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## Expedient Degradation of Dye Methyl Green by Enhanced Photo – Fenton Process : A Green Chemical Approach

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### ABSTARCT

*Photo-Fenton processes are one of the ecologically viable techniques for degradation of textile dyes and industrial wastewater treatment. Relatively simple approach, use of simple chemicals for the degradation, cyclic nature and high reaction yields make this process a green chemical pathway in reducing pollution caused by dyes and other organic compounds. In the present study, the photochemical degradation of dye methyl green by photo-Fenton reaction in presence of catechol as an additive has been investigated. The kinetics of the reaction has been followed spectrophotometrically. The effect of variation of various parameters such as pH, concentration of  $Fe^{3+}$  ion, dye and additive; amount of  $H_2O_2$  and light intensity on the rate of photo degradation has also been analysed.*

**Key words:** Photo-Fenton, AOP's, Wastewater treatment, Methyl green, Catechol.

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### INTRODUCTION

A major onslaught of industrial revolution is being experienced by the entire world. Increasing complexity and the amount of dye wastewater discharged over the years by printing, dyeing and textile industries into the receiving media is causing grave environmental concerns by endangering aquatic – ecosystem. Apart from aesthetic pollution, these compounds lead to high Oxygen Demand (COD) and Biological Oxygen Demand (BOD) and absorb sunlight strongly which adversely affect the photosynthetic activity of aquatic plants thereby causing a grave threat to the whole ecosystem.[1].Moreover, these effluents are toxic, mutagenic and carcinogenic in nature [2]. Various chemical and physical processes, have been employed for the decolorization and degradation of textile effluents but lead to non-biodegradable contaminations which give rise to new types of pollutants and hence require further treatment involving expensive procedure. Conventional techniques such as coagulation/flocculation, membrane separation, absorption by activated carbon are based on phase transfer of the pollutants. These methods transfer the

contaminant from waste water to solid wastes and they are not efficient for removal of dyes. Biological methods are also ineffective for decolourization of azo dyes because of its stability and complex aromatic structure. To resolve such types of problems Advanced oxidation processes are proved to be most promising technique. Advanced Oxidation processes (AOPs), based on the generation of highly reactive hydroxyl radicals ( $\cdot\text{OH}$ ), are the most cost effective chemical methods which are extensively used for treatment of textile effluents and variety of organic pollutants. [3-9]. Faster degradation of dye is achieved due to the high oxidative power of ( $\cdot\text{OH}$ ) radicals (2.83V v/s NHE) and can be further improved by using UV- visible radiations that generate additional hydroxyl radicals.

Fenton and photo-Fenton treatments, involving generation of hydroxyl radicals by  $\text{Fe}^{2+}/\text{Fe}^{3+}$  and  $\text{H}_2\text{O}_2$  have been used for degradation of recalcitrant pollutants [10-14 ] and dyes in wastewater discharge of textile, printing and other industries [15,16]. Heterogeneous Fenton and photo-Fenton reactions have also been employed for the degradation of dyes [17-19 ].

The effect of various reaction parameters, metal ions, organic additive and anions on the photo-fenton process, for degradation of dyes has been studied [20-23]. Kinetic parameters related to the decolourization and mineralization of reactive dyes using Fenton and photo-Fenton processes have also been investigated [24-26]. The effect of quinones on free radical processes of oxidation and fragmentation of hydroxyl containing organic compounds has been studied by Shadyro *et al.*[27].

According to the present studies, a negligible degradation of dye methyl green is possible by photo-Fenton reaction. A very fast and almost complete decolorization of dye can be achieved by addition of catechol as an additive. The present work investigates the effect of an organic additive (catechol) on the rate of photo-Fenton degradation of methyl green with more emphasis on determining the conditions under which this additive showed the maximum rate for the photo degradation reaction. An attempt has also been made to explain their behaviour as a catalyst.

## MATERIALS AND METHODS

Stock solutions of  $1 \times 10^{-3}$  M concentration of dye (methyl green, E.Merck, A.R),  $\text{FeCl}_3$  (LOBA, A.R), and catechol (E.Merck, A.R) were prepared in doubly distilled water. Hydrogen peroxide (30% w/v, E.Merck A.R) was used. The reagents and chemicals were used directly as received commercially without any further purification. For the typical run, 30  $\text{cm}^3$  reaction mixture was prepared by adding all reagents one after the other. A concentration of  $4.00 \times 10^{-5}$  M for dye,  $3.33 \times 10^{-5}$  M for ferric chloride and  $4.66 \times 10^{-5}$  M for catechol was attained in the reaction mixture and 0.08  $\text{cm}^3$  of  $\text{H}_2\text{O}_2$  was added to the solution. The pH of the above solution was set 2.75 and kept under the light of 9000 lux intensity. These specific concentrations were chosen on the basis of optimum concentration value of each reagent. The optimal value for one reagent, once established, was kept constant and other parameters were changed one after the other to get their optimum concentration.

A 200 W tungsten lamp was used for irradiating the solution thereby emitting radiations in the visible range of electromagnetic spectrum. Lux meter (LT Lutron electronics, model no: LX-101A) was used to measure the intensity of light. A petridish filled with water was used as water filter to cut off thermal radiations. A digital pH meter (Equiptronics, Model: EQ611) was used to measure the pH of the system. 0.1 M sodium hydroxide and 0.1 M sulphuric acid (both previously standardised) were used to adjust the pH of the solution to the required value. All the solutions were kept at room temperature. Progress of the photochemical reactions was observed by measuring absorbance at regular intervals of 10 minutes with the help of UV-Visible spectrophotometer (Chemiline, Model CL 340).

## RESULTS AND DISCUSSIONS

The absorbance or O.D. of the dye in the reaction mixture was measured at  $\lambda_{\text{max}} = 660 \text{ nm}$  every 10 minutes using  $3.0 \text{ cm}^3$  of the aliquot. With increasing time of exposure, the absorbance or O.D. of the solution decreased. This indicated a decrease in concentration and hence degradation of the dye. The results presented graphically in Fig 1 A show a linear variation of  $1 + \log_{10} [\text{O.D.}]$  versus time and the reaction was found to follow pseudo-first order kinetics. The value of rate constant was calculated using expression:

$$k = 2.303 \times \text{Slope}$$

It has been observed that the reaction undergoes completion in two stages. The first stage possibly involving the generation of the  $\bullet\text{OH}$  radicals can be identified as induction period; which shows a slight decrease in O.D, probably due to simple photochemical degradation of dye. However at a later stage, after the initial slow stage, a significant decrease in O.D. has been observed. This second stage is photo-Fenton degradation, where  $\bullet\text{OH}$  radicals act as oxidizing agents for the degradation of dye. Effect of variation of various parameters on the rate of degradation was studied by comparing the rates of the second stage of the photo-Fenton reaction.

It was also observed that the first stage (slow step or induction period) gets shorter time span as experimental conditions approached the optimum values. A comparative study of **Fig. A** and **B** indicates faster degradation of dye in presence of additive and light than in dark. A very negligible degradation of dye was observed in the absence of organic additive (catechol) by photo-Fenton process. The observations indicated quite well that the organic additive accelerates reaction rate either by generating  $\bullet\text{OH}$  radicals rapidly or by making available larger of  $\bullet\text{OH}$  radicals. Under optimal conditions of dye,  $\text{FeCl}_3$ ,  $\text{H}_2\text{O}_2$  or additives, the rate of degradation became very fast and induction period diminished to zero.

### Effect of pH

Keeping all the reagents at constant concentrations, the effect of pH on additive catalyzed photochemical degradation was analyzed by varying pH in the range of 2.50 – 4.25. The result of **Fig. C** shows an increase in rates of degradation of dye methyl green with an increase in pH up to 2.75 after which the rate was found to decrease; At higher pH, ferric ions decompose hydrogen peroxide into water and oxygen, instead of generating hydroxyl radicals, the species responsible for the photochemical degradation of dye.

### Effect of Amount of Hydrogen Peroxide

Maintaining all the other parameters constant, the amount of  $\text{H}_2\text{O}_2$  added was varied from 0.04 to  $0.32 \text{ cm}^3$  and its effect on the rate of photochemical degradation was investigated. The result as reported in **Fig. D** shows an increase in the rate of photo - decolourisation of dye with increase in the amount of  $\text{H}_2\text{O}_2$  up to  $0.08 \text{ cm}^3$  only, beyond which a decrease in degradation of dye is observed. This can be attributed to the presence of perhydroxyl radicals ( $\text{HO}_2\bullet$ ) produced as a result of  $\text{H}_2\text{O}_2$  scavenging  $\bullet\text{OH}$  radicals at higher concentrations of  $\text{H}_2\text{O}_2$  and having lower oxidation potential than hydroxyl radicals.

### Effect of Ferric Ion Concentration

The effect of ferric chloride [ $\text{Fe}^{+3}$  ions] concentration on the rate of photochemical degradation was studied by maintaining all other parameters constant. The result shown in **Fig.E**. shows an increase in the rate of photo-Fenton degradation with increasing concentration of ferric ions up to  $3.33 \times 10^{-5} \text{ M}$ . Upto this particular  $\text{Fe}^{+3}$  ion concentration, an increase in the number of  $\text{Fe}^{3+}$  ions enhances

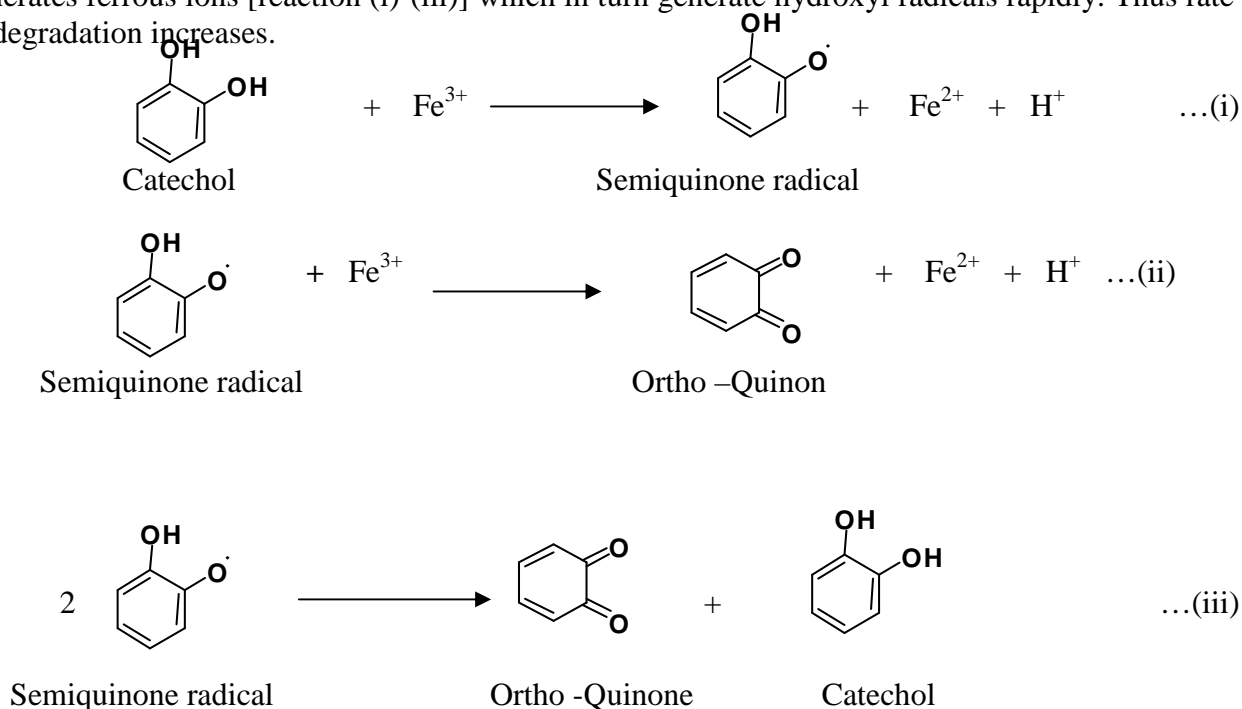
the generation of  $\bullet\text{OH}$  radicals thereby increasing the rate of photochemical degradation. Beyond this concentration rate of reaction is very fast and due to the experimental limitation, it was not possible to record the observations correctly.

### Effect of Dye Concentration

At different concentrations of dye (methyl green) ranging from  $3.33 \times 10^{-5}$  M to  $8.00 \times 10^{-5}$  M and keeping all other factors identical, the effect of the dye concentration on the degradation of methyl green was studied. The result as reported in **Fig. F** indicates an initial increase in rate of photobleaching of dye due to availability of more dye molecules for degradation. A further increase in the dye concentration results in retardation of reaction due to increase in number of collisions between dye molecules themselves while decreasing the probability of collisions between dye and  $\bullet\text{OH}$  radicals. In addition, at higher concentrations the dye molecules themselves may act as quenchers for the photons of the incident light, and hence prevent sufficient light intensity to reach the bulk of the solution. Hence at higher dye concentrations, a decrease in the rate of photobleaching of dye is observed.

### Effect of Additive

The analysis of effect of variation of additive concentration (catechol) on the photochemical degradation of methyl green as reported in **Fig. G** shows that rate of reaction increases with increase in concentration of additive up to  $4.66 \times 10^{-5}$  M. Here catechol acts as a catalyst and generates ferrous ions [reaction (i)-(iii)] which in turn generate hydroxyl radicals rapidly. Thus rate of degradation increases.



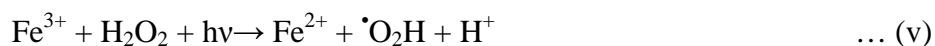
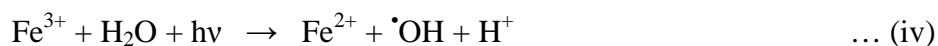
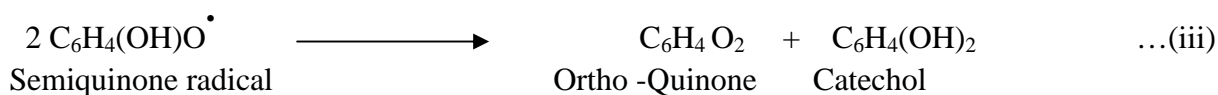
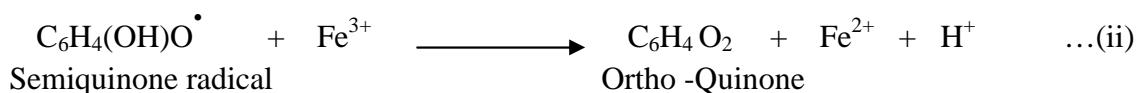
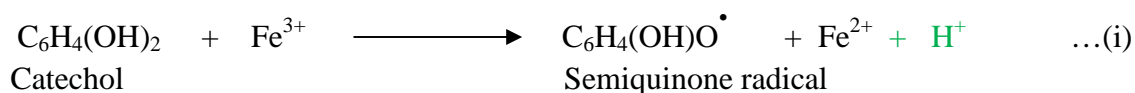
### Effect of Light Intensity

Effect of varying intensity of light on the photo-Fenton degradation of methyl green was studied. The results are reported in **Fig. H** and the rate of degradation of dye shows a linear relationship with intensity of light such that there is an increase in reaction rate with increase in the intensity of light. This can be explained through an increase in the number of photons of light striking the solution

with increasing light intensity, thereby generating more hydroxyl radicals which are responsible for the photochemical degradation of dye.

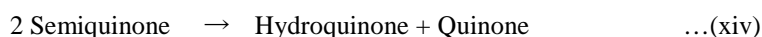
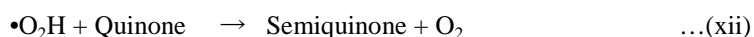
## MECHANISM

On the basis of this multidimensional study and extensive literature survey [23,28 & 29], a tentative mechanism has been proposed for photo-Fenton degradation of methyl green in presence of organic additive.



On exposure to light, the aqueous solution of ferric ions generate protons,  $\bullet\text{OH}$  radicals and ferrous ions (iv). Ferrous ions are also generated by the reactions of ferric ions with hydrogen peroxide as well as with catechol in presence of light (v and i). These ferrous ions react with  $\text{H}_2\text{O}_2$  and produce hydroxyl ions and hydroxyl radicals and are oxidized back to ferric ions (vi). The hydroxyl radicals, thus generated are probably the most active species in the degradation of dye.

Aromatic additive plays an important role in catalyzing the Fenton reaction. Catechol accelerates the Fenton reaction by increasing the regeneration of ferrous ion ((i) and (ii)), which might be the slowest step in the mechanism of the simple Fenton reaction. Further, Catechol like compounds are continuously regenerated from quinone or semi-quinone by reaction with  $\bullet\text{O}_2\text{H}$  radicals and disproportionation reaction of semiquinone [(xii)- (xiv)]. This regeneration of hydroquinone continuously promotes the rate of the reaction.



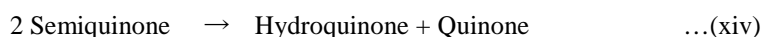
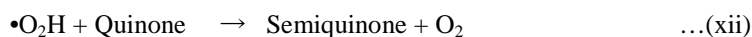
The role of  $\bullet\text{OH}$  radicals as active oxidizing species was confirmed by using isopropanol, a hydroxyl radical scavenger. The rate of photochemical degradation was drastically reduced in

presence of isopropanol.



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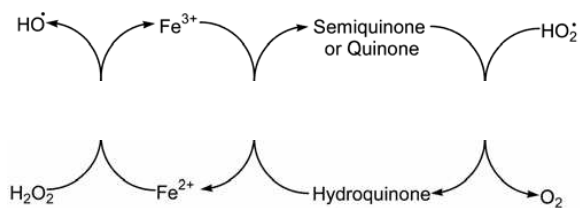
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## CONCLUSION

In order to free wastewater / industry effluent from dye molecules, photo-Fenton reactions are the highly effective, provide a dye decomposition method which is benign to the nature and are economically viable. The efficacy of this reaction can be increased further by adding an aromatic additive like catechol. In the present case, a negligible decolorization of dye was observed in the absence of additive by photo-Fenton process. Moreover, it was found that in absence of light the rate of Fenton reaction is extremely slow, whereas exposure to light accelerated the rate of degradation of dye. The comparative study reveals that the rate of degradation in different conditions shows the following order –

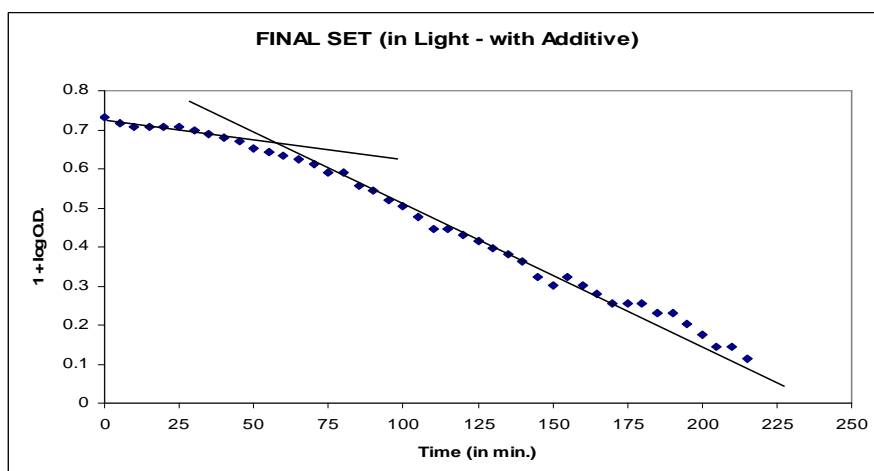
With Additive in Light > With Additive in Dark > Without Additive in Light > Without Additive in Dark

In the presence of light, the rate of regeneration of ferrous ions in the system increases through the photo-Fenton reactions (iv and v) and the process thus becomes cyclic. Aromatic compounds play an important role in catalyzing the Fenton reaction by increasing the regeneration of ferrous ions in the system. The entire process is cyclic and all chemicals used in these reactions are less hazardous to the environment thereby providing an eco- friendly or green chemical pathway for degradation of the dye. The whole process can be depicted diagrammatically as shown below:



**Fig. A :Typical run – Photo-Fenton Degradation of Methyl Green in Light**

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Amount of $\text{H}_2\text{O}_2 = 0.08 \text{ cm}^3$	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	Light Intensity = 9000 Lux



**Fig. B:Typical run – Photo-Fenton Degradation of Methyl Green in Dark**

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Amount of $\text{H}_2\text{O}_2 = 0.08 \text{ cm}^3$	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	<b>IN DARK</b>

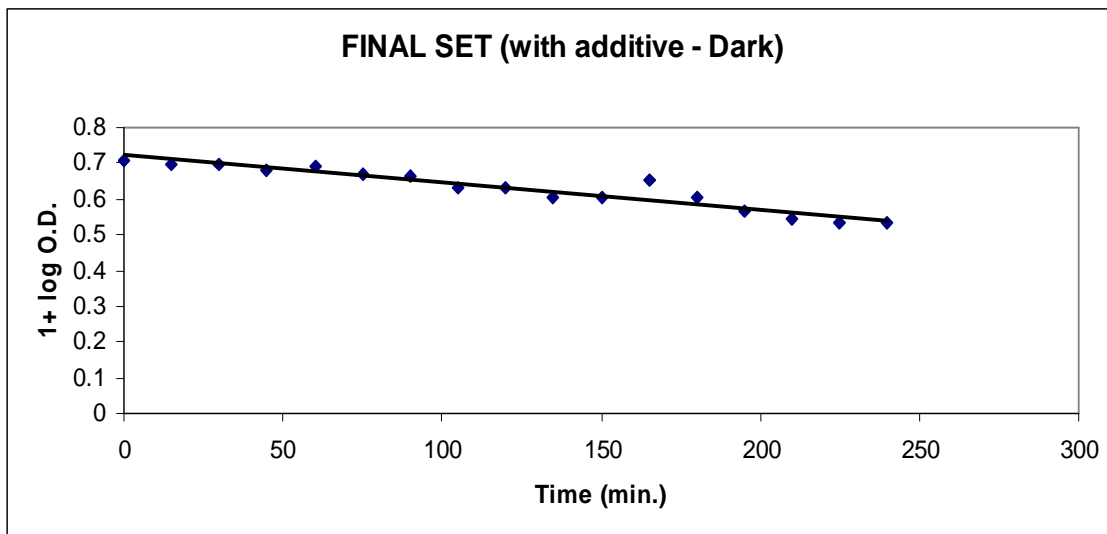


FIG - C : Effect of pH on the photo-Fenton degradation of dye

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
Amount of $\text{H}_2\text{O}_2 = 0.08 \text{ cm}^3$	Light Intensity = 9000 lux
Catechol = $[4.33 \times 10^{-5} \text{ M}]$	

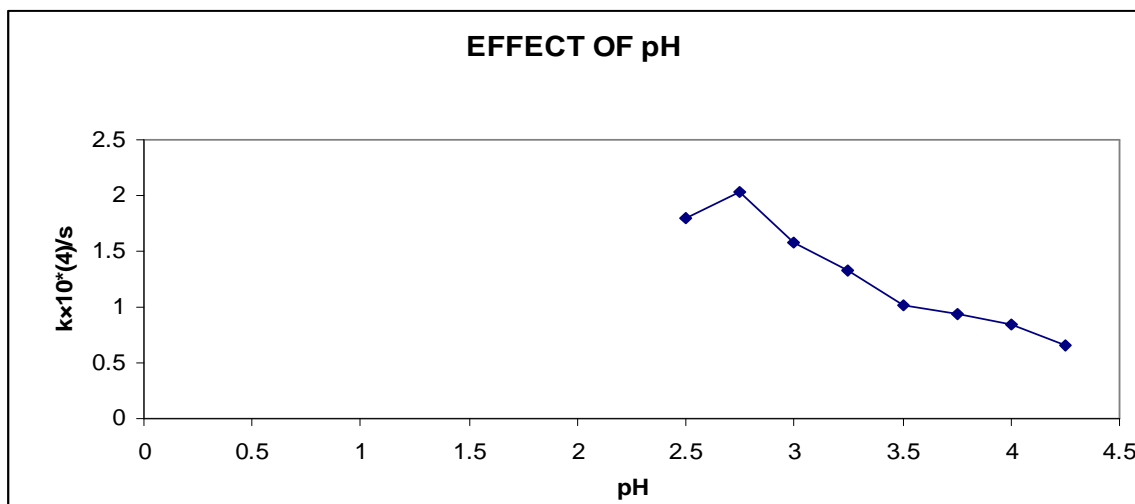
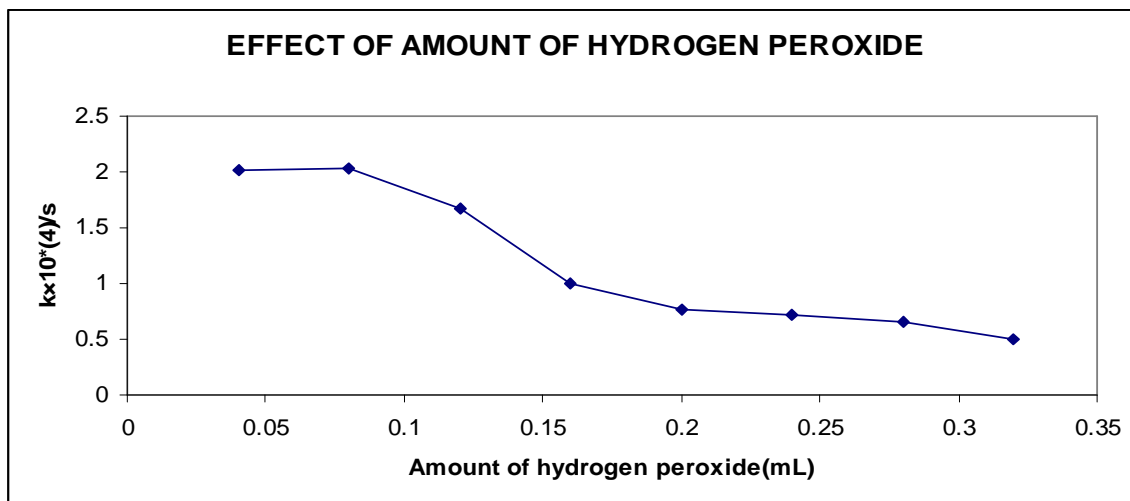


FIG - D : Effect of amount of  $\text{H}_2\text{O}_2$  on the photo-Fenton degradation of dye

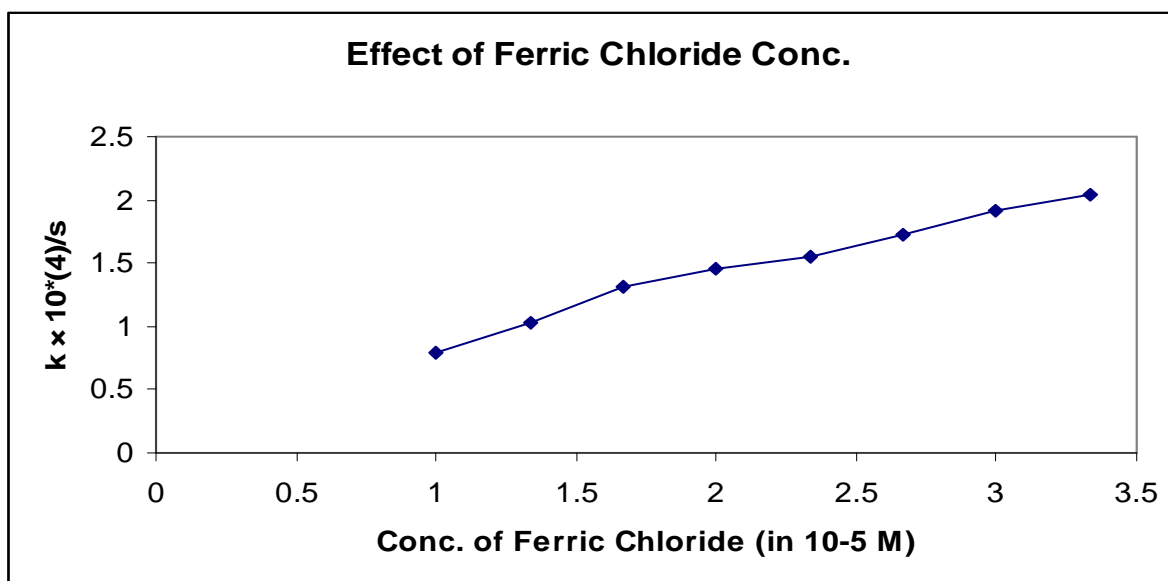
Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
	Light Intensity = 9000 lux





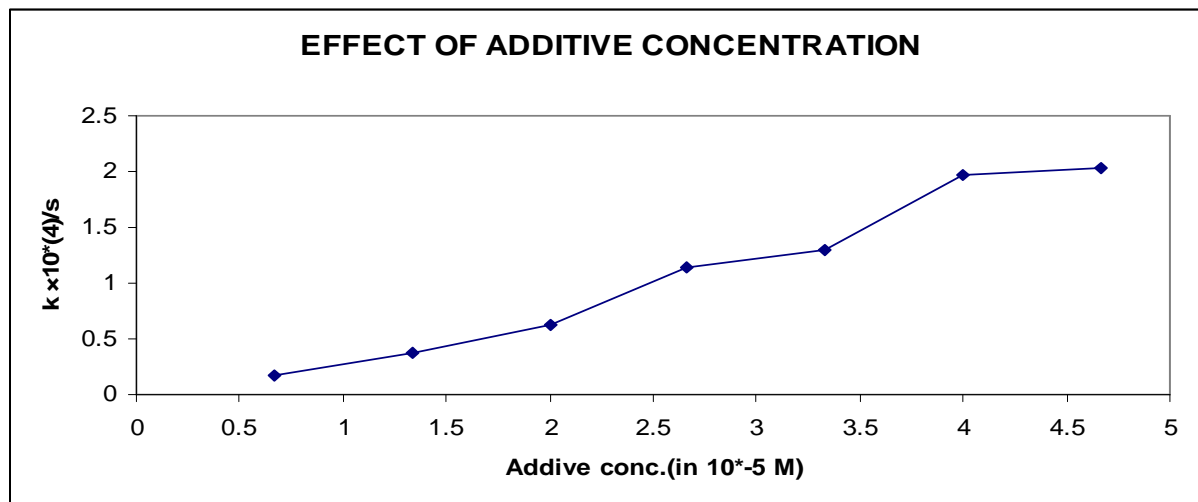
**FIG – E: Effect of FeCl<sub>3</sub> on the photo-Fenton degradation of dye**

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Amount of H <sub>2</sub> O <sub>2</sub> = 0.08 cm <sup>3</sup>	Light Intensity = 9000 lux
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	



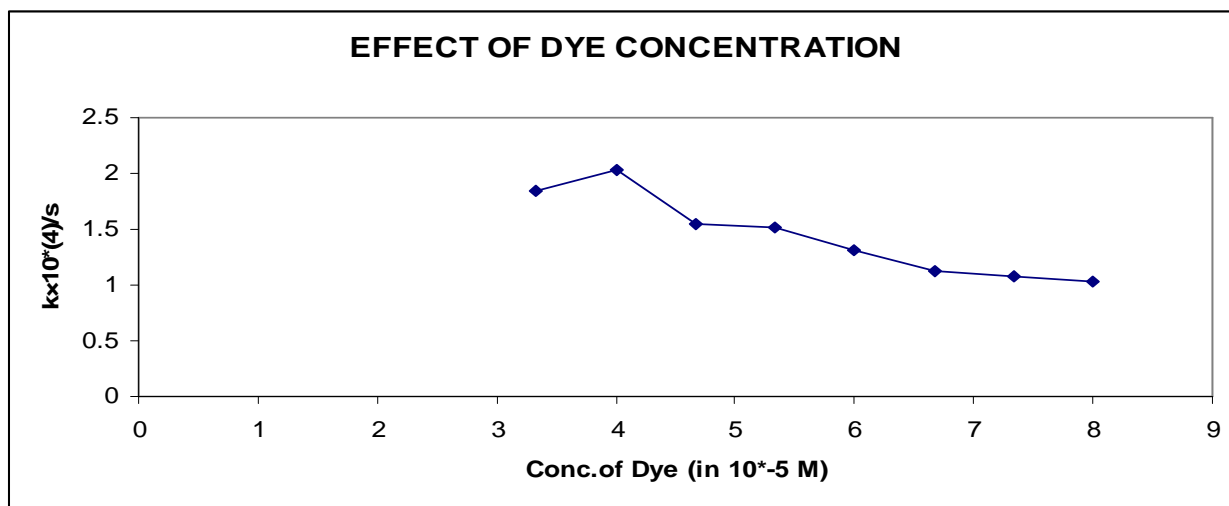
**FIG – G: Effect of additive conc. on the photo-Fenton degradation of dye**

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Amount of H <sub>2</sub> O <sub>2</sub> = 0.08 cm <sup>3</sup>	FeCl <sub>3</sub> = $[3.33 \times 10^{-5} \text{ M}]$
Light Intensity = 9000 lux	



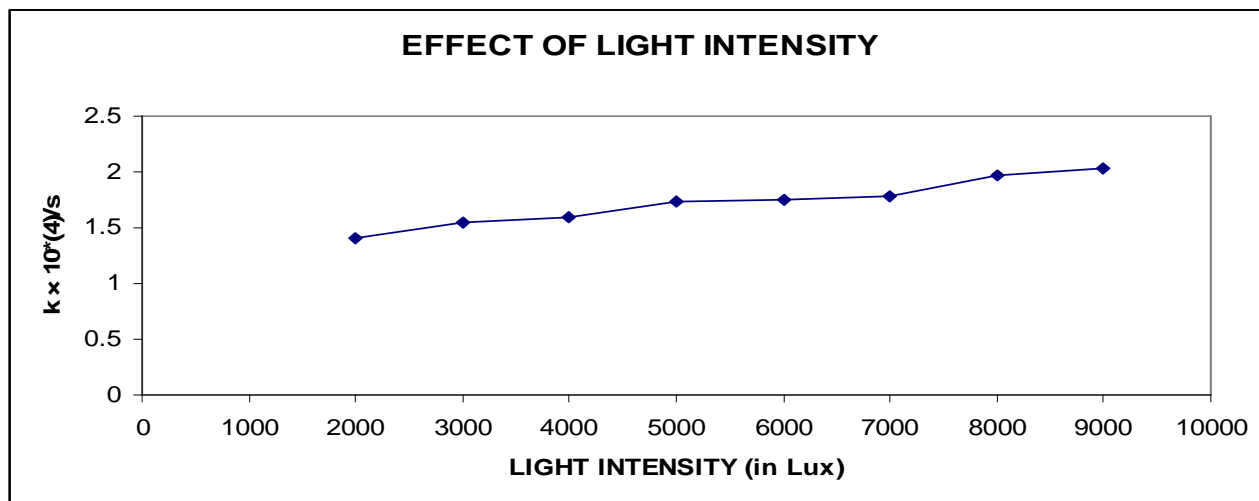
**FIG.F:** Effect of methyl green conc. on the photo-Fenton degradation of dye

pH = 2.75	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
Amount of $\text{H}_2\text{O}_2 = 0.08 \text{ cm}^3$	Light Intensity = 9000 Lux
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	



**FIG – H:** Effect of light intensity on the photo-Fenton degradation of dye

Methyl Green = $[4.00 \times 10^{-5} \text{ M}]$	pH = 2.75
Amount of $\text{H}_2\text{O}_2 = 0.08 \text{ cm}^3$	$\text{FeCl}_3 = [3.33 \times 10^{-5} \text{ M}]$
Catechol = $[4.66 \times 10^{-5} \text{ M}]$	



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