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Treatment of Pollutants in Industrial Wastewater Using Fenton's Oxidation Process: An Overview

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***Abstract*— Nowadays, there has been more emphasis on strict policies regarding the quality of industrial effluents. This is due to the alarming rate of environmental pollution. Besides, there is also need of treatment processes which are efficient and consume less time. Advanced Oxidation Processes (AOPs) are a viable option in such scenarios. Fenton's oxidation reaction is believed to be a prospective method of oxidation for this. The Fenton's reagent is the combination of hydrogen peroxide [H2O2] and a specific ferrous salt [Fe2+]. The wastewater sample is mixed with hydrogen peroxide and the addition of ferrous sulfate [Fe2+] triggers the formation of highly reactive oxidizing species [•OH]; which can degrade the pollutants of water and wastewater. This paper involves a review of studies carried out on laboratory scale treatment for the removal of specific pollutants from water and wastewater of textile, distillery and other such industries. The objective is to provide a clearer idea of the effects of the reagent on the degradation of the pollutants in water/wastewater during treatment.**

***Index Terms*- Advanced oxidation, Industrial effluents, Fenton's reagent**

1. Introduction

There is an increasing amount of awareness in the society regarding the problem of industrial effluent treatment and its qualities. Moreover, the need for constant improvement in technology due to demographic changes has led to more toxic and less eco-friendly effluents. Nowadays, due to the increasing presence of molecules, refractory to the microorganisms in the wastewater streams, the conventional biological methods cannot be used for complete treatment of the effluent and hence, introduction of newer technologies to degrade these refractory molecules into smaller molecules, which can be further oxidized by biological methods, has become imperative [1]. This has provided a push to innovators for finding new ways of treatment. Use of Advanced Oxidation Processes (AOPs) has been an outcome of this outlook towards wastewater treatment. There are numerous AOPs available for a variety of wastewaters generated in the society viz., Fenton's Oxidation, Ozonation, Photocatalysis, Cavitation, etc. Among AOPs, Fenton's oxidation is one of the low-cost and effective methods for organic contamination removal [2]. For that, both laboratory scale and actual on-site applications of this system are studied and observed. This can help in obtaining a clearer idea of the effectivity of the Fenton's reagent. A number of pollutants like 3-Aminopyridine (3-AP), Benzene-Toluene-Ethylbenzene-Xylene (BTEX), Diuron, Methyl tert-Butyl Ether (MTBE) intermediates etc. and textile dyes such as Remazol Red F3B are known to have been removed successfully with the help of the Fenton's reagent. Another reason for evaluating the use of this treatment can be quoted as the simplicity of operation conditions as well as the lesser time requirement of the reaction to complete.

The Fenton reaction was first observed by H. J. Fenton in 1894 and is described as the enhanced oxidative power of Hydrogen Peroxide (H_2O_2) when using iron (Fe) as a catalyst under acidic conditions [3]. Thus, the Fenton's reaction uses the formation of a highly reactive oxidizing species known as hydroxyl free radicals (•OH) which has an oxidizing potential of about 2.8V. The catalytic decomposition of H_2O_2 by a transition metal such as ferrous iron (Fe^{2+}) has been successfully used as the source of hydroxyl free radicals (•OH) [4].

The free radicals react rapidly with most of the organic compounds, either by addition to a double bond or by abstraction of a hydrogen atom from aliphatic organic molecules. The resulting organic radicals then react with oxygen to initiate a series of oxidation reactions leading to mineralization of the organics to produce Carbon dioxide (CO_2) and water (H_2O) [5]. Use of Ferrous Sulfate ($FeSO_4.7H_2O$) is generally done as a source of the transition metal ions i.e. ferrous ions (Fe^{2+}). The normal course of reactions that occur in Fenton's oxidation process is shown in the equations (1-7) below:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO\bullet \quad (1)$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2\bullet + H^+ \quad (2)$$

$$HO\bullet + H_2O_2 \rightarrow HO_2\bullet + H_2O \quad (3)$$

$$HO\bullet + Fe^{2+} \rightarrow Fe^{3+} + OH^- \quad (4)$$

$$Fe^{3+} + HO_2\bullet \rightarrow Fe^{2+} + O_2 + H^+ \quad (5)$$

$$Fe^{2+} + HO_2\bullet + H^+ \rightarrow Fe^{3+} + H_2O_2 \quad (6)$$

$$HO_2\bullet + HO_2\bullet \rightarrow H_2O_2 + O_2 \quad (7)$$

The normal way of understanding the effectivity of the Fenton's reagent on the effluents is comparing its characteristics before and after treatment. These characteristics include COD

(Chemical Oxygen Demand), BOD_5 (Biochemical Oxygen Demand after 5 days), TOC (Total Organic Carbon), Colour, pH, etc.

Also, effectivity is studied by determining the concentration of the target pollutant in the water after the treatment with the reagent. In some papers there is the use of the mass spectrophotometer and gas chromatography for the residual pollutant concentration determination. Use of open as well as closed flask titrimetric methods is also made in some studies for the COD determination of the wastewater. This paper is intended to present an overview of the effectiveness of the Fenton's reagent on various industrial effluents as well as waters which contain specific types of pollutants.

2. MTBE intermediates removal

A. General

Burbano, A. A., *et al* studied effect of Fenton's reagent on degradation of the intermediate by-products that are formed during the reaction of MTBE with Fenton's reagent such as tert-butyl Formate (TBF), tert-butyl alcohol (TBA), methyl acetate, and acetone. The mentioned by-products are separately considered as parent contaminants and treated under the same reaction conditions initially used for MTBE [4]. MTBE is a pollutant found in the groundwater aquifers due to the leakage of underground gasoline pipelines.

B. Experimental Procedure

The experiments were carried out in a 4-litre amber borosilicate glass bottle reactors. The reactors were filled with 3.5 L of deionized water and the pH was adjusted to 3.0 using hydrochloric acid. This was made due to the fact that the system Fe^{2+}/H_2O_2 reaches its maximum catalytic activity at a pH of about 2.8–3.0 based on the referred literature [4]. Under anaerobic conditions of oxygen levels <1%, the Fenton reagent was added to simulate actual underground conditions. A single dose of Fenton's reagent was applied at the beginning of the reaction, using a molar ratio of Fenton's Reagent: Compound of 10:1 [4]. Samples were drawn for analysis at different intervals over a period of 60 minutes. Fig. 1 shows the experimental setup of the reaction.

C. Analysis and Findings

Degradation of MTBE intermediate by-product generation was found to be quick and appreciable during the first 2 to 3 minutes of reaction, and later the rates decreased and finally diminished after 30 minutes. As compared to MTBE degradation, the reduction of TBF was not as high and recorded only 80.4% in 60 minutes of reaction time. The degradation of TBA yielded a reduction percentage of 91.1% after 60 minutes. Methyl acetate reduction was found to be only 29.8% after 60 minutes of reaction time. Acetone presented lower amenability to •OH attack when compared to compounds containing the tert-butyl group, but still a higher degradation rate than that observed for methyl acetate. The extent of degradation reached 64.6% after 60 minutes of reaction [4].

The study concludes that acetone is a major product formed in the degradation of MTBE, TBF and TBA. Another conclusion is that the extent of degradation is inversely proportional to the oxidation state of the compound. Oxidation state is related to the compound structure. Ramifications in the structure such as that of the tert-butyl group show higher degradability than the simpler structures.

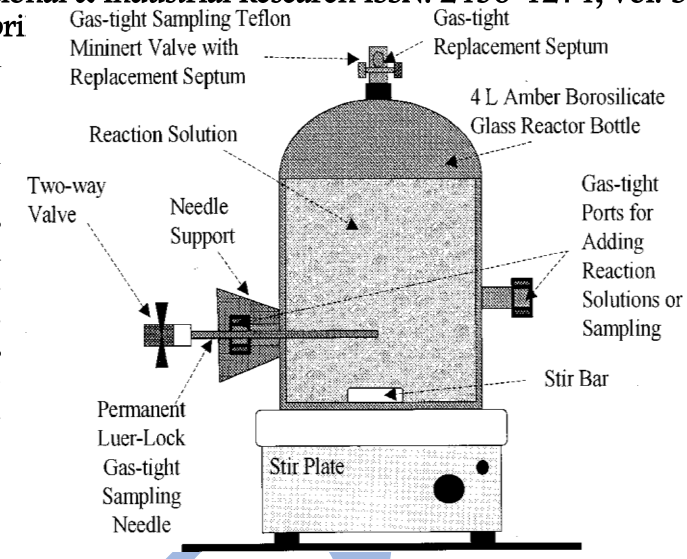


Fig. 1 Reactor setup for MTBE intermediates degradation using Fenton's reagent.

3. Oxidation of petroleum aromatic hydrocarbons

D. General

Fard, M. A., *et al* have shown the feasibility of nanoscale nonzero valent iron (n-ZVI) instead of ferrous sulfate in the degradation of the petroleum aromatic hydrocarbons like the BTEX using Fenton or photo-Fenton process was evaluated. Due to the underground gasoline storages the BTEX compounds can get mixed with the underground water sources and thus pose threat to human health. Hence the degradation of these pollutants is studied under the Fenton's reaction.

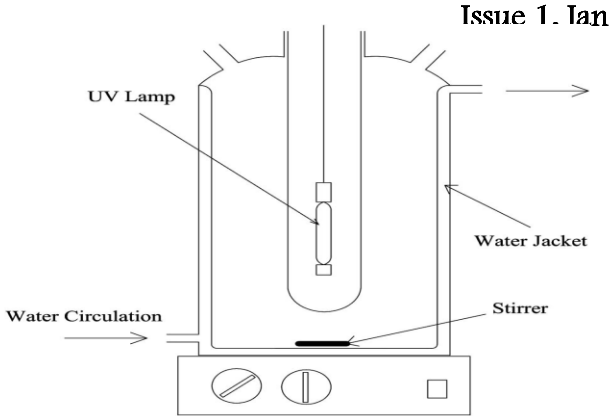
E. Experimental Procedure

All Fenton and photo-Fenton reactions were conducted in a 1000-mL cylindrical quartz four-necked reactor [2]. For the photo-Fenton reactions, mercury lamp of 125W power was used as a source of UV light. The batch method was used for oxidation testing. The schematic of the setup is given in Fig. 2. Addition of n-ZVI was done prior to addition of H_2O_2 and its complete mixing was achieved by using ultrasound. The total detention time of each sample was a maximum of 30 minutes. Samples were analyzed for the efficiency in degradation under different pH, the effect of initial concentrations of H_2O_2 and Fe^{2+}, the effect of inorganic substances and reaction time, TOC removal and total phenols formation.

F. Analysis and Findings

Using gas chromatography and mass spectrometry, the BTEX concentrations were found out. The concentration of residual H_2O_2, total phenols, dissolved oxygen (DO), and electrical conductivity was determined using ammonium metavanadate, 4-aminoantipyrine method, DO meter and EC meter, respectively. Total organic carbon (TOC) was

Fig.2 Reactor setup for photo-Fenton oxidation for BTEX degradation.



measured with a TOC analyzer [2]. Study of effect of different pH values viz. 3.0, 5.0, 7.0 and 9.0 showed that under acidic conditions, the degradation efficiency is the highest for both Fenton and photo-Fenton reactions. Degradation efficiency reduces in the alkaline conditions. This is believed to be due to an increase in the rate of the reaction of oxygen with $Fe(OH)_2$ to form Fe_2O_3, leading to a decrease in production of hydroxyl radicals [2]. The pH at 3.0 had maximum n-ZVI solubility leading to higher degradation efficiency. The degradation of BTEX in both systems was quickly finished within the first 5 min of reactions in which benzene, toluene, ethylbenzene, and xylenes removal efficiencies were 81.5, 82.6, 82.0, and 83.1% in the Fenton system and 91.1, 93.1, 92.2, and 92.9% in the photo-Fenton system, respectively [2]. Cations like Ca^{2+} and Mg^{2+}, as per the study, had the most deteriorating effects on the degradation efficiency. Anions like SO_4^{3-} and CO_3^{2-} played a salient inhibitor role for the purpose. The optimal degradation conditions of BTEX were achieved at pH 3.0 with H_2O_2 9.0 mM (millimoles) and n-ZVI 150 mg/L within 15 minutes for the Fenton process and within 10 minutes for the photo-Fenton process [2]. Use of n-ZVI instead of ferrous salt proved to improve the degradation efficiency of the Fenton and photo-Fenton process in treating the BTEX compounds.

4. Degradation of 3-Aminopyridine from water

G. *General*

3-Aminopyridine (3AP), used in the manufacture of anti-inflammatory drugs and also as a plant growth regulator is one of the emergent contaminants, because of its toxic and carcinogenic potential and hazardous effect on the natural environment [6]. Karale, R.S., *et al* studied the degradation capacity of the Fenton and photo-Fenton process for 3AP in water.

H. *Experimental Procedure*

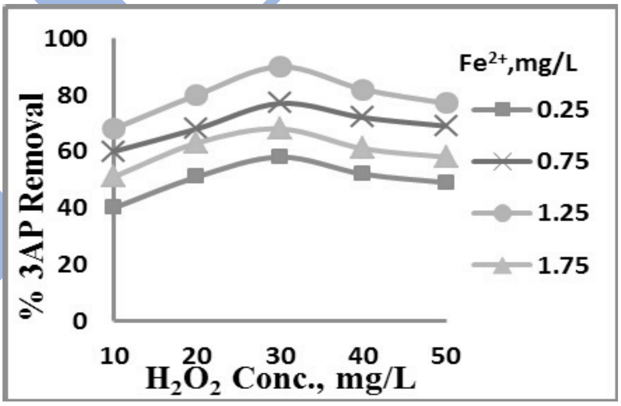
Use of 2000-mL capacity reactors was made for the reaction. The volume of the solution containing the certain concentration of 3AP was 1000-mL. All the tests were carried out at the room temperature of 27°C by using the batch method. Apart from the ferrous sulfate, the iron extracted from the laterite soil of the campus of National Institute of Technology Karnataka, India. he reactor setup had a magnetic stirrer to completely mix the reagent in the solution. H_2O_2 was added finally to initiate the reaction. For the experiments, pH was adjusted after adding appropriate iron and H_2O_2 solution, stirred with magnetic stirrer. pH of the solution was adjusted using 0.1 N H_2SO_4 (Sulfuric Acid) and 0.1 N NaOH (Sodium Hydroxide) [6]. The analysis of 3AP concentration was done using UV-VIS Double Beam Spectrophotometer. The COD analysis was done by adopting closed reflux titrimetric method.

I. *Findings*

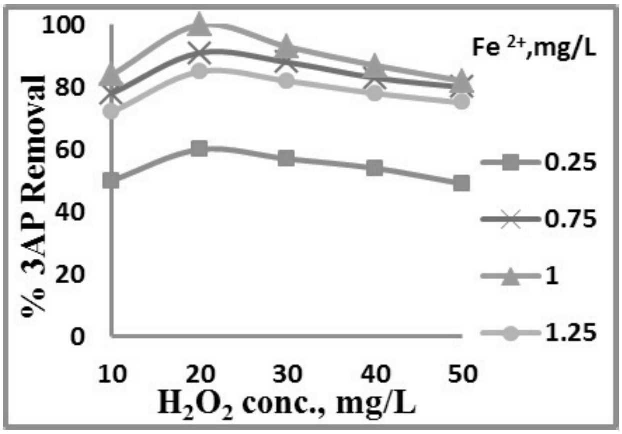
For both ferrous and laterite ions, the optimum pH was found to be 3.0. Drug removal efficiency reduced at other pH values. This may be because at higher pH (above 4.0), ferrous ions get easily converted to ferric ions, which have a tendency to produce ferric-hydroxy-complexes with H_2O_2. The low degradation at pH 2.0 and 2.5 may be due to the hydroxyl radical scavenging by H^+ ions [6]. At pH 3.0, the maximum removal percentage of 3AP and COD was found to be 90% and 84% respectively for optimum H_2O_2 concentrations of 30 mg/L and optimum Fe^{2+} concentrations of 1.25 mg/L. Higher Fe^{2+} concentrations, however, led to the reduced removal of the drug and COD. Use of Iron (Fe^{3+}) extracted from laterite soil showed a slight synergy (data is not shown) in the degradation of 3AP when compared with traditional ferrous iron (Fe^{2+}) in both Fenton's and Photo-Fenton processes. Thus, iron extracted from laterite soil can be a cost-effective option to treat polluted water bodies containing 3AP [6]. Photo-Fenton process is believed to have a more additive effect on the degradation of 3AP as per the results of mass spectrophotometry in the study. Fig. 3 (a) and (b) show the optimum dosage of H_2O_2 for 3AP removal for Fenton and photo-Fenton process respectively.

Fig. 3 (a) Effect of Fenton oxidation using ferrous iron on 3AP Removal.

Fig. 3 (b) Effect of photo-Fenton oxidation using ferrous iron



on % 3AP removal.



5. Remazol Red F3B dye mineralization

J. *General*

The dyes used in textile industry are known to be a pollutant and impart strong color and are recalcitrant in nature. Also, the environmental hazards they pose are related to defective self-purification of river streams as the dyes absorb and reflect the sunlight required for algal growth. Saatci, Y. studied the decolorization of Remazol Red F3B dye, which is a commercial azo-reactive dye, using Fenton and photo-Fenton process.

K. Experimental Procedure

Funnel shaped vessels were used for Fenton reactions. Dye solution of concentration 180 mg/L was adjusted to pH 3.0 using 1 N H_2SO_4 and 1 N NaOH. A known amount of H_2O_2 and $FeSO_4$ were added to 500-mL of the solution. For the Fenton process, different reaction times (5 to 135 minutes) were tested in order to determine the optimal conditions [7]. Use of UV enabled photoreactors of capacity 2500- mL was made for carrying out the photo-Fenton reactions. The radiation source was a low-pressure mercury ultraviolet (UV) lamp which was placed into a quartz sleeve and then into a cylindrical photoreactor [7]. For both Fenton and photo-Fenton processes, a magnetic stirrer was provided to ensure continuous mixing (120 revolutions per minute).

L. Analysis and Findings

Findings show that optimum pH for treatment was 3.0. Maximum decolorization of 96% was achieved in 40 minutes at this pH for Fenton process. For the photo-Fenton process, decolorization was found to be 99% in 15 minutes reaction time.

With respect to the concentration of H_2O_2, the decolorization efficiency increased in direct proportion. For the Fenton process, the amount of decolorization increases from 68.0 to 98.4% within 125 min of raising the H_2O_2 concentration from 100 to 700 mg/L. For the photo-Fenton process, raising the H_2O_2 concentration from 100 to 700 mg/L increases the decolorization from 91.2 to 98.8% [7]. It was remarkable that when the H_2O_2 concentration was increased from 500 mg/L to 700 mg/L, there was no significant change in the decolorization efficiency.

It was also seen that Fe^{2+} also increased the decolorization efficiency with the increase in its own concentration. For the Fenton process, the increase in Fe^{2+} concentration from 2.8 mg/L to 8.4 mg/L in 125 minutes increased the decolorization efficiency from 80.0% to 96.8%. Similarly, for the photo-Fenton process, in 20 minutes, the decolorization efficiency increased from 86.8% to 99.8%. The low degradation capacity of Fe^{2+} at low concentrations is probably due to decreased production of •OH radicals, which contribute to the oxidation [7]. It is found that 6.7 mg/L of Fe^{2+} can be taken as the optimum dosage for both of the processes [7]. Fig. 4 (a) and (b) depict the effect of initial concentration of Fe^{2+} on decolorization efficiency in Fenton and photo-Fenton process respectively. The Irradiation time is the exposure time of UV light to the solution.

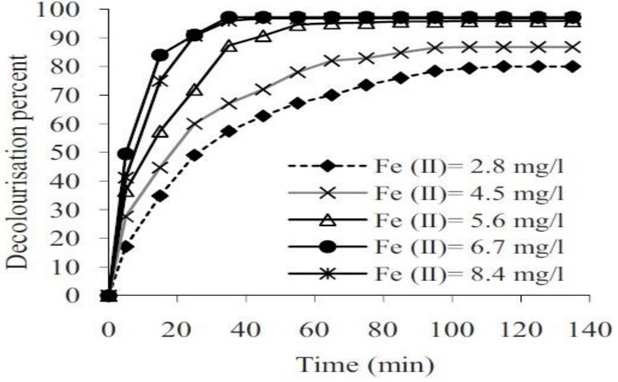
The initial concentration of Remazol RED F3B is also vital in case of analyzing the effectiveness of the Fenton and photo-Fenton process. It is stated in the paper that with an increase in the initial dye concentration in the solution from 40 mg/L to 300 mg/L reduced the reagent's efficiency of color removal from 99.4% to 84.6%. The increase in dye concentration increases the number of dye molecules and not the •OH radical concentration, and so the removal rate decreases [7].

For determining the mineralization capacity of Remazol RED F3B by the process, COD analysis was undertaken and it showed that there was 72.3% COD reduction in 80 minutes for Fenton process and 92.8% COD reduction in 50 minutes for photo-Fenton process. TOC removal was also evaluated and it yielded the result that the TOC removal for the Fenton and photo-Fenton processes was found to be 61.3% and 78.8% respectively under identical operational conditions.

For the Fenton and photo-Fenton reactions, the rate of RRF3B degradation was lower at low temperatures, and the extent of degradation was highest over the range 30-40°C. The dye degradation rate decreased at temperatures over 50°C, due to decomposition of H_2O_2. The obtained color, COD, and TOC removal efficiencies for the Fenton process are 98.6, 87.6, and 72.9%, respectively. For the photo-Fenton process at 40°C, these efficiencies are 99.4, 94.0, and 81.5%, respectively [7]. Above 40°C, the removal efficiencies decreased considerably. This may be due to the decomposition of H_2O_2 at higher temperatures.

The paper concludes that Fenton and photo-Fenton processes can be a powerful means of decolorization and mineralization of azo-dyes, the photo-Fenton process being the better of the two processes. The author also stated that the photo-Fenton process is more beneficial in decolorization than COD and TOC removal.

Fig. 4 (a) Effect of Initial Concentration of Fe^{2+} on the Decolorization Efficiency of the Fenton Process.



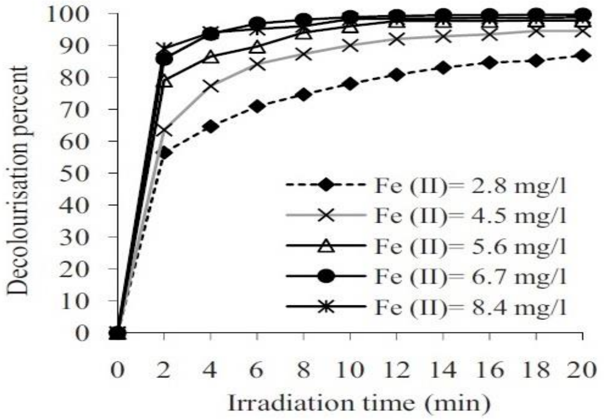


Fig. 4 (b) Effect of initial concentration of Fe^{2+} on the decolorization efficiency of the photo-Fenton process.

6. Fenton's oxidation in the treatment of distillery effluents

M. General

Martins, C.R., *et al* studied the effectiveness of the Fenton's process in treating the wastewaters of distillery industry in combination with flocculation and Ozonation. The distillery wastewater has high organic content. The use of Fenton's process is known to be effective for treatment of industrial effluents.

N. Experimental Procedure

The entire procedure involves three phases- Flocculation, Fenton's process and Ozonation. The process of flocculation was carried out for a sample volume of 200-mL to which 0.1g of anionic flocculant (923PWG, Polyacrylamide) was added. The reactor volume was 500-mL. Stirring was carried out with the help of baffle paddles at 300 revolutions per minute for 5 minutes. After five minutes, the speed was reduced for the next 25 minutes.

Fenton's process was also carried out on a 200-mL sample in a 500-mL reactor. Ferrous sulfate was added and mixed into the sample. The addition of H_2O_2 in the reactor marked the initiation of the reaction. A magnetic stirrer was used to mix the contents thoroughly. H_2O_2 used was of 50% strength and of industrial grade. Use of 3 M NaOH was made to alkalinize the sample to stop the reaction at predetermined times for sample analysis.

For Ozonation, a semi-batch reactor of 500-mL was used. Ozone was supplied in a continuous stream which was generated by a gas generator. The flow was 500 mL/min. with the ozone concentration of 40 g/m^3. A gas diffuser was used to bubble ozone through the liquid bulk of the sample.

O. Analysis and Findings

The analysis included COD, BOD_5, TOC, Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), Total phenolic content (Tph), Respirometry, and Luminescence. The results were taken twice and maximum deviations between analyses were found to be 8%, 20%, 2%, 5%, 10%, 9%, 8%, and 2% respectively.

In case of flocculation it was found that a pH in the range of 2.0 to 3.0 yielded maximum TSS separation and it decreased when the pH was increased further. Alkaline conditions were the most unfavorable for solids separation. TSS and VSS content was reduced to 350 mg/L and 50 mg/L respectively. However, less than 2% COD reduction occurred in this process. The sample still needed oxidation process as the organic charge was found to be 19,650 mg/L of O_2.

As far as Fenton's process is concerned the ideal pH of the solution was considered to be in the range of 3.0 to 4.0. For more acidic conditions, an inhibition on the formation of Fe^{3+} per-oxo-complexes occurs, reducing the ability for catalyst regeneration. Besides, for higher pH, H_2O_2 decomposition into useless water and oxygen is enhanced without significant generation of hydroxyl radicals [8]. The H_2O_2 concentration adopted for COD reduction analysis was 0.25 M to 1.50 M. There are papers which refer to the optimum concentration of H_2O_2 for maximum efficient treatment of distillery wastewater as 1.20 M. In a treatment time of 60 minutes, the authors found out that COD reduction increased with an increase in the concentration of H_2O_2. The experiment involving 1.5 M of H_2O_2 had the best COD removal, and a higher BOD_5 decrease, yet resulted in an effluent characterized by a low biodegradability (BOD_5/COD =0.22) even below the initial value (0.44), which makes the effluent untreatable by biological processes (<0.4) [8]. The effect of $[H_2O_2]/[Fe^{2+}]$ was also studied and it resulted that a ratio of 30:1 gave the most satisfying results. However, the biodegradability i.e. BOD_5/COD of the wastewater was found to be the most in case of $[H_2O_2]/[Fe^{2+}]$ of 10:1. Fig. 5 shows the effect of various ratios of $[H_2O_2]/[Fe^{2+}]$ on the COD degradation efficiency of Fenton's process.

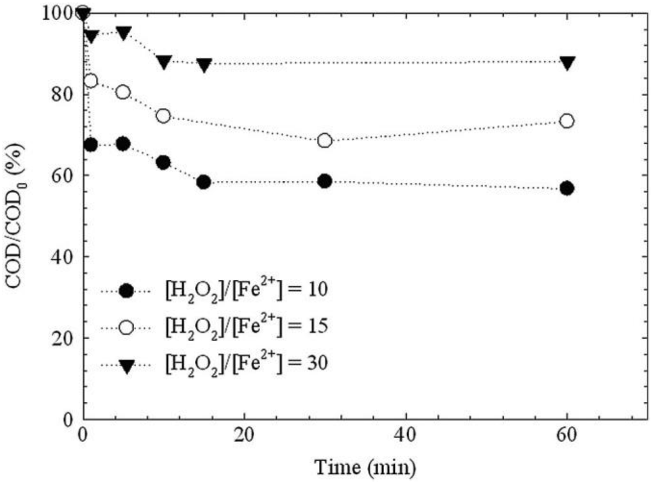


Fig. 5 Effect of $[H_2O_2]/[Fe^{2+}]$ on COD reduction in Fenton's process.

The paper also states that the Fenton's process is much more efficient than Ozonation in reducing the COD. Ozonation caused a COD removal of only less than 10%. The paper concludes that the increase in the $[H_2O_2]/[Fe^{2+}]$ increases the COD abatement, but simultaneously it reduces the biodegradability of the solution thus rendering further biological processes ineffective.

7. Summary

The referred research papers provide good insight in the aspect of chemical oxidation of industrial effluents and also some specific emerging as well as known pollutants present in groundwater using the Fenton's reagent. Almost all the literature agrees upon the requirement of acidic conditions especially of pH 3.0. It is believed that the free hydroxyl radical generation is of maximum order at this pH. The initial concentrations of hydrogen peroxide, ferrous ions, and the molar ratio of hydrogen peroxide and ferrous ions also play important roles in determining the rate of COD reduction as well as degradation rate of the pollutants in water. Another important parameter in determining the effectivity of the reagent is the initial concentrations of the pollutant in the water or wastewater. Every wastewater sample has an optimum pollutant concentration value which can be degraded using the Fenton's reagent. The result of photo-Fenton process is definitely more impressive than the normal Fenton process. However, the biodegradability of the resulting solution after treatment is not evaluated in most of the papers. The effluent after treatment may or may not be suitable for biological degradation. Combination of Fenton’s process with other conventional as well as advanced oxidation treatment methods may yield desired results.

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