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Fenton Process for The Treatment of Wastewater Effluent from the Pharmaceutical Industry

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ABSTRACT

The present study intends to investigate the performance of the Fenton process as one of the most efficient (AOPs) in a batch mode for treating wastewater effluent from the pharmaceutical industry, as well as the parameters that influence reaction such as pH, hydrogen peroxide (H_2O_2) and ferrous sulphate heptahydrate doses ($FeSO_47H_2O$) doses at various reaction times. Fenton process is low cost, highly efficient and eco-friendly technologies in the removal of organic pollutants in wastewater using hydroxyl radicals for oxidation. The obtained results indicated that the authenticated response to the chemical oxygen demand (COD) removal was 92-94 % at optimum values of pH, H_2O_2 dose, $FeSO_47H_2O$ dose and reaction time, 3, 2 mL, 3 g and 60 min respectively. Under these conditions, the residual COD after Fenton oxidation process becomes 100 mg/L. Obtained results have shown that the process is effective for removal of COD and it can be applied for the treatment of pharmaceutical industry's wastewater effluent.

Keywords: *Advanced oxidation, Fenton process, wastewater treatment, pharmaceutical industry*

1. INTRODUCTION

Water is one of the most important essential requirements of life. Recent day's quality of water plays a significant role in the health and hygiene of society. A biggest problem we are facing is because of overuse and contamination of water. Different anthropogenic activities are responsible for spoiling the quality of water. It is estimated that about 67 % of the world's population lacks access to clean drinking water at least one moth in a year (Trinh *et al.*, 2021). Reasons for water scarcity include emissions of harmful pollutants such as organic and inorganic compounds released into water bodies by industry and anthropogenic activities. Water pollution by harmful pollutants has become a key issue in current and future scenarios as it directly and indirectly affects all living things (Varma *et al.*, 2021). Major water pollutants include pharmaceuticals, heavy metals, dyes, phenolic compounds and pesticides (Rashid and Ramshoo, 2012). Amongst these pharmaceuticals are considered to be the most harmful organic compounds. This is because these drugs are not easily degraded and remain in the aquatic environment for long period of time. When the concentration of pollutants in wastewater exceeds permissible limits cause adverse effect on communities, they are termed as water pollutants. These include contaminants such as organic pharmaceutical residue (Brown *et al.*, 2006). Due to its persistence and adverse effects, the pharmaceutical residue has attracted worldwide attention.

Some pharmaceuticals are eliminated without changes in metabolism (Sorensen *et al.*, 1998). The presence of even minute concentrations of such drugs in the environment can have toxic effects on microorganisms. This is because organic compounds are complex in nature; those are difficult to break down into simple end products. Therefore, they are considered one of the most significant pollutants to environment and human health (Kummerer *et al.*, 2000). Several recent studies have shown that conventional treatment methods for pharmaceutical contaminants based solely on biological treatments are ineffective (Trovo *et al.*, 2009). Therefore, it is important to develop efficient treatment methods to limit the presence of pharmaceutical contaminants in the aquatic environment. Aminoglycoside antibiotics are organic compounds containing amino cyclitol units to which amino sugars are glycodically linked. These antibiotics are effective against gram +ve and gram –ve organisms. Streptomycin, Neomycin and Gentamycin are prominent examples of aminoglycoside antibiotics. *Escherichia coli, Enterococcus feecalis, Pseudomonas aeruginosa, Klebsiella pneumoniae, Enterobacter aerogenes, Staphylococcus aureus and Acinetobacter baumannii* are critical pathogens and In India these pathogens have recorded resistance against aminoglycoside antibiotics (WHO, 2017).

However, many antibiotics with different structures have been found in the environment, those are regarded to have poor biodegradability and may affect living organisms. Several physic-chemical, biological and advanced techniques were evaluated to treat water containing contaminants such as antibiotics and their efficiency depends on the type of contaminants to be degraded and related operating parameters (Sievers, 2011).

2. MATERIALS AND METHODS

Chemicals:

Ferrous sulfate heptahydrate ($FeSo_{4.}7H_2O$) and hydrogen peroxide (H_2O_2) were used to conduct the fenton reaction, while sulphuric acid and sodium hydroxide were used for adjustment of pH. All the chemicals were of analytical grade and acquired from Hi media and Qualigens Fine Chemicals Pvt. Ltd. respectively.

Sampling of waste water:

The wastewater sample was collected from the pharmaceutical industry based at five-star MIDC Kagal, Kolhaour and it was stored in incubator at 4 ºC to avoid biodegradation.

Experimental set up

The batch approach was used to conduct an experiment (Disli, 2010). the effect of different variables including pH, reaction time and dosage of fenton reagent on COD reduction efficiency were investigated. The batch methods involves agitation of 100 ml of wastewater. reaction time were kept constant at 60 minutes. To determine the efficiency at different dosages and times, a total of 9 experimental sets were employed in triplicate. Following the initial setup's results, 1 N H_2SO_4 and 1 N NaOH were used to correct the pH at ideal circumstances.

COD reduction efficiency

Both the initial and final COD after completion of experiment were determined using the standard method (APHA, 2005). pH measurements were done with a pH meter. COD reduction efficiency was calculated using the following formula (Syama *et al.*, 2015)

$$\%\ \text{Efficiency} = \frac{Co - Cf}{Co} X100$$

3. RESULT AND DISCUSSION

The experiment was carried out to investigate the ability of Fenton reagent to work as a oxidant at various doses and pH. The treatment efficiency was analysed through COD reduction which further indicates degradation of various organic compounds. Treatment to the pharmaceutical effluent having initial COD 1850 mg/L was conducted using Fenton process (H_2O_2 and $FeSO_47H_2O$) at room temperature under different doses of Fenton reagents and pH. The efficiency of these photochemical methods was determined by monitoring COD which is commonly used to monitor performance of conventional; wastewater treatments. COD reduction indicates the degradation of organic compounds contained in water to be treated.

Characterization of Pharmaceutical effluent plant water

Table 1. represents the initial characterization of pharmaceutical waste water

Sr. No	Characteristics	value
1	pH	7.8
2	EC mS/cm	3.2
3	COD mg/L	1850

Effect of reagent dosage on COD reduction

To observe the effect of H_2O_2 and $FeSO_{4.}7H_2O$ dosage as Fenton reagent, dosage of both were varied from 1 to 3 mL for H_2O_2 and 1-3 g for $FeSO_{4.}7H_2O$ for 100 ml of pharmaceutical effluent. Dosage can play significant role in efficiency of entire degradation process as it is main component that limits the production of hydroxyl radicals during treatment (Bensalah *et al.*, 2018). It has been observed during experiment that during the COD of the solution decreases with the increasing dosage. However excessive dosage can lead to an increase in COD of the effluent, in addition to reaction with hydroxyl radicals it forms hydroperoxyl radicals, a species with lower oxidizing power when compared with hydroxyl radicals who are having high oxidizing power (Babuponnusami *et al.*, 2014).

Table 1: Effect of Fenton reagent in COD reduction Efficiency

FeSO4	H2O2 (ml)	% Reduction efficiency
1 gm	1 ml	58.9
	2 ml	66
	3 ml	68.7
2 gm	1 ml	73
	2 ml	75.4
	3 ml	76.2
3 gm	1 ml	91
	2 ml	94
	3 ml	92.1

Generally, it has been found that the percentage degradation of contaminants increases with respect to dosage of H_2O_2 (Kang *et al.*, 2000). However, the unutilized reagents contribute to rise in COD. The care should be taken in selecting the dosage. When beyond optimum dosage added then there will be increase in COD level due to presence of residual reagents remains in the treated solution (Kuo, 1992).

The obtained results shows that at constant retention time of 60 min, increase in dosage of H_2O_2 and $FeSO_4.7H_2O$ leads to increase in % reduction of COD. At ratio of Fe2+/H2O2 the COD reduction is maximum at 3:2 that is 94 %. Beyond this dosage at 3:3 ratio the COD efficiency decreased as results are negatively affected. Therefore, the optimum ration of fenton reagent (Fe2+/H2O2) is 3:2 per 100 mL of solution to be treated as shown in Table no.2.

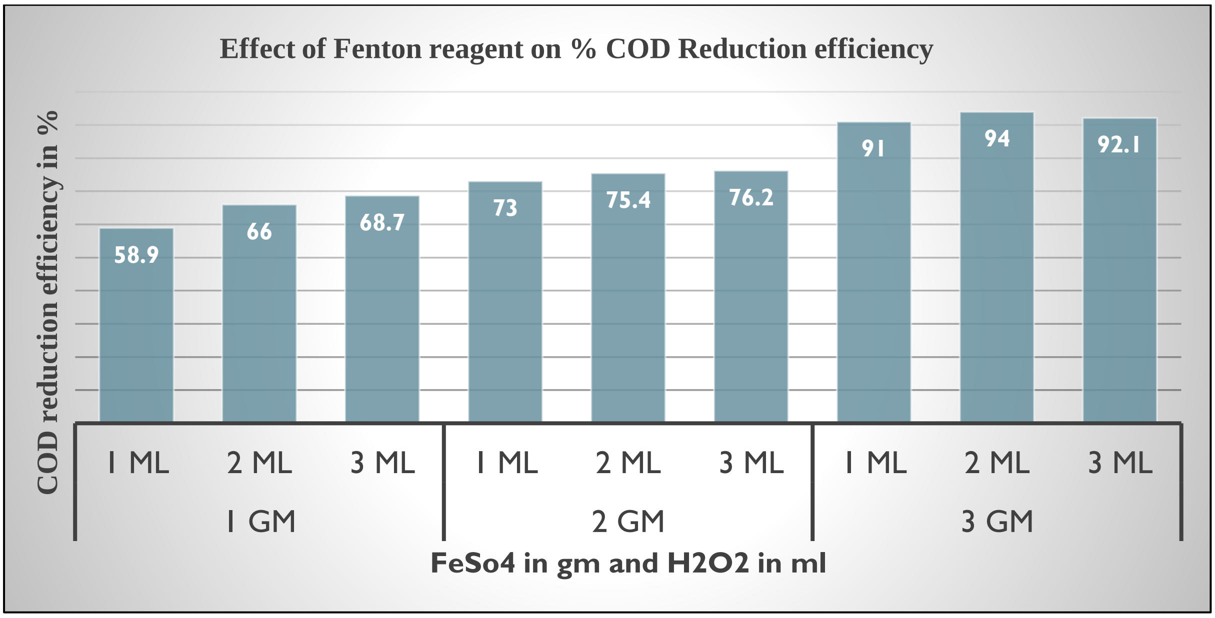


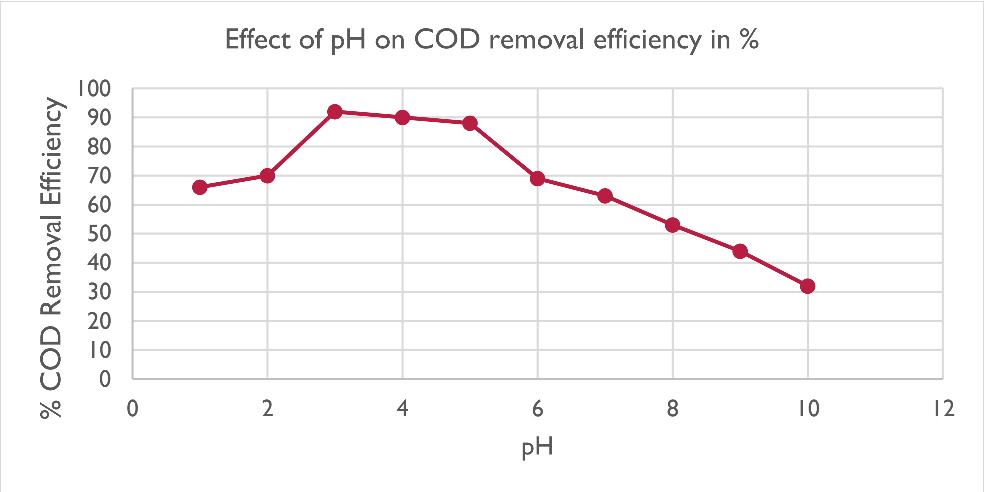
Figure 1: Effect of Fenton reagent on % COD Reduction efficiency

Effect of pH

The pH value has an effect on the oxidation potential of hydroxyl radicals because of the reciprocal relation of the oxidation potential to the pH value (Ebrahiem *et al.*, 2017). The pH value influences the generation of hydroxyl radicals and hence the oxidation efficiency (Elmolla and Chaudhuri 2017). To determine the optimum pH experiments were conducted by varying the pH in the range 1-10. The experimental conditions were 100 mL of pharmaceutical effluent, retention time of 60 min and ration of Fenton reagent Fe2+/H2O2 was 3:2. For this experimental study of Fenton process maximum COD reduction was achieved at pH 3 by keeping constant dose ration of Fe2+/H2O2 to 3:2 at 60 minutes of retention time. It was seen that increase in the pH from 1-3 leads to the reduction in COD but after pH 3 there was substantial decrease in the efficiency of COD with increasing pH.

pH	% Efficiency
1	66
2	70
3	92
4	90
5	88
6	69
7	63
8	53
9	44
10	32

At pH value greater than 7, a rapid decrease in COD reduction was also observed. This is due to rise in pH which makes the solution alkaline. As H_2O_2 is unstable in alkaline condition decomposes to give O_2 and H_2O, also consequently losses its oxidizing capacity (Wang, 2008). By varying pH it was found that at pH 3 COD reduction was maximum, nearly 92 % reduction was achieved. At room temperature by just varying pH alone it was found that COD reduction was maximum at pH 3.



4. CONCLUSION

In this study, Fenton reagent shown its potential for removal of COD from pharmaceutical effluent. The investigation has proven that the use of Fenton reagent in treating pharmaceutical waste water is a good method to reduce the pollutant concentration in wastewater. The removal of COD from batch experiment increases with increase in the dosage of ferrous and hydrogen peroxide, showing that the process is highly dependant on the reagent. The optimum condition for this experiment was 60 min reaction time, 2mL hydrogen peroxide, 3 g ferrous sulphate and 3 pH. It has shown maximum 94% of COD removal efficiency at optimum condition. The treatment method after meeting all the future improvements in the design can be implemented to put up an end to the prevailing unhealthy environment because of pollution by pharmaceutical wastewater

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