

## Study of ZnO-TiO<sub>2</sub> Composite Photocatalyst Mediated Photodegradation of Eosin Yellow

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**Abstract:** Composites of ZnO-TiO<sub>2</sub> powders have been prepared by simple combustion method using TiO<sub>2</sub> (rutile) as support and ZnSO<sub>4</sub>·7H<sub>2</sub>O as precursor for ZnO. To study the effect of constituent and combustion temperature on particle size and shape, two different molar ratios like 1:1 and 2:1 of ZnO-TiO<sub>2</sub> were prepared at 450 °C and 750 °C temperatures. For complete characterization of the prepared composites, Reflectance measurement, Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), FT-IR Spectroscopy, and X-ray powder Diffractometry (XRD) techniques were used. The 35-40 nm needles shaped, and 90-95 nm nano sized spherical composite particles were found at 750 °C and 450 °C respectively. The photocatalytic behavior of the prepared 2:1 ZnO-TiO<sub>2</sub> composites were studied by doing photodegradation experiment of Eosin Yellow as an organic pollutant under UV, and sunlight sources. It was found that the photodegradation follows pseudo first order kinetics. The highest percentage of decolourization was 99.52% under sun light irradiation in needle shaped composite prepared at 750 °C.

**Keywords:** Combustion method, Kinetics study, Organic pollutant, Photodegradation, SEM, ZnO-TiO<sub>2</sub> composite.

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### I. Introduction

Textile industries create wide range of water loaded with various components. The different processes of the fabrication classically used are spinning and weaving, the whitening, dyeing, impression and readiness. From these stages, water highly polluted with organic waste is produced during the dyeing process [1]. About 10-15% of the dyes are lost in wastewater during dyeing operation. This affects the esthetic merit of surface water and reduces light penetration, hampering aquatic lives and hindering photosynthesis. The discharged wastes containing dyes are toxic to microorganisms, aquatic life and human beings [2]. These deleterious effects of chemicals on the earth ecosystems are a cause for serious concern. These components sometimes are not biodegradable and are toxic enough for aquatic ecosystems [3, 4]. The treatment of textile wastewaters has always been a serious problem for these industries. Some classical methods such as: adsorption on activated carbon, ozonation, reverse osmosis, ion exchange on synthetic adsorbent resins, flocculation, decantation, etc. are available for removing dye from water. However, these methods have high operating cost or are not efficient due to complex aromatic structure [5].

Heterogeneous photocatalysis is an attractive advanced technology for removal of organic pollutants from water and air under light illumination. Titania, and zinc oxide are the most popular semiconductor oxides used as photocatalysts due to their unique blend of properties [6]. The coupling of two semiconductors, possessing different energy levels for their corresponding conduction and valence bands, provides an approach to achieve a more efficient charge separation, an increased lifetime of the charge carriers and an enhanced interfacial charge transfer to the adsorbed species favoring their photooxidation and further mineralization [7]. There is significant interest in the scientific research on nanostructured ZnO-TiO<sub>2</sub> composites with different configurations and morphologies in order to obtain more efficient photocatalytic degradation [8, 9]. In the present article, ZnO-TiO<sub>2</sub> nanocomposites of various molar ratios of ZnO-TiO<sub>2</sub> were synthesized through

thermal decomposition of their corresponding salt. The morphological properties of prepared nanocomposites were studied and then the as-prepared nanocomposites were used as photocatalysts to decolorize the Eosin Yellow, an organic dye, under two different light sources.

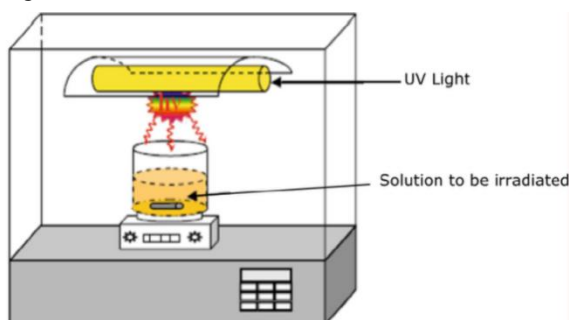
## II. Materials and Methods

**2.1 Preparation of nanocomposites:** ZnO-TiO<sub>2</sub> Composites were prepared the ratios 2:1 & 1:1. For preparation of composite ZnCO<sub>3</sub> was prepared in situ by using aqueous solution of ZnSO<sub>4</sub>·7H<sub>2</sub>O (Merck, India) and (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>. For this purpose 10.0 g ZnSO<sub>4</sub>·7H<sub>2</sub>O was dissolved in 500 mL of deionized water in a borosilicate beaker and required amount (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> (Merck, India) solution was also prepared in 250 mL volumetric flask. The solution of (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> was drop wise added to beaker until complete conversion of ZnSO<sub>4</sub> to ZnCO<sub>3</sub>, during this time 2.12 g TiO<sub>2</sub> (Merck, Germany) was added slowly with continuous stirring until solid layer formation bottom of the beaker. To accelerate the conversion it was heated moderately during addition [10]. Complete conversion was tested by adding excess ammonium carbonate solution. Finally suspended liquid was decanted off and portion was transferred quantitatively into crucible then it was heated in a muffle furnace at 750°C about 9.0 hours. Then product was weighted and 74.6% yield was found. This composite was the 1:1 ratio of ZnO-TiO<sub>2</sub> and 2:1 composite also prepared at 750°C by same way [10, 11]. In a similar way the composites of ZnO-TiO<sub>2</sub> ratios of 1:1 and 2:1 were prepared at 450°C temperature heated about 3 hours. Following similar procedure, The ZnO nanoparticle was also prepared from ZnSO<sub>4</sub>·7H<sub>2</sub>O without adding TiO<sub>2</sub> [12, 13].

**2.2 Characterization Techniques:** SEM (JSM-6490LA, JEOL, Tokyo, Japan) was used to study the particle sizes, XRD (RIINT 2100, Rigaku Corp., Tokyo, Japan) & FT-IR (Shimadzu, Japan) were used to study the structural characteristics, EDS 6490-LA, JEOL, Japan) was used for the chemical (elemental) characterization, and then reflectance measurement was used to determine band gap energy of the as-prepared samples.

**2.3 Photodegradation experiment:** 10.0 mL of de-ionized water was taken in 100 mL borosilicate beaker & 0.2 g of ZnO-TiO<sub>2</sub> composite was taken in it and kept overnight. After that it was made 4 × 10<sup>-5</sup> M adding 0.4 mL of 0.01 M Eosin Yellow solution & 89.40 mL water. The pH of suspension recorded. The prepared solution then carried out 40 minutes adsorption, the suspension was stirred magnetically while irradiating & it was continued for two hours. Afterwards suspension was centrifuged and ready for taking absorbance using UV-spectrophotometer (UV-1800 PC, Shimadzu, Japan). The percent degradation was calculated as:

$$\% \text{ Degradation} = (A_0 - A_t/A_0) \times 100$$



(Assuming degradation follows the same trend as that of decolourization)

Where  $A_0$  is the absorbance at time zero minute &  $A_t$  is the absorbance at time  $t$  minute.

The irradiation experiments were studied under sunlight (month of January) and UV-source at same concentration of EY dye & same amount (0.2 g) of (2:1) composite prepared at 450°C & 750°C temperatures.

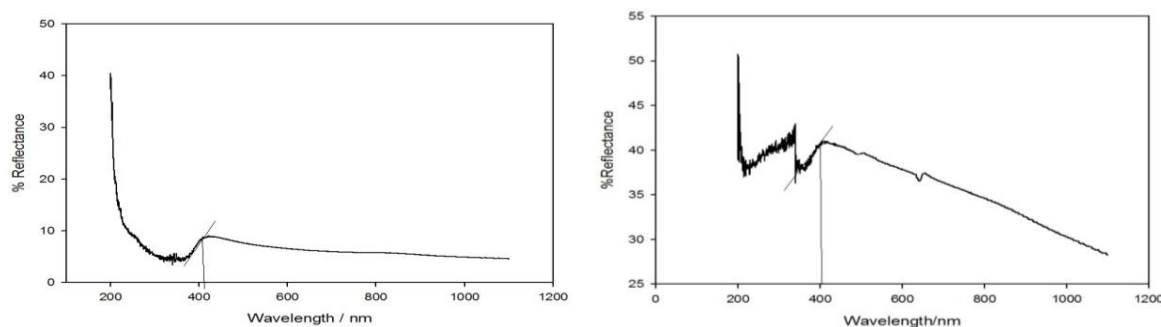
**2.4 Derived data for kinetics study:** The first order rate equation:  $\ln \frac{C_0}{C_t} = kt$

Where,  $C_0$  &  $C_t$  are the concentration of dye at zero minute and any time  $t$  minute respectively,  $k$  is the pseudo first order rate constant. Derived data for  $C_0$  and  $C_t$  were calculated from respective absorbance ( $A$ ) versus time data (Fig-7). The beer-lambert law ( $A = \epsilon cl$ ) was used to calculate the derived data when the molar extinction coefficient ( $\epsilon$ ) was calculated from calibration curve. Then  $\ln \frac{C_0}{C_t}$  versus time curve was constructed for kinetics study.

### III. Results and Discussions

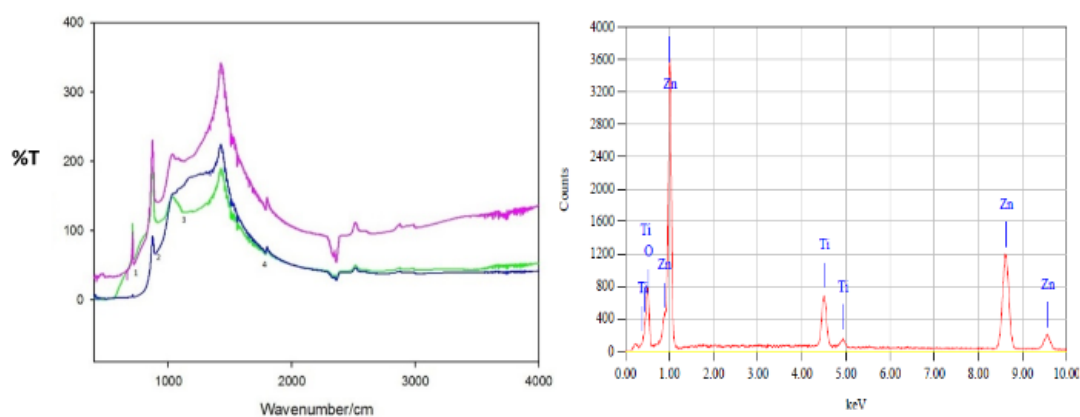
**3.1 Band gap energy:** Band gap energy of prepared ZnO-TiO<sub>2</sub>(1:1 & 2:1) composites was determined by reflectance measurement. The best fit points correspond to 407nm and 401.2 nm for 1:1 & 1:2 ZnO-TiO<sub>2</sub> composites respectively (Fig-1). Hence, absorption edge,  $\lambda = 407\text{nm}$  for ZnO-TiO<sub>2</sub> (1:1) composite. We know,  $E = hv = hc/\lambda$ ; where,  $h = 6.626 \times 10^{-34} \text{ Js}$  and  $c = 3 \times 10^8 \text{ ms}^{-1}$ . So,  $E = 4.88 \times 10^{-19} \text{ J}$ , Or,  $E = 3.05 \text{ eV}$

Similarly for ZnO-TiO<sub>2</sub> (2:1) nano-composite band gap energy is 3.10 eV. The band gap energy of TiO<sub>2</sub> is 3.23-3.32 eV and for ZnO 3.30-3.39 eV [12,13]. The band gap energy of prepared composites are smaller than that of both TiO<sub>2</sub> and ZnO. Hence it is expected that the composite would be better photocatalyst either TiO or ZnO.



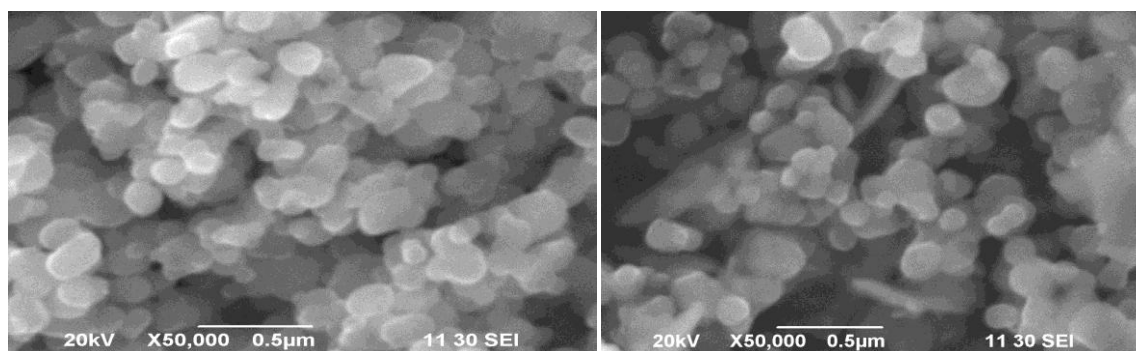
**Fig-1:** Measurement of reflectance as a function of wavelength for 1:1 & 1:2 ZnO-TiO<sub>2</sub> composites respectively.

**3.2 FT-IR and EDS spectrum:** Characteristics IR peaks of ZnO-TiO<sub>2</sub>(1:1) composites were identified in 435cm<sup>-1</sup>, 540cm<sup>-1</sup>, 610cm<sup>-1</sup>, 700cm<sup>-1</sup>, 860cm<sup>-1</sup> and 1200cm<sup>-1</sup>. The peak 435cm<sup>-1</sup> is for vibration of Zn-O-Zn bond, peaks 540cm<sup>-1</sup>, 610cm<sup>-1</sup> are for bending vibration of Ti-O-Ti bond and, peak at 1200cm<sup>-1</sup> is for stretching vibration of Ti-O-Ti bond [14-16]. The peaks 2 & 4 of composite are common both in TiO<sub>2</sub> and ZnO, the peaks 1 & 3 of composite are common in ZnO only (Fig-2). Thus from this observation it is concluded that the prepared composites were composites of TiO<sub>2</sub> and ZnO. From the elemental analysis of EDS (Energy Dispersive Spectroscopy) spectrum (Fig-2), it was shown that there no foreign elements were found except desired Zn, O, and Ti. Molar ratio of analyzed 2:1 composite also verified from the calculation of EDS analysis.

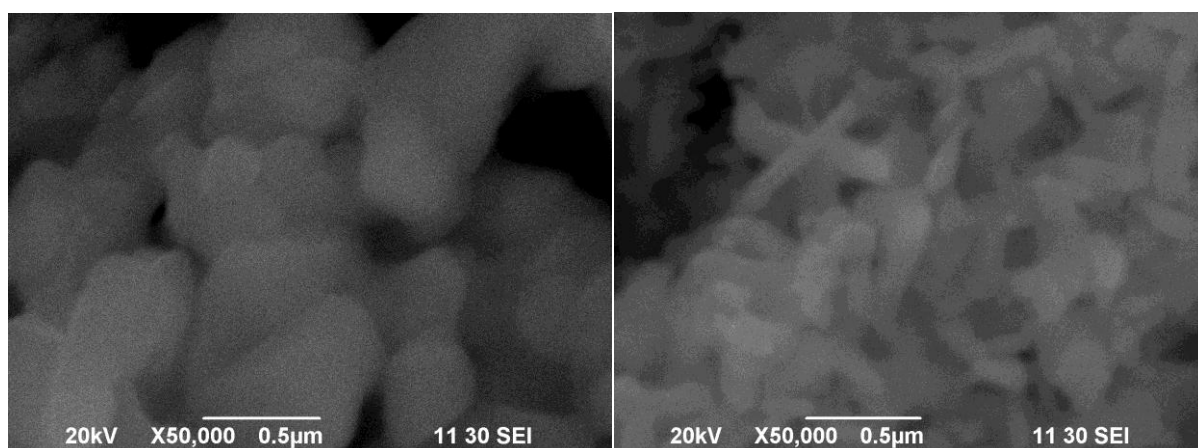


**Fig-2:** FT-IR spectrum (left side; green for 1:1 composite, Pink for prepared ZnO and blue for TiO<sub>2</sub>) & EDS spectrum of 2:1 composite (right side)

**3.3 SEM:** The SEM (Scanning Electron Microscope) images of ZnO-TiO<sub>2</sub>(1:1&2:1) particles prepared at 450°C & 750°C were taken at different magnification & different working voltage (Fig-3). Average particle size of 1:1 composite prepared at 750°C was found to be in the range of 200-210nm, in case of 2:1 composite needle shaped particles was found with 35-40 nm dimension, and the average particles size of composite prepared at 450°C was about 90nm to 95nm. Due to agglomeration of particles at higher temperature the larger particles size was found at 750°C (1:1 composite). Hence, the higher percentage of ZnO and higher preparation temperature both the factors are responsible for needle shapes [13] ZnO-TiO<sub>2</sub>(2:1) composite.

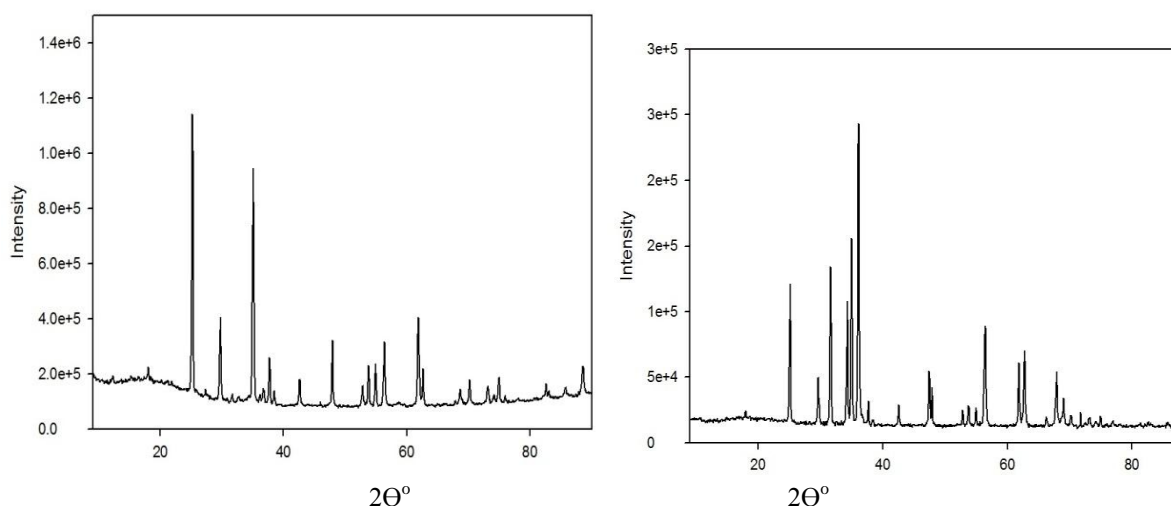


**Fig-3:** SEM images of ZnO-TiO<sub>2</sub> composite prepared at 450°C temperature at ratios 1:1(left)& 2:1(right).



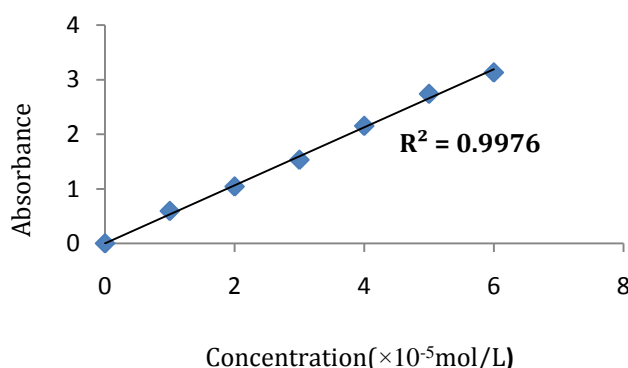
**Fig-4:** SEM image of 1:1 & 2:1(ZnO-TiO<sub>2</sub>) composites prepared at 750°C respectively.

**3.4 X-ray Diffraction:** X-ray diffraction (XRD) is one of the most important characterization tools used in solid state chemistry and materials science, which could provide most definitive structural information (e.g. interatomic distances, bond angles, crystallinity, and etc.). The extensive use of X-rays for the analysis of atomic structural arrangements is based on the fact that the wavelength of the X-ray is in the  $1 \times 10^{-10}$ m range, which is the same order magnitude of the atomic spacings in crystalline solids. When x-rays interact with crystal lattice, a simple model called Bragg's law can be used to understand the required conditions for diffraction. The Bragg's law can be expressed as  $n\lambda = 2d\sin\theta$  where  $\lambda$  is the wavelength of X-ray,  $d$  is the spacing between layers of atom,  $\theta$  is angle between incident X-ray beam and scattering plane, and  $n$  is integer. Thus, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms if the atoms are arranged in a periodic fashion in crystals. And the structural information of the crystals can be revealed based on the diffraction peaks. The phase compositions of prepared ZnO-TiO<sub>2</sub> composites have been identified by XRD analysis (Fig-5). The appearance of composites were crystalline and have diffraction peaks at  $2\theta = 31.67^\circ, 34.43^\circ, 36.25^\circ, 47.54^\circ, 56.61^\circ, 62.73^\circ, 67.96^\circ, 69.03^\circ,$  and  $72.48^\circ$  corresponds to miller indices (100, 002, 101, 102, 110, 103, 112, 201 and 004) corresponding to polycrystalline ZnO of wurtzite phase which is excellent agreement with JCPDS 36-1451. In addition  $2\theta$  values  $27^\circ, 36^\circ$  and  $55^\circ$  indicating the rutile phase of titania (JCPDS 88-1175). Moreover  $25^\circ$  and  $48^\circ$  values of  $2\theta$  indicating the anatase phase of TiO<sub>2</sub> (JCPDS 84-1286)[13-16]. Finally it can be concluded that the analysis of FI-IR, EDS, XRD, SEM and reflectance measurement results confirmed the formation of nanocomposites.



**Fig-5:** XRD-spectrum of ZnO-TiO<sub>2</sub> of 1:1 & 1:2 composite respectively.

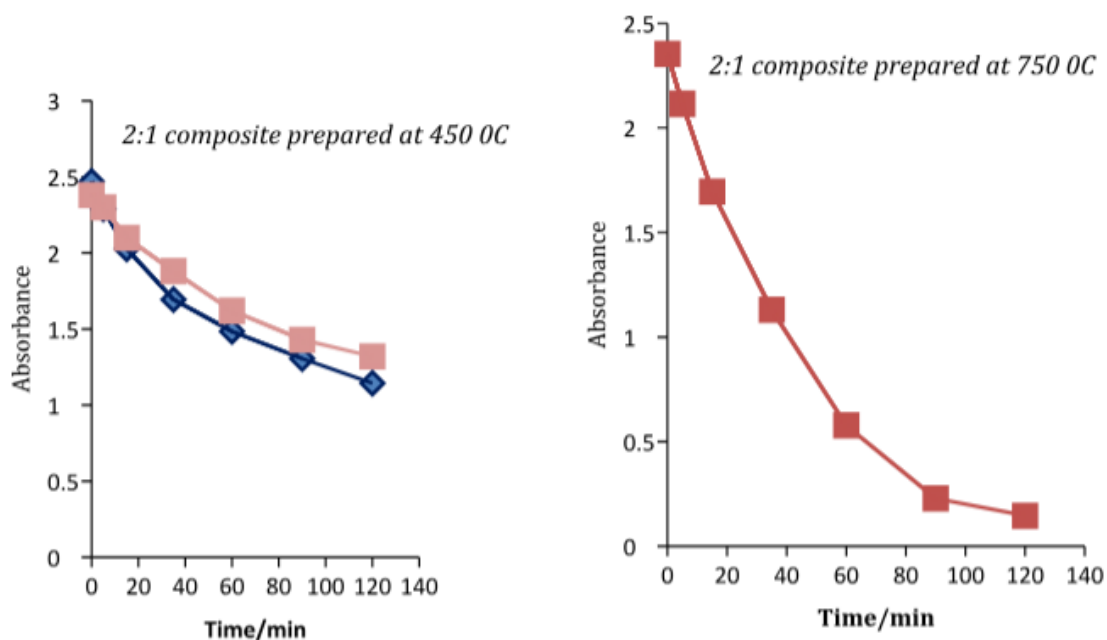
**3.5 Photodegradation:** Absorbances of Eosin Yellow at different solution concentrations at pH 6.67 were determined UV-Visible spectrophotometer at absorption maximum ( $\lambda_{max}=516$  nm). From the data of calibration curve the molar extinction co-efficient ( $\epsilon$ ) of Eosin Yellow in aqueous solution at room temperature (30°C) at  $\lambda_{max}$  (516 nm) was  $52050 \text{ L mol}^{-1} \text{ cm}^{-1}$ . Photodegradation experiments of EY dye using ZnO-TiO<sub>2</sub> (2:1) composite photocatalysts prepared at 750 °C and 450°C two different light source taking same initial concentration of EY and same concentration of composite-water suspension were carried out and results are shown in (Fig-7). Comparing the results of both figures it was found that the composite prepared at higher temperature is the better photocatalyst than that of lower one. It also was found that sunlight is the better irradiation source than UV light source. The percentage of degradation was found to be higher than 90% in both the cases. However, it was observed that after 90 minutes of degradation 96.9% of EY was degraded by composite prepared at 750°C and 89.99% of dye was degraded by composite prepared at 450°C (Fig-8). It is well known that at higher temperature due to agglomeration of the particles size become larger and surface area decreases and then photocatalytic activity decreased [14]. In the previous study of our laboratory it was found that nanoparticles prepared at lower temperature show higher photocatalytic activity for their smaller particle size [17-20]. Most interesting fact was that composite prepared at higher temperature show higher photocatalytic activity than that of lower temperature. This unusual phenomenon was found due to larger surface area of needle-shaped composite. Due to its needle shape more active sites available for adsorption of dye molecules.



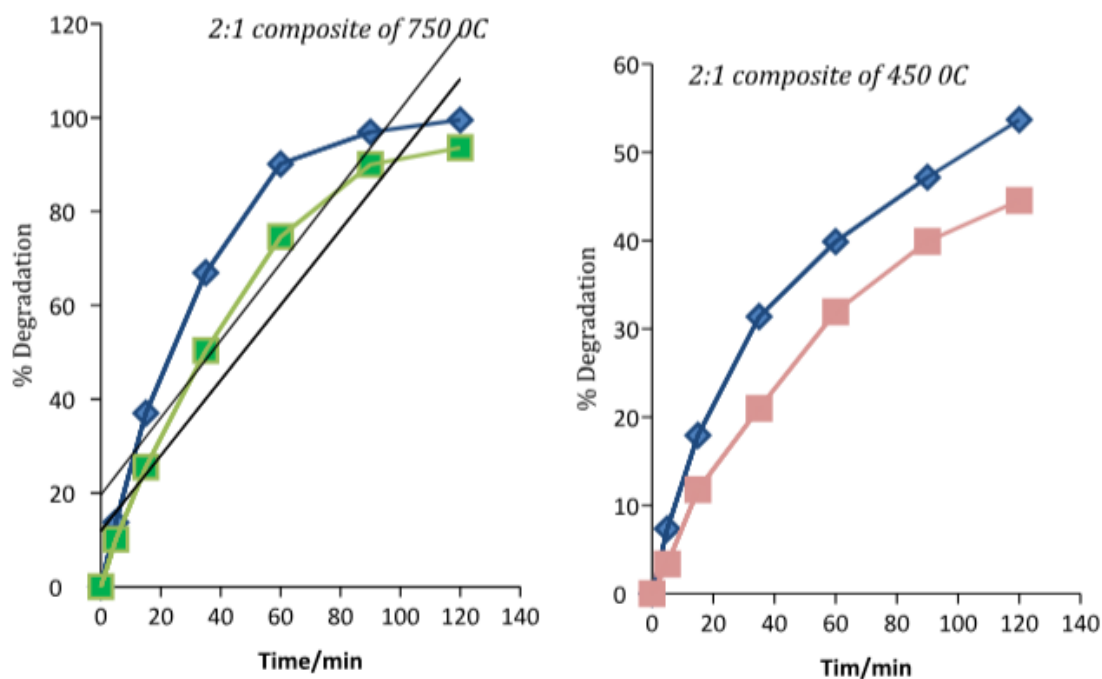
**Fig-6:** Calibration curve of Eosin Yellow

**Table-1:** Absorbance of EY solutions at  $\lambda_{max}$

Concentration of EY solutions (mol/L) × 10 <sup>5</sup>	Absorbance at $\lambda_{max}$
0.0	0.00
1.0	0.593
2.0	1.041
3.0	1.531
4.0	2.152
5.0	2.739
6.0	3.131



**Fig-7:** Decreases of absorbance with time during photodegradation of  $6 \times 10^{-5}$  M EY under UV & sun light sources.



**Fig-8:** Variation of percent degradation of 2.59 mg/L EY with time during photodegradation of 2 g/L composite under sun light (left side) and UV light (right side) sources.

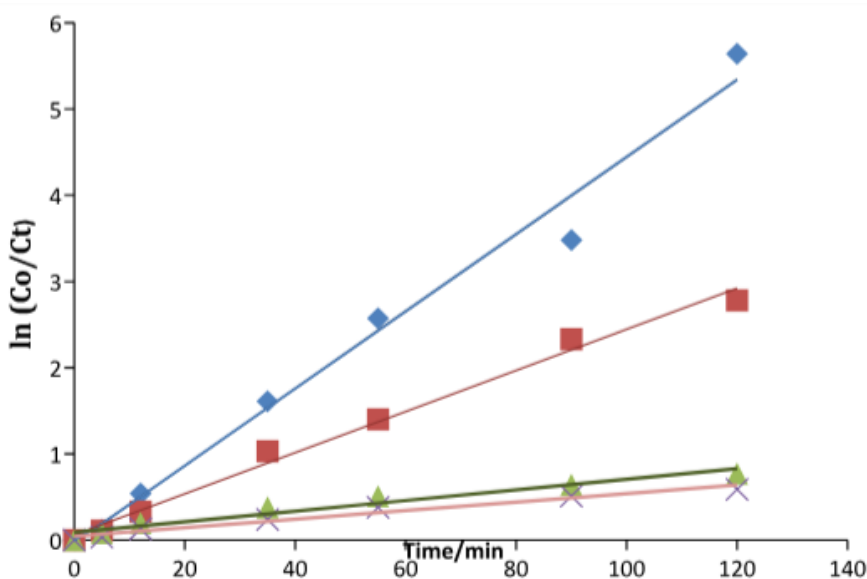
**3.6 Kinetics study of photodegradation:** The first order rate equation is  $\ln \frac{C_0}{C_t} = kt$ . Where  $C_0$  &  $C_t$  are the concentration of dye at zero minute and any time  $t$  minute respectively and  $k$  is pseudo first order rate constant. To determine the pseudo first order rate constant ( $k$ ) different values of  $\ln \frac{C_0}{C_t}$  at different time are calculated from respective absorbance vs time plot (Table-2).

**Table-2:** Derived data for  $\ln \frac{C_o}{C_t}$  at different time for composites from (Fig-7)

$\ln \frac{C_o}{C_t}$	Composite at 750 °C		Composite at 450 °C	
	Sun light irradiation	UV light irradiation	Sun light Irradiation	UV light irradiation
Time/min				
00	0.00	0.00	0.00	0.00
05	0.12	0.08	0.11	0.034
12	0.54	0.20	0.33	0.13
35	1.61	0.38	1.03	0.24
55	2.57	0.51	1.41	0.38
90	3.48	0.64	2.33	0.51
120	5.64	0.77	2.78	0.59
Rate constant	k=48.0×10 <sup>-3</sup>	k=23.6×10 <sup>-3</sup>	k=6.5×10 <sup>-3</sup>	k=4.9×10 <sup>-3</sup>

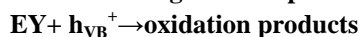
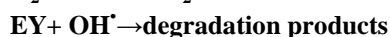
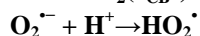
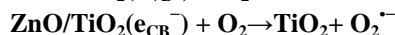
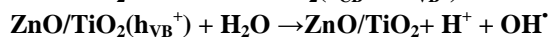
The plot of  $\ln \frac{C_o}{C_t}$  versus time (t) for composites under both sunlight and UV degradations follow first order integrated rate equation. Thus under sunlight & UV light degradation of EY using prepared composites follow pseudo first order kinetics [14,17, 21]. The values of rate constant k decreased when irradiation source was changed from sunlight to UV light (Table-2). The values revealed that the highest rate constant was found to be 48.0×10<sup>-3</sup>/min suggesting that the needle shaped composite is the most efficient photocatalyst and sunlight is the most effective irradiation source for EY mineralization.

This could be due to intensity of used light and different energy of sources.



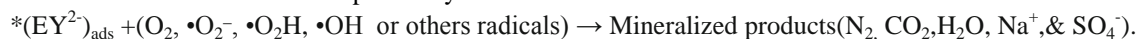
**Fig-9:**  $\ln \frac{C_o}{C_t}$  versus time plot to determine pseudo first order rate constant at above mentioned conditions

**3.7 Mechanism of photodegradation:** It is well established that conduction band electrons (e<sup>-</sup>) and valence band holes (h<sup>+</sup>) are generated when aqueous ZnO/TiO<sub>2</sub> suspension is irradiated with light energy greater than its band gap energy (eg 3.05 eV). The photogenerated electrons could reduce O<sub>2</sub> adsorbed on the composite-surface or dissolved in water, producing superoxide radical anion O<sub>2</sub><sup>-•</sup>. The photogenerated holes can oxidize H<sub>2</sub>O producing OH• radicals. OH• radicals react with organic molecules purely. Further radicals like .OOR, .OOH, .OR, .R are also formed [7, 21, 22]. According to this, the relevant reactions at the semiconductor surface causing the degradation of dyes can be expressed as follows.



The resulting •OH radical, being a very strong oxidizing agent (standard redox potential +2.8 V) can oxidize dyes finally can mineralize. EY decolourization is initiated by the attack of •OH and fragmenting the dye molecule.

Decolourization of EY is accompanied by its mineralization:



#### IV. Conclusion

Nanodimensional composites ZnO-TiO<sub>2</sub> in the different ratios and at the different temperatures were carried out successfully by simple combustion method (SCM), a cost effective, very simple but efficient technique. The different particle size and shape were found varying both prepared temperature and molar ratios of ZnO and TiO<sub>2</sub>. The shape of the particles of the composites 1:1 & 2:1 calcined at 750<sup>0</sup>C are spherical (200-210nm) & needle shaped (35-40 nm) respectively, but the particles shape becomes spherical in both composites when calcined at 450<sup>0</sup>C. The particle size of composite both 1:1 & 2:1 appears to be affected by temperature of calcination when calcined at 450<sup>0</sup>C, the size of the particles both cases fall in the range of 90-95nm. Increases of particles size could be due to agglomeration at high temperature. In the composite anatase and rutile phases of TiO<sub>2</sub> and Wurtzite phase of ZnO were confirmed from XRD analysis. Degradation of Eosin Yellow (EY), a typical organic dye has been carried out by ZnO-TiO<sub>2</sub> (2:1) composite under UV and Sun light irradiation. The maximum photodegradation was observed under sunlight using needle shaped composite at pH 6.67. Photodegradation of Eosin Yellow (EY) by ZnO-TiO<sub>2</sub> composite has been found to be affected by the nature of light sources. The percentage degradation of EY increases with the increase of intensity of light. Most interesting feature is that the composite prepared at high temperature can decolorize EY more efficiently than the composite prepared at low temperature. This unusual phenomenon was found due to larger surface area of needle-shaped composite. Since ZnO-TiO<sub>2</sub> composite can absorb light up to 516nm which is available in sunlight, it can cause photodegradation. In conclusion, it can be shown that ZnO-TiO<sub>2</sub> composite is an effective photocatalyst under sunlight irradiations and more effective of causing degradation than that of UV. It was found that the composite prepared following the same procedure having different composition shows different photocatalytic efficiency. Therefore, ZnO-TiO<sub>2</sub> nanocomposites may extensively use for industrial effluent mineralization especially organic dye degradation under sunlight irradiation.

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