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A Comparative Study over Degradation of Direct Green 6 by using Synthesized Magnesium Aluminate and Magnesium Zincate Nanoparticles

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Abstract: *Magnesium Aluminate ($MgAl_2O_4$) and Magnesium Zincate ($MgZnO_4$) nanoparticles were synthesized by solution combustion method, which were used as photocatalyst for the degradation of water containing direct green 6 (DG6). These synthesized nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), UV-absorption spectroscopy and FT-IR spectra studies. It was calculated that the corresponding band gap was found to be 2.9eV and 3.07eV respectively and also average crystalline size was found to be 13nm and 28nm. Photocatalytic degradation was determined against DG6 by varying parameters such as, catalyst concentration, pH, and dye concentration. From these experimental results we know that, the maximum degradation was found to be 97.99% for $MgAl_2O_4$ nanoparticle and 94.64% for $MgZnO_4$ nanoparticle. Hence the efficiency of photodegradation of DG6 dye by using $MgAl_2O_4$ and $MgZnO_4$ nanoparticles was ascertained.*

Key Words: *Direct Green 6, $MgAl_2O_4$, $MgZnO_4$, degradation.*

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I. Introduction

Dyes are the major sources for environmental contamination, these dyes are majorly used in textile industries and industrial waste water consists of these dyes, mainly azo dyes (1). More than 50% of textile dyes is azoic dyes which are recognized by nitrogen π-bound (2). Moreover, these azo dyes are non biodegradability due to their carcinogenicity, complex structure and high molecular weight (3). However, dyes are highly soluble in water contaminates water and also poisonous, this depletes water quality of water, but it also impacts on flora and fauna of water (4) and in developing countries (e.g., Bangladesh, West Bengal (India), Sri Lanka etc.) water bodies are highly polluted and causes environmental problems due to without any treatment of waste water (5).Direct green 6 (DG6), has diazo group, it has toxicity and carcinogenic nature (6). In the earlier studies other researcher have investigated the degradation of DG6 by different nanoparticles such as, Sr and Ag co-doped TiO_2, nano-strontium titanate (7) (8) and also removal of dye from activated carbon prepared by agro waste Traditional treatment methods are not able to remove complete colour from the waste water due to their aromatic groups in dye, but it can transfer pollutants from one phase to another phase, these methods are inefficient and costly. But in recent years, researcher has developed advance oxidant process (AOPs) to eliminate or destruction of dyes in aqueous medium. Some of the AOPs are Fenton reaction, ozonation, sonophotocatalysis, electro-chemical oxidation and heterogeneous photocatalysis (9). Among these heterogeneous photocatalysis is an effective tool for destroy dyes present in waste water (10). Out of these methods heterogeneous photocatalysis has emerged as a very efficient tool for the eradication and mineralization of dyes (11). In photocatalysis, light energy from a light source excites an electron from the valence band of the catalyst to the conduction band with a series of reactions which results in the formation of hydroxyl radicals. The hydroxyl radicals have high oxidizing potential and therefore can attack most organic structures causing oxidation. However, this method is cost effective and doesn't produce any secondary pollutants (12).

In our studies mainly focused on the photocatalytic degradation of direct green 6 dye by using synthesized magnesium aluminate and magnesium zincate nanoparticles and effect of pH, dye concentration and the catalyst concentration were studied.

II. Experimental

2.1. ***Materials:***

The commercially available water soluble dyes Direct Green 6 were obtained from Panam Biochemicals, Bangalore and used without further purification. Its structure is given in Fig. 1. The chemicals Magnesium Nitrate **(**$Mg(NO_3)_2$), Zinc Nitrate (Zn(NO3)2. 6H2O), AR, Aluminium Nitrate (Al(NO3)3.9H2O) were obtained from Loba Chemicals, Mumbai and Glycine ($C_2H_5NO_2$) were obtained from Hi-Media Chemicals, Mumbai. Sodium hydroxide (LR, ≥ 97%), which are used for adjusting the pH of the dye solution, were procured from RFCL Limited, New Delhi. The visible spectrophotometer (Elico, SL 177) has been used for recording absorbance at 493nm (λ_{max}). Later the absorbance with respect to time was recorded by visible spectrophotometer (Elico, SL 177).

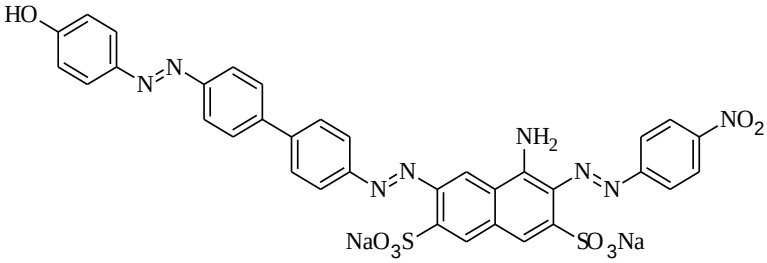


Fig 1: Chemical structure of Direct Green 6

2.2. Synthesis of Nanoparticles**:** The synthesis of Magnesium Aluminate and Magnesium Zincate nanoparticles by simple solution combustion method using glycine has a fuel.

2.2.1. Synthesis of $MgAl_2O_4$ nanoparticles**:**

The $MgAl_2O_4$ nano-particle was prepared by solution combustion method using $Mg(NO_3)_2$, $Al(NO_3)_3.9H_2O$ and $C_2H_5NO_2$(as fuel). Initially, $Mg(NO_3)_2$, $Al(NO_3)_3.9H_2O$ and $C_6H_{12}O_6$ were dissolved in 2:2:3 molar ratio with minimum quantity of water in a silica crucible. The crucible was introduced to the pre heated muffle furnace then furnace rises as heat from room temperature to 600° C. The solution boils and undergoes dehydration followed by decomposition along with the release of the certain amount of gases, it swells forming foam which ruptures with a flame. The product formed after combustion is a voluminous and foamy $MgAl_2O_4$. After the complete combustion, the crucible was taken out and allowed to cool.

2.2.2. Synthesis of $MgZnO_4$ nanoparticles:

The $MgZnO_4$ nano-particle was prepared by solution combustion method using $Mg(NO_3)_2$, $Zn(NO3)_2$. $6H_2O$, and $C_2H_5NO_2$ (as fuel). Initially $Mg(NO_3)_2$, Zn(NO3)2.6H2O and $C_2H_5NO_2$ were dissolved in 2:2:3 molar ratio with minimum quantity of water in a silica crucible. The crucible was introduced to the pre heated muffle furnace then furnace raises as heat from room temperature to 600° C. The solution boils and undergoes dehydration followed by decomposition along with the release of the certain amount of gases, it swells forming foam which ruptures with a flame. The product formed after combustion is a voluminous and foamy $MgAl_2O_4$. After the complete combustion, the crucible was taken out and allowed to cool. [13]

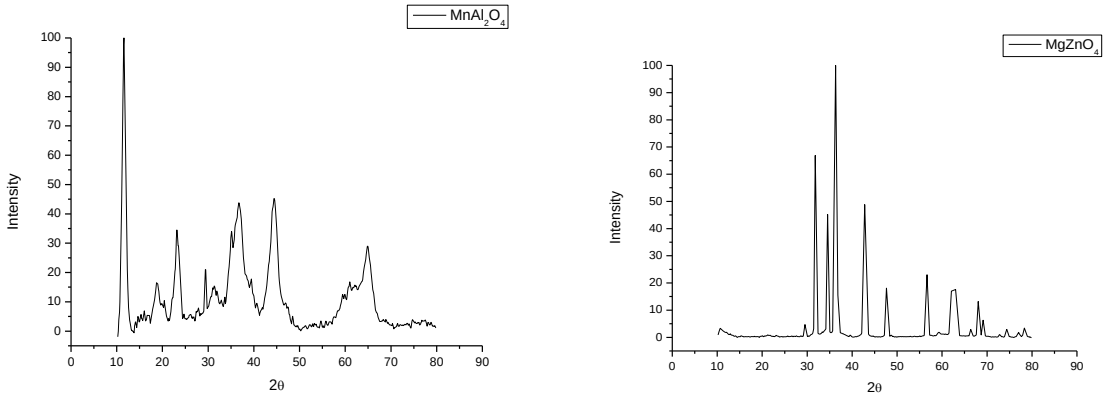
2.3. Characterization of Synthesized Nanoparticle

2.3.1. X-Ray Diffraction (XRD):

The XRD patterns of $MgAl_2O_4$ and $MgZnO_4$ nanoparticles reveal that, the presence of orthorhombic and Hexagonal structure and the 2θ peaks were observed which related to Magnesium Aluminium , (23.17°, 29.42°, 36.73°, 44.46°, 64.97°) (JCPDS card No.01-075-0278) for MgAl2O4 and for $MgZnO_4$ the peaks were related to Magnesium Zinc (34.54°, 36.2°, 47.82°, 68.02°, 78.30°) (JCPDS card No.03-065-4596) and the XRD was performed by powder X-ray diffraction (Rigaku diffractrometer) using Cu-Kα radiation (1.5406 Å) in a θ-2θ configuration. The pattern obtained from the XRD analysis of the prepared $MgAl_2O_4$ and $MgZnO_4$ nanoparticles is presented in Figure 2a and 2b.

According to the Debye Scherrer's formula: $D=K\lambda/\beta \cos\theta$ (4)

In the present work, the powdered sample of newly synthesized $MgAl_2O_4$ and $MgZnO_4$ nanoparticles were examined by XRD studies and found that $MgAl_2O_4$ nanoparticle size varies from 4 nm to 24 nm and henceforth the average crystallite size was found to be 13 nm. Whereas $MgZnO_4$ nanoparticles found to be varied from 22 nm to 35 nm and its average size was achieved on 28 nm respectively.



(a) (b)

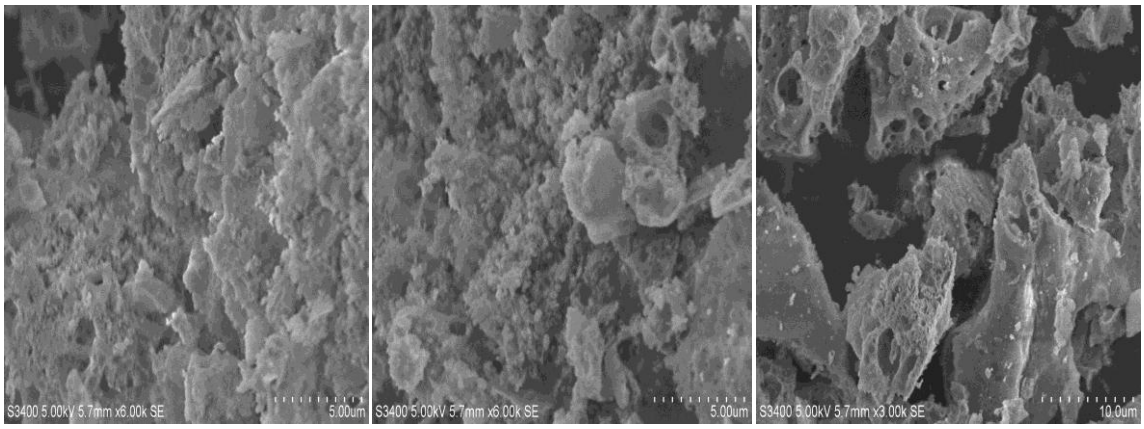
Fig 2: XRD of the synthesized Nanoparticles (a) $MgAl_2O_4$ (b) $MgZnO_4$

2.3.2. Scanning Electron Micrograph (SEM)

In the present study powdered sample of $MgAl_2O_4$ and $MgZnO_4$ nanoparticles were examined by using SEM technique for the study of external morphology of nanoparticles.

SEM study of $MgAl_2O_4$:

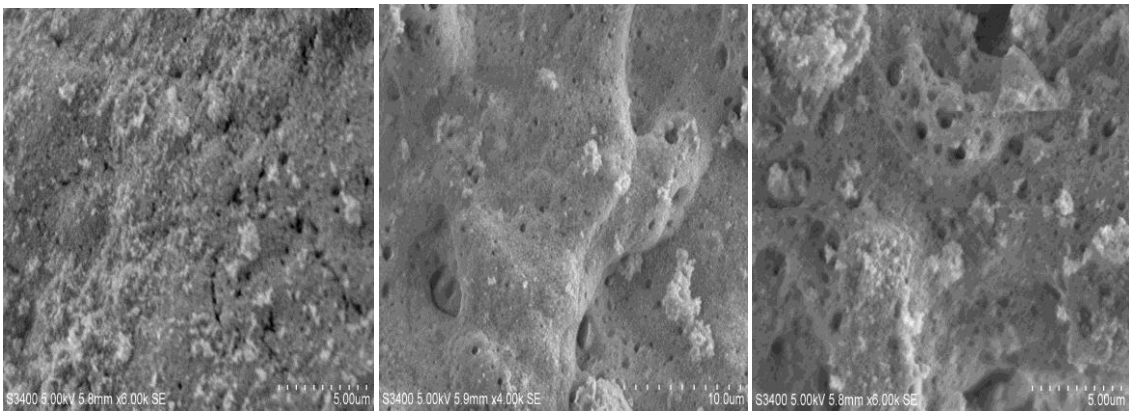
The SEM images of prepared $MgAl_2O_4$ nanoparticles exhibited irregular, dense crystals like appearance and non-uniform flakes like structure



(a)

SEM study of $MgZnO_4$:

The SEM images of prepared $MgZnO_4$ compound exhibited irregular, dense uniform cluster like appearance and non-uniform flakes like structure.

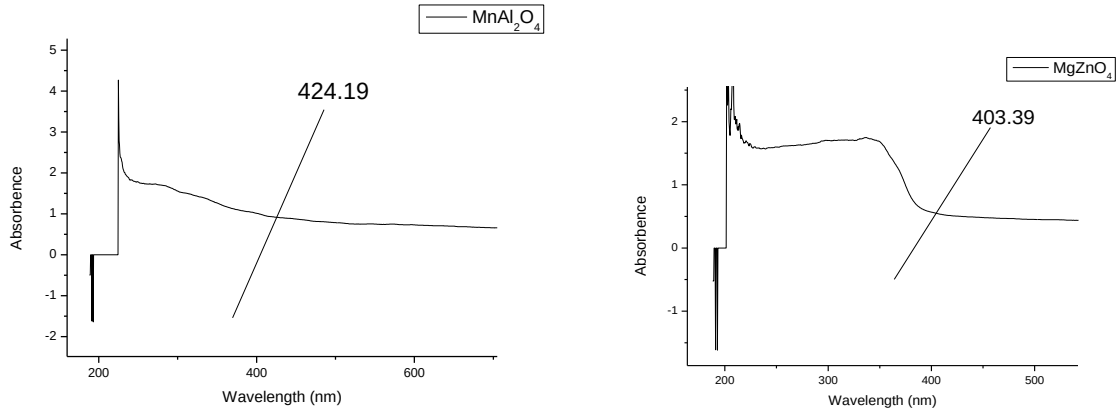


(b)

Fig 3: Scanning Electron Micrographs of synthesized (a) $MgAl_2O_4$ (b) $MgZnO_4$

2.3.3. UV-Vis Absorption Spectroscopy

The absorption spectrum of synthesized $MgAl_2O_4$ and $MgZnO_4$ nanoparticles were recorded using UV-VIS spectrophotometer of make Ocean Optics DH-2000, as taking synthesized nanoparticles directly into the spectroscopy over the wavelength range 200-1200nm.The spectral data showed the strong cut off appears at 424.19nm for $MgAl_2O_4$ and 403.39nm for $MgZnO_4$ [14, 15]. The band gap energy of the nanoparticles was calculated using the Planck's equation $E = hC/\lambda$ -- (Eq. 5) where h=Planck's constant, c=velocity of light and λ=wavelength. It was noticed that the band gap energy of nanoparticles was found to be 2.92eV for $MgAl_2O_4$ and 3.07eV for MgZnO4. The UV-absorbance spectra of synthesized MgAl2O4 and $MgZnO_4$ are presented in Figure 4a and 4b.

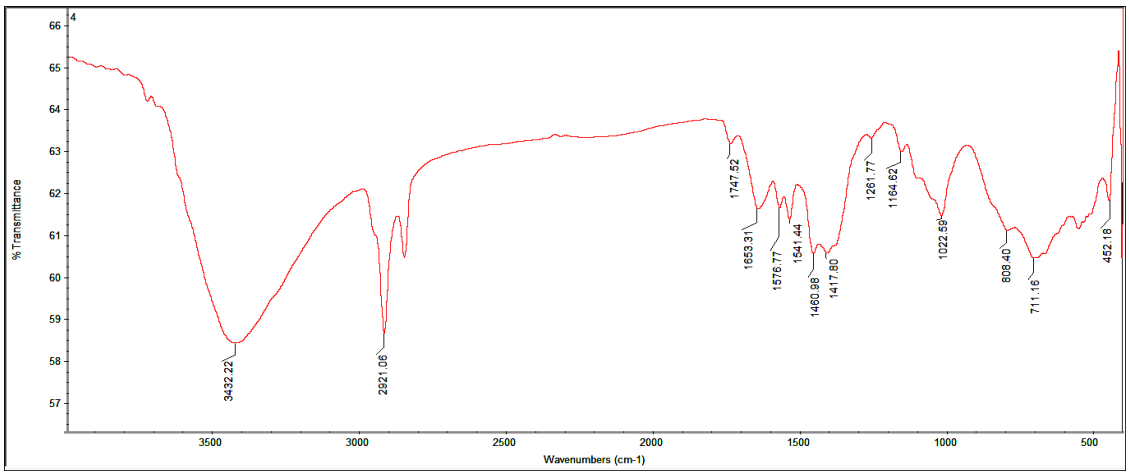


(a) **(b)**

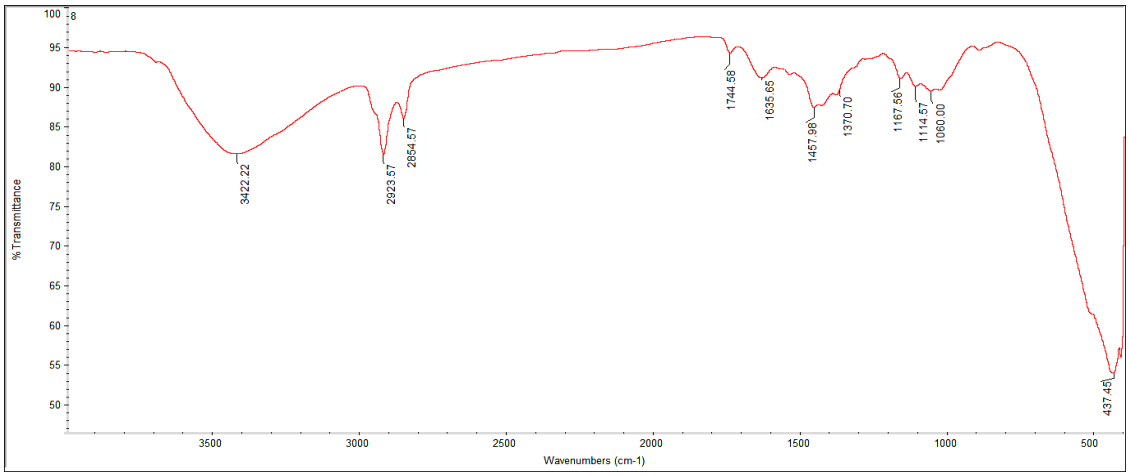
Fig 4: UV-absorption spectra of synthesized Nanoparticles (a) $MgAl_2O_4$ (b) $MgZnO_4$

2.3.4. FT-IR Spectroscopy

Fig: 5 showed the infrared spectrum of the synthesized nanoparticle such as $MgAl_2O_4$ and $MgZnO_4$ were ignited at 600°C. When these nanoparticles ignited while synthesis the absorption peaks are centred at 3432.22 cm-1was assigned to the O-H stretching band and the band 1460.98 cm-1was O-H bending band of $MgAl_2O_4$are featured. Respectively $MgZnO_4$ nanoparticles absorption peaks are centred at 3432.22 cm-1was assigned to the O-H stretching band and the band 1457.98 cm-1was O-H bending band. These bands are the evidence for the presence of water in prepared nanoparticle. However, the strong peak observed at 711.16 cm-1 and 437.45 cm-1 were assigned to Mg-Al-O and Mg-Zn-O stretching band respectively, which indicating the presence of $MgAl_2O_4$ and $MgZnO_4$ nanoparticle in ignited compound [16].



(a)



(b)

Figure 5: FT-IR Spectra of synthesized (a) $MgAl_2O_4$ (b) $MgZnO_4$ nanoparticles

2.4. Experiment Procedure:

The experiments were conducted in presence of sunlight. Primarily 30ppm of DG6 dye solution was prepared by dissolving 0.03 g of dye with 1000ml double distilled water and transferred to glass reactors for further proceedings. Aqueous photocatalyst dispersion was prepared by adding catalyst dosages (0.1g to 0.6g) to a 100ml dye concentrations. Prior to irradiation, the solution were magnetically stirred in darkness for 2 to 3 min to establish adsorption equilibrium. Irradiation was carried under natural sunlight between 11 a.m. and 2 p.m. and the average intensity of sunlight during this period is 892×100 lux unit using lux meter. To study the reaction of different pH level (pH 3 to pH 12), the initial pH of the dye solution was adjusted by addition of NaOH solution. Here for pH media experiment catalyst dosage remains optimum. Further, the experiments were repeated at optimum catalyst dosage for the study of different dye concentration solution. At each 30-minute interval of irradiation time, a 5-ml of sample was taken, centrifuged and concentration of Direct Green 6 was monitored spectrophotometrically by recording absorbance of the supernatant at a wavelength of 493nm (λmax).

The percentage was calculated by equation,

The percentage of decolourization = [(Ao- At) ÷ Ao]× 100

Where, Ao is the initial absorbance of the dye solution At is absorbance at time interval‘t’

Mechanism of Photodegradation

$MgAl_2O_4$ and $MgZnO_4 + h\nu \rightarrow (e^-_{CB} + h^+_{VB})$ (Eq. 4)

Step 1: The nanoparticles under sunlight irradiation get excited and transfer electrons to the conduction band.

$e^-_{CB} + O_2 \rightarrow O_2^{\bullet-}$ (Eq. 5)

Step 2: It can reduce molecular oxygen and produce the super oxide radical.

$H_2O + O_2^{\bullet-} \rightarrow OOH^\bullet + OH^-$ (Eq. 6)

$2OOH^\bullet \rightarrow O_2 + H_2O_2$ (Eq. 7)

$O_2^{\bullet-}$ + Direct Green 6 → Direct Green 6 -$OO^\bullet$ (Eq. 8)

Step 3: Molecular oxygen, adsorbed on the surface of the photocatalysts prevents the hole-electron pair recombination process [17, 18]. Recombination of hole-electron pair decreases the rate of photocatalytic degradation. This radical may form hydrogen peroxide or organic peroxide in the presence of oxygen and organic molecule.

$O\ OH^\bullet + H_2O + e^-_{CB} \rightarrow H_2O_2 + OH^-$ (Eq. 9)

Step 4: Hydrogen peroxide can be generated in another path.

$H_2O_2 + e^-_{CB} \rightarrow OH^\bullet + OH^-$ (Eq. 10)

$H_2O_2 + O_2^{\bullet-} \rightarrow OH^\bullet + OH^- + O_2$ (Eq. 11)

Step 5: Hydrogen peroxide can form hydroxyl radicals which are powerful oxidizing agents.

$OH^\bullet$ / $O_2^{\bullet-}$/ $MgAl_2O_4$ and $MgZnO_4^{\bullet+}$+ Direct Green 6 → Direct Green 6 degradation (Eq. 12)

Step 6: The radicals produced are capable of attacking dye molecules and degrade them.

III. Results and Discussions

3.1. Effect of Catalyst Concentration:

The effect of catalyst concentration was study by varying the catalyst concentration range from 0.1g to 0.6g/100ml of DG6 dye. The experimental results illustrated that, the efficiency of $MgAl_2O_4$ nanoparticles record 95.31% for 0.4g in 90min and for $MgZnO_4$ shows 92.18% for 0.5g in 90min (Fig.6). The highest

photocatalytic degradation shows in $MgAl_2O_4$ when compare to $MgZnO_4$ nanoparticles. Beyond the optimum catalyst concentration increase in the amount of catalyst which increases the number of dye molecules adsorbed on the dye surface and above catalyst concentration i.e. 0.4g for $MgAl_2O_4$ and 0.5g for $MgZnO_4$ nanoparticles was decreased in the photocatalytic degradation due to the overcrowding and collision of nanoparticles. However, the degradation was decreased due to the light penetration to the suspension (19).

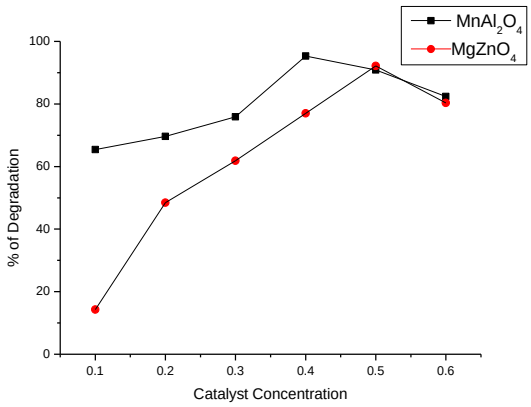


Fig 6: Effect of catalyst concentration on the photocatalytic degradation of DG6

3.2. Effect of pH: The pH parameter plays an important role in the degradation and pH varies from 3 to 11. The percentage of degradation of DG6 dye for $MgAl_2O_4$ (Fig. 7) increased from 75.66% to 97.99% from pH 3 to pH 10 and decreased to 97.76% at pH 11 in 90 minutes for 0.4g/100ml. For $MgZnO_4$ (Fig. 7) the percentage of degradation of the DG6 increased from 65.17% to 94.64% from pH 3 to pH 10 and decreased 80.13% at pH 11 in 90 minutes for 0.5g/100ml. From experimental results, the $MgAl_2O_4$ nanoparticles record maximum degradation when compared to $MgZnO_4$ nanoparticles due to the interaction of DG6 dyes on the surface of the catalyst increasing the generation of OH• radicals. These OH- ions will generate more •OH radicals by combining with the hole of the semiconductor and the OH• radicals are the main oxidizing species responsible for photocatalytic degradation. After the optimum pH (above pH 10) the degradation efficiency was decreased, can be explained on the basis of amphoteric nature of the catalyst. Here the catalyst surface becomes negatively charged for higher pH value, which causes the electrostatic repulsion between the catalyst and negatively charged dyes [20]. The maximum percentage of degradation for the two different nanoparticles was achieved at pH 10. The alkaline condition is more suitable when compared to acidic condition as it gives effective and good results in smaller dosages of catalyst (21, 22, 23).

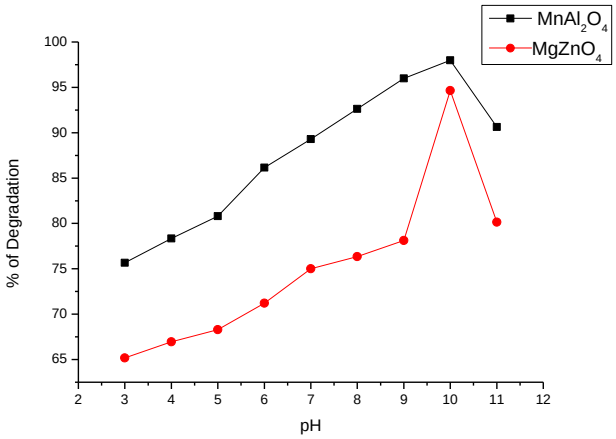


Fig 7: Effect of pH on the photocatalytic degradation of DG6

3.3. Effect of Initial Dye Concentration:

Dye concentration is a very important parameter in treatment of waste water. The effect of initial concentration of dye on the degradation was performed by varying the initial dye concentration from 30ppm to 50ppm with constant catalyst loading and pH. The results obtained for $MgAl_2O_4$ (Fig. 8) was 97.99% for 30ppm, 85.71% for 40ppm and 72.99% for 50ppm. And for $MgZnO_4$ (Fig. 8) is 94.64% for 30ppm, 83.25% for 40ppm and 69.86% for 50ppm, these experiments illustrated that the degradation efficiency was directly affected by the concentration.

When the concentration of the dye solution increases, this leads to the amount of dye adsorbed on the catalyst surface increases and affects the photocatalytic activity of the $MgAl_2O_4$ and $MgZnO_4$ nanoparticles, this also decreases the path length of photons entering into the dye solution. The degradation is mainly related to the formation of OH radicals, these OH radicals are the main critical species in the degradation process and The path length of photons entering the solution decreases, and in low concentration the reverse effect is observed, the number of photon absorption by the surface of catalyst in lower concentration when increase the dye concentration. Hence, the rate of degradation decreases with increase in the dye concentration (24, 25, 26, 27).

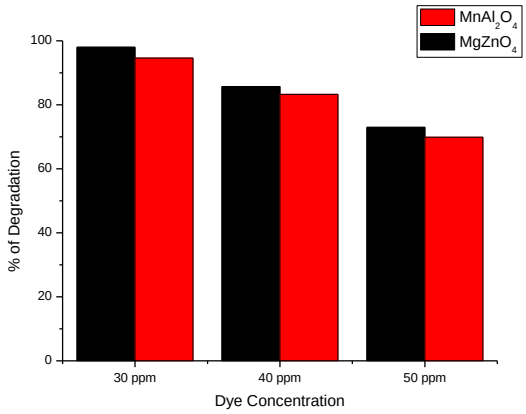


Fig 8: Effect of initial dye concentration on the photocatalytic degradation of DG6

IV. Conclusion

In the present study mainly focused on the degradation of DG6 dye under natural sunlight by synthesized $MgAl_2O_4$ and $MgZnO_4$ nanoparticles. The degradation depends on the size and band gap of the nanoparticle, $MgAl_2O_4$ nanoparticles achieved maximum degradation (97.99%) at 0.4g/100ml for pH 10 and for $MgZnO_4$ nanoparticles (94.64%) at 0.5g/100ml for pH 10 in a short interval time (90 min). Hence this can apply to the industry for the efficient treatment of the effluent which is hazardous to the environment and it is cost-effective too.

Acknowledgments

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