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Distillery Waste Water Treatment by Electro Fenton Process

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ABSTRACT

Sugarcane industry (Distilleries) are one of the most polluting industries which generating large volumes of high strength wastewater. Waste water produced from distillery containing highly color, COD, BOD, TDS and other organic matter. During fermentation of molasses 8 -10 L of spent wash generated per liter ethanol production. AOPs are efficient over the conventional methods for distillery and other industrial waste water treatments. AOPs like Electro Fenton and Photo Fenton are used for distillery waste water treatment for the removal of color and COD from the waste water and both processes are efficient. Electro Fenton process is the combination of Fenton and Electro Chemical oxidation process. EF process suitable for the all types of industrial waste water treatments. Various analysis shows the effect reaction time, voltage, pH, Fenton reagent dosage and current density etc. on the various characteristics of waste water. Distance between the electrodes in this method is selected between 3 cm and 3.5 cm which causes a 4% increase in the removal efficiency. Optimum value of 12-30 V DC after that rate of degradation reduced. optimum reaction time is 90 minutes demonstrated that the color and 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. Reaction to be conducted under the conditions of reaction time 30-120 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. ***Keywords: AOPs, Decolorization and COD, Reduction, Electro Fenton Process, Sugar Cane Distillery Waste Water Treatment.***

1. INTRODUCTION

Waste water from distillery contain mostly dark brown colored recalcitrant compounds collectively termed as melanoidin polymers which are the product of maillard reaction between the amino acids and carbonyl groups present in molasses. Distillery waste water have very high Chemical Oxygen Demand (COD) and these effluents are environmental hazards when released in water bodies they cause oxygen depletion and associated problems. Spent wash produce from the distillery poses a serious threat to water quality in several regions of the country. After some treatment disposal on land is equally detrimental causing a reduction in soil alkalinity and inhibition of seed germination. Distillery spent wash owing to the presence of melanoidins and color. For production of 1 l alcohol (ethanol) around 8 – 15 l of spent wash generated. Characteristic of waste water generated from the distillery industry is depending up on the quality of molasses and fermentation process.

Various Conventional Methods

1. Biological flocculation
2. Nano filtration
3. Activated carbons
4. Bio electrochemical process
5. Ozonation-based process electro oxidation
6. Membrane-based Nano filtration
7. Reverse osmosis.
8. Aerobic and Anaerobic process
9. Electro Coagulation
10. Chemical Coagulation

Environmental Hazards of Distillery Wastewater

1. Waste water from distillery has very high BOD and COD.

2. High BOD/COD ratio.

3. Large amount of inorganic substances (Nitrogen, Potassium, Phosphates, Calcium)

4. Spent wash disposal in environment ishazardous and has high pollution potential.

5. Distillery effluent toxic effect on common guppy.

6. It also impact of distillery effluent on carbohydrate metabolism of fresh water fish.

7. Distillery effluent disposed on land is equally hazardous to the vegetation.

8. Spent wash also inhibit seed germination, cause soil manganese deficiency

9. Distillery effluent caused damage agricultural crops.

Types of AOP

Non-Photochemical	**Photochemical**
Homogeneous Processes	
Alkaline media ozonation (O_3/HO^-)	Vacuum ultraviolet (VUV)
Ozonation with Hydrogen peroxide ($O3/H_2O_2$)	UV/H_2O_2
Fenton (Fe^{2+} or Fe^{3+}/H_2O_2)	UV/O_3
Electro-oxidation (EF)	$UV/O_3/H_2O_2$
Wet air oxidation (WAO)	Photo-Fenton (Fe^{2+} or $Fe^{3+}/H_2O_2/UV$)
Supercritical water oxidation (SCWO)	
Heterogeneous Processes	
Catalytic wet air oxidation (CWAO)	SnO_2/UV, TiO_2/UV, $TiO_2/H_2O_2/$ UV

Electro-Fenton Process

Advanced oxidation process used in wastewater treatment technology which is Electro Fenton process. EF 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.

Advantages of the EF Process over Fenton

1.Controlled generation of Fenton's reagent.

2. Avoiding thus the risks related to transport, storage and handling of H_2O_2

3. Eliminate parasitic reactions that wasting OH.

3. Total master ship of the processing by current or potential control.

4. Possibility of controlling the degradation kinetics and performing mechanistic studies.

5. Almost total mineralization of organics including the intermediates.

Electro-Fenton Method is Used for

1. To removal of many kinds of recalcitrant pollutants

2. To treatment of landfill leachate

3. Phenol degradation

4. Reduction of Turbidity, BOD, COD and Color from various types of wastewater.

5. Treatment of various types of industrial waste water.

Application of EF for Wastewater Treatment

1. Chemical Industry

Nowadays problem of industrial wastewaters, not only in terms of discharge volumes but also looking at the hazardous nature of many of the pollutants found in the effluents. Due to the increase in various rules and regulation regarding waste treatment have enforced the application of advanced technologies.

2. Pharmaceutical Industry

From these industrial wastewaters has always been troublesome owing to the wide variety of chemicals used in drug manufacturing which leads to wastewaters of variable composition and fluctuations in pollutant concentrations. The pharmaceutical industry is in most complex organic chemicals that are resistant to biological degradation.

3. Pulp and Paper Industry

Number of effluents resulting from the different stages of paper making and some of these pollutants are naturally occurring wood extractives (tannins, resin acids, lignin, etc.), others are xenobiotic compounds that are formed mostly in pulp manufacture (chlorinated lignin, phenols, dioxins and furans, among others).Paper and pulp waste water / effluents are highly colored and contain high organic loads and Fenton oxidation is effective for the treatment of pulp bleaching effluents.

4. Textile Industry

This sector known for its high-water consumption as well as the amount and variety of chemicals used throughout the different operations. Textile effluents are in a great part due to color associate large environmental problem. Nature of textile wastewaters from the dyeing and finishing stages is mainly attributable to the extensive use of various dyestuffs and chemical additives (such as polyvinyl alcohol, surfactants, etc.). Textile effluent characterized by high organic matter content like COD and color. By the various studies Fenton process is effective in removing COD and color.

5. Food Industry

Waste water treatment of food industry by the Fenton technology has also proved to be effective and includes wastewaters from olive oil extraction plants, commonly named „olive mill wastewaters" and wastewaters generated by the table olive producing industry. The oily juice is extracted from the fruit through simple milling or, more recently, by centrifugation. Previous treatment requires tabling olive production in order to eliminate the bitterness of the fruit, due to the presence of poly

6. Landfill Leachates

Landfill leachates vary greatly depending on the type of wastes and the age of the landfill. For this type of waste treatment biological processes including anaerobic and aerobic processes have shown to be very effective in the early stages when dealing with domestic wastes because the BOD/COD ratio of the leachate has a high value.

7. Biomedical Application

There is important application of Fenton reaction in biology because it involves the creation of free radicals by chemicals and metal ions such as iron and copper donate or accept free electrons via intracellular reactions and help in creating free radicals. Fe^{+3} form and must be reduced to the Fe^{+2} form to take part in Fenton reaction.

8. Dye-Process Industrial Waste

Development of on-site wastewater treatment technologies suitable for dye process industries such as the wood-floor sector and nature of their activities these industries generate lower volumes of highly polluted wastewaters after cleaning activities. EF AOPs process is potentially feasible options for treatment of these wastewaters. EF is effective in treating various industrial wastewater components including aromatic amines, a wide variety of dyes, pesticides, surfactants, explosives as well as many other substances and effective for the destruction of toxic wastes.

9. Pre-Treatment to wastewater, Sludge or Contaminated Soil

1. Organic pollutant destruction
2. Toxicity reduction
3. Biodegradability improvement
4. BOD / COD removal
5. Odor and color removal

2. LITERATURE REVIEWS

In 2015, Experimental studied by Charles David, M. Arivazhagan and Fazaludeen Tuvakara EF process, a maximum of 44% color reduction was noticed at pH 3 for a treatment time of 4 h. 4 A current was supplied to the electrodes which are placed with 0.5 cm of inter-electrode distance and the batch reactor was agitated at 400 rpm for proper mixing of the reagents. With increasing the pH to neutral, color removal efficiency decreased. Fenton process is more efficient at acidic conditions. Most studies have also reported that the optimum pH of electro Fenton process was at pH 2.8–3. The color removal gradually increased and attained constant after 3 h of electrolysis time.[1] **The decolorization efficiency directly depends on the concentrations of hydroxyl** and metal ions generated by the electrodes and longer electrolysis time leads to more generation of ions. The generation of H_2O_2 at the cathode depends on current intensity. Higher current intensity increases the amount of H_2O_2 produced, thus increasing the number of hydroxyl radicals in the electrolyte which are highly reactive and responsible for the degradation of recalcitrant pollutants. The efficiency increased from 21% to 44% with an increase in Fe^{2+} dosage from 5 to 20 mg/L. [1] **Treatment of the wastewater was carried out by an electrochemical batch reactor** with two iron electrodes. EF trials for different H_2O_2 doses 313, 626, 940 and 1253 mg/L which are equal to 25%, 50%, 75% and 100% of the theoretical H_2O_2 dose were performed electrical power 24.0W as 0.75 A and 31.5 V with pH=3.0. and electricity consumption for decreased COD mass was estimated. The optimum result in terms of the energy consumption and the chemical consumption of the dose of 313 mg H_2O_2 /L, the efficiency of COD removal was 86%. EF process can be used as an alternative treatment for the textile wastewaters. In the case of the presence of 1253 mg/l H2O2 (100% theoretical dose) and the application of electrical power of 24.0 W, the removal of COD was 75% in 30 min, 67% in 20 min and 25% in 10 min. the efficiency of COD reduction is changed between 41-99% based on different variables such as wastewater type, energy consumption, reaction time.[3] **EF is a promising method and it is more economical, efficient, environmental friendly to remove organic matters.** EF is good for removal of COD, TOC, Color and it is alternative method for treatment of wastewater containing synthetic dyes due to this efficient and low operating cost and environmentally friendly method. The distances decrease between the electrodes cause to decrease of the ohmic drop through the electrolyte and then an equivalent decreases the electrical conductivity and voltage. The electrode distance between 1.3 and cm. H_2O_2 Concentration is one of the most important factors in EF process and H_2O_2 Concentration dosage increases the hydroxyl radicals are also increased.[4] **The efficiency of Fenton process combined with coagulation for treatment of** wastewater. Parameters affecting the Fenton process, such as pH, dosages of Fenton reagents and the contact time were determined at pH 4, 0.8 ml/L H_2O_2, 25 mg/l FeSO4 and 40 minutes contact time. The coagulation using fenugreek and ferrous sulfate ($FeSO_4$) was beneficial to improve the Fenton process treated effluent in reducing the flocs settling time, enhancing turbidity, COD and BOD removal. The overall turbidity, COD and BOD removal reached 99%, 63% and 39.5% under selected conditions.[5] **By experimental analysis shows the feasibility of the electro-Fenton process to** generate reagent species (Fe^{2+}/H_2O_2) was assessed as a potentially more economical to produce reclaimed water. Combined wastewater was treated in discontinuous and continuous reactors at pH = 3.5. The discontinuous reactor was a 2 L electrochemical laboratory cell fitted with concentric graphite and iron electrodes. Several tests were carried out at different conditions of reaction time (0–60 min) and electrical current values (0.2–1.0 A) in the discontinuous reactor. The best operating conditions were 60 min and 1 A without filtration of effluents COD, turbidity and color removal were 65–74.8%, 77–92.3% and 80–100% respectively.[6] **The EF system showed up to 96% of COD removal with increasing current density yet a current density of 16 mA/cm2 was used.**

COD removal efficiencies of 65, 91 and 96% were achieved for 8, 16, and 24 mA/cm2 of current density. EF and anodic oxidation at pH 3 was used to produce •OH radicals and for effective with initial concentration of 5 mM was used for 25 min. [7] **The best removal efficiencies were obtained when the treatment time was 40** minutes. COD were achieved 86.84% removal efficiency. the effect of distance between the electrodes on the removal efficiency of COD. If the distance between the electrodes in this method is selected between 3 cm and 3.5. cm this causes a 4% increase in the removal efficiency and gives rise to energy consumption and operational cost of the method. Thus, the minimum distances should be selected. The optimum distance between the electrodes was 3 cm, which resulted in 68% of COD removal at a pH of 3, voltage of 15 volt, an H_2O_2 concentration of 150 mol/l and a treatment time of 60 min. [13].

Characteristics of Distillery Waste Water

Parameter	Value
pH	4 - 4.5
TDS	65000-100000 mg/l
COD	80000 -125000 mg/l
BOD	40000- 50000 mg/l

Table 3.1 Characteristics of DWW

Materials

1. Hydrogen Peroxide (H_2O_2)

This is the strong oxidant and its application in the treatment of various inorganic and organic pollutants. 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} is 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 amount 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.

4. Electrodes

Iron or Aluminum electrode are used for the electro Fenton process.

Electro Fenton 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. 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. Start the DC current supply. (Readings can take at different DC supply 12 and 24 V)

5. After fixed reaction time, before carrying out COD tests, pH was adjusted to 8 to remove residual Fe^{2+}.

6. Settlement was achieved for 30 minutes.

7. For every 30 min time of intervals withdrawal of sample and examination of % of Color and %COD should be done.

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

9. In between continuous stirring process will require.

4. RESULTS AND DISCUSSION

Effect Of 12 V and 1 Amp On DWW

After collecting the samples of waste water and giving treatment EF methods for every 30 minutes interval are results obtained shown in table 5.2. Experiment carried out at 12 V and 0.5 Amp and calculate % reduction in COD and Color for distillery waste water. Table 5.2 shows the effect of 12V and 1 Amp on % reduction of COD and Color at different time intervals 30 min up to 120 min.

Sample No.	Time (Min)	% Reduction Color	% Reduction COD
1	30	32	36
2	60	48	52
3	90	56	68
4	120	72	76

Table 4.1 Effect Of 12 V and 1 amp on DWW

4.2 Effect Of 24 V and 2 Amp On DWW

After collecting the samples of waste water and giving treatment EF methods for every 30 minutes interval following are results obtained for all the parameters values. Experiment carried out at 24 V and 2 Amp and calculate % reduction in COD and Color for distillery waste water. Table 5.4 shows the effect of 24 V and 2 Amp on % reduction of COD and Color at different time intervals 30- 120 min.

Sample No.	Time (Min)	% Reduction Color	% Reduction COD
1	30	36	40
2	60	54	64
3	90	68	75
4	120	80	86

Table 4.2 Effect Of 24 V and 2 Amp On DWW

Effect of Various Parameters

Effect of pH value

Reaction to be conducted under the conditions of reaction time 30-120 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.

Effect of Reaction Time:

Reaction time is the important factor for treatment process by Fenton. As per experimental studies optimum reaction time is 90-120 minute demonstrated that the color and COD decreased gradually to 90-120 minutes reaction time and then increased. Ferrous iron and hydrogen peroxide with the production of hydroxyl radical was almost complete in 90-120 minutes.

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.

Effect of Fe^{2+} on COD and Color 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 COD and Color content of the effluent stream.

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.

Effect of Voltage

As applied voltage increase the rate of photolysis of H_2O_2 increase. Optimum value of 12-30 V DC after that rate of degradation reduced. Value of DC voltage should be less than 40V DC supply.

4.3.7 Effect of Distance between the Electrodes

Effect of distance between the electrodes on the removal efficiency of COD and Color. Distance between the electrodes in this method is selected between 3 cm and 3.5 cm which causes a 4% increase in the removal efficiency and gives rise to energy consumption and operational cost of the method. Thus, the minimum distances should be selected. As distance between two electrodes as 3cm which resulted in 68% of COD removal at a pH of 3, voltage of 15 volt, an H_2O2 concentration of 900 mg/l and treatment time of 60 min. The distance between the electrodes is an important operation condition for the electrodes which affects the removal of pollutants. If distance between decreases the electrodes leads to a decrease in the ohmic drop through the electrolyte and then decreases the cell voltage and energy consumption.

CONCLUSION

There is complete mineralization of organic matter and no need for any processing units on the surface. EF 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. Electro Fenton process is a relatively economical method other AOPs and both iron and hydrogen peroxide are relatively cheap and safe. The reactions are efficient at low pH-levels which is difficult to maintain. In some cases, chemical oxidation may even lead to increased toxicity due to the formation of even more toxic oxidation by-products. Electro Fenton Process for waste water treatment shows better results over the conventional method. Electro Fenton process can be used as a tertiary treatment to waste water. The other parameters such as TDS, COD, BOD and color shows effective changes over conventional method.

Various analysis shows the effect reaction time, voltage, pH, Fenton reagent dosage and current density etc. on the various characteristics of waste water. Distance between the electrodes in this method is selected between 3 cm and 3.5 cm which causes a 4% increase in the removal efficiency. Optimum value of 12-30 V DC after that rate of degradation reduced. optimum reaction time is 90 minutes demonstrated that the color and 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. Reaction to be conducted under the conditions of reaction time 30-120 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.

SCOPE AND BENEFITS

Future Scope

- Electro Fenton can be adopted to treatment of waste water.
- To improve the efficiency of conventional method.
- Electro Fenton can be used as an additional treatment to treat waste water.
- Electro Fenton process can make waste water for reusable.

System Capabilities

- Removes heavy metals.
- Removes suspended and colloidal solids.
- Destabilizes oil and other emulsions.
- Removes complex organics.
- Remove COD and Color.

BENEFITS

- Treats multiple contaminants.
- Sludge minimization.
- Capital cost significantly less than conservative technologies.
- Operating cost significantly less than conservative technologies.
- Low maintenance.
- Minimal operator attention.
- Consistent and reliable results.

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NOMENCLATURE

AOP – Advanced Oxidation Process

BOD – Biological Oxygen Demand

COD – Chemical Oxygen Demand

DWW – Distillery Waste Water

EF – Electro Fenton

SS – Suspended Solids

TDS – Total Dissolve Solids

WHO – World Health Organization?