

# Colour and COD removal of Distillery spent wash by using Electro coagulation

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**Abstract** - Distilleries are one of the backbone industries of India contributing a fair amount to nations GDP. It also contributes to India's export. Distillery industry consumes 12-15 l of water for one liter of alcohol production, generating  $4.04 \times 10^{10}$  liter of unwanted residual liquid waste called spent wash. Spent wash is a dark brown colour and very high organic loading, very high chemical oxygen demand (COD) and high Biochemical Oxygen demand (BOD). Thus distillery industries have a great adverse impact on the environment. Numbers of clean up technologies have been worked out to efficiently treat the distillery spent wash. In this paper, electro coagulation with aeration is carried out by using Aluminum and Iron electrodes in a batch reactor. The effects of operating parameter such as current density, pH, agitation speed, aeration were studied. By using iron electrodes, the maximum colour removal 99.78 % was observed at a 25 Voltage for pH 8. UV spectrophotometer was used to find out colour removal. Maximum COD removal was found to be 85.71% at pH 3 for an electro coagulation time of 150 minutes.

**Keywords:** - Electro coagulation, distillery spent wash, molasses, melanoidin, UV Spectrophotometer, Caramelized, Recalcitrant

## 1. INTRODUCTION

Distillery spent wash is apparently one of the serious pollution problems of the countries, especially who producing alcohol from the fermentation and subsequent distillation of sugar cane molasses. In India there are approximately 319 sugar industries producing alcohol  $3.25 \times 10^9$  liters and generating  $40.4 \times 10^9$  liters of spending was annually [1,2,3]. The distillery spent wash is characterized as one of the caramelized and recalcitrant wastes containing extremely high COD, BOD, SS, inorganic solids, colour and low in pH typically 10-15 liters of effluent are produced for each liter of alcohol [4,5]. It is the common example that distillery effluent is disposed without proper treatment causing serious hazard to surrounding land and water bodies because they prevent sunlight penetration into an aquatic system thereby dissolved oxygen concentration is depleted. This has occurred due to the presence of dark brown pigment melanoidin, generated through maillard reaction between sugar and amino compounds [6, 7, 8]. Economical and eco-friendly distillery spent treatment is a great challenge to environmentalists and scholars. There are a number of methods to treat distillery spent wash, especially decolourization and removal of COD such as physical, chemical and biological methods. Bio-methanation of distillery spent wash followed by aerobic treatment is the commonly used treatment to treat distillery [9, 10]. Aerobic treatment reduces the chemical oxygen demand (COD), and Biological oxygen demand up to 50 to 70 %, but till 100% color, COD and BOD are not reduced [11].

**Table 1:** Characteristics of Untreated and aerobically treated distillery effluent [2, 12, 13, 14]

Parameter (All values in mg/l except pH)	Untreated distillery effluent	Aerobically treated effluent

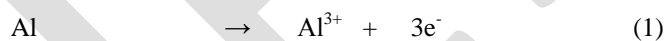
pH	3.0-4.5	7.5-8.0
BOD <sub>5</sub>	50,000- 60,000	8000-10,000
COD	1,10,000- 1,90,000	45,000-52,000
Total Solid (TS)	1,10,000-1,90,000	70,000-75,000
Total Volatile Solid (TVS)	80,000-1,20,000	68,000-70,000
Total suspended solid (TSS)	13,000-15,000	38,000-42,000
Total dissolved solids (TDS)	90,000-1,50,000	30,000-32,000
Chlorides	8000-8500	7000-8000
Phenols	8000-10,000	7000-8000
Sulphate	7500-9000	3000-5000
Phosphate	2500-2700	1500-1700
Total nitrogen	5000-7000	4000-4200

## 2. LITERATURE REVIEW

Electro coagulation is an efficient method to remove colour and reduce COD. Manisankar et al., (2004) in their work concludes that by using Graphite - Graphite electrodes 85.2 % COD removed at pH 6.9 – 7.2 and duration is 180 minutes. Krishna et al., (2010) reported that by using Al - Al electrode 72.3 % COD removed in 2 hours when pH is 3. Khandegar and Saroha et al., (2012) state that by using Al - Al electrode 81.3 % COD removed successfully. They also prove that by using Al - Fe electrode 71.8 % COD removed in 2 hour duration when the pH of the solution is 3. Khandegar et al., (2014) revealed that 98 % COD removal efficiency was obtained by using Al – Al electrode when the pH of the waste water is 7.2. They also concluded that the electro - coagulation technique can be successfully employed for the treatment of distillery effluent. Electro coagulation (EC) has the potential to treat distillery spent wash. Electro coagulation reaction occurring at the electrodes for aluminum and iron electrodes is as follows

For Aluminum electrodes [15]

At anode

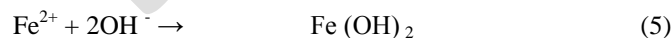
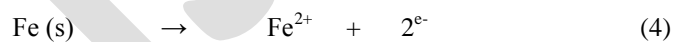


At Cathode

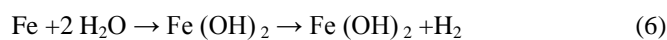


For Iron electrodes [15]

At Anode



At Cathode



Distillery spent wash contains chlorides. By the passing the electric current generation of chlorine and hypo chloride ions occurs. It reacts with organic matter present in spent wash, and oxidation starts. Due to formation of hypochlorous acid and hypochlorite ions the organic matter decomposes because of their high oxidative potential. Following reaction takes place at anode and cathode.

At anode



### 3. MATERIALS AND METHODS

In present work Spent wash was collected from a shri dnyeshwar distillery, bhenda Ahmednagar. As per the distillery laboratory, characteristics of spent wash are mentioned in table no.2. In practice these parameters are usually measured in situ.

**Table No 2:- Spent Wash Parameters**

Parameter	Untreated (Raw spent wash)	Homonization tank	Digester tank	Digester Outlet
pH	3.50-4.20	6.6	7.20-7.60	7.5-8.0
Temperature	35 - 40 <sup>0</sup> C	40 <sup>0</sup> C	37 - 39 <sup>0</sup> C	37- 40 <sup>0</sup> C
TVA(Total Volatile Acid)	2500-3500 mg/l	15,000 mg/l	3,500 – 5,000 mg/l	5014
COD	80,000-1,44,200 mg/l	35,000-45,000 mg/l	30,000 – 34,600 mg/l	35432

### 4. EXPERIMENTAL SETUP

The experiment was conducted in the batch mode of operation. In each run 1000ml of sample was taken for electro coagulation in a beaker. Two different types of electrodes i.e. Iron and Aluminium are used and the most effective electrode in the treatment process is analyzed. A pair of electrodes as Fe-Fe or Al-Al is used at a time. Each electrode having cross sectional area 100mm×28mm×3mm are used. The dipping area for each the electrode was 75mm×28mm×3mm. DC power supply having a capacity 7V-30V and 0-1.2Amp was used. For each run the voltage applied is varied to determine the efficiency of the treatment at different voltage ranges.

Magnetic stirrer of 1 Liter capacity was used and the agitation speed was kept 500rpm throughout the experiment. For aeration a motor is provided and the pipe of aeration is inserted in the beaker. The total time required for each run is 150 minutes. A sample is taken out every 30 minutes using a 25ml glass pipette. Total 6 samples are analyzed in each run. A sample is taken in a conical flask and then the difference between colour and COD is measured for different time intervals.

$$\% \text{ Colour removal efficiency} = \frac{\text{Initial} - \text{Final}}{\text{Initial}} \times 100$$

Organic strength of the spent wash is nothing but Chemical oxygen demand, which was determined by the dichromate method (Open reflux titrimetric method).

#### 4.1. Colour Removal

The treated sample is observed using spectrophotometer to measure the percentage colour removal by electro coagulation process. The spectrophotometer used was "Spectrochem NV-201" having wavelength range 400-700  $\lambda$ . Firstly instrument is turned on and warm up time of 15-20 minutes is given. Then any wavelength is selected and with the sample compartment closed empty the % transmittance (zero percent transmission of light) to read 0% T is set using front dial. Then clean and dry cuvette filled approximately  $\frac{3}{4}$  full of distilled water is placed in a sample compartment. The compartment is closed and % transmittance is adjusted to 100% (100% transmission of light) using front dial. This is the calibration process for spectrophotometer.



Figure No 1. Spectrophotometer (Spectrochem NV 201)

For obtaining the most suitable wavelength of the sample further adjustment is required to be done. Sample is taken in the cuvette and placed in the sample compartment. By changing the wavelength  $\lambda$ , the % absorbance of the sample is noted. The frequency at which the % absorbance is maximum should be selected as the most suitable frequency for the given sample. The wavelength  $\lambda$  suitable for distillery wastewater was 425  $\lambda$ .

% Absorbance at different wavelength is given in the following table.

Wavelength $\lambda$	% Absorbance
400	1.710
425	2.020
450	1.610
475	1.190
500	0.870
525	0.580
550	0.390
575	0.690

600	0.330
625	0.350
650	0.320
675	0.150
700	0.440

#### 4.2. COD Removal

The chemical oxygen demand (C.O.D) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. The COD of the sample is determined by Dichromate reflux method. The reagents used for the COD process are as follows-Silver Sulphate  $\text{Ag}_2\text{SO}_4$ .

- Standard Ferrous Ammonium Sulphate  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ .

39gm FAS powder is added to 20ml of concentrated  $\text{H}_2\text{SO}_4$  and diluted with 1 liter distilled water.

- Ferroin Indicator
- Mercuric Sulphate  $\text{HgSO}_4$  crystals.
- Silver Sulphate  $\text{Ag}_2\text{SO}_4$
- Sulphuric acid conc.  $\text{H}_2\text{SO}_4$ . (96% pure)
- Standard potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) 0.25N

Firstly 15gm of  $\text{K}_2\text{Cr}_2\text{O}_7$  powder is heated in the oven at  $105^\circ\text{C}$  for 2hrs. Then after cooling 12.25gm powder is added in 1 liter distilled water.

For COD process firstly 5 conical flasks are taken in which four flasks are for the sample and the fifth flask is for Blank correction. For COD process the dilution factor 10 is taken, i.e. 1 ml of the sample is added to 9 ml of distilled water. 1 gm of  $\text{HgSO}_4$  and  $\text{Ag}_2\text{SO}_4$  are added to each flask. 10 ml of  $\text{K}_2\text{Cr}_2\text{O}_7$  and 30 ml of  $\text{H}_2\text{SO}_4$  is added to the flask and the solution is stirred. Then the flasks are placed on COD hot plate at  $80^\circ\text{C}$  for 2Hrs.



Figure No 2. COD Hot Plate with Temperature Regulator

After 2 Hrs sample is cooled for 1 Hr. then 40 ml of distilled water is added to each flask. Ferroin indicator is added 2-3 drops. The solution is titrated against Ferrous Ammonium Sulphate Till the end point i.e. reddish brown is achieved and the burette readings are noted. Electro coagulation (EC) efficiency was determined in terms of colour and COD removal

**Formula for COD calculations**

$$\frac{(a-b) \times N \times 8 \times 1000}{\text{ml of sample}} \times \text{D.F.}$$

Where,

a = ml of titrant used for blank correction

b = ml of titrant used for sample

N = Normality of FAS =  $\frac{2.5}{\text{Blank reading}}$

D.F. = 200

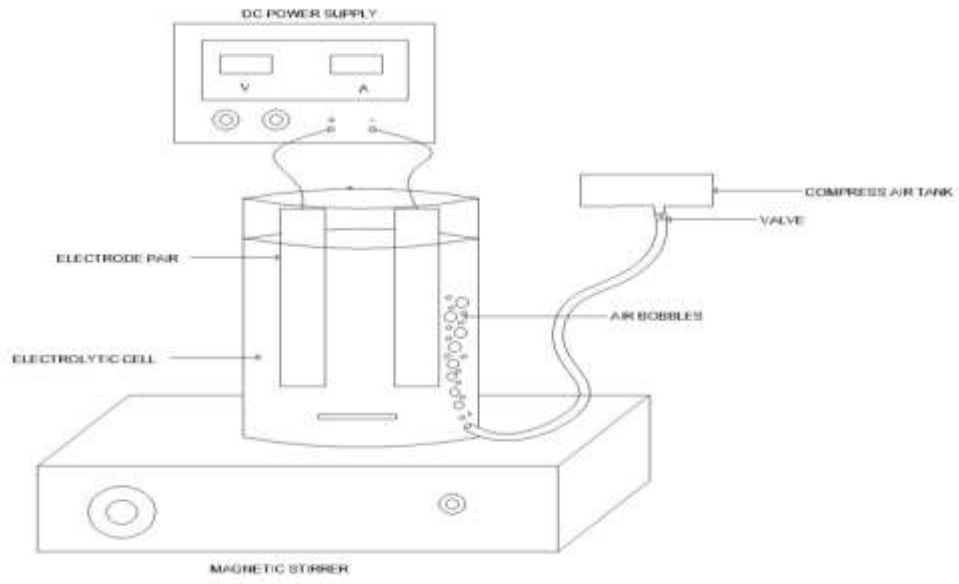


Figure No .3. Electro- Coagulation Setup



Figure No 4. Experimental setup of Electro Coagulation Process with aeration



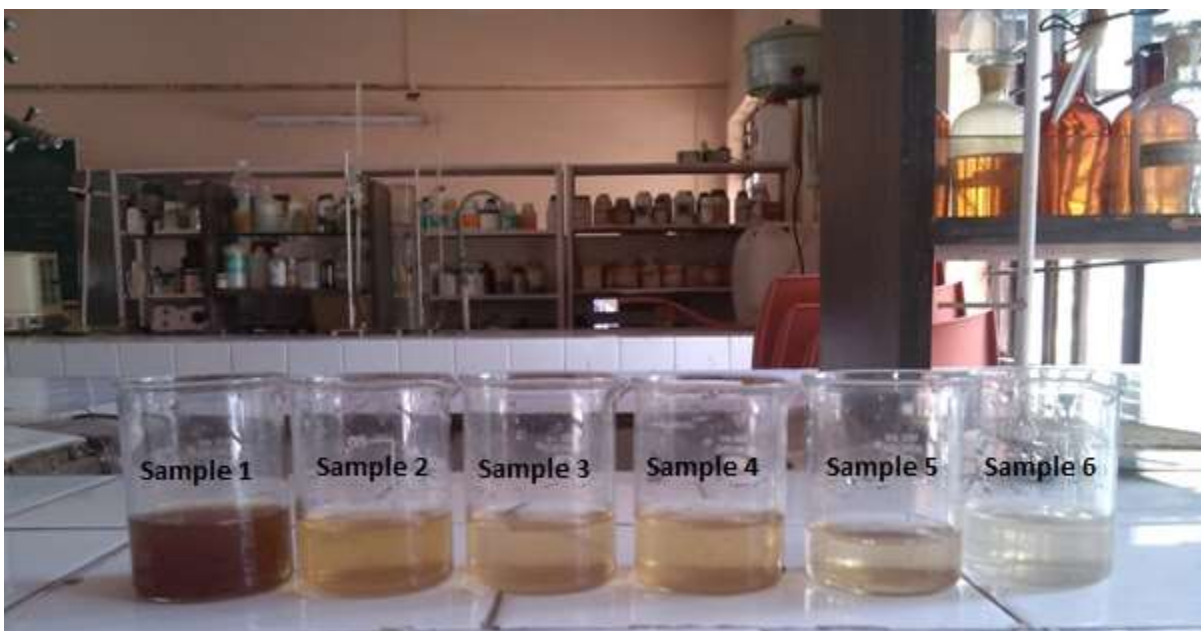
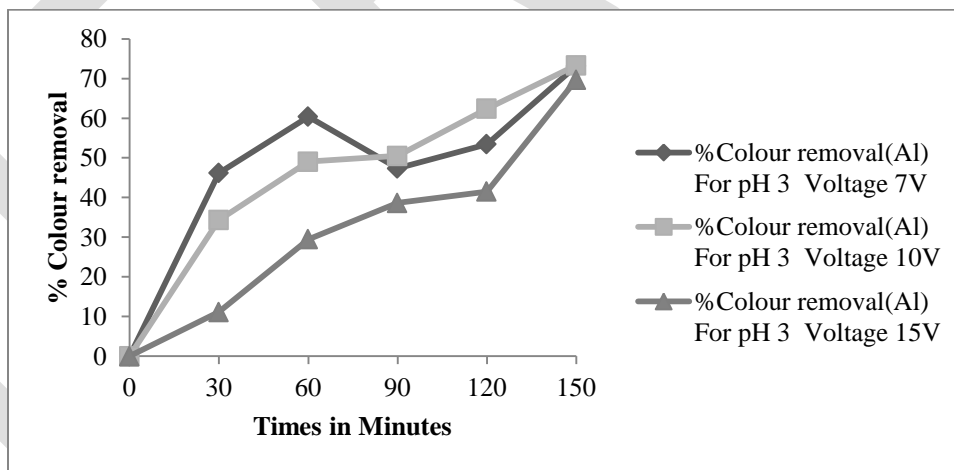


Figure No 5. Colour Removal Efficiency by using Electro coagulation

## 5. RESULT AND DISCUSSION

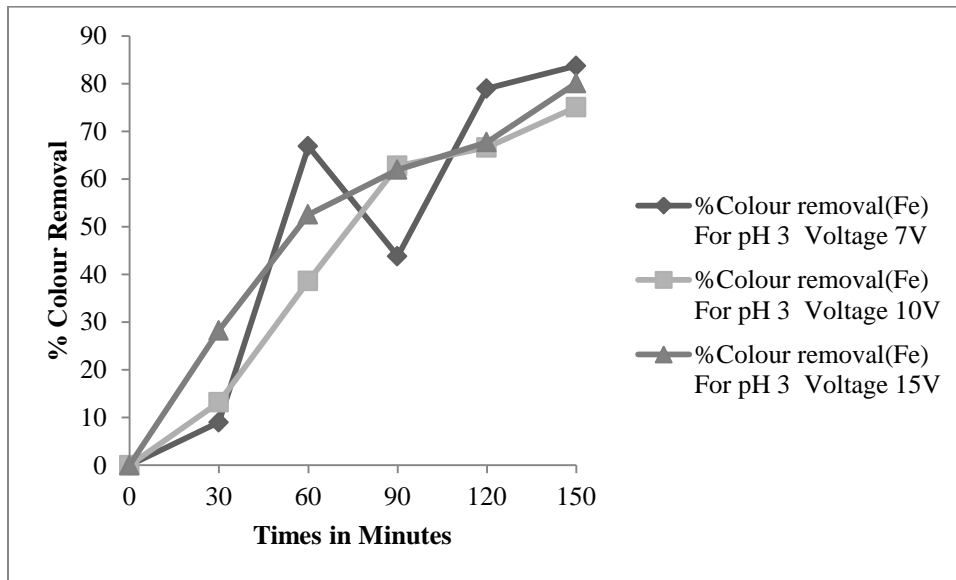
### 5.1 Effect of pH of the solution

Initially experiments were conducted on original solution pH 8. The colour removal efficiency 99.78 % and 97.42 % for Fe – Fe electrodes and Al-Al electrodes respectively. This efficiency is achieved by electro coagulation (EC) followed by filtration of sample. In electro coagulation process aluminum electrodes are dissolved and more oxidation of Iron electrodes.

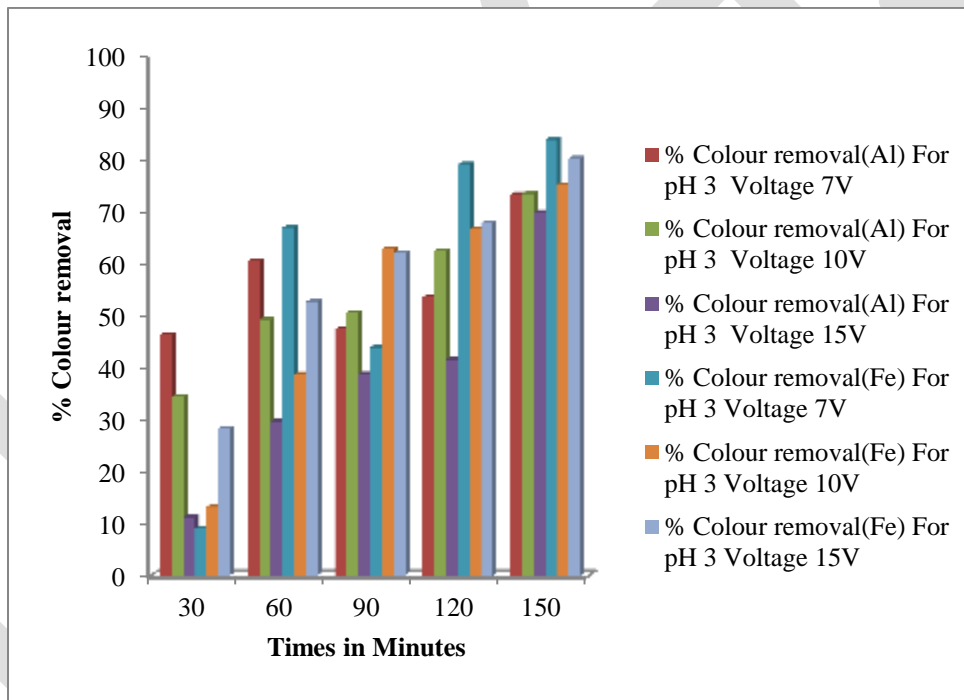


Graph No 1. % Colour Removal for Al –Al Electrodes at pH 3

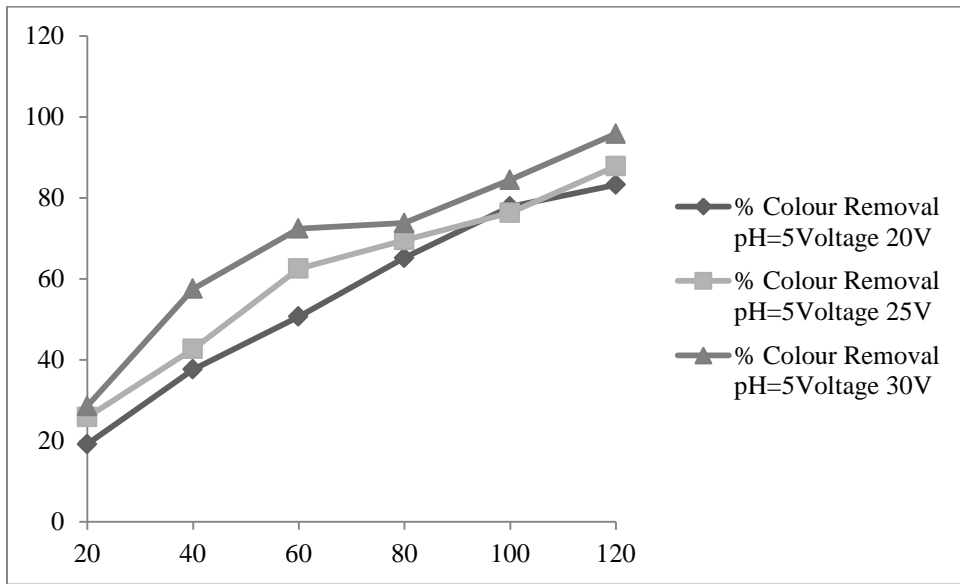




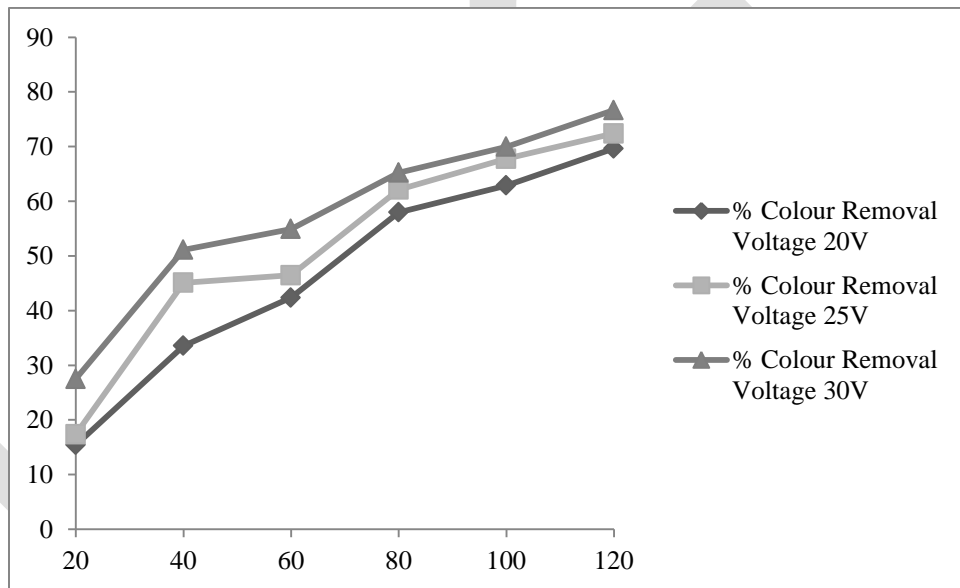
Graph No 2. % Colour Removal for Fe –Fe Electrodes at pH 3



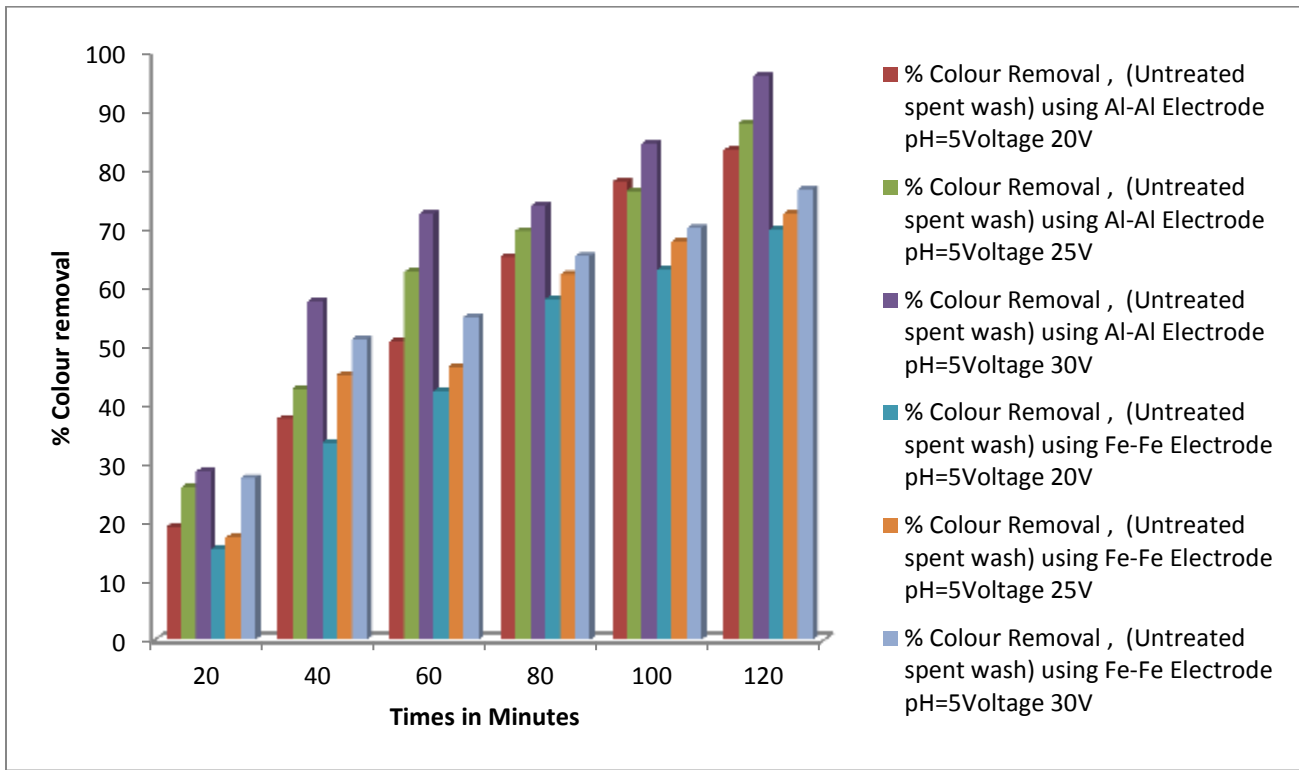
Graph No 3. % Colour Removal for Al - Al and Fe –Fe Electrodes at pH 3



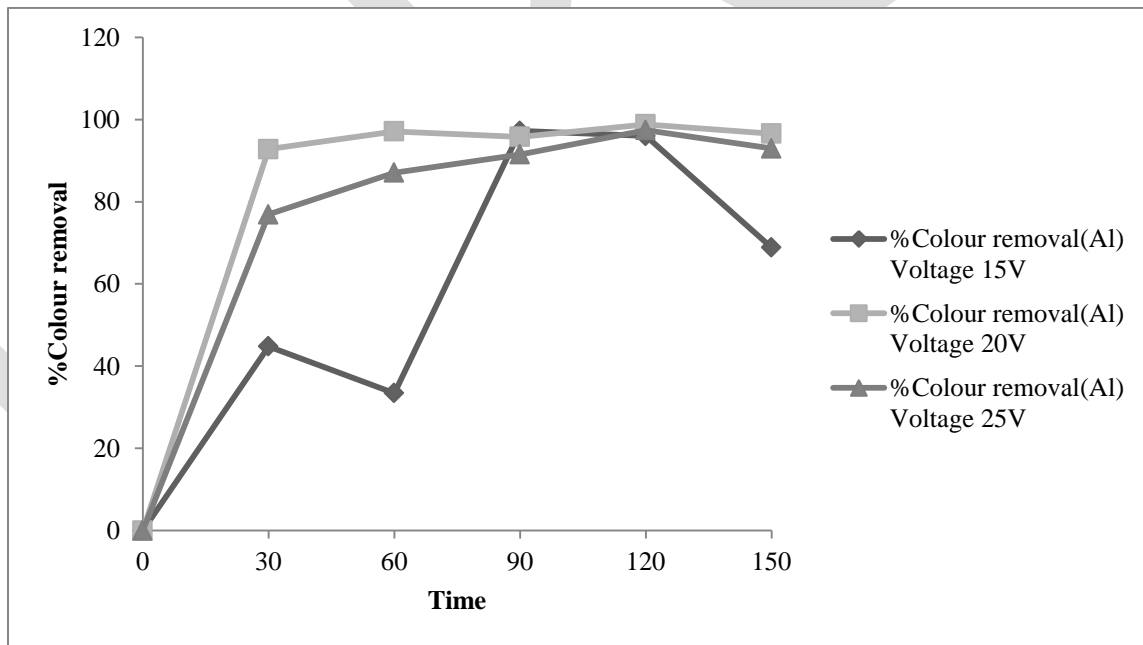
Graph No 4. % Colour Removal for Al - Al Electrodes at pH 5



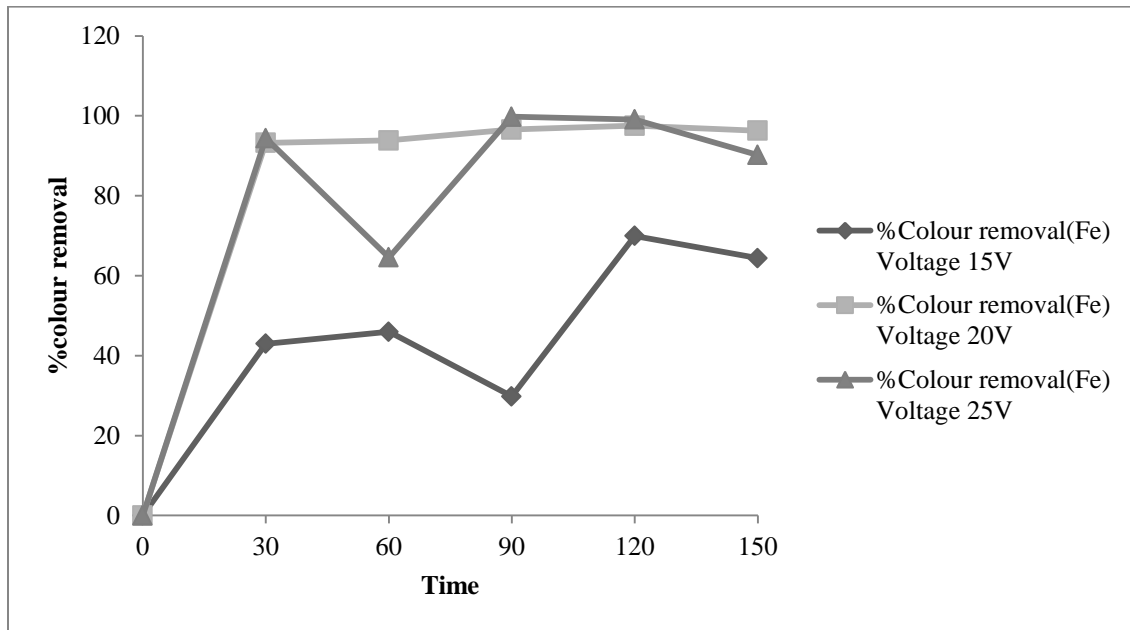
Graph No 5. % Colour Removal for Fe -Fe Electrodes at pH 5



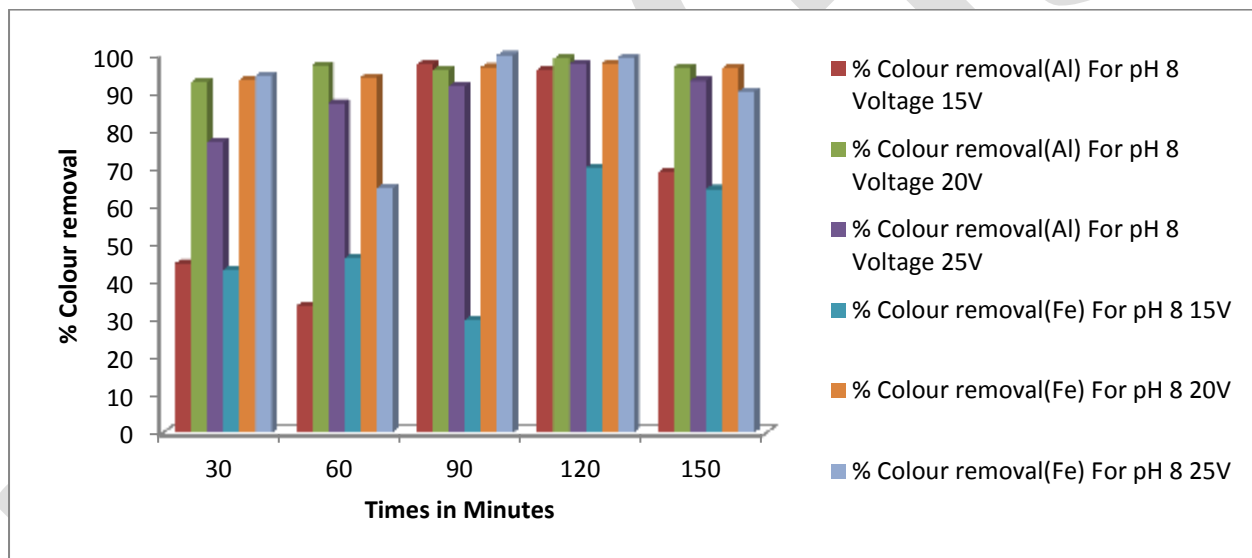
Graph No 6. % Colour Removal for Al - Al and Fe -Fe Electrodes at pH 5



Graph No 7. % Colour Removal for Al -Al Electrodes at pH 8



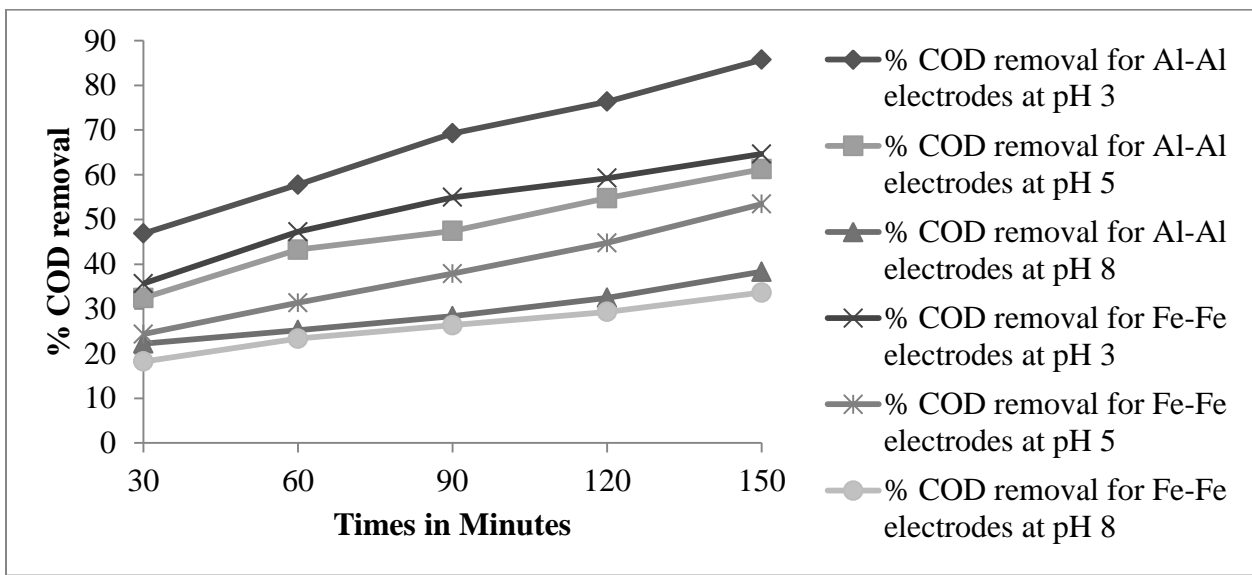
Graph No 8. % Colour removal for Fe-Fe Electrodes at pH 8



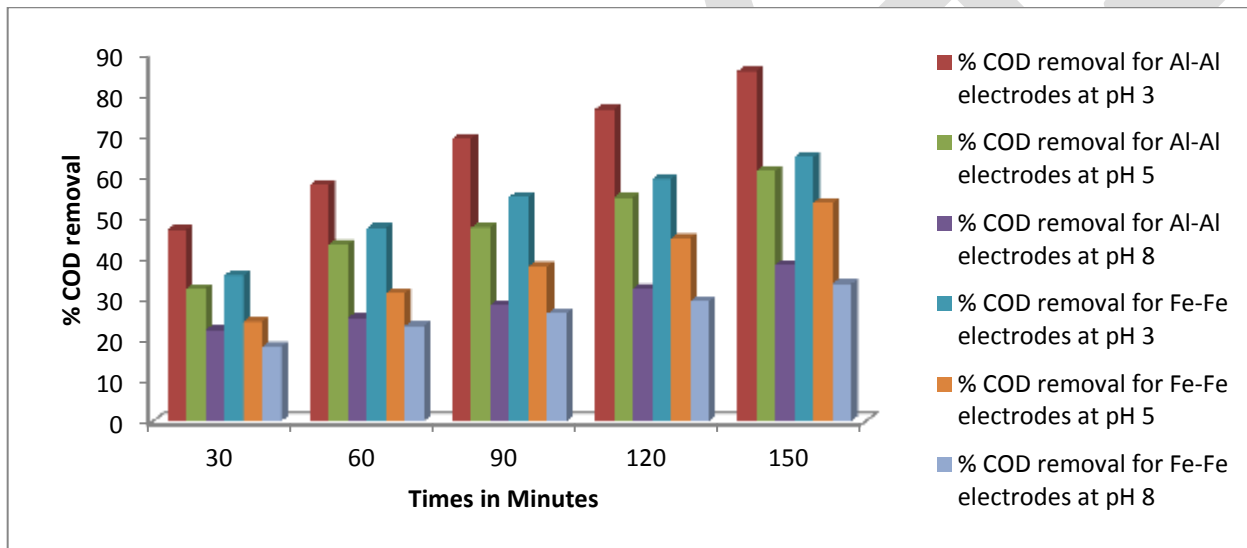
Graph No 9. % Colour removal for Al-Al and Fe-Fe Electrodes at pH 8

#### 4.2. Effect of pH on COD removal

Experiments were performed to check effect of different pH on COD removal. It is found that at lower pH maximum COD removal occurs because acidic condition is more favorable for COD removal. In acidic condition chlorine is present in the spent wash in the form of hypochlorous acid which has more oxidation potential. Al –Al electrodes remove maximum COD removal as compared to Fe-Fe electrodes.



Graph No 10. % COD removal by Al-Al and Fe- Fe electrodes at different pH



Graph No 11. % COD removal by Al-Al and Fe- Fe electrodes at different pH

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## 5. CONCLUSION

Treatment of distillery spent wash was carried out by using electro-coagulation in batch mode operation. Aluminum and iron type electrodes are used in the electro-coagulation (EC) process and optimum values of various parameters were obtained. The maximum 99.78 % removal of colour was obtained by using iron electrodes (Fe-Fe) at 25 volts for pH 8. For aluminum electrodes (Al-Al) maximum colour removed was 98.81 % at 20 volt for pH 8. In both cases this maximum colour removal efficiency was obtained by

electro coagulation followed by filtration by using watman paper number 42. Optimum COD removal found to be 85.71 % for pH 3 for an electro coagulation time of 150 minutes.

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