ISSN (Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

*An ISO 3297: 2007 Certified Organization* *Volume 4, Special Issue 1, February 2015*

THIRD NATIONAL CONFERENCE ON ADVANCES IN CHEMISTRY (NCAC – 2015)

On 18th February 2015

Organized by

Department of Chemistry, Easwari Engineering College (SRM Group of Institutions), Chennai-600089, India.

# Solar Light Aided Photobleaching of Congo red Dye Using Spions: a Novel Route for Complete Dye Degradation

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**ABSTRACT:** In this study super paramagnetic iron oxide nano particles (SPIONs), ZnO and Ce-$TiO_2$ were synthesized. The synthesized catalysts were characterized using X-ray diffraction, Fourier transform infra-red spectroscopy, diffuse reflectance UV-Visible spectroscopy, scanning electron microscopy and transmission electron microscopy. The photocatalytic efficiency of all the synthesized catalysts was investigated for the degradation of Congo red dye under solar light. Among the catalysts tested, SPIONs was found to be most active for the complete degradation of the dye. Other parameters influencing the dye degradation like effect of $Na_2CO_3$, hydrogen peroxide, pH and recyclability of the catalyst were also studied for the optimum degradation and the results have been discussed.

**KEYWORDS:** photodegradation, super paramagnetic iron oxide nano particles (SPIONs), ZnO, Congo red dye.

## I. INTRODUCTION

Textile, paint, pharmaceutical and cosmetic industries were the major source for contamination of both water and soil over the past decades. In industrial effluents, synthetic organic dye was found to be a significant environmental threat [1, 2]. Synthetic organic dyes are the harmful materials present in water effluents and are carcinogenic to human beings. The dyes present in the water effluents block the penetration of oxygen and sunlight which are essential for aquatic life [3, 4].

Degradation of azo dye, as reported by various papers can be dealt with techniques such as ultrafiltration, reverse osmosis, coagulation, sedimentation, ion floatation, adsorption etc. All these methods are versatile techniques to treat waste water. But the disadvantage is that it gives a secondary pollutant product which has to be further treated. It is time consuming and cost demanding [5-7]. The photocatalysis is the best and most promising method to remove the dyes present in the water effluents.

Azo dyes are a well known class of coloured organic compounds which are most widely used for its industrial applications. Congo red dye belongs to a group of azo dyes derived from benzidine. It has a very stable structure (Scheme 1). The stable structure makes it highly resistant towards degradation by micro organism and it is a suspected mutagen and carcinogen.

The degradation of Congo red dye have been so far carried out using ZnO under ultra violet radiation [8], ozonation process [9], and also utilizing certain plants like chara vulgaris[10], and aloe barabadensis [11]. So far, there has been no report showing the complete degradation of Congo red dye. Since a very low concentration of the dye is also proved to be a major environmental threat [12, 13], an alternative, convenient and efficient method using solar light for complete dye degradation is essential.

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Over the past years, the semiconductor oxides have been widely used in photocatalytic reactions. In the present study, the catalysts used are Ce-$TiO_2$, ZnO and SPIONs. SPIONs, with a band gap of 2.10 eV [14] have wide applications in nano-fields such as Ferro fluids, magneto caloric refrigeration, biotechnology, and in vivo bio-medical field using visible radiation [15]. ZnO, an n-type semiconductor having a band gap of 3.2 eV [16] absorbs light with a wavelength equal (or) less than 385 nm and is capable of generating hydroxyl radicals. $TiO_2$ has a band gap of 3.2 eV [17] and has properties like strong oxidizing power, photostablity and is non toxic. Metal loaded Titania displayed high photocatalytic activity [18].

Hence the present work deals with the synthesis and characterization of SPIONs, ZnO, cerium loaded titania and to study the degradation of Congo red dye using the synthesized catalysts along with pure titania in the presence of sunlight. Various factors influencing the photocatalytic activities like the effect of time on stream, inhibitors, electron acceptors, pH and recyclability were also studied.

## II. EXPRIMENTAL

**2.1 MATERIALS:** Cerium nitrate hexahydrate (Merck), sodium Hydroxide (Merck), Zinc sulphate heptahydrate and ferrous sulphate heptahydrate (Merck), Ferric chloride (Merck). Congo red dye (Merck) and titanium dioxide (Merck) was used without purification.

### 2.2 SYNTHESIS OF CATALYSTS:

**2.2.1. SYNTHESIS OF ZnO:** ZnO was prepared by the incipient method. $ZnSO_4.7H_2O$ was used as the starting material and NaOH as the precipitant in the mole ratio 1:2. The resulting slurry was continuously stirred for 5 h, filtered, and washed with deionized water. The wet powder was dried in the oven at 120 °C for 2 h to 4 h and finally the ZnO was obtained.

**2.2.2. WET IMPREGNATION METHOD OF Ce-$TiO_2$:** Ce-$TiO_2$ was synthesized by incipient wet impregnation method using the heating magnetic stirrer. 300 mL of $CeNO_3$ (0.01N) was taken in a round bottom flask and 3 g of $TiO_2$ was added to it, and continuously stirred for 3 h at 85 °C. It was then filtered and dried in the oven at 120 °C for 6 h. The dried catalyst was used for further characterization.

**2.2.3. SYNTHESIS OF SPIONs:** The synthesis of magnetic nanoparticles has been carried out using a controlled co-precipitation method. The $Fe^{2+}$ and $Fe^{3+}$ used were in the mole ratio 3:2. 25 mL of ferric solution was added drop wise to the 250 mL alkali solution under vigorous mechanical stirring for 30 min at room temperature. The colloidal solution containing nanoparticles was then separated and used for further characterization.

$$2Fe^{3+} + Fe^{2+} + 8OH^- \rightarrow Fe_3O_4$$

**2.3. EXPERIMENTAL PROCEDURE FOR PHOTODEGRADATIO OF CONGO RED DYE:** 100 mL of 1 x $10^{-4}$ M solution of Congo red dye was taken with 0.5 g of the prepared catalyst. The degradation reaction was carried out under solar light with the dye solution containing the catalyst being continuously stirred using a magnetic stirrer. 5 mL of the sample was withdrawn at required time intervals for analysis.
The experiment was carried out in sunlight between 9 am to 3 pm in the months of june, july and august. The digital lux meter LX-101 was used to measure the intensity of the sunlight. During this period, the average intensity of sunlight was 1.2371 x $10^5$ lux unit.

**2.4. CHARACTERIZATION:** X-ray diffraction study of the catalysts was done by Philips X'Pert model no. PW 3040/60, using Cu Kα radiation (λ = 1.5060 Å). FT-IR study was done by using Nicolet (Avatar 360) instrument using the KBr pellet technique by making 50 scans at 2 $cm^{-1}$ resolution. Diffuse reflectance UV–visible spectra of the catalysts were carried out using DRS spectrophotometer Jasco V 650 model with integrating sphere. $BaSO_4$ was used

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as a standard. The spectra were recorded at room temperature in the spectral range of 200 nm to 800 nm. The morphology of the catalyst was studied with scanning electron microscopy using a Hitachi S-4200 electron microscope. Transmission electron microscopy measurements were performed on JEM-2100 electron microscope, operated at an accelerating voltage of 80 KV. The photocatalytic degradation of Congo red dye was monitored by using a double beam spectrometer-2203, Systronics.

## III. RESULTS AND DISCUSSION

### 3.1. X-RAY DIFFRACTION ANALYSIS:

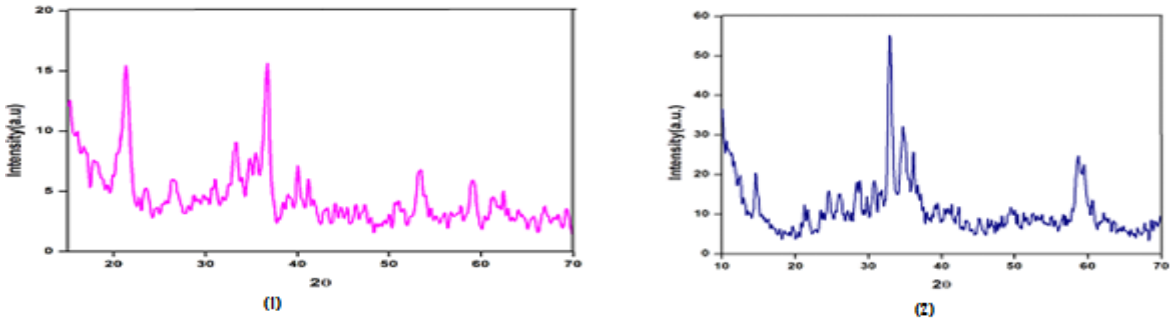


Fig.1. XRD of ZnO and Fig.2. XRD of SPIONs

The XRD pattern of the synthesized nano ZnO showed major reflections between 30° and 40° (2 θ values) indicating more crystalline regions in the zinc oxide sample. It is polycrystalline and fits well with the hexagonal (wurtzite) crystal structure with orientation along (002) and (101) reflections. The reason for relatively lower peak intensities is the formation of mixed amorphous and nano crystalline phases. The data is analyzed by using standard diffraction data from Joint Committee for Powder Diffraction Standards (JCPDS) card no.05-0664. Some weak reflections such as (1 0 2), (1 0 3) and (2 0 1) was observed with small intensities (Fig. 1).

The structure and crystallinity of the synthesized SPIONs were analyzed by XRD. The pattern fits well with magnetite $Fe_3O_4$ JCPDS file, No. 00-011-0614. The catalyst exhibited a strong peak in the 2 θ range of 33˚ [19]. The sharp peaks represent the crystallinity of SPIONs. The pattern clearly indicates the synthesized particles have a spinal cubic structure of magnetite nanoparticles (Fig. 2).

**3.2. FT-IR ANALYSIS:** A surface hydroxyl group plays an important role in the photodegradation method through their interaction with photogenerated holes.

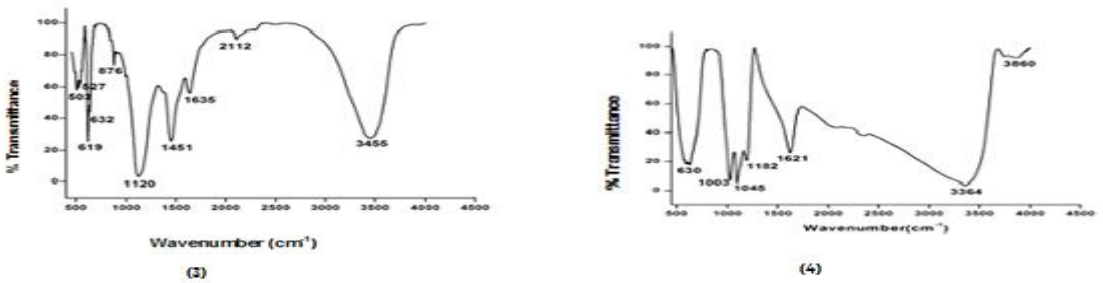


Fig.3. FT-IR of ZnO and Fig.4. FT-IR of SPIONs

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FT-IR transmittance spectra of the samples were recorded in order to obtain the structural information of the catalyst surface (Fig. 3 and Fig. 4). The peak at 619 $cm^{-1}$ was related to the stretching vibrations of the Zn-O bonds [20]. The peak at 1120 $cm^{-1}$ and 3455 $cm^{-1}$ indicates the H-O-H bending vibration and the presence of - OH group (Fig. 3). The strong absorption band at 630 $cm^{-1}$ is due to the stretching vibration of iron oxide nano particles in SPIONs (Fig. 4).

### 3.3. DIFFUSE REFLECTANCE UV-VISIBLE SPECTROSCOPY:

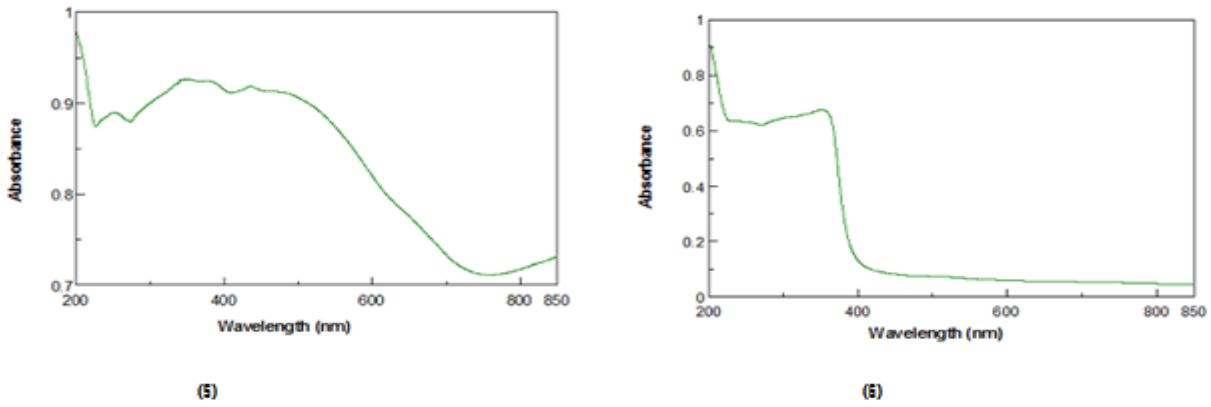


Fig.5. DRS-UV if SPIONs and Fig.6. DRS-UV of ZnO

Diffuse Reflectance UV-Visible Spectroscopy (DRS-UV) spectrum of SPIONs and ZnO were given in Fig.5 and Fig.6. This characterization technique showed the absorption of the synthesized ZnO nanoparticles was in the range of 300 nm to 400 nm and the absorption for synthesized SPIONs was mostly in the visible region. The SPIONs showed the strong absorption throughout 400 nm to 700 nm. The pattern of the synthesized SPIONs confirmed it to be magnetite material [21].

### 3.4. SEM ANALYSIS:

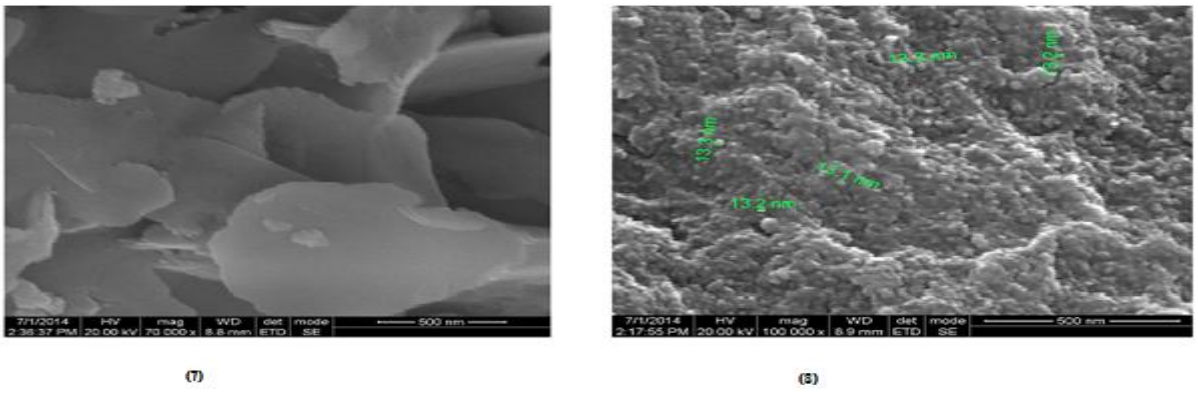


Fig.7. SEM of ZnO and Fig.8. SEM of SPIONs

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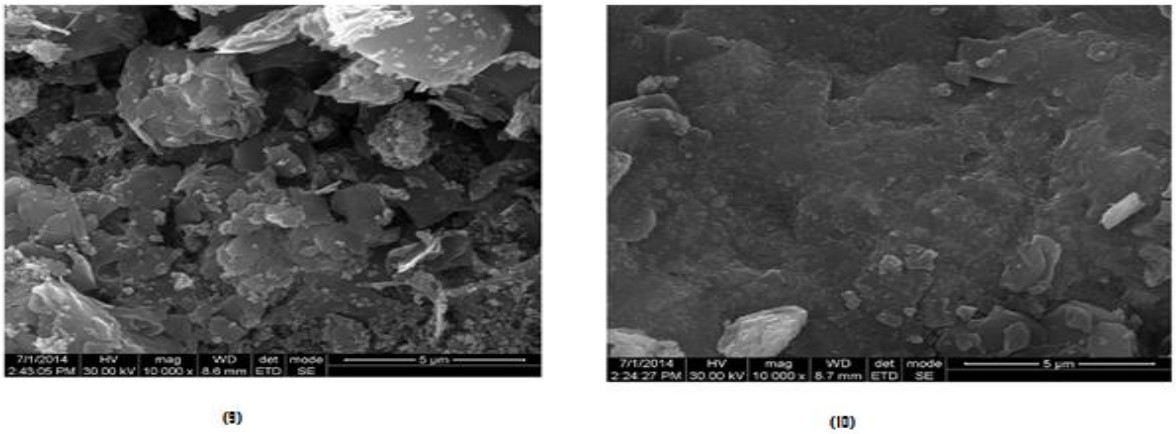
The surface morphology of the ZnO and SPIONs were obtained from SEM and shown in the photograph (Fig.7 and Fig.8). The particle size of the synthesized SPIONs catalyst was found to be in nano range. SEM image of ZnO showed various sizes of sheet like particles [22].

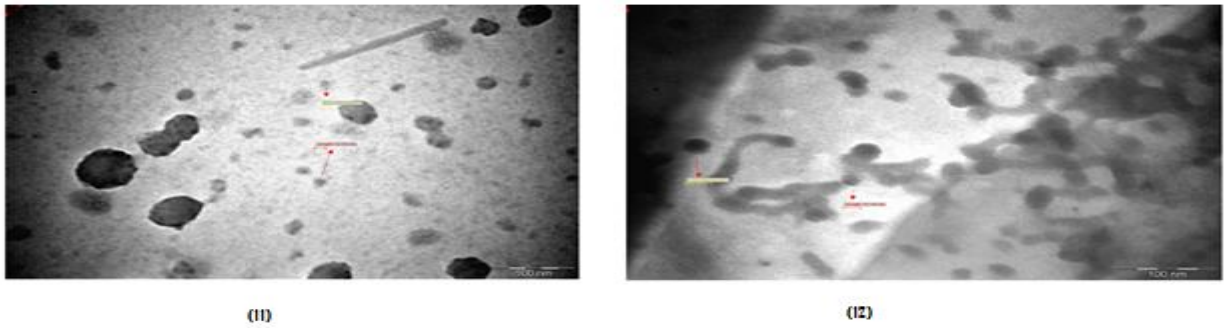


(9) (10)

Fig.9. SEM of ZnO and Fig.10. SEM of SPIONs

The SEM of SPIONs (Fig 9 and Fig 10) clearly displayed the presence of spherical shaped iron oxide nanoparticles with narrow particle size distribution. The particle size of SPIONs was observed to be in the range of 13.1 nm to 13.9 nm.

**3.5. TEM ANALYSIS:**



(11) (12)

Fig.11. TEM micrograph of ZnO and Fig.12. TEM micrograph of SPIONs

The morphologies of the ZnO and SPIONs shown by TEM photograph (Fig. 11 and Fig. 12) were consistent with SEM. The particle size of the SPIONs was found to be in nano range. The TEM of SPIONs (Fig. 12) clearly showed the presence of spherical shaped iron oxide nanoparticles with narrow particle size distribution. [23, 24]

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### 3.6. PHOTOCATALYTIC ACTIVITY OF VARIOUS CATALYSTS:

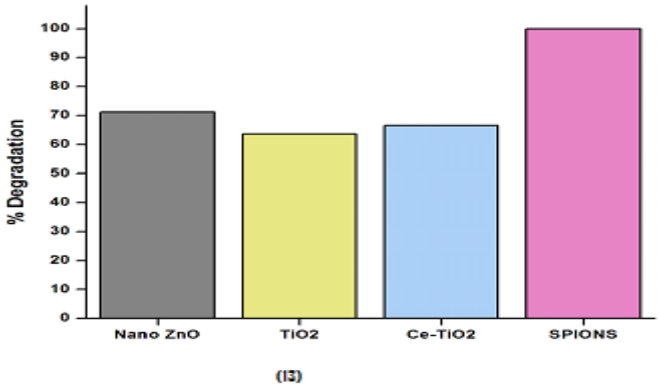


Fig.13. Comparison of photo degradation of Congo red dye with different photocatalysts
[Dye] = 1 x $10^{-4}$ M, Time = 2 h, under solar light.

The photocatalytic activity of the synthesized catalysts iron oxide nanoparticles, ZnO, and Ce-$TiO_2$ were studied by degrading Congo red dye. In 100 mL of $10^{-4}$ M concentrated dye, 0.5 g of the catalyst was added, degradation was carried out as mentioned above and the results were shown in Fig. 13.

SPIONs have shown the maximum activity exhibiting 100% dye degradation. Under investigation, its activity was noticeably higher than the other catalysts. It was recorded previously that the effective surface area and the particle size of the semiconductor played a major role in shifting the absorption in visible region [25]. It is highly evident from the present study that lower the particle size of the synthesized catalyst, higher the efficiency.

The particle size and the total effective surface area of the semiconductor play a major role in percentage degradation. In accordance [26] to previous study, $Fe^{3+}$ present in SPIONs suppress the undesired recombination of the photogenerated electrons and holes, accelerating the desired degradation. Therefore, high activity SPIONs catalysts were observed. The percentage degradation slightly increased when $TiO_2$ is doped with $Ce^{2+}$ which gives Ce-$TiO_2$, an n-type semiconductor that increased the particle size and also had a corresponding influence in reducing the band gap having an extended photo response of the formal to the visible region enhancing its activity.

### 3.7. TIME ON STREAM:

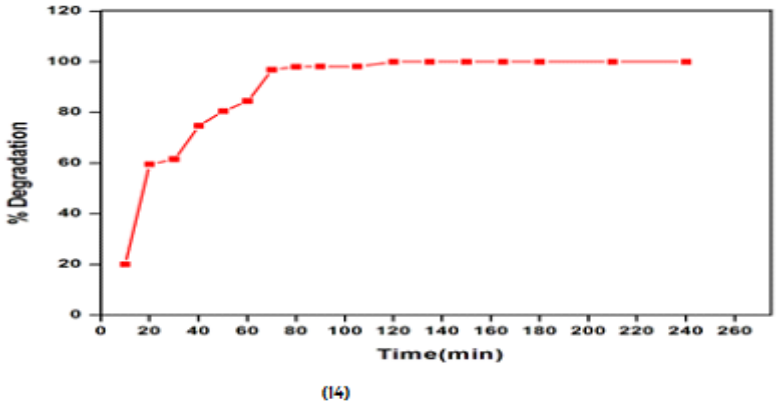


Fig.14. Effect of % degradation of Congo red dye in presence of SPIONs

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[Dye] = 1 x $10^{-4}$ M, Time = 2 h, under solar light.

The catalytic activity of the iron oxide nano particles were studied for 4 h. The analysis showed 100% degradation in 120 min. The gradual increase in the percentage of degradation with time is due to the gradual increase in the active site on the catalysts with time as shown in Fig 14.

$Fe_2O_3$ + photon → $Fe_2O_3^*(e_{CB}^- + h_{VB}^+)$ (Formation of electron-hole pair)

$h_{VB}^+$ + dye → Direct oxidated dye (Formation of hydroxyl radical)

$h_{VB}^+ + H_2O \rightarrow H^+ + {}^{\cdot}OH$

$e_{CB}^- + O_2 \rightarrow O^-_2$

$O^-_2$ + dye → dye-$OO^{\cdot -}$

The above scheme shows the formation of radicals by scavenging off their electron hole pair using molecular oxygen and water. It shows the formation of electron hole pair and the formation of directed oxidized dye and hydroxyl formation. Electron in the conduction band is also responsible for the mineralization of the dye.

### 3.8. EFFECT OF INHIBITORS: SODIUM CARBONATE ($Na_2CO_3$):

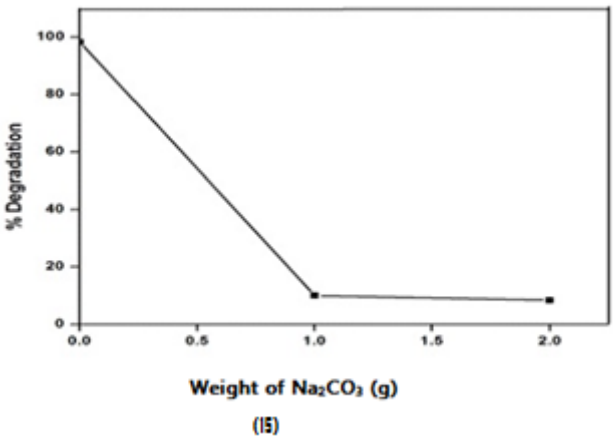


Fig.15. Effect of Sodium Carbonate on the degradation of Congo red dye
[Dye] = 1×$10^{-4}$ M, SPIONs = 5 $gL^{-1}$ at neutral pH.

Sodium carbonate is widely used in printing and dyeing industries as it adjusts the pH of the bath which makes the dye adhere to the fabric and the fast fixing of colours. This influences the degradation of the dye in optimum condition. The dye of 1×$10^{-4}$ M concentration was added with 0.5 $gL^{-1}$ of the photocatalyst SPIONs under sunlight along with different quantities of sodium carbonate in the range of 0-2 $gL^{-1}$ and the results obtained are as shown in the Fig.15.

The decrease in the percentage is due to the hydroxyl radical inhibiting property of sodium carbonate as shown in the following equations

$OH^{\cdot} + CO_3^{2-} \rightarrow OH^- + CO_3^{\cdot -}$

$OH^{\cdot} + HCO_3^- \rightarrow H_2O + CO_3^{\cdot -}$

In the presence of sodium carbonate, the degradation of the dye was reduced by 10% due to the carbonate ion adsorption on the surface of the catalyst. The dye adsorption on the surface of the catalyst can be prevented by the neutralization of $Na^+$ ions which was also proved by the pH studies.

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**3.9. EFFECT OF ELECTRON ACCEPTORS: HYDROGEN PEROXIDE ($H_2O_2$):** The effect of hydrogen peroxide was studied because it is widely used as a bleaching agent. The hydrogen peroxide is added in the concentration of 0-20 $mmolL^{-1}$ to the dye. One strategy to inhibit electron/hole pair recombination is to add other electron acceptors to the reaction. They have several different effects to increase the number of trapped electrons and consequently avoid recombination to generate more radicals and other oxidizing species to increase the oxidation rate of intermediate compounds and to avoid problems caused by low oxygen concentration.

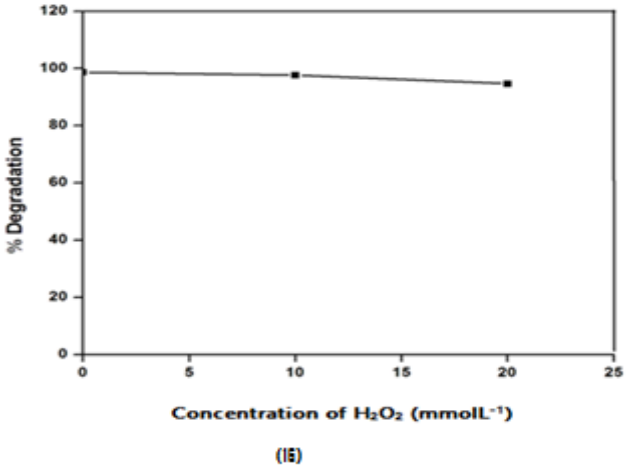


Fig.16. Effect of $H_2O_2$ on the photodegradation of Congo red dye
[Dye] = $1\times10^{-4}$ M, SPIONs = 5 $gL^{-1}$ at neutral pH.

$H_2O_2$ can also become a scavenger of valence band holes and ·OH radicals when present in high concentrations decreasing the dye degradation. Therefore, it shows slight decrease in the degradation of dye. (Fig. 16)

**3.10. EFFECT OF pH:**

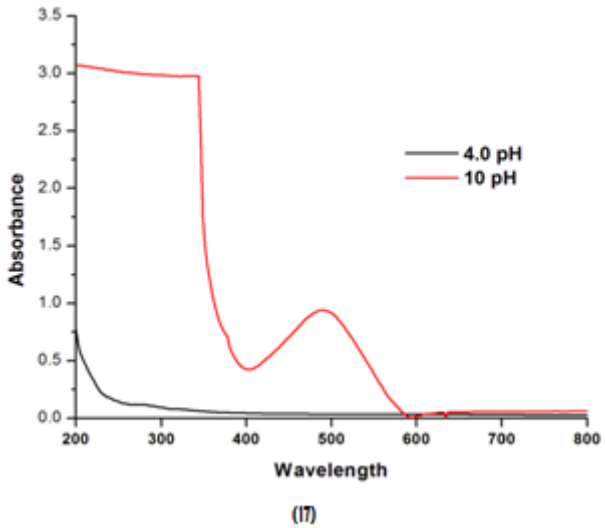


Fig.17. Effect of pH on the degradation of Congo red dye dye

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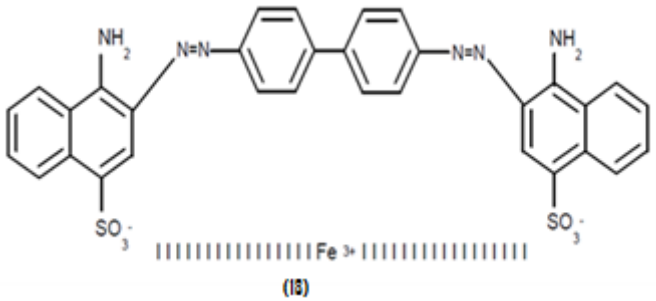
[Dye] = $1\times10^{-4}$ M, SPIONs = 5 $gL^{-1}$, Time = 2 h, under sun light.

The Fig. 17 shows the pH study of the Congo red dye. The acid solution pH = 4.0 and base solution pH = 10.0 of the dye were prepared by adding HCl and NaOH respectively. The degradation at low pH was due to the adsorption of dye on the surface of the catalyst. In basic solutions, when the pH is 10.0, the adsorption decreased due to the influence of the $Na^+$ ions present in the dye solution which reduces the adsorption of the dye on the surface of the catalyst.

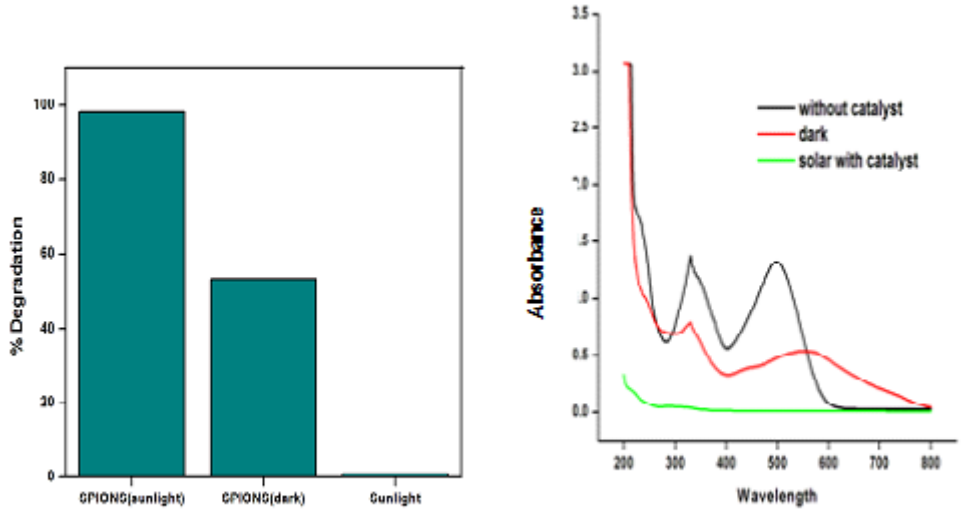
### 3.11. PHOTO DECOLOURISATION OF CONGO RED DYE WITH SPIONs WITH DIFFERENCT ENVIRONMENTS:




(18)

Fig. 18. Adsorption of dye on $Fe_3O_4$.

Photo decolourisation of the dye in the presence and absence of the catalyst were studied. There was no observable colour loss in the absence of the catalyst and sunlight. In dark, there was a slight degradation in the dye solution with the presence of the catalyst which was due to the adsorption of Congo red on the surface of the iron oxide nano particles through the two oxygen atoms in the sulphonate group present in the dye (Fig. 18)




(19)

Fig.19. Degradation of Congo Red dye with various environments.

In the presence of sunlight, the degradation of Congo red dye was significantly higher when compared to the other two factors. This clearly explains that the role of SPIONs with sunlight produces Photo-Kolbe like reaction which facilitates

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the mineralisation of dye to 100%. It concludes that the reaction is photocatalytic and the comparison is as shown in Fig. 19.

### 3.12. RECYCLABILITY OF THE CATALYST:

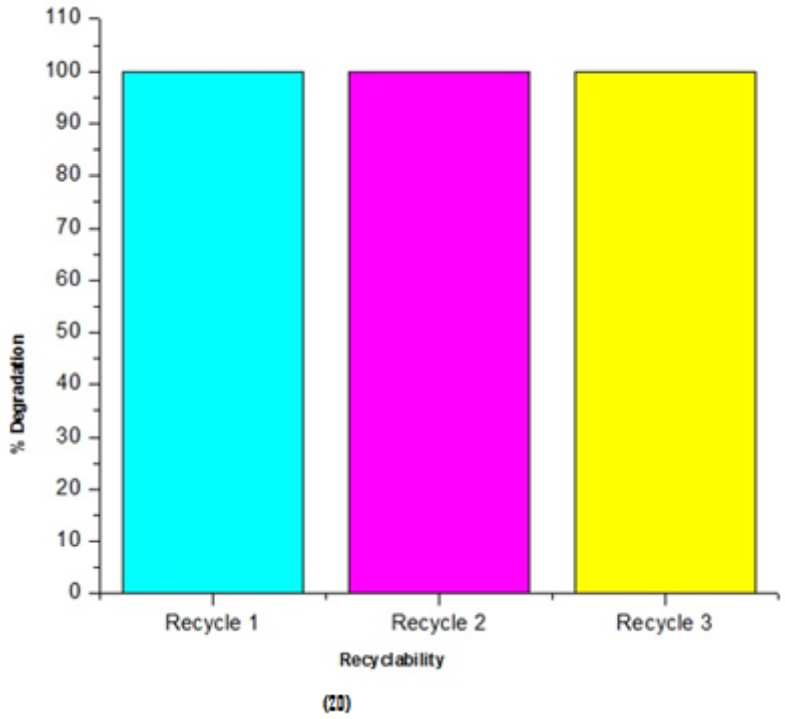


Fig.20. Effect of catalyst recycling on the degradation of Congo red dye
[Dye] = $1\times10^{-4}$ M, SPIONs = 5 $gL^{-1}$, Time = 2 h, under sun light.

Photocatalysis is a clean technique which is very useful and is easy to dispose hazardous materials from the effluents. The photodegradation efficiency of SPIONs was tested by using 0.5 g of the catalyst in 100 mL of $1x10^{-4}$ M Congo red dye under solar irradiation with constant stirring for 120 min. The Congo red dye was completely degraded and the catalyst was filtered and reused with another 100 ml of $1x10^{-4}$ M Congo red dye following the same procedure. There was complete degradation of azo dye in 120 min. Even after the catalyst was filtered and reused for the third time, there was still complete degradation of Congo red dye as shown in the Fig. 20. Hence, SPIONs are found to be recyclable three times allowing complete dye degradation.

## IV. CONCLUSION

ZnO, Cerium incorporated $TiO_2$ and SPIONs were successfully synthesized. The synthesized catalysts were characterized by XRD, FT-IR, DRS-UV, TEM and SEM techniques. The photocatalytic activity of these synthesized compounds and $TiO_2$ have been tested for the degradation of Congo red dye under solar light irradiation. Among the catalysts tested, SPIONs was found to be the most active. The optimum condition for 100 % degradation of Congo red dye was found to be 100 mL of $1\times10^{-4}$ M of dye solution along with 0.5 g of SPIONs under solar light irradiation for 120 min. There was a decrease in degradation of the Congo red dye with increase in concentration of inhibitors like sodium carbonate. Electron acceptors like hydrogen peroxide slightly decrease the percentage degradation of the Congo

ISSN (Online) : 2319 - 8753
ISSN (Print) : 2347 - 6710

International Journal of Innovative Research in Science, Engineering and Technology

*An ISO 3297: 2007 Certified Organization* *Volume 4, Special Issue 1, February 2015*

**THIRD NATIONAL CONFERENCE ON ADVANCES IN CHEMISTRY (NCAC – 2015)**

**On 18th February 2015**

**Organized by**

**Department of Chemistry, Easwari Engineering College (SRM Group of Institutions), Chennai-600089, India.**

red dye. The optimum degradation was not attained in the basic condition of pH but in acidic and neutral condition the degradation was significantly high. The active catalyst SPIONs, were found to be efficiently recyclable several times.

## ACKNOWLEDGEMENT

The authors acknowledge DST-SERB India for supporting the above work.

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