.

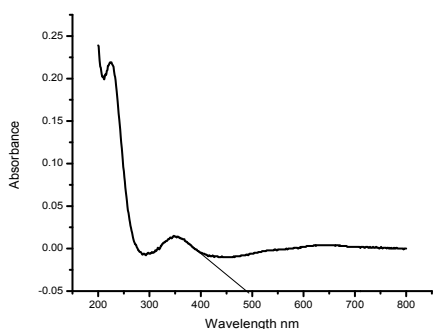


Fig 3 UV-Vis diffuse reflectance spectra of CuBi₂O₄

Microstructural investigation studies

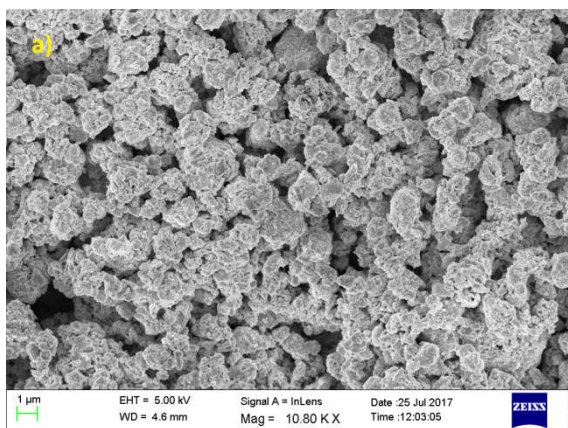


Fig 4 a) SEM image and b) EDS spectra of CuBi₂O₄

The microstructures of the obtained samples were studied by SEM analysis as can be seen from Fig 4. a) The SEM image show that the photocatalysts consist of a large number of hierarchical nanostructures like submicro-flowers (Zhang *et al.* 2015) with the size less than 100 nanometers and two-dimensional crystalline nanoparticles. These particles exhibit agglomerated (Zhang *et al.* 2015) morphology due to the ultrafine nature of the sample. EDS analysis of areas in Fig 4. b) was performed with its spectrum. It can be seen that the sample was composed of Bi, O elements and Cu respectively. The atomic percentages of Cu, Bi and O in CuBi₂O₄ are listed in Table.1 according to the EDS data. The quantifications of Cu:Bi:O is 1:1.97:4.19. The atomic ratio of Cu:Bi:O is close to 1:2:4, which closely agrees with the stoichiometric composition of CuBi₂O₄ (Zhang *et al.* 2015) (Zhang and Jiang 2015). This result is consistent with the XRD pattern presented above.

Table 1 The atomic percentage (mol %) of Cu, Bi and O in CuBi₂O₄.

Compound	Cu	Bi	O	Atomic Ratio
CuBi ₂ O ₄	13.95	27.53	58.52	1:1.97:4.19

FT-IR studies

The IR spectra of the calcined powder of CuBi₂O₄ shown in Fig. 5. After drying at 120°C, the spectrum is complex due to the existence of lots of organic compounds. The broad one around 700 ~ 400 cm⁻¹ originates from the metal-oxygen (Bi-O) vibration. The absorption band observed at 866 cm⁻¹ indicates the symmetric stretching of Bi-O bond in CuBi₂O₄ (Varaprasad *et al.* 2014). Bending vibrations of Cu-O at 574.79 (Hosseini-Sarvari and Moeini 2014) supports monoclinic structure of the catalyst. Stretching frequencies at 478.36 belongs to Cu-O (Hosseini-Sarvari and Moeini 2014).

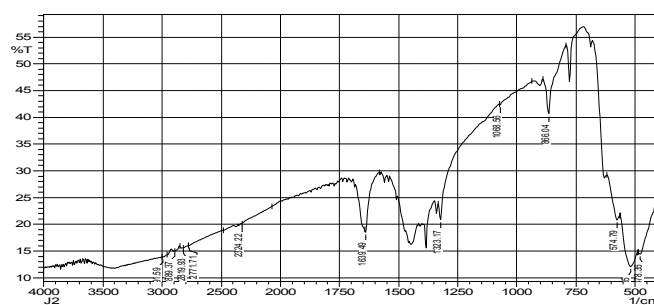


Fig 5 FT-IR spectrum of CuBi₂O₄.

Photocatalytic degradation

The decoloring curves of the prepared photocatalyst for RB under visible light were shown in Fig 6. to test the adsorption ability. At the beginning of the adsorption experiment, the photocatalyst rapidly adsorbed layers of dye molecules on their surface due to the high specific surface area and porosity, but the adsorption tended to be saturated after 30 min. First, the solutions were stirred for approximately 30 min in the dark to sufficiently adsorb dye molecules, and then irradiated under a 400 W xenon lamp with a 420 nm cutoff filter and a 500 nm filter to assess the visible photocatalytic properties. Activity of CuBi₂O₄ photocatalyst prepared at 300 °C is reaching approximately 90 % within 2 hours. We also found that CuBi₂O₄ photocatalyst can decolor RB dye solution without adding trace amount of H₂O₂, which can overcome the reported literature (Wei *et al.* 2010), that no obvious dye decoloration is observed for CuBi₂O₄ (Zhang and Jiang 2015) photocatalyst without H₂O₂ even after 1200 min. So the CuBi₂O₄ sample prepared by this co-precipitation method is the real photocatalyst not just the so called assisted-photocatalyst. The mechanism can be predicted by the following equations (A.M. Umabala, P. Suresh 2016a).

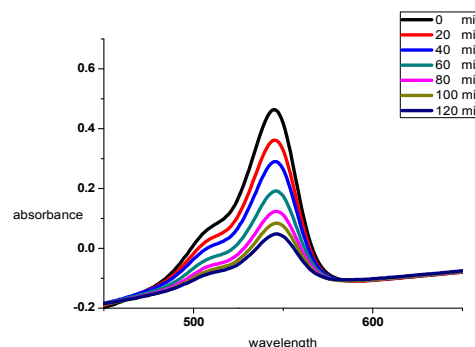
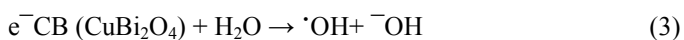
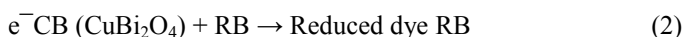
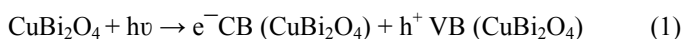


Fig 6 Photo catalytic degradation of RB over CuBi₂O₄.

Possible photocatalytic mechanism involved is suggested as follows;



Rapid formation of $\cdot\text{OH}$ free radicals through steps (3) and (4) is the important in accumulation of more $\cdot\text{OH}$ free radicals which used for disintegration of RB. In order to confirm the generation $\cdot\text{OH}$ free radicals during irradiation of CuBi_2O_4 , photoluminescence spectroscopy was used with Terphthalic acid (TPA) as a probe molecule. TPA combines preferentially with $\cdot\text{OH}$ to form hydroxy terphthalic acid (HTPA) which shows a characteristic luminescence peak at 422 nm. Fig 7. depicts photoluminescence spectra for CuBi_2O_4 + TPA prior to and after irradiation. Intense luminescence peak after irradiation confirms formation of $\cdot\text{OH}$ free radicals due to irradiation.

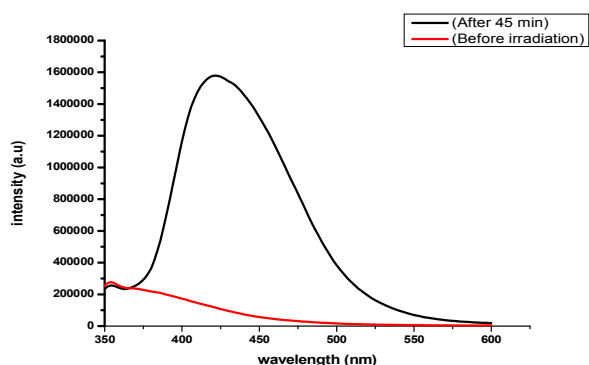


Fig 7 Photoluminescence spectra for CuBi_2O_4 +TPA prior to and after irradiation for 45 minutes.

Effects

Effects of pH Variation

Table 2 Effect of variation of pH on reaction rate

Effects of Variation of pH	$\text{CuBi}_2\text{O}_4 = 0.1 \text{ g}$ Temp = 308 k
pH	$10^{-4} \times k \text{ (s}^{-1}\text{)}$
6.0	2.102
6.5	2.452
7.0	3.024
7.5	3.425
8.0	3.928
8.5	3.99
9.0	2.962
9.5	2.489
10.0	2.192

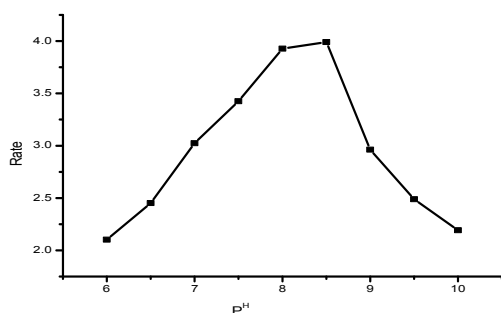


Fig 8 Effect of variation of pH on reaction rate

The effect of pH change on the rate of Photocatalytic degradation of RB was investigated in the pH range 6.0 -10.0. The dye solution was degraded and semi-conductor CuBi_2O_4 dissolved in highly acidic media and therefore photocatalytic degradation could not be investigated at lower pH range. The rate constants (k) for this reaction were determined using the expression $k=2.303 \times \text{Slope}$. The results were reported in above table. The rate of the degradation of RB was found to increase with increase in the pH value of the medium. In alkaline medium there is a greater probability for the formation of hydroxyl radicals (OH^\cdot), which can act as oxidant. Thus the rate of the photocatalytic degradation of the dye increases. But after certain pH value 8.5 a further increase in pH of medium decreases the rate of photocatalytic degradation. It may due to the fact that the dye does not remain in its cationic form. Due to greater concentration of OH^- and as such the reaction rate decreases.

Effect of amount of CuBi_2O_4

The results of effect of amount of semi-conductor CuBi_2O_4 powder on the rate of degradation are reported in table

Table 3 Effect of variation of amount of catalyst on reaction rate

pH = 8.5	[RB] = $3.0 \times 10^{-5} \text{ M}$ Temp = 308 k
Amount of CuBi_2O_4 g	$10^{-4} \times k \text{ (s}^{-1}\text{)}$
0.05	3.100
0.1	3.772
0.15	3.763
0.2	3.763
0.25	3.763
0.3	3.763

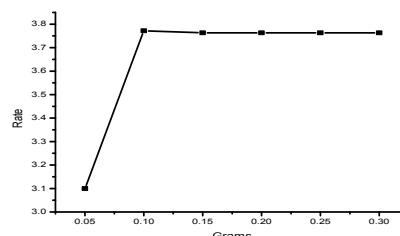


Fig 9 Effect of variation of amount of catalyst on reaction rate

As evident from the above data, the value of k increases with the increase in the amount of CuBi_2O_4 but time taken for degradation of RB decreases with the increase in the amount of CuBi_2O_4 . This increase in the rate of degradation may be attributed to increase in the exposed surface area of the CuBi_2O_4 . But after certain limit (0.1 g) if the amount of CuBi_2O_4 is increased further, there will be no increase in the exposed surface area of the Photo catalyst. It may be considered like a saturation point, above which increase in the amount of CuBi_2O_4 has no additional or negligible effect on the rate of photo catalytic degradation of RB.

Effects of Rose Bengal Concentration

Table 4 Effect of variation of [RB] on reaction rate

pH = 8.5	$\text{CuBi}_2\text{O}_4 = 0.1 \text{ g}$ Temp = 308 k
[RB] in ppm	$10^{-4} \times k \text{ (s}^{-1}\text{)}$
5	3.221
10	3.761
15	3.450
20	3.224
25	2.882

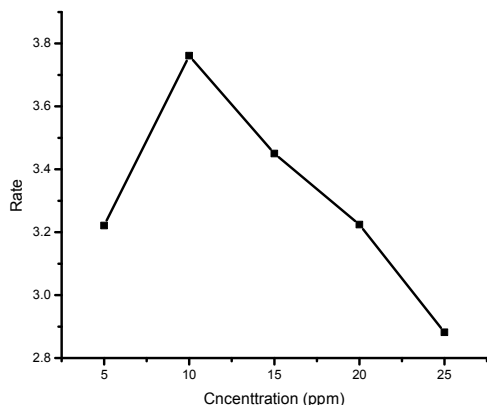


Fig 10 Effect of variation of [RB] on reaction rate

It is evident from the data that with the increasing [RB], reaction rate increases due to the increase in number of molecules participating in the reaction but after the optimum value of concentration 10 ppm, the rate of the reaction decreases. It can be explained on the basis that as the concentration of the dye was increased, it may start acting like a filter for the incident light and does not allow light to reach the semi-conductor surface.

CONCLUSION

Photocatalytic degradation of Rose Bengal was performed in the presence of the semiconductor CuBi_2O_4 . The degradation rate increased with increasing pH because more hydroxyl ions were present (generating more hydroxyl radicals). It attains maximum rate at pH 8.5; a further increase in pH above 8.5 results in a decrease in the rate of the reaction, because of decreasing attraction between the neutral form of the dye and the negatively charged semiconductor surface. Increasing the concentration of Rose Bengal also increased the rate up to a certain value due to the increase in the number of dye molecules, but it shows a declining behaviour on further increase of the concentration of dye. This decrease may be attributed to the fact that at higher concentration, the dye may act as an internal filter for the incident radiations, which decreases the intensity of the incident radiation on the semiconductor particles. The results indicate that initially the rate increases with increasing amount of semiconductor but after 0.10 g, the rate becomes virtually constant (saturation behavior). This may be due to the complete coverage of the bottom of the reaction vessel by the semiconductor. Any further increase will not add to an increase in the exposed surface area but only increases the thickness of the layer. An increase in the light intensity will increase the number of photons striking semiconductor CuBi_2O_4 powder per unit area per second and as a consequence, the reaction rate increases almost linearly with the increase in light intensity. The optimum reaction conditions were obtained as: pH = 8.5; [Rose Bengal] = 10 ppm; CuBi_2O_4 = 0.10 g. Formation of $\cdot\text{OH}$ free radicals was confirmed by photoluminescence spectroscopy using Terphthalic acid.

Acknowledgements

The authors would like to thank University Grant Commission (UGC), New Delhi, INDIA, for their financial support of this

research under RGNF No. "F1-17.1/2016-17/RGNF-2015-17-SC-AND-6314/(SA-III/Website)"

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How to cite this article:

Jagadeesh Ch., Suresh P and Sailaja B.B.V (2017) 'Photocatalytic properties of CuBi_2O_4 prepared by the co-precipitation method: Degradation of rose bengal and possible reaction mechanism under visible light irradiation', *International Journal of Current Advanced Research*, 06(12), pp. 8605-8610. DOI: <http://dx.doi.org/10.24327/ijcar.2017.8610.1392>
