Treatment of Pharmaceutical Sludge by Fenton Oxidation Process

Rajesh Nithyanandam and Raman Saravanane

Abstract—Fenton Oxidation process was used to treat the pharmaceutical sludge having the COD value of 118, 400 mg/L for the safe disposal. By varying the ratio of ferrous to hydrogen peroxide for different molar ratio (0.1M, 0.2M, 0.3M, 0.35M,0.4 M) of Fenton's reagent the pharmaceutical sludge was treated and COD reductions were calculated and optimum ratio was found out. Sludge was also treated by physical treatment by varying the pH and temperature and COD reductions were calculated and the optimum point was found out. The untreated and treated sludge was subjected to GC-MS for the quantitative analysis of non-biodegradable compounds. Scanning Electron Microscope image acquistation and elemental analysis were also studied for both treated and untreated samples. The purpose of the study is to find the suitable end-use method for reusing the treated and oxidized products.

Index Terms—Antibiotics, chemical treatment, Fenton Oxidation, pharmaceutical sludge.

I. INTRODUCTION

Pharmaceutical industry often generates high strength wastewater and sludge changing, in character and quantity depending upon the used manufacturing processes and season [1]. The effluents originated from the formulation of antibiotics have low biodegradability since they contain almost only active substance. As per chemical equilibrium principle, the compounds which are present in effluent will also present in sludge. Hence treatment is necessary for pharmaceutical sludge. Several studies have observed antibiotic agents in the aquatic environment, including groundwater, surface water, and wastewater treatment plant effluent [2]. Despite knowing the sources and pathways of pharmaceuticals into the environment, actual concentrations and availability are unknown. Antibiotics generally have low biodegradability since they are biocidal substances and the degradation of these substances cannot be accomplished in the natural environment or biological treatment plants [3]. Pharmaceuticals released in the environment may impose toxicity virtually on any level of the biological hierarchy, i.e. cells, organs, organisms and population [4]. Hence chemical treatment such as AOP (Advanced Oxidation Process) is necessary.

In recent years, there is a wide concern by Scientist and researchers towards recalcitrant compounds (xenobiotic or non-biodegradable) which is discharged from industry as effluent or sludge. The hazardous waste discharged from industries such as pharmaceutical industry is primary concern in the recent decades. In Italy, France, Greece and Sweden different therapeutic pharmaceutical classes were found in the effluent of sewage treatment plant [5].

In spite of the regulations of Environmental Protection Agency (EPA) and Federal Agency, the effluent from industry is not adequately treated [6]. Several methods have been found for treating the recalcitrant compound found in waste water or in sludge. But the methods were found to be ineffective in respect of complying with the final disposal standards. From the literature survey it is very well clear that among Advanced Oxidation Process (AOP), Fenton's reagent is the effective method to treat the recalcitrant compound because the hydroxyl radical formed during the reaction of ferrous sulphate to hydrogen peroxide, have the tendency to degrade the aromatic and heterocyclic ring [7]-[8].

It was found that advanced oxidation had not been widely applied yet because the chemical processes behind advanced oxidation is not completely understood [9]. Specific operating conditions and reactant doses are necessary for complete mineralization of the organics, and for effective wastewater and sludge treatment. AOP is expensive to install and operate; they may be unavoidable for the tertiary treatment of refractory organics present in industrial effluents and sludge to allow safe discharge of industrial contaminants. AOP includes Fenton reagent, O_3 , O_3/H_2O_2 , O_3/UV .

The application of several AOP has also been tested in some pilot-plant studies. In spite of the enormous physical, chemical and technical knowledge about AOP experts still disagree about the most efficient method for dealing with a specific wastewater problem. Among AOP, Fenton treatment as the following advantages: The Fenton reaction has a short reaction time among all advanced oxidation processes and Iron and H_2O_2 are cheap and non-toxic, there is no mass transfer limitations due to its homogenous catalytic nature, there is no energy involved as catalyst and the process is easily to run and control [10].

Fenton treatment, however, appears to be technically very complicated, as the pH should be near 2 during the reaction, and the dissolved iron has to be precipitated as Fe(OH), by neutralization at the end of the process. There is evidence that the mechanism of the Fenton reaction is considerably more complicated. However this method is the effective way of treating the recalcitrant compounds. Nevens et al., (2002 & 2003) studied the effects of pH, temperature, reaction time and H_2O_2 concentration with considerable reduction in organic concentration [13].

The objective of the present study is to treat the pharmaceutical sludge by advanced oxidation process using

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Fenton oxidation method. The scope of the study is (a) to find the effective ratio of ferrous to hydrogen peroxide from different molar ratio of Fenton's reagent (b) to find the elemental analysis and the compound degradation, before and after treatment.

II. MATERIALS AND METHODS

A. Waste Sludge

The sludge was collected from the pharmaceutical industry, "Orchid Chemicals and Pharmaceuticals Limited, SIDCO Industrial Estate, Alathur", South India and it was stored in incubator at 4°C to avoid biodegradation. It was produced by the pharmaceutical industry from secondary treatment during effluent treatment process. Before analysis, sludge was diluted and used for all the experiments. The experiments were conducted at a room temperature of 35°C. The sludge was diluted to 25% by means of distill water. 200ml of diluted sludge sample was taken in five different canes (5 liters capacity).

B. Fenton Oxidation Experiment

200 ml of diluted sludge was poured into the each 5 liter cane. The pH of the diluted sludge was tested. It is very well clear from the literature Fenton's reaction is optimum at pH 2 to 3 [7]. The diluted sample has the pH of 7.8. In order to drop the pH from 7.8 to 2 -3, 20 ml of 20 % H₂SO₄ was added. During this addition, the sample raised with bubbles and the pH was reduced to this range. Afterwards, for 0.1 molar ratio of Fenton reagent, different ratio of ferrous to hydrogen peroxide (1:2, 1:4, 1:8, 1:10) was added to each cane and allowed to react for half an hour. The 5th cane was used as control to that there is neither addition of sulphuric acid, to control pH, nor Fenton's reagent. This was done to find the normal degradation rate of the compound within half an hour in the atmospheric environment.

After half an hour, sample from each cane was tested for the COD (Chemical Oxygen Demand) reduction and Total solid. COD measurements were used to determine the efficiency of Fenton's Reagent in order to remove the organics during chemical oxidation process.

The same procedure was repeated for different molar ratio of Fenton's Reagent such as 0.2, 0.3, 0.35, and 0.4. The reaction was fast and exothermic, at a room temperature of 35°C.The aqueous solution of Fenton reagent and diluted sample was stirred during the reaction period of 30 minutes. At the end of half an hour the samples were withdrawn and COD was determined immediately.

C. Sample Preparation Procedure for GC-MS

2 ml of 25% diluted sludge was taken in a test tube. 0.05 N of NaSO₄ (2ml) was added to remove the moisture content. Sample was kept for 1 day. Then the sample was centrifuged. Settled particles were subjected to high pressure vacuum filter. Settled particles were kept at -17 °C (snow) for 15 min and then they were subjected to high pressure vacuum filter. Then the moisture less compound was subject to the addition of methanol (2ml). Finally, the solution was centrifuged at 11000 rpm for 20 min. Supernatant was taken and subject to FID (Flame ionization detector) and GC-MS (AGILENT Technologies; Make: JEOL GC mate; SI.No.6890N).

D. Scanning Electron Microscope

For SEM ,the liquid sample was first soaked in phosphate buffered 6% glutaraldehyde for 1 hour and at less than 20° C, washed with phosphate buffer and dehydrated with acetone. Then these samples were mounted on a sample stub and cooled with Gold (200 A °thick) in a sputter coating unit. (Ion sputtering device IPC 1100E, Japan). The samples were then Scanned using SEM (JOEL-JSM 5300, Japan) at a resolution of 4.5nm at 30 KVA at a working distance of 8 mm.

E. Elemental Analyzer

X-ray fluorescence analysis was used to determine the elements of the sample. Rhodium was used as the standard anode material. The tube and generator were designed for a permanent output of 4 kW. The detector was scintillation counter and proportional counter. A 0.077 $^{\circ}$ collimator was used for high resolution measurements with LiF.

F. Variation of pH and Temperature

1 ml of the sludge was taken and it was diluted to 100ml distill water to get 1% dilution. From that 10ml was taken and it was diluted to 100 ml to get 0.1% dilution. The 100 ml solution was subject to the addition of 1% H_2SO_4 to attain the pH of 2.5. Then the solution was heated to 37 °C, 42 °C, 47 °C, and 50 °C respectively. For different temperature COD value was determined. The same procedure was repeated for pH 4 and 6. For increase the pH to alkaline condition pH buffer 9.2 solutions (1 tablet/100ml) was added and the same procedure was repeated for the determination of COD at different temperature. Without changing the pH, the 0.1% diluted sample (pH = 7.1) was subject to different temperature and COD was determined.

III. RESULTS AND DISCUSSION

A. Characterization of Pharmaceutical Effluent Plant Sludge

Table I presents the characterization of secondary pharmaceutical waste sludge from Orchid Pharmaceutical Effluent treatment Plant.

S.	Characteristics	Values
No		
1	pH	7.8
2	Electrical Conductivity, mS/cm	5.13
3	Alkalinity, mg/L	7721.4
4	Total solids, mg/L	302900
5	Total Dissolved Solids, mg/L	6650
6	Total Suspended solids, mg/L	296250
7	Total Volatile Solids, mg/L	76200
8	Total Volatile suspended Solids, mg/L	75100
9	Total Chemical Oxygen Demand, mg/L	118400
10	Soluble Chemical Oxygen Demand, mg/L	16000
11	Volatile Fatty Acid, mg/L	1380
12	Total Kjeldahl Nitrogen, mg/L	1014
13	Phosphorus, mg/L	5900
14	Specific gravity at 32 °c, mg/L	1.106
15	Biochemical Oxygen Demand, mg/L	-
16	Alkalinity/VFA	5.6

TABLE I: CHARACTERIZATION OF SECONDARY PHARMACEUTICAL WASTE SLUDGE WITH 25% DILUTION

B. COD and Total Solid Variation for Different Molar Ratio of Fenton Reagent at Different Ratio of Fe^{2+}/H_2O_2

Pharmaceutical sludge was treated with different molar ratio of Fenton Reagent for 30 minutes. During the treatment of sample with 0.1M of Fenton reagent the foam generated in the cane is very less when compared to the higher molar ratio of Fenton reagent (Fig. 1). At 0.1M of Fenton reagent, COD is increased drastically, when compared to the original sample. This implies generation of hydroxyl radical is not sufficient at this molar ratio and COD is contributed by ferrous sulphate and hydrogen peroxide as well. Total solid is minimum at 1:8, when compared to the other results.

At 0.2M of Fenton reagent, COD reduction is not appreciable because it is slightly higher than that of original sample but COD values are lesser than 0.1M concentration of Fenton reagent. Among all the canes the foam generated in 1:8 cane is more. When compared to the other ratios of ferrous to hydrogen peroxide (1:2, 1:4 and 1:10) COD is minimum for 1:8. Total solids were minimum value at 1:8.

C. COD and Total Solid Variation for Different Molar Ratio of Fenton Reagent at Different Ratio of Fe^{2+}/H_2O_2

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Fig. 1. COD reduction for different ratio of Fe^{2+}/H_2O_2 at different molar ratio.



Fig. 2. Total solid reduction for different ratio of Fe^{2+}/H_2O_2 at different molar ratio.

At 0.2M of Fenton reagent, COD reduction is not appreciable because it is slightly higher than that of original sample but COD values are lesser than 0.1M concentration of Fenton reagent. Among all the canes the foam generated in 1:8 cane is more. When compared to the other ratios of ferrous to hydrogen peroxide (1:2, 1:4 and 1:10) COD is minimum for 1:8. Total solids were minimum value at 1:8.

At 0.3M the foam generation is large in all the ratios of ferrous to hydrogen peroxide. Especially at 1:8 &1:10 the foam generated is maximum. The COD reduction is maximum in 1:10 ratio of ferrous to hydrogen peroxide. Nearly 65% of COD reduction is achieved in this ratio. In 1:8 ratio COD reduction is nearly about 35% only. Total solid is minimum at 1:8, when compared to the other results. For the control COD reduction is only 3 %.

At 0.35M ratio of Fe^{2+}/H_2O_2 the COD reduction is maximum at 1:4 & 1:8 .But Total solid is minimum at 1:8 only. Nearly 60% of COD is reduced at 1:4 & 1:8. At 1:10 COD reduction is only 35%. During hydrogen peroxide consumption at 1:8 &1:10 the foam generated is maximum and lot of heat energy is generated, it shows the reaction is exothermic. At 1:10 COD value is higher than that of 1:8. This is because large amount of generation of hydroxyl radical consume dissolved oxygen in the sample. The production of hydroxyl radical within a short period of time results in the depletion of dissolved oxygen and this may reduce the efficiency of the mineralization of organics [14]. By comparing the results of 0.1M, 0.2M, 0.3M & 0.35M for a particular ratio of ferrous to hydrogen peroxide COD is reduced drastically. This implies higher the molar ratio COD reduction is maximum [15]-[16].

D. Sludge Characteristic Analysis Before and After Treatment

S.NO.	Characteristic	Sludge (before treatment)	Sludge (after treatment)
1	COD	29600	10400
2	Volatile fatty acids	840	1680
3	Phosphate	226750	148750
4	Total Solid	121400	253350
5	Total Volatile Solid	29100	172650
6	Fixed Solid	92300	80700
7	Total Dissolved solid	1850	4950
8	Total dissolved volatile solid	1550	2600
9	Total suspended solid	119550	248400
10	Volatile suspended solid	27550	170050

TABLE II: SLUDGE CHARACTERISTIC BEFORE AND AFTER TREATMENT

25% diluted sludge was taken and subject to various characteristic analysis before and after treatment. From the results it was clear that there is a drastic reduction in COD, Phosphate, and Fixed solid. On the same time Total solid, total volatile Solid increases rapidly. Increase in the Value of Total solid is due to addition of Fenton reagent (Ferrous sulphate).That can be reduced by further treatment. The ultimate aim is to reduce the COD value that was achieved by this process (48% reduction) [17].

E. GC-MS Analysis for Treated and Untreated Sample

From the chromatogram plots (not shown) it was found that 4 compounds are present for untreated sample by four different peaks. The peaks of different retention time indicates different individual compound. From GC-MS all the four compounds was found. The retention time of 4.591 minutes indicates the compound 1,4 benzene dicarboxylic acid. The retention time of 5.442, 7.352, and 8.21 minutes indicates the compound Benzene 1, 1' (bromo methylene) bis, phenylthioacetic acid and phenyl 3- pyridyl ketone respectively. There is only one peak for treated sample that indicates that the other three compounds were degraded by Fenton process. From the chromatogram plots it was clear that the compound having the retention time of 8.21 minutes is not degraded fully. But mostly it was degraded and it was up to the disposal limit.

By GC-MS technique the compounds which were present before and after treatment was found out. By the above technique it was sustain that COD value of the sample is high due to all the four recalcitrant compounds.

F. SEM and Elemental Analysis for Fenton Treated (Optimum) Sample

From the elemental analysis it was clear that the elements which are present considerably more is oxygen, iron, Phosphorus and Molybdenum. It was clear that iron content was increased drastically from 6% (by weight) to 28.12 % (by weight) due to Ferrous sulphate. The iron content was reduced by treating the Fenton treated sample with sodium hydroxide. Hence further treatment is necessary. This is the main disadvantage of this process.



Fig. 3. Image acquistation for Fenton treated sample



Fig. 4. Elemental analysis for Fenton treated sample

Element	Net	K-Ratio	Weight %	Atom %
	Counts			Error
0	1032	0.214	34.26	+/- 3.33
Na	368	0.041	6.94	+/- 0.80
Si	245	0.021	1.83	+/- 0.25
Р	2014	0.207	16.76	+/- 0.77
Fe	1208	0.384	28.12	+/- 0.56
Мо	970	0.133	12.09	+/- 0.38
Total			100.00	

G. SEM for pH and Temperature Optimum Variation Sample

From the elemental analysis of pH and Temperature variation sample it was found that the oxygen element is found to be more. It was clear that there is no further treatment necessary. But considerable amount of carbon is present here.



Fig. 5. Image acquistation for pH and temperature sample



Fig. 6. Elemental analysis for pH and temperature variation sample

TABLE IV: ELEMENTAL ANALYSIS FOR PH AND TEMPERATURE VARIATION

Element	Net Counts	K-Ratio	Weight %	Atom % Error
С	105	0.132	25.37	+/- 7.06
0	573	0.199	35.37	+/- 2.92
Mg	493	0.062	4.64	+/- 0.34
Si	1186	0.172	10.04	+/- 0.25
P	534	0.092	5.53	+/- 0.22
Са	581	0.157	8.10	+/- 0.36
Fe	217	0.115	6.45	+/- 0.28
Мо	308	0.071	4.49	+/- 0.13
Total			100.00	

H. COD Variation by Varying pH and Temperature



Fig.7. COD variation for different pH and temperature.

By varying pH and Temperature it was found that at pH 4 and 42 C COD reduction was maximum, nearly 48% reduction was achieved. At room temperature by just varying pH alone it was found that COD reduction was maximum at pH 4.



Fig. 8. COD variation for different pH at room temperature.

IV. CONCLUSIONS

The hydroxyl radical formed during the reaction is efficiently degrading the antibiotic which is present in the sample, within the detention time of half an hour. For different ratio of ferrous to hydrogen peroxide (1:2, 1:4, 1:8, 1:10) in different molar ratio of Fenton's reagent(0.1M, 0.2M, 0.3M, 0.35M, 0.4M) the COD reduction was determined. It has been found that COD reduction is maximum in 1:10 ratio of ferrous to hydrogen peroxide at 0.3M of Fenton's reagent. In this ratio nearly 65% reduction in COD was achieved. In the same ratio Total solid was also minimum. From the GC-MS results it was found that the compounds like 1, 4 benzene dicarboxylic acid, Benzene 1, 1' (bromo methylene) bis and phenylthioacetic acid were removed totally after treatment. The compound phenyl 3- pyridyl ketone was removed mostly after treatment. And by physical treatment, varying the pH and Temperature, it was found that COD is minimum at pH=4 and T=42 °C. At this critical point 48% COD reduction was achieved, without adding any chemicals.

From SEM, elemental analysis was found for both Fenton (optimum ratio) treated sample as well as optimum pH and Temperature variation sample. The antibiotic which is present in the sludge is 7Amino cephalosporanic Acid (7ACA) which is the intermediate of cephalosporin C, which is determined by HPLC. And the further study is needed to determine the degradation pathway of 7 ACA by Fenton process.

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NOMENCLATURE

GC-MS: Gas chromatography – mass spectroscopy COD: Chemical oxygen demand SEM: Scanning electron microscope 7ACA: 7Amino cephalosporanic acid

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