Distillery Waste Water Treatment by Advanced Oxidation Process (Fe^{3+}/H_2O_2 or H_2O_2/UV or Fe^{3+}/H_2O_2/UV))

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ABSTRACT

Advanced Oxidation Processes (AOP) involving hydroxyl radicals, which are one of the strongest inorganic oxidants next to elemental fluorine, have been extremely effective in the destruction of organic pollutants. These advanced oxidation process (AOP) generally use a combination of oxidation agents (such as H_2O_2 or O_3), irradiation (such as UV or ultrasound), catalysts (such as metal ions or photo catalysts) and radiolysis (such as gamma irradiation or electron beam) as a means to generate hydroxyl radicals ..

The hydroxyl radicals can be generated by different oxidation processes, such as ozone (O3), ultraviolet (UV), Hydrogen peroxide combined with ultraviolet radiation (H_2O_2/UV), Fenton reagent (Fe^{2+}/H_2O_2) , photo- Fenton process UV + Fe II + H_2O_2 and heterogeneous photo catalysis using semiconductors such as zinc oxide (ZnO) but mainly titanium dioxide (TiO_2) - TiO_2/UV and (H_2O_2/UV) process Ultrasonic technology as an innovative technology may be used for water and wastewater treatment for pollution removal. This technology acts as an advanced oxidation process.

Application of this technology leads to the decomposition of many complex organic compounds to much simpler compounds during physical and chemical compounds during cavitation process. Advanced Oxidation Processes (AOP) using ozone, H_2O_2, ultrasound (US), ultraviolet radiation (UV), Fenton's reagent (FeII+H_2O_2) alone or in combination involving hydroxyl radicals are considered as possible methods of clean and ecologically safe remedial treatment for the degradation of organics.

Keywords - ***Advanced oxidation process, Photo Penton Process, wastewater Treatment, Fenton reagent.***

1. INTRODUCTION

Sugarcane molasses is the byproduct of sugar industry which generated during sugar production, sugarcane molasses contain 50 % fermentable sugar and about 4 to 10 kg of molasses which is required for 1 l of alcohol production. Molasses is the dark brown, putrid, viscous liquid. Sugar molasses is the most common feed stock for industrial fermentation processes, molasses are diluted 1- 3 fold for effective fermentation process and purification of spirit. Spent wash is highly acidic, having strong odor, variety of recalcitrant coloring pigment as melanoidins, metal sulfides and phenolics are responsible for dark brown color of spent wash.

Due to rapid growth of population and industrialization the requirement of water increase but the natural source of water which is useful for the domestic and industrial uses is very limited. From the industrial process the large amount of waste water is coming out treatment of this waste is necessary to protection of environment and human being from harmful effect.

1.1 Necessity of wastewater treatment

1. Protection Of the Environment from pollution.
2. Protection Of Human Health by harmful effect.
3. Avoid Shortage Of the Water for Domestic as well as Industrial use.
4. Save the Earth life.
5. Increasing awareness about the environment.

Heterogeneous (UV + Fe II + H_2O_2) and UV + H_2O_2 homogeneous photochemical reactions ultrasonic irradiation combined son chemical and photochemical techniques with variable results. Photo-Fenton and TiO_2- mediated photo catalytic degradation of several surfactants using solar energy has been reported. . . . . [1].

AOPs like photo Fenton and Electro Fenton treatment was found to be feasible for soil remediation at natural soil pH. Soil consists of iron able to catalyze hydrogen peroxide without addition of extra Fe2+ in the Fenton treatment. By the previous studies combined chemical and biological treatment (the Fenton treatment or ozonation with moderate doses of chemical oxidants and biodegradation) was more effective than either one alone and can be used as a successful treatment technology for contaminated soil remediation. . . . . [2].

In 1894 by its inventor H.J.H. Fenton. Many metals have special oxygen transfer properties which improve the utility of hydrogen peroxide. The most common of these is iron which, when used in the prescribed manner, results in the generation of highly reactive hydroxyl radicals (OH). . . . . [7].

Photon catalytic oxidation and Fenton oxidation process by Grcic et al. (2010) investigated for the treatment of wastewater containing reactive azo dyes, Reactive Violet 2 and Reactive Yellow 3 and inferred that Fenton process rate is faster than the photo catalytic process.

In 2004 experimental study by Bali et al. Various dye solutions like Reactive Black 5, Direct Red 28 and Direct Yellow 12 for treatment using UV, UV/ H_2O_2 and UV/H_2O_2/Fe^{+}_2 process and reported that photo- Fenton process increases the decolorization and mineralization within short irradiation time and distillery effluent treatment using AOP.

Experiment in 2012 observed by Asaithambi et al. ozone assisted electro coagulation for the treatment of effluent, and they observed ozone-assisted electro coagulation was more effective than electro coagulation and ozonation alone.

Study of efficiency of AOPs by Vineetha et al. (2013) the photo degradation of effluent in the presence of solar radiation and the result shows 79 % color removal under the optimum conditions of H_2O_2, pH and catalyst.

In 2008 Experimental study Sreethawong and Chavadej experimented treatment in the presence of immobilized iron catalyst using ozonation, and the result shows that the presence of catalyst enhances the color and COD removal. . . . . [17].

2 .Treatment Process

2.1. Adsorption

Activated carbon is a well-known adsorbent due to its extended surface area, microporous structures, high adsorption capacity and a high degree of surface reactivity. Adsorption techniques are considered to be most effective and proven technology having potential application in both water and waste water treatment. Most of processes industries are prefer this method for waste water treatment.

Sugarcane bagasse and charcoal use an adsorbent for decolorization of distillery spent wash. Activated carbon is a widely used adsorbent for the removal of organic pollutants from wastewater, but the relatively high cost restricts its usage.

2.2.Membrane Treatment

Electro dialysis has been explored for desalting spent wash using cation anion exchange membrane gives 50 -60 % reduction in potassium content. Nanotechnology is effective for COD and Color removal, by using nanotechnology 100 % color is removed and about 97.1 % COD is removed. Nanotechnology is more effective but high capital cost and effect on human health and the environment. Electro dialysis and reverse osmosis process are less economical and pretreatment is required for reverse osmosis.

2.3.Oxidation process

Different oxidation process is used for treatment of waste water, such as ozone, single hydrogen peroxide, Fenton's reagent and ozone combined with hydrogen peroxide. Ozone treatment was able to reduce 76 % of color, a combination of ozone with a low concentration of hydrogen peroxide was able to increase the color removal efficiency up to 89%.

2.4.Electrocoagulation

Color removal efficiency of electro-coagulation is decreases with increase in concentration of melanoidin; he also mentioned that electrodes consumption increases with increase in concentration of melanoidin.

Efficiency of the chemical oxygen demand (COD) removal retarded with increases in pH, spacing between electrodes plays important role in decolorization of melanoidin, COD removal efficiency accelerate with increase in the distance between electrodes . Acidic condition is more favorable for treatment of distillery spent wash due to decreased production of chlorine or hypochlorite at higher pH.

2.5.Aerobic Treatment

Anaerobic treatment is the primary treatment to treat distillery spent wash but still contain high concentration of COD, suspended solids, chloride, and biochemical oxygen demand. Effluent also contain high ration of C: N which affect the fertility of the land as C: N ratio reduces mineral nutrients so cannot be disposal and discharge directly.

The followings are the various goals of waste water treatment

1. To control pollution
2. Prevention of infectious, chronic and hazardous diseases
3. Protecting environment
4. Reusing water for gardening and agriculture purpose.
5. Increase the water resources

2.5 Fenton process

This is the most important oxidation processes in effluent treatment of recalcitrant compounds and the process of oxidation of organic substrates by iron (II) and hydrogen peroxide is called the Fenton chemistry and it was first described by H.J.H. Fenton. Simple reaction (of Fe2+ions with H_2O_2) which was observed by H.J.H. Fenton over 110 years ago proves to be very difficult to describe and understand. He first observed the oxidation of tartaric acid by H_2O_2 in the presence of ferrous iron ions. In 1894 that several metals have a special oxygen transfer properties which improve the use of hydrogen peroxide.

The production of iron sludge can be reduced. Photo-Fenton reaction does not produce new pollutants and only small amount of iron salt is needed. The radiation sources of the Photo-Fenton process Using UV or solar light can increase the mineralization degree and make dark Fenton process more efficient by the photoreduction of $Fe(OH)^{2+}$ which leads to additional $\cdot OH$ production and continuous regeneration of Fe^{2+}.

2.5.1 Theory of Fenton Process

Catalytic oxidation of tartaric acid in the presence of ferrous salts and hydrogen peroxide was reported by Fenton and hydroxyl radicals are generated from the reduction of hydrogen peroxide.

$$\mathbf{Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\bullet} + OH^{-}}$$

High concentrations of Fe2+, the hydroxyl radicals formed can oxidize other ferrous ions to ferric ion as follows

Fe^{2+}+ $OH^{•}$ → $Fe_3{}^{+}$+ OH^{-}

Hydrogen peroxide (H_2O_2) as the oxidation agents and Fe salts acts as catalyst for Fenton process and high concentration of certain refractory contaminants and the low rate of reactions at reasonable H_2O_2 concentration.

Using transition metal salts, ozone and also UV-light and use H_2O_2 and metal salts are classically known as Fenton process.
The reaction between $H_2O_2$and iron salts it will results in the formation of hydroxyl radicals, HO•.

Effects of various parameters on the Fenton Oxidation Process

1. Mass Ratio & Dosage of H2O2/Fe2+
2. Iron type (Ferrous Fe2+ – Ferric Fe3+)
3. Temperature.
4. pH
5. Reaction Time.
6. Adding chemicals in steps.
7. The Reaction is followed by neutralization.
8. Characteristic of Wastewater treated.

Types of Fenton Process

1. Electro-Fenton Process
2. Photo-Fenton Process

2.5.2 Electro-Fenton Process

Advanced oxidation process used in wastewater treatment technology which is Electro Fenton process. This process is modification of conventional Fenton process. It consists of electrolysis cell that regenerates Fenton reagent by electrochemical reaction between anode and cathode.

In this electrochemical reaction which is combination of two reaction Fenton reaction and electrochemical oxidation (EO) and the reaction works in single reaction chamber. EO is electrochemical reaction as Fenton is a chemical reaction who oxidizes the pollutant by electrochemical process.

Anode Reaction

$Fe^{+3} + e^{-} = Fe^{+2}$

Cathode reaction

$O2 + 2H^{+} + 2e^{-} = H_2O_2$

2.5.3 Photo-Fenton Process

There are many photochemical processes using iron compounds and H_2O_2 that provide alternative ways of generating hydroxyl radicals. Photo-Fenton system and its modifications are very efficient regarding the destruction of organic pollutants. The photo-Fenton reaction is based on the applying of the $Fe^{2+/}Fe^{3+}$ and H_2O_2 reagents with near-UV and visible light.

The production of iron sludge can be reduced. Photo-Fenton reaction does not produce new pollutants and only small amount of iron salt is needed. The radiation sources of the Photo-Fenton process Using UV or solar light can increase the mineralization degree and make dark Fenton process more efficient by the photoreduction of $Fe(OH)^{2+}$ which leads to additional ·OH production and continuous regeneration of Fe^{2+}.

2.6 Effect of various parameters on Photo Fenton Process

1. Effect of pH value

Reaction to be conducted under the conditions of reaction time 60 minutes, $H_2O_2/FeSO_4$ (Fe salt) =1:1 and different pH values results found that low pH has effective for Fenton's reagent, and the best removal efficiency is obtained at a pH =3. At the lower value of pH is better to remove inorganic carbons from waste water as they can scavenge hydroxyl radicals.

As the value of higher pH COD is increasing, the decomposition rate decreases. At high pH formation of Fe (II) complexes with the buffer occurs inhibiting the formation of free radicals Precipitation of ferric oxy hydroxides inhibits the generation of ferrous ions and the oxidation potential of hydroxyl radical is known to decrease with increase in pH.

2. Effect of Reaction Time:

Reaction time is the important factor for treatment process by Fenton. As per experimental studies optimum reaction time is 90 minute demonstrated that the COD decreased gradually to 90 minutes reaction time and then increased. Ferrous iron and hydrogen peroxide with the production of hydroxyl radical was almost complete in 90 minutes.

3. Effect of Fe_2+and H_2O_2 addition:

For this AOP process iron and hydrogen peroxide are two major chemicals determining operation costs as well as efficiency and the dosage of H_2O_2 depends on initial COD. If COD is higher the requires more H_2O_2 and if COD lower than the less H2O2 required.

As per experimental study optimum amount of H_2O_2 obtained is 600-900 mg/l of waste water treated. As value or amount of H_2O_2 contributes to residual H_2O_2 leading to increase in COD. Amount of excess hydrogen peroxide is harmful to many microorganisms and will affect the overall efficiency and hydrogen peroxide present in large quantities acts as a scavenger for the generated hydroxyl radicals. Amount of hydrogen peroxide is to be adjusted so that the entire amount is utilized.

4. Effect of Fe2+ on COD removal:

Usually the rate of degradation increases with an increase in the concentration of ferrous iron but an enormous increase of ferrous iron leads to an increase in the unutilized quantity of ferrous irons, which will contribute to an increase in the TDS content of the effluent stream.

5. Temperature effect

The value of temperature increase rate of degradation also increase at specific value of temperature after some value of temperature degradation stops. So the optimum value of temperature is important. The degradation is better and faster as demonstrated at 60°C.

6. Effect of UV light

As intensity of UV light increase the rate of photolysis of H2O2 increase. Optimum value of UV 400-450 nm after that rate of degradation reduced. Value of UV intensity should be $\lambda < 450$ nm.

Table 2.1 Characteristics of Pre-treated distillery waste water

Parameter	Value
pH	3.67
Total Solids	66,980 mg/l
Total Dissolved Solids (TDS)	14,660 mg/l
Chemical Oxygen Demand (COD)	34000 mg/l

3. MATERIAL AND METHODOLOGY

3.1 Materials

1. Hydrogen Peroxide (H_2O_2)

This is the strong oxidant and its application in the treatment of various inorganic and organic pollutants is well established. H_2O_2 consist of two hydrogen molecules and two oxygen molecules.

2. Fenton's Reagents (Fe salt/ $FeSO_4$ Solution).

Metal salts (e.g. iron salts) which are strong oxidants that is the Fenton's process. Fe^{+3} and Fe^{+2} are used to oxidation of H_2O_2 which decompose or cause of degradation of waste water. The amount of this Fenton reagent is based on the amount used of H_2O_2.

3. Acid Or Alkali

H2SO4 acid or NaOH alkali to be used for Ph maintain of waste water. The optimum Value of pH necessary for the Fenton process.

3.2 Reaction Mechanism Photo Chemical Process

Formation of hydroxyl radical also occurs by the following reactions.

$$H_2O_2 + UV \rightarrow OH\bullet + OH\bullet$$

$$Fe^{3+} + H_2O + UV \rightarrow OH\bullet + Fe^{2+} + H^+$$

This process is combination of Fenton process and UV light could be an interesting allied in dye decolorization due to its capacity to influence the direct formation of •OH radicals. Pollutant degradation could be increased by irradiation of Fenton with UV light (photo-Fenton process).

3.3 Fenton Treatment Procedures

Fenton treatment procedure of waste water was carried out at ambient temperature in the following sequential steps.

1. Waste water sample was put in a beaker and stirred for mixing.
2. The scheduled Fe_2^+ dosage was achieved by adding the necessary amount and Fenton Agent add 1:1 proportion of H_2O_2.
3. A known 200-900 mg/l volume of 35% (w/w) H_2O_2 solution was added in a single step.
4. After fixed reaction time, before carrying out COD tests, pH was adjusted to 8 to remove residual Fe $_2^+$.
5. Settlement was achieved for 30 minutes, and then examination of COD should be done.

6. After settlement check COD of sample per 15 min interval of time.

7. In between continuous stirring process will require.

3.4 Photocatalytic oxidation with UV/H_2O_2 Treatment Procedures

Treatment procedure of waste water was carried out at ambient temperature in the following sequential steps.

1. Waste water sample was put in a beaker and stirred for mixing.
2. Check the parameters of waste water before treatment pH, COD and Color.
3. A known 200-900 mg/l volume of 35% (w/w) H_2O_2 solution was added in a single step.
4. Start the UV light by supply Ac current.
5. After fixed time of measure the take samples and calculate COD.
6. Also calculate reduction in Color from waste water with the help of spectrophotometer.
7. Settlement was achieved for 30 minutes.
8. Sample should take after settlement for COD and Color measurement.
9. In between continuous stirring process will require

3.5 Photocatalytic oxidation with Fe^{2+}/UV/H_2O_2 Treatment Procedures

Treatment procedure of waste water was carried out at ambient temperature in the following sequential steps.

1. Waste water sample was put in a beaker and stirred for mixing.
2. Check the parameters of waste water before treatment pH, COD and Color.

3. A known 200-900 mg/l volume of 35% (w/w) H_2O_2 solution and Fenton reagent was added in a single step.
4. Fenton reagent and H2O2 should added as per proportions.
5. Start the UV light by supply Ac current.
6. After fixed time of measure the take samples and calculate COD.
7. Also calculate reduction in Color from waste water with the help of spectrophotometer.
8. Settlement was achieved for 30 minutes.
9. Sample should take after settlement for COD and Color measurement.
10. In between continuous stirring process will require.

4.Result Analysis

4.1 Effect of pH on % COD Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$

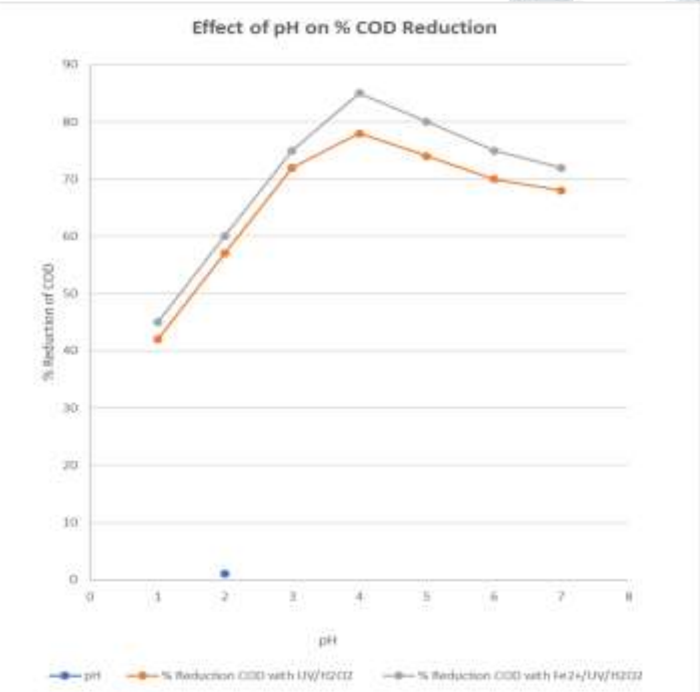


Fig. 4.1 Effect of pH on % COD Reduction

Fig. shows the Effect of pH on % COD Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$. % COD Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 lower than the $Fe^{2+}/UV/H_2O_2$. The optimum value for pH is 4 for both processes.

4.2 Effect of pH on % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$

Fig. shows the Effect of pH on % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$. % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 lower than the $Fe^{2+}/UV/H_2O_2$. The optimum value for pH is 4 for both processes.

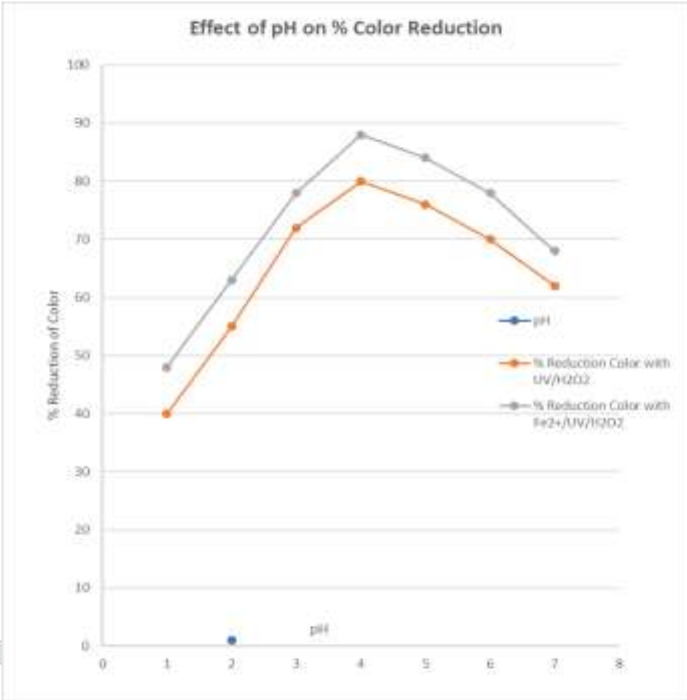


Fig. 4.2 Effect of pH on % Color Reduction

4.3 Effect of time on % COD Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$

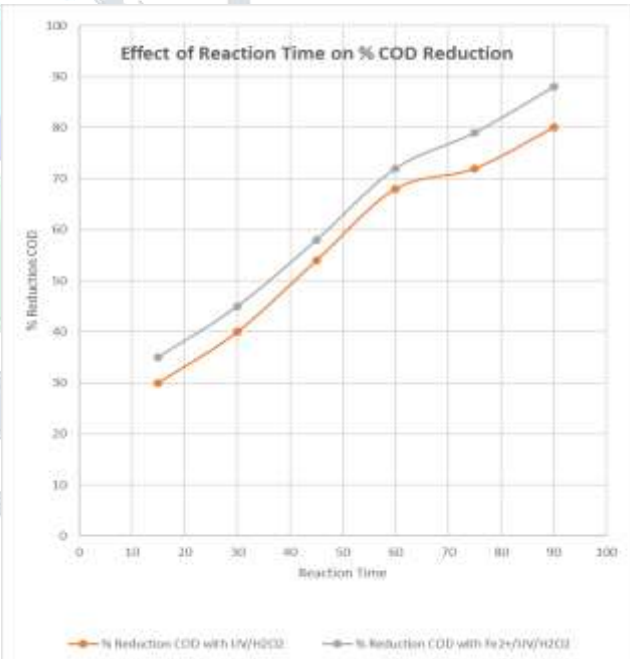


Fig. 4.3 Effect of Reaction Time on % COD Reduction

4.4 Effect of time on % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$

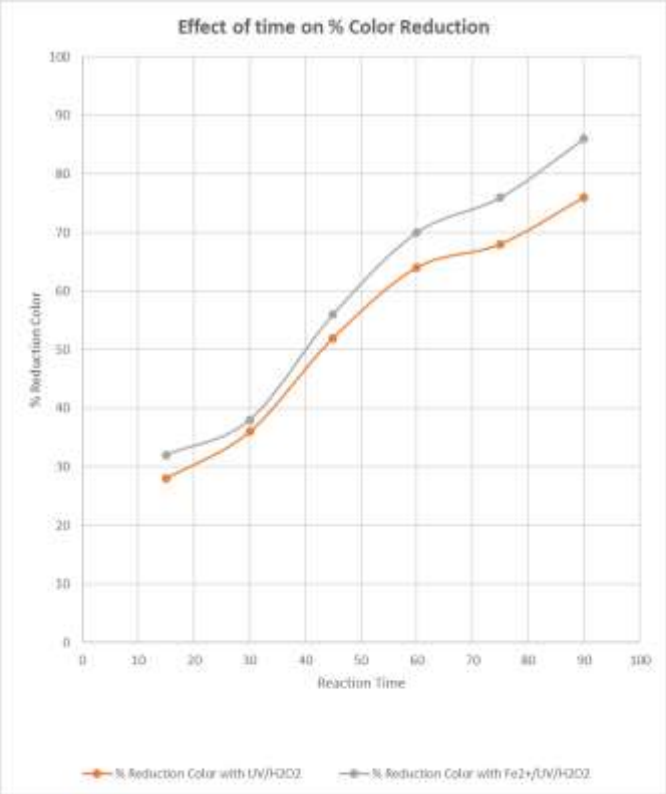


Fig. 4.4 Effect of Reaction Time on % Color Reduction

Fig. shows the Effect of Reaction Time on % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 and $Fe^{2+}/UV/H_2O_2$. % Color Reduction on distillery waste comparing as Photocatalytic oxidation with UV/H_2O_2 lower than the $Fe^{2+}/UV/H_2O_2$. The optimum value for 90 min for both processes.

4.5 Effect of 0.5:1 proportion of H_2O_2: Fenton's Reagent (450:900 mg/l) % COD and Color Reduction on distillery waste for Photocatalytic oxidation with $Fe^{2+}/UV/H_2O_2$.

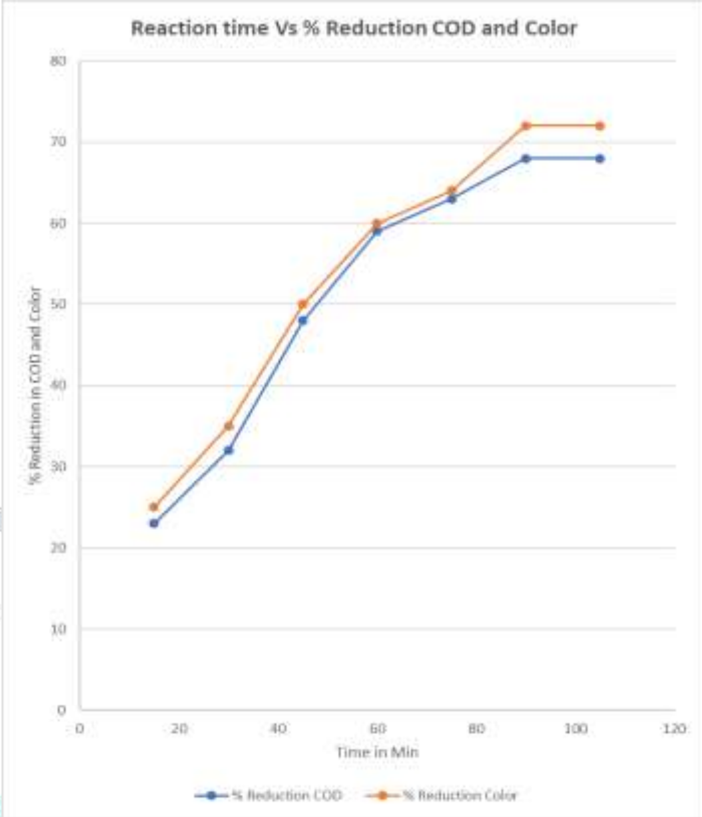
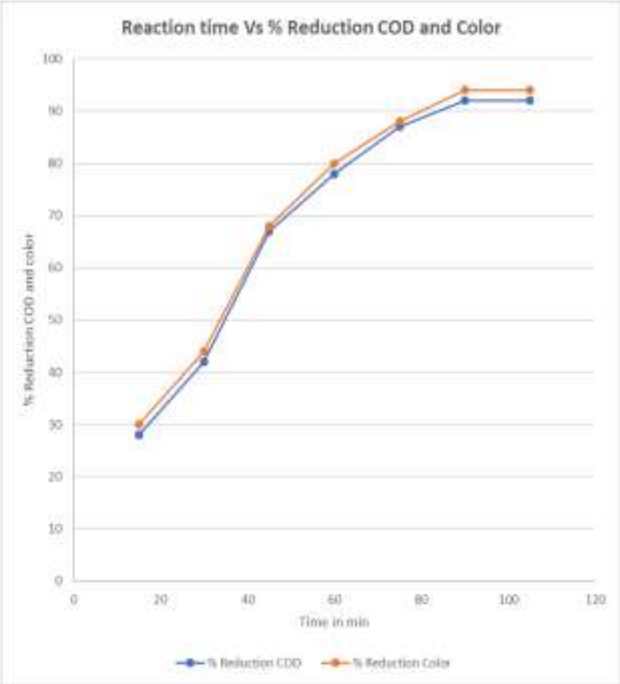


Fig. 4.5 Reaction time Vs % Reduction COD and Color for 0.5:1 proportion of H_2O_2: Fe^{+2}

Fig. shows the % Reduction of COD and Color vs reaction time for 0.5:1 proportion of H_2O_2: Fe^{+2}. From Graph it's clear that % Reduction of COD and Color increase with reaction time. The optimum reaction time is 90 min.

4.6 Effect of 1:1 proportion of H_2O_2: Fenton's Reagent (900:900 mg/l) % COD and Color Reduction on distillery waste comparing as Photocatalytic oxidation with $Fe^{2+}/UV/H_2O_2$

Fig. shows the % Reduction of COD and Color vs reaction time for 1:1 proportion of H_2O_2: Fe^{+2}. From Graph it's clear that % Reduction of COD and Color increase with reaction time. The optimum reaction time is 90 min.



Graph 4.6 Reaction time Vs % Reduction COD and Color for 1:1 proportion of H_2O_2: Fe^{+2}

CONCLUSION

1. There is complete mineralization of organic matter.
2. There is no need for any processing units on the surface.
3. This process reduces organic loading in terms of chemical oxygen demand and done the removal of recalcitrant and toxic pollutants thus allowing for further conventional biological treatment.
4. Fenton process is a relatively economical method since it requires no additional energy when compared to many other AOPs and both iron and hydrogen peroxide are relatively cheap and safe.
5. The reactions are efficient at low pH-levels (<6) - which is difficult to maintain.
6. In some cases chemical oxidation may even lead to increased toxicity due to the formation of even more toxic oxidation by-products.
7. The Fenton Process for waste water treatment shows better results over the conventional method.
8. The Fenton Process can be used as a tertiary treatment to waste water.
9. The other parameters such as TDS, COD, BOD shows effective changes over conventional method.
10. Waste water samples are collected from industry after giving primary and secondary treatment and Fenton Process with any convectional method gives better results.

FUTURE SCOPE AND BENEFITS

Future Scope

•AOPs can be adopted to treat waste water.
• AOPs improve the efficiency of conventional method.
• AOPs can be used as an additional treatment to treat waste water.
• This process can make waste water for reusable as process water by removal of COD and Color from waste water.
• Destroys and removes bacteria, viruses and cysts.

Benefits:

• Capital cost significantly less than conservative technologies.
• Operating cost significantly less than conservative technologies.
• Low power requirements.
• Low maintenance.
• Minimal operator attention.
• Consistent and reliable results.

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