

Study of Photocatalytic activity of ZnO and TiO₂ nanoparticles

Fauzia Khan, Seraj Anwar Ansari, Anees Ahmad

Abstract— Dye discharge from the textile industries are one of the major aquatic pollutant which was investigated by photocatalytic methods. ZnO and TiO₂ nanoparticles are the photocatalysts for the degradation of different dyes i.e. Methylene blue, Amido black 10B, Methyl red and Rose Bengal. A comparative study of photocatalytic degradation of different dyes under solar light was monitor up to 1 hour duration. It was found that Amido Black 10 B shows 99.90 % degradation with ZnO nanoparticle while 39.65 % with TiO₂. The percentage degradation for methylene blue was found to be 88.83% and 47% with ZnO and TiO₂ NPs respectively. Rose Bengal shows a highest percentage of degradation of 88.8% and 62% with ZnO NPs and TiO₂ NPs respectively. The percentage degradation of Methyl Red was found to be 50.5% and 3.11% with TiO₂ and ZnO NPs. Comparison of result shown that ZnO nanoparticle is more efficient photocatalyst than TiO₂.

Index Terms—Pollution, ZnO, TiO₂, Photocatalyst,

I. INTRODUCTION

Disposal of dyes from different small or large scale industries such as textiles, plastics, paper, leather, cosmetics, food industries etc. generally takes place in water bodies deporting ample of hazard to the environment. All through in dyeing processes about 1-20% loss occurs of the total world dye production and is released in the textile effluents [1]-[4]. These industrial dyes are responsible for the destruction of the ecosystem due to the slow biodegradation and their toxicities and also generate hazardous byproducts from many side by side chemical reactions occurring in the wastewater [5], [6]. To prevent the living organisms, including human being which are affected by these effluent of dyes there is a dire need to improve the existing processes of the water treatment to make them more eco-friendly. Photocatalysis has attracted a lot of attention in recent years as an operational process in the extenuation of environmental pollution due to totally mineralize the board pollutants [7]. Basically, photocatalysis is light induced catalytic process in which organic molecules are reduced or oxidized through redox reactions which occurs on the surface of the catalysts by numerous steps and many researchers described the mechanism of photocatalysis[8], [9] in detail. Zinc Oxide (ZnO) and Titanium dioxide (TiO₂) are supposed to be the noble photocatalysts for the elimination of numerous organic compounds owing to their high oxidation efficiency, chemical stability, inexpensive and eco-friendly nature [10]. Solar light is renewable source of energy which is available in

abundant amount, can be used for purification of semiconductors in the photodegradation of pollutants and make the procedure economically feasible [10].

The aim of this research work is the comparative study of the photocatalytic degradation of Methylene Blue (MB), Methyl Red (MR), Amido Black 10B (AB 10B) and Rose Bengal (RB) dyes by ZnO and TiO₂ nanoparticles in presence of solar light.

II. EXPERIMENTAL

1.1. CHEMICALS

Methylene Blue (MB) (493-52-7) Sigma-Aldrich, Amido Black 10B (AB 10B) (C.I.20470) Merck, Methyl Red (MR) (C.I. 13020) Merck, Rose Bengal (RB) (211-183-3) Merck, Zinc Oxide Nanoparticles (ZnO NPs), Titanium Oxide Nanoparticles (TiO₂ NPs), Distilled Water (DMW).

2.2 SYNTHESIS PROCESS OF NANOPARTICLES

2.2.1. ZINC OXIDE NANOPARTICLE

The method of preparation of ZnO nanoparticles was based on the wet chemical precipitation method. 0.2M of freshly prepared aqueous Zn(NO₃)₂·H₂O was taken as precursor and aqueous (1M) NaOH solution was added to it slowly in drops under continuous stirring for 4 hours and then the solution was filtered after 24 hours, washed with DMW until it become neutral. It was then dried for 3 hours at 60°C in an oven.

2.2.2. TITANIUM OXIDE NANOPARTICLE

Solution A-7.21 mL dibutyl phthalate was added to 20 mL absolute ethyl alcohol and stirred for 0.5 h using magnetic stirrer.

Solution B- 1.50 mL de-ionized water and 20 mL absolute ethyl alcohol were mixed together. 0.5 ml of concentrated hydrochloric acid was added to it to make liquid B.

Solution B was added to A and stirred intensely. After 0.5 h a gel is formed which is a little bit milky white in colour. The mixture was stirred continually for 12 hours (there will be twice ultrasonic dispersions in-between). The gel was then dried at 80 °C constant-temperature for 7 h in an oven. The product was porphyzied and kept for baking for 2 h at 550 °C in a muffle furnace. It was then cooled and stored in a desiccator for further use.

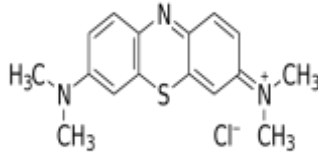
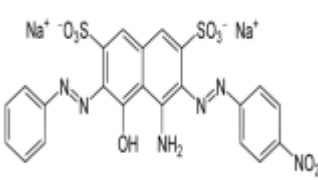
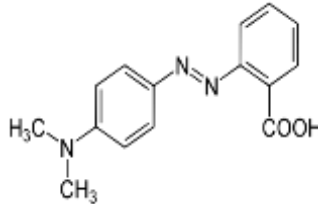
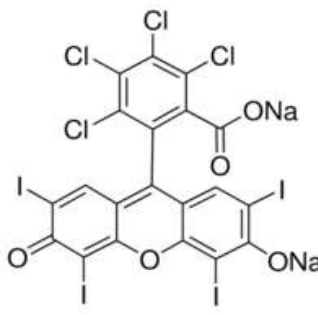
2.3. PREPARATION OF DYES SOLUTION

Stock dye solutions of 1000 ppm were prepared by dissolving 1.0 mg of a dye in 1000 mL volumetric flask with distilled water. 10 ppm was used as working solution prepared from stock solution. 100 mL of dye solution was measured and poured into beakers to study the photocatalytic degradation of ZnONPs and TiO₂ NPs. The samples were taken at every 10 min intervals of time to estimate the % degradation of each dye by the two catalysts. Table 1 shows the structure and physical properties of dyes

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	Chemical Name	Methylene Blue
	Molecular formula	C ₁₆ H ₁₈ ClN ₃ S
	IUPAC name	[7(dimethylamino)phenothiazin-3-ylidene]dimethylazanum;chloride]
	λ_{max} (nm)	668
	M_w (g/mol)	319.85
	Chemical Name	Amido Black 10B
	Molecular formula	C ₂₂ H ₁₄ N ₆ Na ₂ O ₉ S ₂
	IUPAC name	disodium;(6Z)-4-amino-3-[(4-nitrophenyl)diazenyl]-5-oxo-6-(phenylhydrazinylidene)naphthalene-2,7-disulfonate
	λ_{max} (nm)	620
	M_w (g/mol)	616.487
	Chemical Name	Methyl Red
	Molecular formula	C ₁₅ H ₁₅ N ₃ O ₂
	IUPAC name	2-[[4-(dimethylamino)phenyl]diazenyl]benzoic acid]
	λ_{max} (nm)	428
	M_w (g/mol)	269.30
	Chemical Name	Rose Bengal
	Molecular formula	C ₂₀ H ₂ Cl ₄ I ₂ Na ₂ O ₅
	IUPAC name	potassium;2,3,4,5-tetrachloro-6-(3-hydroxy-2,4,5,7-tetraiodo-6-oxoxanthan-9-yl)benzoic acid
	λ_{max} (nm)	549
	M_w (g/mol)	973.67

2.4. DEGRADATION STUDY OF METHYLENE BLUE AND AMIDO BLACK 10B DYE USING ZNO AND TiO₂ NANOPARTICLES AS A FUNCTION OF TIME

To observe the color removal of dyes by determining its absorbance at λ_{max} of dyes using UV-visible spectrophotometer 100 mg of ZnO and TiO₂ NPs was added into the beakers containing 100 ml of 10 ppm dye solution and to stir in darkness for 30 minutes to reach adsorption equilibrium between the dye and the photocatalyst. After 30 minutes, the dye solutions with nanoparticles were placed under the exposure of solar light irradiation. Since the different areas will receive sunlight at different intensities at different locations. So every replication was carried out at the same location to decrease the effect of variable sunlight intensity. At a regular 10 min time intervals, aliquot of dye sample was taken and centrifuged (centrifuge 5430, operate at maximum revolutions per minutes: 7830 rpm) for 20 minutes. Centrifugation is done to separate ZnO and TiO₂ NPs from degraded dye solution by avoiding the scattering of

nanoparticles. Extra precaution was taken when withdrawing the sample from centrifuge tube as to escape disturbing the unseating of nanoparticles on the wall of centrifuge tube. A minor move can cause resuspension of nanoparticles in dye solution. By doing this, a clear supernatant solution was obtained which is free from traces of nanoparticles in the sample which otherwise could lead to inaccurate result wherein presence of nanoparticles can contribute to amount of light scattering during the absorbance measurement. After centrifugation, the sample was subjected to UV-Vis Single Beam Spectrophotometer (PerkinElmer λ45UV-Visible spectrophotometer) scanning in the range of 200 to 800 nm. The maximum wavelength of Methylene blue, Amido black 10B, Methyl red and Rose Bengal dyes was recorded as 660nm, 618nm, 410 nm and 549nm respectively.

2.5. DETERMINATION OF DEGRADATION PERCENTAGE

Degradation percentage of dye solution was calculated by taking the absorbance values recorded at λ_{max} before and after photocatalytic degradation.

The equation was provided as shown:

$$\text{Degradation \%} = \frac{(C_0 - C_t)}{C_0} \times 100\%$$

Where,

C₀ = initial absorbance of dye solution before expose to sunlight

C_t = absorbance of dye solution at different time intervals (t)

III. RESULT AND DISCUSSION

1.2. DEGRADATION OF METHYLENE BLUE DYE UNDER SOLAR IRRADIATION

Figure 1 and 2, showed the UV-Vis spectra of dye degradation of Methylene blue solution by ZnO and TiO₂ NPs as a function of contact time. At different time intervals, the absorbance measurements of Methylene blue solution was recorded at λ_{max}=660 nm.

Based on the spectra, it was clearly seen that the intensity of absorption peak at λ_{max} decreases as reaction time increases from 0 minutes to 60 minutes. The absorbance depends on the number of molecules present in solution. That is the highest absorption peak of the dye under study contains highest concentration of Methylene blue dye solution which is yet to be degraded. From the UV-Vis spectra, the wavelength range was measured at the range between 200 to 800nm. Two absorption peaks were observed. A new peak appeared which served as an evident for the destruction of Methylene blue dye molecules through the photodegradation. Decolourization was attributed to the photocatalytic degradation with two outcomes either the peak corresponds to the maximum wavelength completely flattened or being shifted to another wavelength giving rise to generation of new peak in the spectrum [11]. Formation of new peak was due to the different intermediate metabolites formed during degradation (decolourization) of Methylene blue. However, the degraded products that give rise to this new peak cannot be defined exactly as the degradation mechanism of Methylene blue upon solar irradiation has not been devised. It was observed that a large gap appeared in between the control and the first time interval, the degradation efficiency was shooting up to 47.21 % at the first 15 minutes. The rapid removal of dye from

solution at the initial stage leading to a drastic decrement in absorbance reflects the degradation capability of nanoparticles for dye molecules [12]. After the addition of ZnO NPs and TiO₂ NPs, the solution was stirred for 30 minutes and reached equilibrium between adsorption and desorption. The dye molecules were allowed to adsorb on the surface active sites where adsorption occurs at ZnO and TiO₂ particles surface. As time increases, the surface active sites of ZnO and TiO₂ NPs was gradually occupied by dye molecules and reached a saturation point.

Consequently, less number of surface active sites were be available for further adsorption then followed by photodegradation which was a relatively slow process. This explained why there is only a slight increase in degradation percentage over contact time up to 60 minutes. From the calculation, the highest degradation percentage achieved by ZnO and TiO₂ NPs on Methylene blue was 88.83 % and 45.5% as shown in figure 3 and 4 respectively.

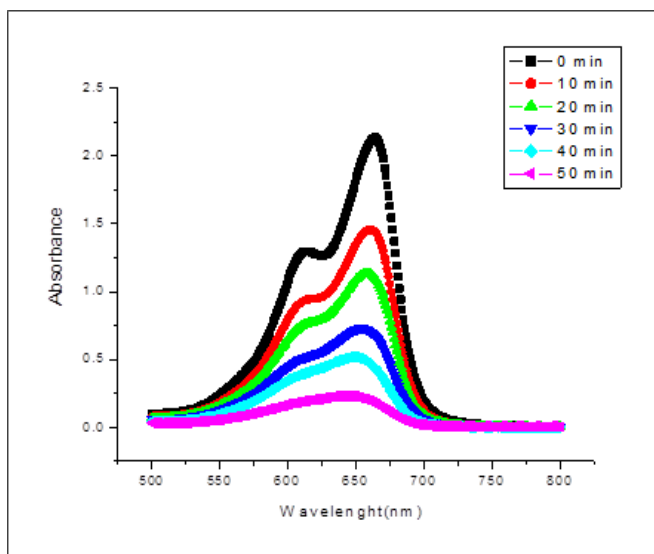


Figure 1. UV-Vis spectra of Methylene blue dye degradation with ZnO NP as a function of time

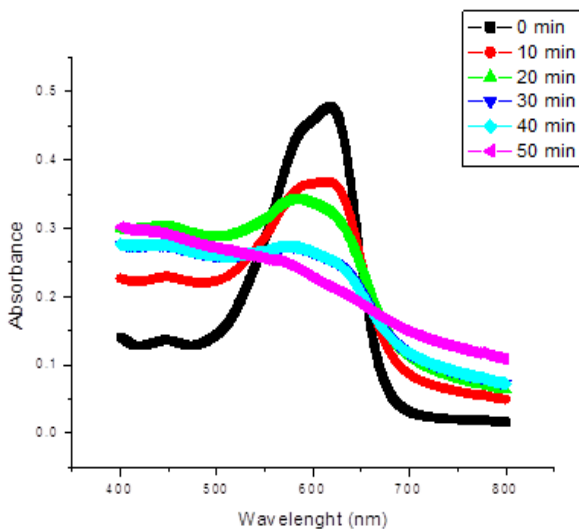


Figure 2. UV-Vis spectra of Methylene blue dye degradation with TiO₂ NP as a function of time

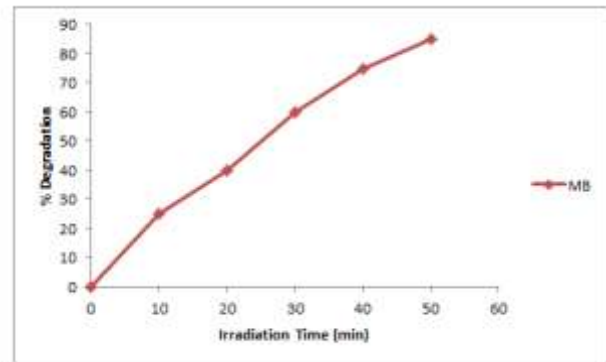


Figure 3. Degradation percentage of Methylene blue with ZnO NP at different time interval

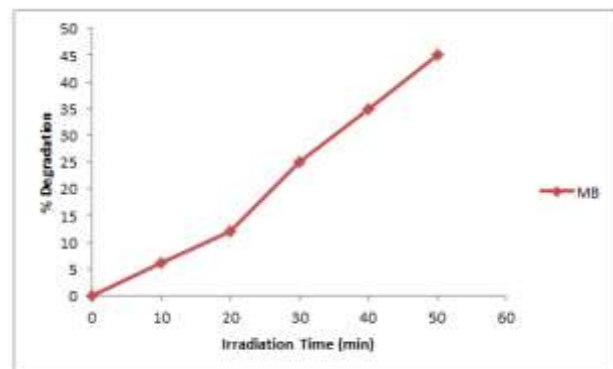


Figure 4. Degradation percentage of Methylene blue with TiO₂ NP at different time interval

3.2. DEGRADATION OF AMIDO BLACK 10B DYE UNDER SOLAR IRRADIATION

In this study, degradation of Amido black 10B by ZnO and TiO₂NPs was also investigated as shown in Figure 5 and 6 respectively. Aliquot of each of the sample were taken at different time intervals and the absorbance of the samples were measured spectrophotometrically at λ_{max} = 618 nm which similar to study done by which reported maximum wavelength of 620 nm.

It was observed that the absorbance of dye solution decreases with time. Thereafter, the concentration of dye solution decreases as reaction time increases. This can be clearly indicated by the decoloration of Amido black 10B from blackish-blue to almost colorless solution. The UV-Vis spectra of Amido black 10B dye degradation showed the similar trend as what we obtained for Methylene blue dye solution. The intensity of adsorption peaks at 618 nm decrease with increasing irradiation time. At the beginning of the reaction, dye degraded tremendously as compared with the degradation rate for the subsequently time intervals thereby the rate of degradation was seen to be very slow after the first 10 minutes. The degradation at the initial stage was very rapid but no hypsochromic shift was seen in this spectrum. This demonstrated that photodegradation mechanism of cleavage at the whole conjugated chromophore group of Amido black 10B structure was more favored. At the same time, it can be observed that a new peak has arised over time. This can be explained by random decomposition of Amido black 10B in many ways as destruction of polyaromatic rings or formation of intermediate products without conjugated system can give rise to the appearance of this new peak. There is another

possible mechanism which happened during the irradiation. Amido black 10B dye molecules absorbed energy and formed excited singlet state under which intersystem crossing (ISC) to triplet state. The degradation effect on Amido black 10B was not that prominent after the first 10 minutes exposure to sunlight. At the beginning stage, adsorption is more likely to happen (rapid process). Thereafter, dye degradation is at slow rate after adsorption throughout the whole photodegradation process. This is due to the surface active sites of ZnO NPs and TiO₂ NPs were already being occupied by previous dye molecules hence the number of available, free surface active sites of nanoparticles for the subsequent adsorption will be greatly reduced. This indirectly influences the rate of degradation because adsorption must occur first then followed by degradation of the dye molecules. In this case, the highest degradation percentage achieved in presence of ZnO and TiO₂ NPs are 99.90 % and 43.47% as shown in figure 7 and 8 respectively.

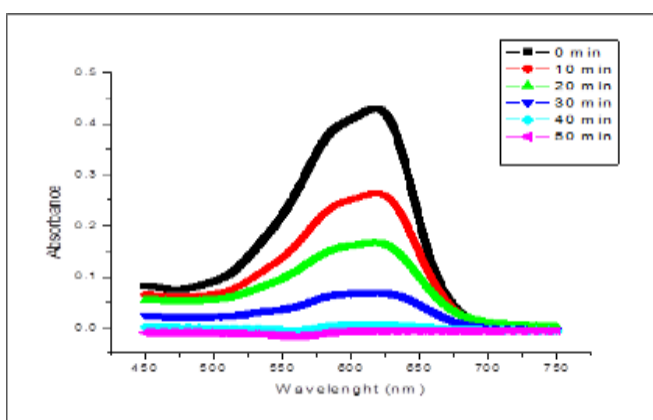


Figure 5. UV-Vis spectra of Amido black 10B dye degradation with ZnO NP as a function of time.

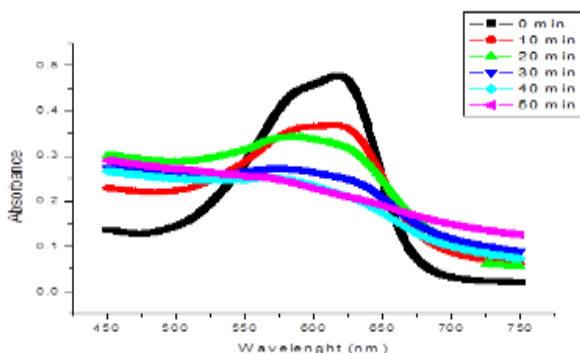


Figure 6. UV-Vis spectra of Amido black 10B dye degradation with TiO₂ NP as a function of time.

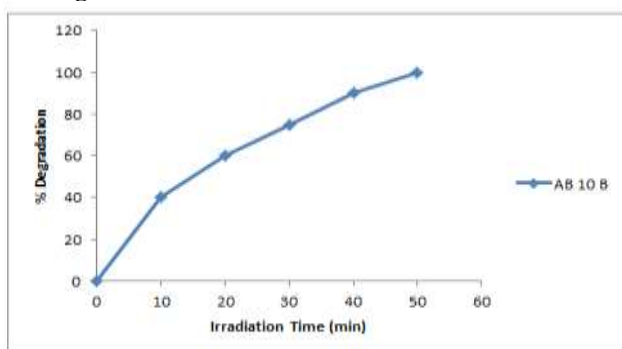


Figure 7. Degradation percentage of Amido black 10B with TiO₂ NP at different time.

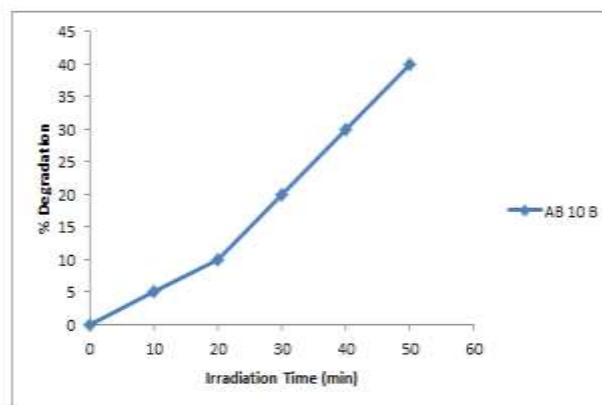


Figure 8. Degradation percentage of Amido black 10B with TiO₂ NP at different time

3.3. DEGRADATION OF METHYL RED DYE UNDER SOLAR IRRADIATION

Figure 9 and 10 showed the UV-Vis spectra of dye degradation of Methyl Red solution by TiO₂ and ZnO NPs respectively as a function of contact time. At different time intervals, the absorbance measurements of Methylene blue solution was recorded at λ_{max} = 410 nm. The λ_{max} of Methyl red was found closely matched with the λ_{max} of 428 nm [13]. Based on the spectra, it was clearly seen that the intensity of absorption peak at λ_{max} decreases as reaction time increases from 0 minutes to 60 minutes in case of TiO₂ as shown in figure 1 but it almost remains same for ZnO as shown in figure 2. The absorbance depends on the number of molecules reacted with it. In other words, the highest adsorption peak of degraded dye contains highest concentration of Methyl red dye solution yet to be degraded. From the UV-Vis spectrum, the wavelength range was measured in the range between 200 to 600 nm. Decolourization was attributed to photodegradation with two outcomes either the peak corresponds to the maximum wavelength completely flattened or being shifted to another wavelength. It was observed that a large gap appeared in between the control and the first time interval in case of TiO₂ whereas no such gap is observed when using ZnO NPs. The rapid removal of dye solution at the initial adsorption stage leading to a drastic decrement in absorbance reflects the adsorption capability of dye molecules to the nanoparticles surface. After adding of TiO₂ and ZnO NPs, the solution was stirred for 30 minutes and reached equilibrium between adsorption and desorption. The dye molecules were allowed to adsorb on the surface active sites of NPs. As time increases, the surface active sites of TiO₂ and ZnO NPs was gradually occupied by dye molecules and reached a saturation point. Consequently, less number of surface active sites were available for further adsorption then followed by photodegradation which was a relatively slow process. This explains why there is only a slight increase in degradation percentage over contact time up to 60 minutes. From the calculation, the highest degradation percentage achieved by TiO₂ and ZnO NPs with Methyl Red was 50.8% and 3.11% as shown in figure 11 and 12 respectively. Furthermore, the dye degradation can also be visually detected by the gradual colour change from red to almost colorless with TiO₂ NPs whereas no such colour change is observed with ZnO NPs

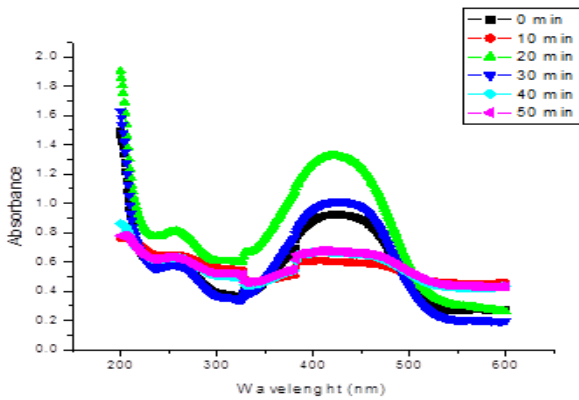


Figure 9. UV-Vis spectra of Methyl Red dye degradation with TiO₂ NPs as a function of time

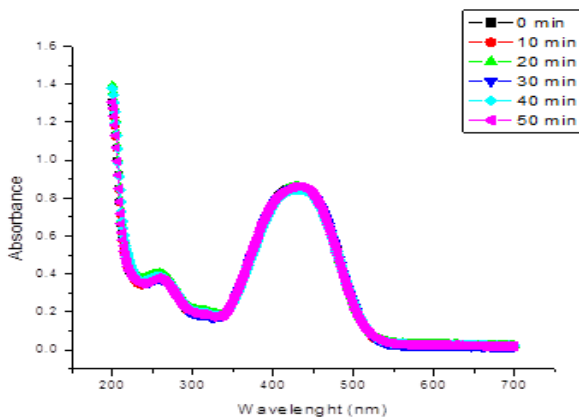


Figure 10. UV-Vis spectra of Methyl Red dye degradation with ZnO NPs as a function of time

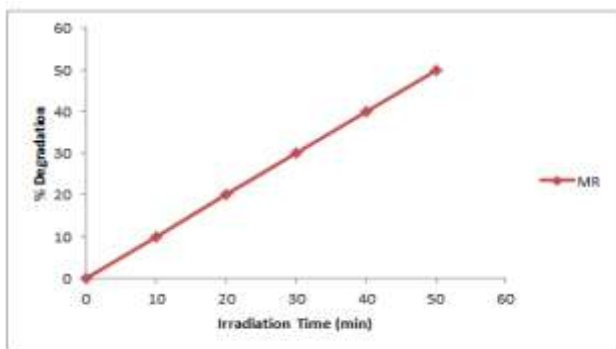


Figure 11. Degradation percentage of Methyl red using TiO₂ NPs at different time

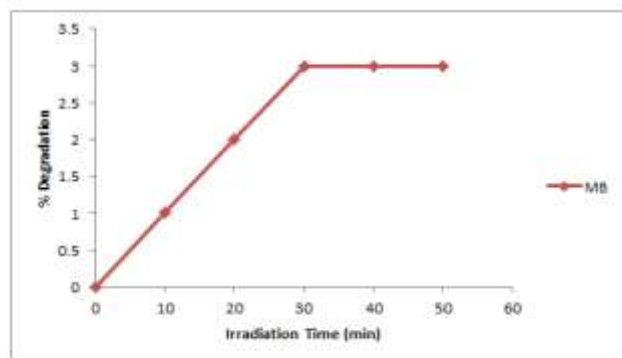


Figure 12. Degradation percentage of Methyl red using ZnO NPs at different time

3.4. DEGRADATION OF ROSE BENGAL DYE UNDER SOLAR IRRADIATION

In this study, degradation of Rose Bengal by TiO₂ and ZnO NPs was also investigated as shown in figure 13 and 14 respectively. Aliquot of each of the sample will be taken at different time intervals and the absorbance of the samples were measured spectrophotometrically at λ_{max} = 540nm which is similar to study done which reported maximum wavelength of 549 nm [14].

It was observed that the absorbance of dye solution decreases against time in both the cases. Thereafter, the concentration of dye solution decreases as reaction time increases. The UV-Vis spectra of Rose Bengal dye degradation showed a different trend as what we obtained for Methyl red dye solution. The intensity of adsorption peaks at 540 nm decreases with increasing irradiation time. In case of degradation of Rose Bengal with ZnO NPs the decrease in intensities of a dye with time is almost uniform as shown in figure 14. The degradation effect on Rose Bengal with TiO₂ NPs was not that prominent after the first 30 minutes exposure to sunlight, as shown in figure 13. In the beginning, adsorption is more likely to happen (rapid process). Thereafter, dye degraded with slow rate after adsorption throughout the whole photodegradation process. This is probably due to the surface active sites of TiO₂ NPs were already being occupied by previous dye molecules hence the number of available, free surface active sites of nanoparticles for the subsequent adsorption were greatly reduced. This indirectly influenced the rate of degradation because of adsorption which occurred first then followed by degradation of the dye molecules. The highest degradation percentage achieved for Rose Bengal with TiO₂ NPs is 62 %, shown in fig 15, whereas in case of ZnO NPs it is 88.8%, shown in fig 16.

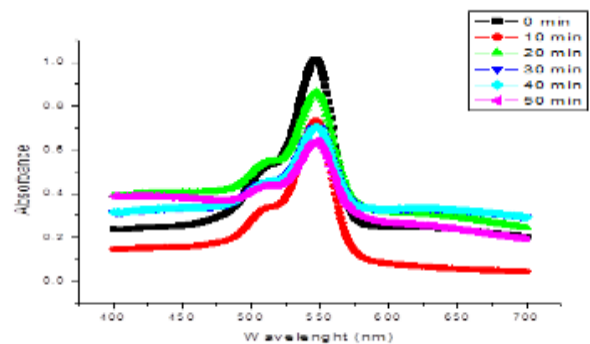


Figure 13. UV-Vis spectra of Rose Bengal dye degradation with TiO₂ NPs as a function of time

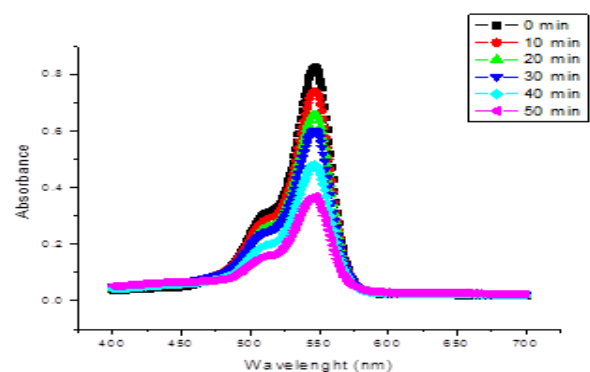


Figure 14. UV-Vis spectra of Rose Bengal dye degradation with ZnO NPs as a function of time

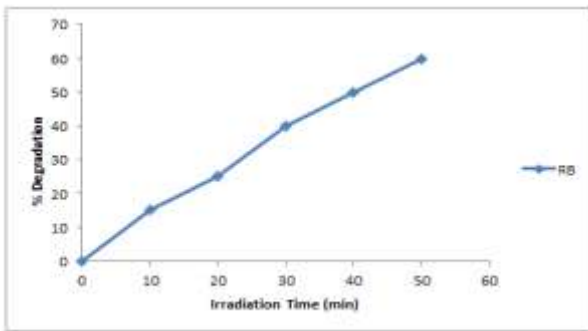


Figure 15. Degradation percentage of Rose Bengal with TiO₂ NPs at different time

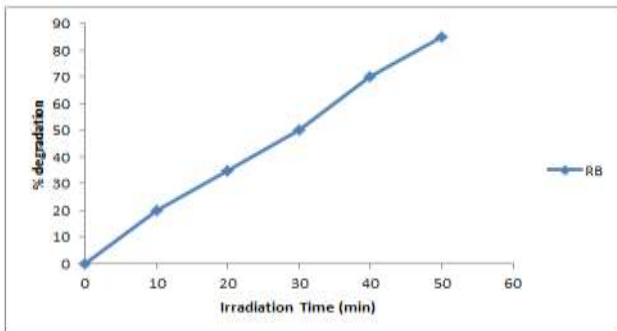


Figure 16. Degradation percentage of Rose Bengal with ZnO NPs at different time

3.5.1. DEGRADATION COMPARISON OF METHYLENE BLUE AND AMIDO BLACK 10B DYE SOLUTION IN ZNO NP.

Photocatalytic activity of ZnO NP on Methylene blue and Amido black 10B dye solution were evaluated. On the contrary, degradation efficiency of Methylene blue and Amido black 10B were found to be 88.83 % and 99.90 % respectively. It was observed that the degradation of Amido black 10B was higher as compared to Methylene blue as shown in figure 17

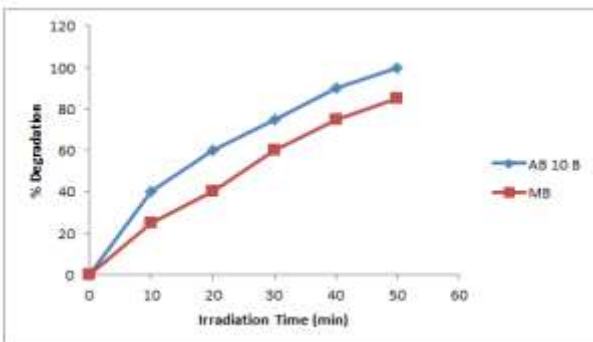


Figure 17. Degradation percentage comparisons by using ZnO NPs for MB and AB10B

3.5.2. DEGRADATION COMPARISON OF METHYLENE BLUE AND AMIDO BLACK 10B DYE SOLUTION IN TiO₂ NP

Photocatalytic activity of TiO₂ NP on Methylene blue and Amido black 10B dye solution were evaluated. On the contrary, degradation efficiency of Methylene blue and Amido black 10B were found to be 47 % and 39.65 % respectively. It was observed that the degradation of Methylene blue was higher as compared to Amido black 10B as shown in figure 18.

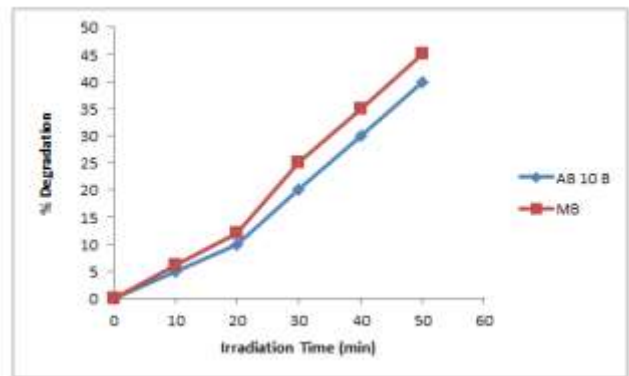


Figure 18. Degradation percentage comparisons by using TiO₂ NPs for MB and AB10B

3.5.3. DEGRADATION COMPARISON OF METHYL RED AND ROSE BENGAL DYE SOLUTION WITH TiO₂ NPS

Photocatalytic activity of TiO₂ NPs on Methyl red and Rose Bengal dye solution were evaluated. On the contrary, degradation efficiency of Methyl red and Rose Bengal were found to be 50.8% and 62% respectively as shown in figure 19

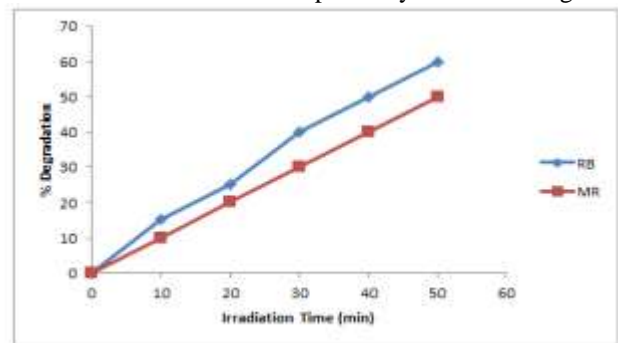


Figure 19. Degradation percentage comparisons by using TiO₂ NPs for MR and RB

3.5.4. DEGRADATION COMPARISON OF METHYL RED AND ROSE BENGAL DYE SOLUTION WITH ZNO NPS

Photocatalytic activity of ZnO NPs on Methyl red and Rose Bengal dye solution were evaluated. On the contrary, degradation efficiency of Methyl red and Rose Bengal were found to be 3.11% and 88.8% respectively as shown in Figure 20.

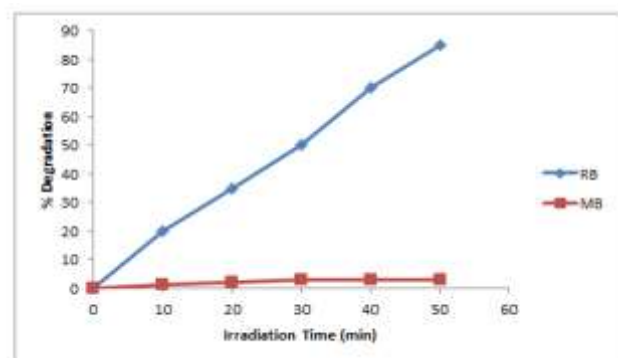


Figure 20. Degradation percentage comparison by using ZnO NPs for MR and RB

IV. CONCLUSION

The present research is focused on comparing the photocatalytic degradation of different dyes under solar light using nanoparticles.

A very simple method was used for the degradation of 10ppm solution of Methylene blue, Amido black 10B, Methyl red and Rose Bengal by using 100mg of ZnO and TiO₂ in the presence of sunlight. Table 2 shows the comparisons of percentage degradation of different dye in presence of ZnO and TiO₂.

A comparative study has been made, in which Amido black 10B shows a highest percentage of degradation of 99.90% with ZnO NP, while in presence of TiO₂ NP its degradation efficiency becomes 39.65%. The percentage degradation for methylene blue was found to be 88.83% and 47% with ZnO and TiO₂ NPs respectively.

Rose Bengal shows a highest percentage of degradation of 88.8% and 62% with ZnO NPs and TiO₂ NPs respectively. The percentage degradation of Methyl Red was found to be 50.5% and 3.11% with TiO₂ and ZnO NPs. Thus the removal of Rose Bengal can be achieved photocatalytically with the help ZnO and TiO₂ NPs whereas these nanoparticles are not as much efficient in removing Methyl Red from their aqueous solution.

Table 2. Comparisons of percentage degradation of different dye in presence of ZnO and TiO₂

	AB 10B	MB	RB	MR
ZnO	99.90 %	88.83 %	88.8 %	3.11 %
TiO₂	39.65 %	47.0 %	62.0 %	50.5 %

Above table shows that in general ZnO NPs is more efficient photo catalyst than TiO₂ NPs.

It is probably due to large surface area of ZnO NPs where there is more adsorption followed by more degradation by solar irradiation. The efficiency can be further enhanced if a reactor is designed for counter current adsorption/desorption cycles with simultaneous exposure to sun light.

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