IJNRD.ORG ISSN : 2456-4184
INTERNATIONAL JOURNAL OF NOVEL RESEARCH AND DEVELOPMENT (IJNRD) | IJNRD.ORG
An International Open Access, Peer-reviewed, Refereed Journal

"Synergistic Purification of Distillery RO Reject Water: Enhanced Treatment via Fenton-Based Advanced Oxidation and Electrocoagulation with Aluminum and Iron Electrodes"

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Abstract: This research delves into the enhancement of an integrated treatment process that merges Fenton based advanced oxidation with electrocoagulation to effectively address distillery condensate RO reject. The Fenton-based AO process involves the generation of hydroxyl radicals **(-OH**) through the reaction of iron salts with hydrogen peroxide **(H_2O_2)**, facilitating the degradation of organic pollutants. The study scrutinizes the impact of these factors on the breakdown of organic pollutants, the decrease in chemical oxygen demand **(COD)**, and the overall energy consumption of the treatment process. The integrated system performance is assessed through a series of batch experiments. Experimental assessments demonstrate improved removal efficiencies compared to individual treatments, emphasizing the synergistic effects of oxidation and coagulation. Insights into the mechanisms of aluminum and iron electrodes provide valuable information on sustainable water treatment technologies.

The findings reveal that the optimized advanced oxidation followed by electrocoagulation process leads to substantial reductions in COD & Colour removal, making the treated effluent suitable for discharge or potential reuse. The study suggests that combining AOPs with EC using aluminum and iron electrodes is an effective strategy for addressing water treatment challenges and ensuring to achieve "Zero Liquid Discharge." (ZLD)

Keywords- **Advanced oxidation processes, Fenton reaction, Electrocoagulation, Synergistic approach, Environment Sustainability.**

I. INTRODUCTION

Access to clean and safe drinking water is a critical global challenge, with increasing concerns over the presence of emerging contaminants in water sources. Reverse osmosis **(RO)** technology has become instrumental in providing high-purity water by effectively removing dissolved salts and most organic contaminants. However, residual pollutants such as organic dyes, pharmaceuticals, and trace metals often necessitate supplementary treatment to meet stringent water quality standards. Achieving Zero Liquid Discharge **(ZLD)** in distillery operations is a critical goal for sustainable and environmentally friendly industrial practices. One of the significant challenges in this process is the management of distillery condensate RO (Reverse Osmosis) reject water. This reject water, rich in organic contaminants and characterized by high Chemical Oxygen Demand (COD) and colour, poses a significant treatment and disposal challenge. The RO technology, while effective in reducing wastewater volume, generates a substantial amount of concentrated brine. This brine concentrate, if not properly managed, is often discharged into natural reservoirs such as rivers, lakes, and ponds, leading to environmental pollution. Additionally, the high treatment costs associated with the brine concentrate discourage further processing, making its proper disposal even more challenging. Reintroducing the brine concentrate into the RO system is not a feasible solution, as it can severely damage the RO membranes, reducing the efficiency and lifespan of the system. Proper management and treatment of RO brine concentrate are crucial to mitigate its environmental impact and ensure sustainable operations within distillery units.

Effective strategies for handling distillery condensate RO reject water are essential to prevent environmental degradation and achieve ZLD. This involves advanced treatment processes that can handle the high organic load and salinity of the reject water, ensuring compliance with environmental regulations and promoting sustainable industrial practices.

To address these challenges, advanced oxidation (AO) and electrocoagulation (EC) have emerged as promising techniques for enhancing the purification of RO reject water. AO processes, such as the Fenton reaction, harness hydroxyl radicals (•OH) generated from the reaction between iron catalysts and hydrogen peroxide (H2O2). These radicals are highly reactive and capable of degrading a wide range of organic contaminants into simpler, less harmful by-products.

On the other hand, EC involves the use of sacrificial electrodes (typically aluminium and iron) to destabilize suspended particles and dissolved pollutants through electrochemical reactions. The coagulation and subsequent flocculation of contaminants enable their removal via sedimentation or filtration processes. The combination of Fenton-based AO and EC techniques presents a synergistic approach towards improving the efficiency and efficacy of water purification. By integrating these processes, synergistic effects can be leveraged to achieve enhanced removal of contaminants compared to individual treatments alone. The oxidative degradation capabilities of AO complement the physical removal mechanisms of EC, thereby addressing a broader spectrum of contaminants in RO reject water. In recent years, there has been growing interest in exploring hybrid water treatment strategies that capitalize on the strengths of multiple technologies to achieve superior water quality outcomes.

This research aims to contribute to this field by investigating the synergistic purification of RO water using Fenton-based AO and EC with aluminium and iron electrodes. The objectives of this study include optimizing operational parameters such as COD, Colour, Contaminant Concentration, Reaction time, and Electrode configuration to maximize purification efficiency. Additionally, the study will assess the effectiveness of the combined AO-EC approach in removing organic pollutants, pharmaceutical residues, and heavy metals from RO water samples. Through systematic experimentation and analysis, this research seeks to provide valuable insights into the feasibility and potential applications of integrating Fenton-based AO and EC techniques for sustainable water treatment. The findings are expected to contribute towards developing cost-effective and environmentally friendly solutions for improving water quality in both industrial and domestic settings.

II. NEED OF THE STUDY

- Proper treatment of this wastewater is crucial to minimize environmental pollution and safeguard water resources.
- Conventional methods of treating wastewater, such as biological treatment, may not be adequate to eliminate persistent pollutants found in distillery RO reject water. There is a demand for advanced treatment technologies that can effectively break down and eliminate these contaminants.
- Combining Fenton-based advanced oxidation and electrocoagulation offers potential advantages, including improved pollutant removal efficiency, reduced chemical usage, and the potential to separate a wide range of pollutants.
- Developing effective and sustainable methods for treating industrial wastewater contributes to broader efforts in water conservation and sustainable water management.
- Stringent environmental regulations necessitate industries to adopt advanced treatment technologies to meet discharge standards. This research aims to provide an innovative treatment solution that can assist distilleries in complying with regulatory requirements and avoiding penalties.

III. MATERIALS

a) **Water Samples:** Reverse osmosis (RO) permeates containing typical contaminants such as organic dyes, pharmaceutical residues, and heavy metals.
b) **Instruments:** Magnetic stirrer, Spectrophotometer, DC power supply, Hot air oven, COD Digester (close reflux), Hot water bath, pH meter.
c) **Chemicals**: Potassium Dichromate Solution, Silver Sulphate, Mercuric Sulphate, Ferroin Indicator, Hydrogen peroxide (H_2O_2), iron salts (e.g., ferrous sulfate), Magnesium Sulphate, Aluminum sulfate, and other reagents of analytical grade.
d) **Electrodes:** Aluminium (Al) and iron (Fe) electrodes for electrocoagulation.
e) **Analytical Instruments:** UV-Vis's spectrophotometer, pH meter, conductivity meter, and other relevant laboratory equipment.

IV. RESEARCH METHODOLOGY

The methodology section outlines the plan and method that how the study is conducted. The details are as follows:

4.1. RO Reject Water Sample Collection

An RO (Reverse Osmosis) reject water sample was collected from Loknete Sunder Rao Solanki SSK Ltd, a sugarcane molasses-based distillery situated near Beed, Maharashtra, India. The distillery, located in Telegaon, produces significant volumes of RO reject water as part of its operational processes. This sample collection is critical for understanding the composition and environmental impact of the reject water, facilitating effective waste management and compliance with environmental regulations.

4.2 Experimental Set Up

4.2.1. Preparation of Experimental Setup

The experimental setup is illustrated in photo fig 1.1, showing a 500 ml glass beaker used for batch mode experiments. Aluminum sheets measuring **10 cm × 2.5cm × 0.3 cm** (length× width× thickness) and iron rods measuring **10 cm ×0.8 cm** (length × diameter) was utilized as electrodes, with an area of **50 mm× 18 mm** submerged in the solution.

Various combinations of electrodes were tested, including **Al-Al, Fe-Fe**, and **Al-Fe**, all connected in a monopolar mode. A direct current source provided the necessary current, while a magnetic stirrer (**REMI MODEL NO RNS16BC, INDIA**) was used to prevent concentration gradients. The electrodes were positioned **1 cm** above the bottom of the beaker for easy stirring. Each experimental run involved charging **300 ml** of RO REJECT in the glass beaker, followed by continuous stirring at a fixed speed using the magnetic stirrer. The electrocoagulation process began with the start of the power supply, and the efficiency was determined based on the COD of the initial and treated samples. The reaction took place at room temperature **(25°C)** in a 500 ml beaker, with 300 ml of RO REJECT at different pH levels **(3, 5, 7)** and varying electric currents **(3, 6, 9, 12, 18 watts)** passed through an aluminum rod submerged in the sample, continuously stirred at 500 rpm.

The electrochemical reactions taking place at the aluminum and iron electrodes are as outlined below:

4.2.2. Mechanism of Experimental Set-Up
Electrochemical reaction occurring at the Electrodes are as follows;
For aluminium electrodes:

At anode: Al Al3 + + 3e- ……… (1)
At cathode 3 H2O + 3e 3/2H2 + 3OH –……… (2)
Al3+ Al (OH)n (3-n) Al 2(OH) $_2{}^4$+ …. (3)

For iron electrode:
At anode:
2Fe (s) Fe (aq) 2e 2Fe(aq) +2e + - (4)
2OH (aq) Fe (OH)2(s) + + - (5)
At cathode:
$2H_2O$ (1) + 2e- 2OH (aq) + H_2(g)……… (6)
Overall:
Fe(s) + $2H_2O$ (1) Fe $(OH)_2$(s) + H2(g)………. (7)

The distillery condensate RO reject undergoes COD reduction in the EC process through two mechanisms.

a. Firstly, in situ generation of coagulants (Al (OH)3 and Fe (OH)2) in the EC process aids in the coagulation of organic content.
b. Secondly, the presence of chlorides in the distillery condensate RO reject , along with the application of electric current, leads to the production of chlorine and hypochlorite ions, which then react with organic molecules and oxidize them. The high oxidative potential of hypochlorous acid and hypochlorite ion allows them to decompose organic matter.
c. The reactions occurring at the anode and cathode are as follows...

$2H_2O$ + 2e- H_2 + 2OH -…….. (9)
Bulk solution.
Cl_2 + H_2O HOCl + H + + Cl –……. (10)
HOCl H + + OCl -…….. (11)

Fig 1: Experimental setup of electro- coagulation treatment

4.2.3. Optimization of Fenton-Based Advanced Oxidation and Electrocoagulation for Reduction of Parameters
The Advanced Oxidation Process (AOP) and Electrocoagulation (EC) techniques, based in Fenton, are commonly utilized for treating wastewater with a range of organic and inorganic pollutants. This research aims to optimize these methods to improve their effectiveness in lowering contaminant concentrations. Electrocoagulation employs electrical currents to dissolve sacrificial electrodes, usually made of aluminum or iron, which then release ions to create coagulants for the removal of contaminants.
Electrolysis treatment, commonly referred to as electrochemical water treatment, is a versatile and effective method for purifying water and wastewater. This technique involves the use of electrical currents to drive chemical reactions that remove contaminants. The process can be divided into several methods, including Electrocoagulation (EC), Electrooxidation (EO), and Electro flotation (EF), each with specific applications and mechanisms.

The electrolysis reactions took place at a temperature of 25°C, within a 500ml beaker with a RO REJECT volume of 300 ml at a pH of 7. During the treatment, an electric current was applied to an aluminum rod submerged in the sample and constantly agitated.

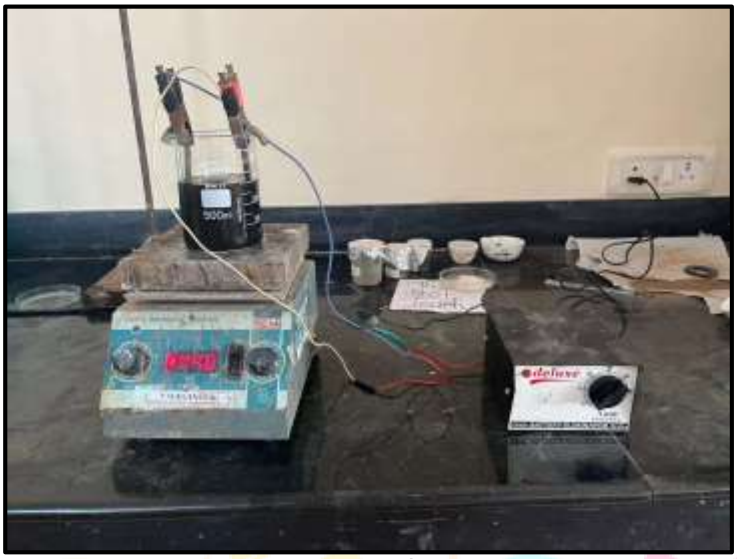

Fig 2: Experimental setup of Electro-Fenton setup

4.2.4. Study of the Effect of Ozonation Treatment

The ozonation experiments involved varying the ozone dosage and were carried out in the laboratory at a temperature of 25°C. Three ozone traps, each containing 100 ml of sample, were connected in series to maximize the use of ozone gas. Ozone was introduced into the reactor through an aeration port using an air diffuser. The ozone gas was allowed to react with the sample for different time intervals (18, 30, 45, 60 min) and doses of 30, 60, 90, and 120 liters. The treated samples were then analyzed for COD and color to assess the impact of the treatment on the degradation of oxidizable compounds in RO REJECT.

Fig 3: Experimental set up of the ozonation treatment

4.2.5. Analytical Methods

pH and Temperature: pH and temperature were determined using a digital pH meter for the samples.

Colour: For color measurement, 10ml of the sample were diluted 100 times for color reduction measurement. The absorbance was measured at 475 nm using a UV-spectrophotometer. The decolorization yield was expressed as the percentage decrease in absorbance at 475 nm related to the initial absorbance at the same wavelength.

Chemical Oxygen Demand: The organic matter present in the sample was completely oxidized by $K_2Cr_2O_7$ in the presence of H_2SO_4 to form CO_2 and H_2O. The excess $K_2Cr_2O_7$ remaining after the reaction was titrated with ferrous ammonium sulfate. The $K_2Cr_2O_7$ gives the oxygen required for oxidation of organic matter. The organic matter was oxidized with a hot sulfuric solution of potassium dichromate, with silver sulfate as a catalyst. Chloride was masked with mercury sulfate. The concentration of unconsumed yellow $Cr_2O_7^{2-}$ ions or, respectively, of green Cr^{3+} ions was then determined photometrically. COD was determined as given in APHA AWWA WEF, (2005): PP 5-18.

Biological Oxygen Demand: The Biochemical Oxygen Demand (BOD) test quantifies the amount of oxygen needed by microorganisms to break down organic matter into carbon dioxide and water. The BOD value was calculated using the 3-day BOD test at 27°C through the 'Wrinkle's Iodometric method' outlined in APHA AWWA WEF, (2005), page 5-2.

(TS), (TDS) & (TSS): A well-mixed sample was evaporated in a weighed dish and dried at a constant weight in an oven at 1030-1050C to determine total solids (TS), total dissolved solids (TDS), and total suspended solids (TSS). The increase in weight over that of the empty dish represented the total solids. These parameters were analyzed following methods as given in APHA AWWA WPCF, (2005). PP 2-55 to 2-59.

Chlorides: In a neutral or sunlight alkaline solution, potassium chlorate can indicate the endpoint of the silver nitrate titration of chloride. Silver chloride is precipitated quantitatively before.

Chloride concentrations were measured utilizing the 'Argentometric method' described in APHA AWWA WEF, (2005) pages 4-70 to 4-71.

Sulphate: The precipitation of sulphate ion (SO4 2-) occurs in an acetic acid solution when barium chloride (BaCl2) is added, resulting in the formation of uniform-sized barium sulphate (BaSO4) crystals. The light absorbance of the BaSO4 suspension is then quantified using a photometer, allowing for the determination of SO4 2- concentration through comparison with a standard curve. The 'Turbid metric method' outlined in APHA AWWA WEF, (2005). PP 4-188 was utilized for sulphate determination.

V. RESULTS & DISCUSSION

RO REJECT was analysed for different parameter.

Results are given in following table.

Table 1: Characteristics of (RO REJECT)

#	Parameters	Inlet Parameter	Treated Parameter
1	pH	7.40	7.33
2	EC (µmhos/cm)	9000	4500
3	**Solids**		
	TS (mg/lit)	7200	3700
	TSS (mg/lit)	100	100
	TDS (mg/lit)	7000	3500
4	COD (mg/lit)	18,000	4500
5	BOD (mg/lit)	6000	2000
6	Chloride (mg/lit)	113	25
7	Sulphate (mg/lit)	25	11

5.1 Effect of Different pH on COD Reduction of RO Reject @ 18 Watts, 2 hrs, and 500 rpm

The study investigates the effect of different pH levels on the reduction of Chemical Oxygen Demand (COD) in Reverse Osmosis (RO) reject water through electrolysis treatments using different electrode combinations at a constant power of 18 watts, duration of 2 hours, and a stirring speed of 500 rpm. The electrode combinations tested were Aluminum-Aluminum (Al-Al), Iron-Iron (Fe-Fe), and Aluminum-Iron (Al-Fe).

The effectiveness of COD reduction in RO reject water through electrolysis is significantly influenced by the pH level and the type of electrodes used. Among the three pH levels tested, pH 7 consistently yielded the highest COD reduction for all electrode combinations. Specifically, the Al-Fe electrode combination demonstrated the most substantial COD reduction across all pH levels, with a peak performance of 64.3% at pH 7. On the other hand, the Fe-Fe combination was particularly effective at the acidic pH of 3. The Al-Al combination showed a moderate reduction across the pH range, with the highest performance observed at pH 7.

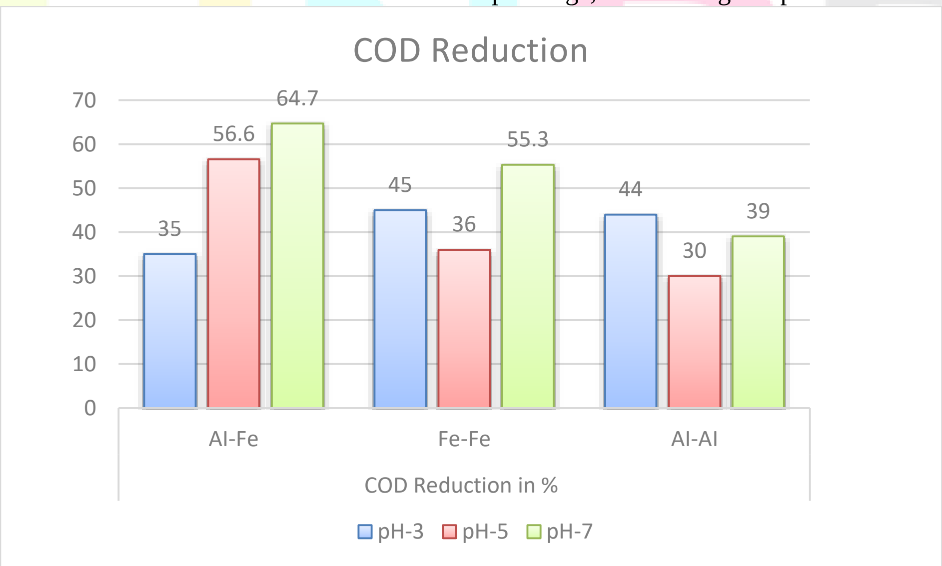

Fig 4: Effect of Different pH on COD Reduction of RO Reject

5.2 Effect of Different pH on Color Removal of RO Reject @ 18 Watts, 2 hrs, and 500 rpm

Electrolysis treatments were applied to RO reject water at pH levels of 3, 5, and 7 using different currents. Aluminum electrodes were used for Al+ ion generation, and iron electrodes were used for Fe+ ion generation. Below are the results for the three electrode combinations—Aluminum-Aluminum (Al-Al), Iron-Iron (Fe-Fe), and Aluminum-Iron (Al-Fe). Experiments were conducted at varying pH levels of distillery condensate RO reject (3, 5, and 7) to investigate the impact of pH on the electrocoagulation process. The results are presented in Figure 4.2.1. Electrolysis was performed for 2 hours with a constant current density of 18 watts, an agitation speed of 500 rpm, and an electrode spacing of 3 cm. The pH of the spent wash was adjusted to the desired level using 0.1N HCl.

Figure 4 and Figure 5 illustrate that the pH of the condensate distillery RO reject significantly influenced the COD and color removal efficiency. The highest color reduction was achieved at pH 3 for all three electrode combinations: Al–Al (64.4%) and Al–Fe (55%). Similarly, the maximum COD reduction was observed at pH 3 for the Al–Mg electrode combination. The COD removal efficiency decreased with increasing pH, indicating that acidic conditions are more favorable for treating distillery condensate RO reject.

The optimal pH value was determined to be 3, and subsequent experiments were conducted at this pH level. Among the electrode pairs tested, the Al–Al electrode pair demonstrated the highest COD and color removal efficiency compared to the Al–Fe and Fe–Fe combinations.

The effectiveness of color removal in RO reject water through electrolysis is influenced by both pH level and electrode type. The data indicates that:

- **Fe-Fe electrodes** generally exhibit the highest color removal efficiency, particularly at pH 5 with an **87%** reduction and at pH 7 with an **82%** reduction.
- **Al-Fe electrodes** also perform well, with notable reductions of **79%** at pH 5 and **79.6%** at pH 7.
- **Al-Al electrodes** are most effective at pH 5 with a **73%** reduction but show the least effectiveness compared to the other electrode combinations at pH 7.

Therefore, for optimal color removal in RO reject water, **Fe-Fe electrodes** are recommended, especially at pH 5 and pH 7. The **Al-Fe electrodes** also provide substantial color removal and can be considered as an alternative. **Al-Al electrodes** may be less effective but still contribute significantly to color reduction, particularly at pH 5.

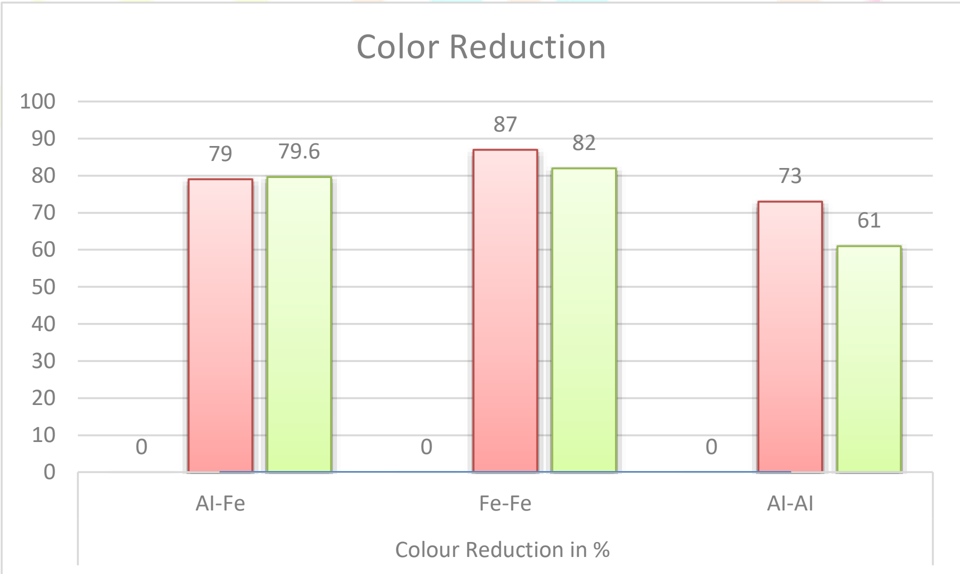

Fig 5: Effect of Different pH on Color Removal of RO Reject

5.3 Effect of Current Density on Electrolysis Treatment pH 7, 2 hrs. And 500 rpm

Electrolysis treatment was applied to RO reject water at different current densities (3, 6, 9, 12, and 18 watts). Aluminium electrodes were used for Al+ ion generation, and iron electrodes were used for Fe+ ion generation. The results indicated that aluminium electrodes achieved greater COD reduction at higher current densities, specifically at 18 watts.

From the data presented in the table, it is evident that increasing the current density improves the COD reduction efficiency for all electrode combinations. Among the electrodes tested, the Al+Al combination consistently exhibited the highest COD reduction, particularly at the highest current density of 36 watts, achieving a COD reduction of **57%.** The Fe+Fe electrodes also showed significant COD reduction, reaching **58%** at 36 watts. The Al+Fe electrodes demonstrated lower performance in comparison, with a maximum COD reduction of **43.7%** at the same current density.

In conclusion, the electrolysis treatment's efficiency in reducing COD from RO reject water is significantly influenced by the current density and the type of electrodes used. Higher current densities result in greater COD reduction, with aluminum electrodes outperforming iron electrodes under similar conditions.

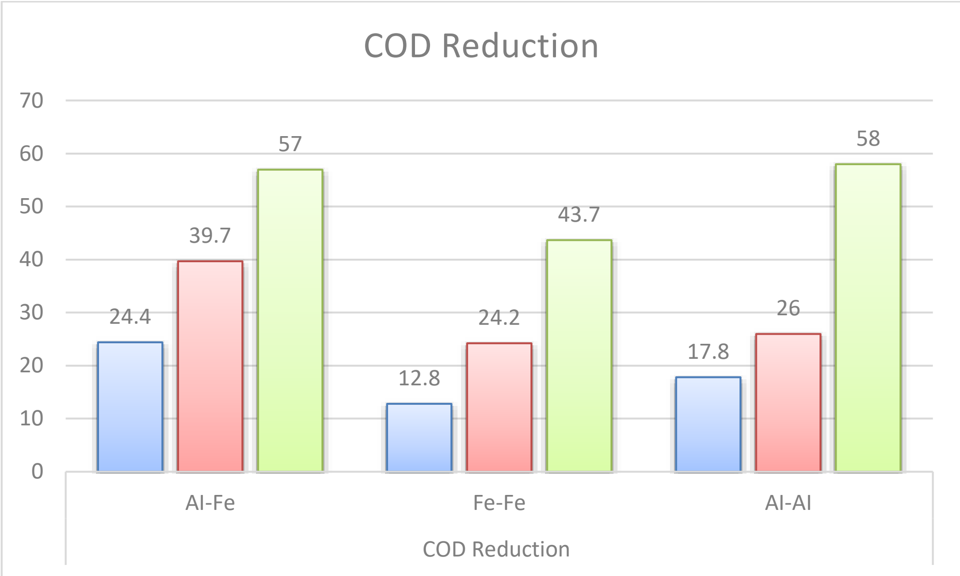

Fig 6: Effect of Current Density on Electrolysis Treatment

5.4 Effect of Current Density on Color Reduction in Electrolysis Treatment at pH 7, 2 hours, and 500 rpm

Electrolysis treatment was conducted on RO reject water at different current densities (18, 27, and 36 watts) using aluminum and iron electrodes. The impact on color reduction was observed, with the detailed results.

From the data presented in **Fig 7**, it is clear that the current density significantly influences the color reduction efficiency in the electrolysis treatment of RO reject water. The Fe+Fe electrodes demonstrated the highest color reduction efficiency, achieving a maximum reduction of 90% at 27 watts. Even at the highest current density of 36 watts, the Fe+Fe electrodes maintained a high color reduction of 87.7%.

In comparison, the Al+Al electrodes showed a more moderate improvement in color reduction efficiency, with a maximum reduction of 41.4% at 36 watts. The Al+Fe electrodes displayed consistent performance with a color reduction of around 33% at lower current densities, increasing slightly to 37.6% at the highest current density.

In conclusion, the electrolysis treatment's effectiveness in reducing the color of RO reject water is highly dependent on the current density and electrode material. Iron electrodes (Fe+Fe) are particularly effective, achieving the highest color reduction across different current densities. Aluminum electrodes (Al+Al) also show improved performance at higher current densities, but their overall color reduction efficiency is lower compared to iron electrodes.

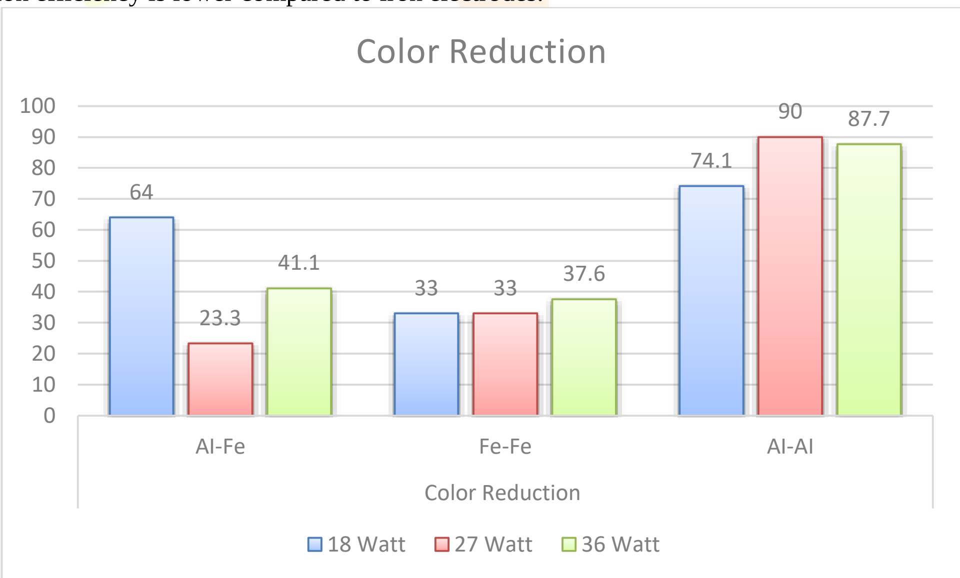

Fig 7: Effect of Current Density on Color Reduction in Electrolysis Treatment

5.5 Effect of different time duration on COD of RO REJECT @ pH7 27 watt and 500 rpm.

Electrolysis treatment was conducted on diluted RO reject water at different time durations (30, 60, 90, and 120 minutes) using aluminum and iron electrodes. From the data in Fig 6, it is evident that increasing the duration of electrolysis enhances the COD reduction for all electrode combinations. The Al+Al electrodes showed the highest COD reduction over time, achieving a 43.4% reduction after 120 minutes. The Fe+Fe electrodes also demonstrated significant COD reduction, reaching 37.7% after 120 minutes. The Al+Fe electrodes exhibited moderate performance, with a maximum COD reduction of 29.5% at the same duration.

The effectiveness of electrolysis treatment in reducing COD from diluted RO reject water is significantly influenced by the duration of treatment. Longer electrolysis durations result in greater COD reduction, with the Al+Al electrode combination showing the highest efficiency. The Fe+Fe electrodes also perform well, particularly at longer treatment times, indicating their suitability for extended electrocoagulation processes.

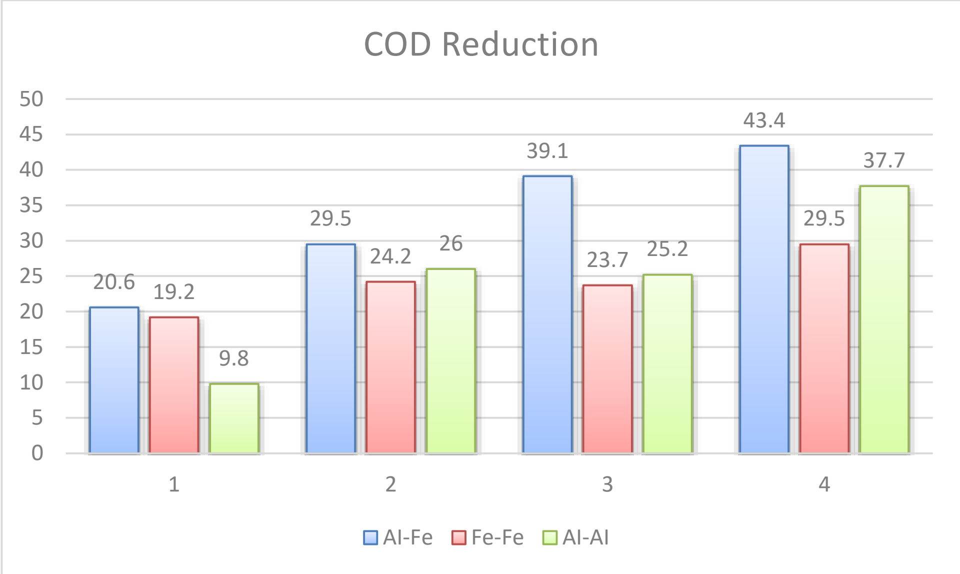

Fig 8: Effect of different time duration on COD of RO REJECT

5.6 Effect of Different Time Durations on Color Reduction in Electrolysis Treatment at 7 pH, 36 watt and 500 rpm.

Electrolysis treatment was conducted on RO reject water at different time durations (30, 60, 90, and 120 minutes) using aluminum and iron electrodes. Increasing the duration of electrolysis significantly improves color reduction for all electrode combinations. The Al+Al electrodes showed the highest color reduction over time, achieving a 73.8% reduction after 120 minutes. The Al+Fe and Fe+Fe electrodes also demonstrated significant color reduction, with reductions of 64.4% and 59%, respectively, after the same duration. The effectiveness of electrolysis treatment in reducing the color of RO reject water is significantly influenced by the duration of treatment. Longer electrolysis durations result in greater color reduction, with the Al+Al electrode combination showing the highest efficiency. The Al+Fe and Fe+Fe electrodes also perform well, particularly at longer treatment times, indicating their suitability for extended electrocoagulation processes.

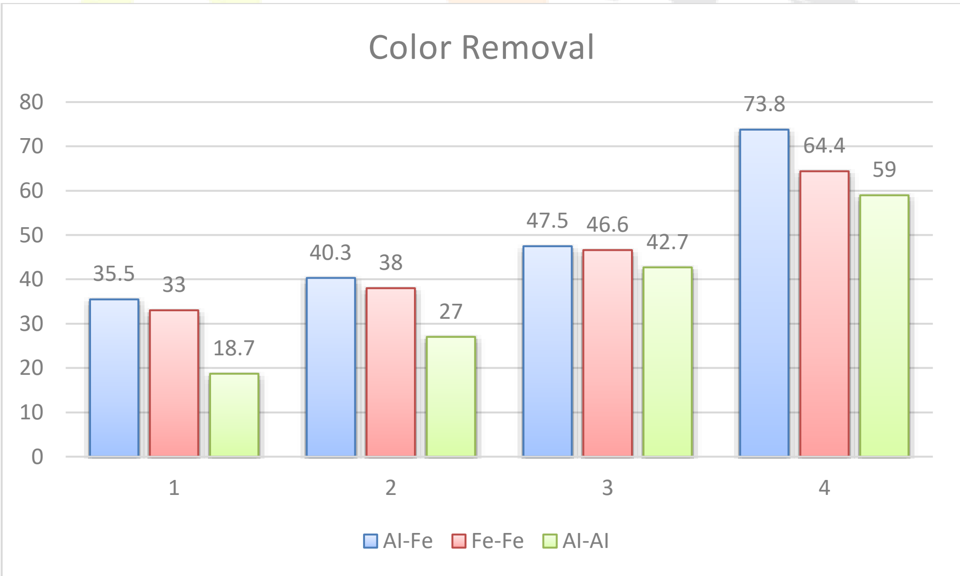

Fig 9: Effect of Different Time Durations on Color Reduction

VI. NCLUSION

The treatment of RO reject from distillery operations, known for its high organic content, was investigated using Electrolysis and Advanced Oxidation Processes (AOP) in batch mode.

pH Variation: Among the pH variations studied (pH 7), it was found that maintaining a neutral pH was most effective for the treatment process.

Time Variation: Extending the treatment time to 120 minutes proved most effective in reducing both COD and color from the RO reject.

Current Variation: At a current density of 27 watts, the treatment showed optimal effectiveness compared to other current densities (18 and 36 watts).

Electrode Combination: The combination of Al + Al electrodes demonstrated superior performance in reducing both color and COD compared to Al + Fe and Fe + Fe combinations.
Combined Treatment: Combining ozonation with Electro Fenton showed promising results, achieving a 70% reduction in COD and a 60% reduction in color. However, Electro Fenton treatment led to an increase in color, possibly due to iron dissolution into the RO reject.
Effluent Quality: Despite significant reductions in organic content, the effluent after treatment still contains residual organic matter that requires further treatment before discharge into aquatic ecosystems.
In conclusion, Electrocoagulation, Electro Fenton, and Ozonation are effective treatments for RO reject containing high organic content. These processes can be employed sequentially to achieve substantial reductions in pollutants, making the effluent suitable for further processing or discharge after additional treatment steps.

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