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Treatment of Distillery Spent Wash by Using Chemical Coagulation (CC) and Electro - coagulation [EC]

Manoj. P. Wagh[1,*], P. D. Nemade[2]

[1]D. Y. Patil College of Engineering and Technology Pimpri, Pune, Savitribai Phule Pune University, Pune, Maharashtra, India
[2]Department of Civil Engineering, S. B. Patil College of Engineering, Indapur, Dist: Pune-413106, Savitribai Phule Pune University, Pune, Maharashtra, India

*Corresponding author: profmpwagh@gmail.com

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Abstract There is an urgent need to find best suitable economic technology to knock out the problems due to distillery industries creating pollution and ecological imbalance. In the present study electro-coagulation treatment is carried out by using different combination of aluminum and iron electrodes in a batch reactor. Also chemical coagulation treatment is carried out by using alum and lime dose to treat distillery spent wash. Maximum 96.09% colour removal was obtained by using Al-Al electrodes for pH 8 and maximum COD removal was obtained 85.7 % by using Al-Al electrodes for pH 3. Further experiments are carried out by using alum and lime coagulant dose to treat distillery spent wash maximum 66. 27 % COD was removed by using alum. Alum is more effective than lime to remove chemical oxygen demand.

Keywords: *distillery spent wash, electro-coagulation, coagulation, melanoidin, recalcitrant, maillard reaction*

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1. Introduction

Distillery industries contributing the lion's share in the economic growth of the country. For the ethanol production Maximum Distillery industries present in the world are sugar base industries. Ethanol production in the distillery industry is only about 5 to 12 % by volume, it means 88 to 95 % waste water contain by volume of alcohol distilled [1,2,3,4]. Distillery spent wash contains dissolved impurities, nutrients added during the molasses fermentation, by-products of fermentation and decomposition products. The suspended impurities like dust, cellulosic fibers, etc. are usually removed before the concentration of the juice. However, water soluble hemicelluloses, proteins, gums, organic non-sugars and minerals present in the cane juice are present in the stillage in the original or converted forms exerting an oxygen demand during its treatment [5]. Typical BOD and COD values for a batch distillery spent wash are 35,000–50,000 and 80,000–100,000 mg/L, respectively, whereas for a continuous process, they are in the range of 60,000–100,000 and 160,000–200,000 mg/L, respectively [5]. These industries required large amount of water for the production of alcohol and also generate a large amount of spent wash. Spent wash is the effluent generated during the alcohol production. Spent wash is dark brown, highly acidic, having very high chemical oxygen demand and biochemical oxygen demand [6,7]. Spent wash contains 2% melanoidin dark brown recalcitrant pigment, caramel, furfural and having high molecular weight nitrogenous brown polymer form by Maillard reaction between the amino acid and sugar [8,9,10]. Melanoidin is highly putrid and prevent penetration of sunlight due to which dissolved oxygen present in aquatic is affected and indirectly photosynthesis process is retarded hence animal and aquatic plants are cannot live more, so a safe disposal method is requiring [11,12]. Economical and eco-friendly the distillery spent treatment is a great challenge to environmentalists and scholars. There are a number of methods to treat distillery spent wash specially 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 [13]. Aerobic treatment reduces the chemical oxygen demand (COD), and Biological oxygen demand up to 50 to 70 %, but till 100% colour, COD and BOD are not reduced. Implementation of physical, chemical and biological method to reduce the colour and chemical oxygen demand of spent wash generates significant amount of secondary sludge [14]. Table 1 show the similar result has been got by different researcher by chemical coagulation and electro coagulation.

Table 1. Electro coagulation Treatment Modified table Khandegar et al., 2014 [18]

Times in minutes	pH	Currents density A/cm^2	Anode – Cathode	% COD Removal	References
120	3	0.817	Al-Al, Fe-Fe	81.3 52.4	14
120	3	0.03	Al- Al	72.3	15
130	6.75	0.146	SS-SS	63.1	16
180	6.9	0.06	Pb - Pb	85.2	17
140	3	0.10	Fe-Fe	56	19
60	7.2	0.71	Al-Al	99	20

This work investigates the feasibility of comparing electro-coagulation and chemical coagulation treatment to remove the colour and chemical oxygen demand (COD) of distillery spent wash. Electro coagulation reaction occurring at the electrodes for aluminum and iron electrodes is as follows

For Aluminum electrodes [21]

At anode

$$Al \rightarrow Al^{3+} + 3e^- \quad (1)$$

At Cathode

$$Al + 3H_2O \rightarrow Al(OH)_3 + 3H^+ + 3e^- \quad (2)$$

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \quad (3)$$

For Iron electrodes [21]

At Anode

$$Fe(s) \rightarrow Fe^{2+} + 2e^- \quad (4)$$

$$Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2 \quad (5)$$

At Cathode

$$Fe + 2H_2O \rightarrow Fe(OH)_2 \rightarrow Fe(OH)_2 + H_2 \quad (6)$$

$$Fe^{2+} + 1.5H_2O + 0.25O_2 \rightarrow \gamma\text{-FeOOH(s)} + 2H^+ \quad (6a)$$

Fe was released to solution as Fe(II) [Reaction (4)] and was then immediately oxidized to Fe(III) due to water and oxygenated environment or by the dissolved oxygen. The Fe(III) then precipitated in the formation of iron oxides particularly Lepidocrocite (γ-FeOOH) at nanoscale [reaction (6a)] which is responsible for removal organic and inorganic COD and other metals ions, ultimately gives removal of colour and COD from wastewater. Distillery spent wash contains chlorides. By the passing the electric current generation of chlorine and hypo chloride irons occurs. It reacts with organic matter present is spent wash, and oxidation starts. Due to formation of hyphochorous acid and hyphochloride irons the organic matter decomposes because of their high oxidative potential. Following reaction takes place at anode and cathode. at anode

$$2Cl^- \rightarrow Cl_2 + 2e^- \quad (7)$$

$$2H_20 + 2e^- \rightarrow H_2 + 2OH^- \quad (8)$$

2. Materials and Method

Distillery spent wash was collected from Sanjivani S.S.K. (Sanjivani Sahakari Sakhar Karkhana) limited, Kopargaon, Dist: Nagar, Maharashtra State, India. The characteristics of distillery spent wash are shown in Table 2. Chemical oxygen demand (COD), measure the organic strength of spent wash, was determined by dichromate method (closed reflux, titrimetric method) [15]. Colour of spent wash is measured by using spectrophotometer NV-201 having wavelength range 400-700 λ. Maximum absorbance wavelength 425 λ was selected for experiment % colour removal efficiency is calculated by using equation.

$$C_R(\%) = \frac{C_i - C_f}{C_i} x100 \quad (A)$$

Where, C_R is the colour removal efficiency, C_i and C_f are initial and final colour removal of the sample.

Table 2. Characteristics of distillery spent wash

Parameter	Values
Colour	Dark brown
Odour	Unpleasant burn
pH	4.80
TDS (mg/L)	91700
TSS (mg/L)	26560
TS (mg/L)	118260
BOD (mg/L)	43000
COD (mg/L)	128000
Organic Carbon (%)	3.7
Nitrogen (mg/L)	1460
Phosphorus (mg/L)	326
Potassium (mg/L)	14300
Calcium (mg/L)	6800
Magnesium (mg/L)	4384
Chloride (mg/L)	10650

Figure 1 shows the graphical (Schematic) representation of electro coagulation treatment which consist pair of electrodes, aeration system and D.C. supply.

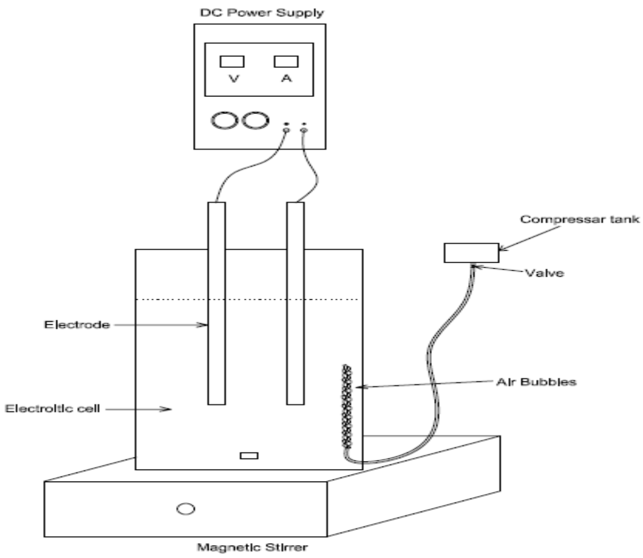

Figure 1. Electro coagulation Treatment

Figure 2. Experimental setup

Experiment was performed by taking 1000 ml of distillery spent wash in a beaker, combination of different electrodes are used such as aluminum- aluminum (Al-Al), iron- iron (Fe-Fe), aluminum iron in a batch mode operation. Aluminum and iron electrodes are having dimension 135x25x2mm, and dipping area is 110 x 25mm^2, the spacing between electrodes is kept 3 cm apart. Direct current was applied in range from 20 V to 30 V the content in the beaker was agitated by a magnetic stirrer (Make REMI) , electrodes were keep 1.5 cm above for easy stirring , agitation speed is kept constant 500 rpm Figure 2 shows laboratory working experimental setup. Initially 20 V. Power supply is applied started at t =0 and at regular interval of 20 minute sample was collected. The efficiency of electro- coagulation was determined in terms of COD removal of raw wastewater and treated wastewater after 20 minute interval. COD removal efficiency is calculated by using equation

$$Cc_R\,(\%) = \frac{Cc_i - Cc_f}{Cc_i} x100 \qquad \text{(B)}$$

Where, Cc_R is the COD removal efficiency, Cc_i and Cc_f are initial and final COD removal of the sample.

3. Results and Discussions

Electrocoagulation

3.1. Effect of pH on COD Removal

Figure 3 and Figure 4 shows variation in COD removal for different pH and experimental plan shown in Table 3. It is found that acidic condition is more favorable to remove the chemical oxygen demand (COD) of distillery spent wash due to decreased production of chlorine or hypochlorite at higher pH [15]. Al-Al electrodes are more effective than Fe-Fe electrodes because due to acidic condition iron electrodes are dissolved during performing experiments, maximum COD removal was 85.7% for Al-Al electrodes and for Fe-Fe 73.17%. Similar results are found by [18]. Figure 2 shows the experimental setup and colour removal by EC.

Table 3. Experimental Plan

electrodes	Voltage	pH	Agitation
Al-Al and Fe-Fe	20 V to 30 V	3,5,6,8	500 rpm

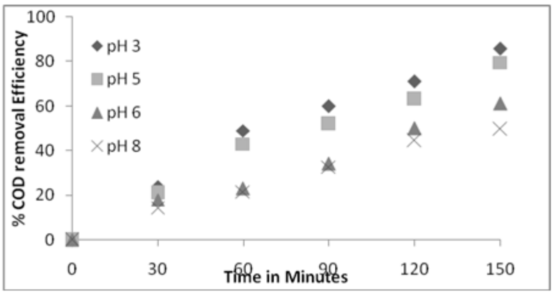

Figure 3. Effect of pH on efficiency of COD removal for Al-Al Electrodes

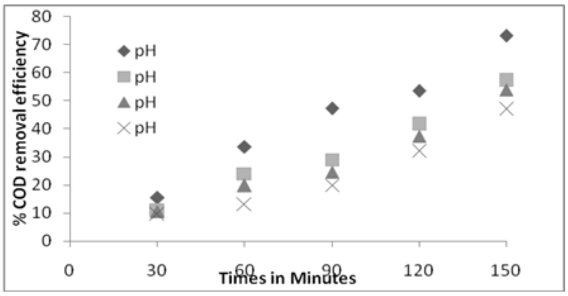

Figure 4. Effect of pH on efficiency of COD removal for Fe-Fe Electrodes

3.2. Effect of pH on Efficiency of Colour Removal

Figure 5 and Figure 6 shows colour removal efficiency of electro-coagulation which is decreases with increase in concentration of melanoidin, electrodes consumption increases with increase in concentration of melanoidin. Decolourization efficiency of melanoidin is increases with increase in conductivity [9,14]. Acidic condition is not favorable to removal colour of distillery spentwash, maximum colour removal can be achieved at pH 8 for Al-Al electrodes. Aluminium electrodes are remove maxium colour upto 96.09 % Aluminum electrodes are more effective than iron electrodes.Colour removal efficiency by using Al-Al electrodes are shown in figure 7. Appearance of electrode before and after Effect of [EC] is shown in Figure 8.

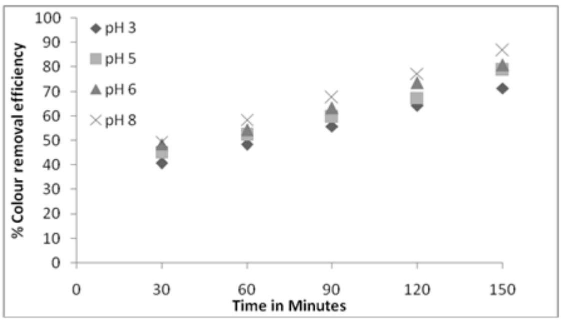

Figure 5. Effect of pH on efficiency of Colour removal for Fe-Fe Electrodes

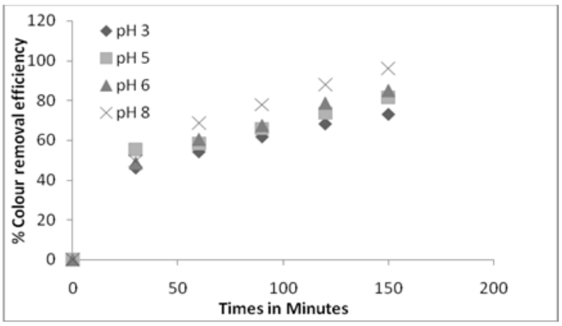

Figure 6. Effect of pH on efficiency of Colour removal for Al-Al Electrodes

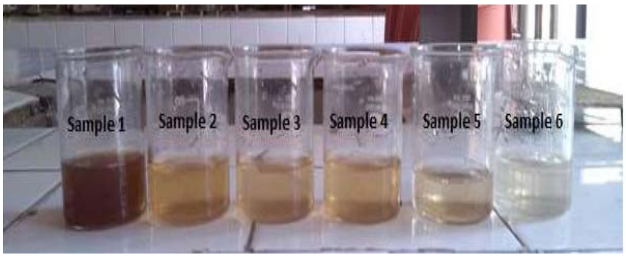

Figure 7. Colour Removal Efficiency by using Electro coagulation (EC)

Figure 8. Electrodes before and after [EC]

Operating cost is the essential parameter, on which application of treatment is depend; operating cost includes cost of material (electrodes) and cost of electricity consumption. In this paper cost of electrode is very less as compared to electric consumption. EEC is shown in Table 4.

Operating Cost = a x Electrode consumption + b x Electricity Consumption

Where a and b is current market price of electrode material and current electricity price Rs/Kwh respectively.

Electric consumption is increases with increase in concentration of COD; approximate operating cost in this work is around 385 Rs / m^3

Table 4. Electrical Energy Consumption (EEC) for Electrodes

Electrode Types	Volume Treated	Currents density A/cm^2	[EEC] $[kWh/m^3]$	% COD Removal
Fe – Fe	1000 ml	0.032	0.0016	73.1
Al - Al	1000 ml	0.04	0.002	85.7

3.3. Chemical Coagulation Treatment

Alum

Figure 9 shows removal of COD using alum as a coagulant to treat distillery spent wash. Different coagulant dose such as 1 gm, 2 gm, 3gm and 4 gm of alum is mix with raw distillery effluent then mixture is allowed to stirrer rapidly for 10 minutes then slow agitation is carried out for half hour in a jar test. Then sample is filtered then its COD is determined by dichromate open reflex method maximum COD removal is 66.27 % at pH 5.

$Al_2(SO4)_3.18H_2O + Ca(HCO_3)_2 \rightarrow 2Al(OH)_3.18H2O\downarrow + 3CaSO_4 + 6CO_2$ ---------- (C)

$Al_2(SO4)_3.18H_2O + 3mg(HCO_3)_2 \rightarrow 2Al(OH)_3 + 3mgSO4 + 6CO_2$ ----------- (D)

Reaction A and B indicate removal of excess oxygen by precipitating with hydroxide and forms as an aluminum hydroxide.

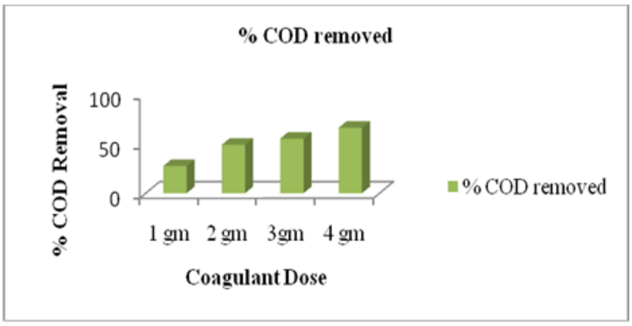

Figure 9. Effect of (Alum) coagulant dose on the COD removal efficiency

Lime

In another experiment lime is used as a coagulant to treat distillery spent wash with varying dose of 3gm, 6 gm, 9gm and 12 gm dose of lime was applied and allowed to mix rapidly in a jar test for 10 minutes then after 30 minutes for slow agitation, maximum COD removal is 51.43% as shown in Figure 10. Alum is more effective than lime to remove the chemical oxygen demand (COD) of distillery spent wash because aluminum present in Al^{+3} state, while calcium exists in Ca^{+2} state. Hence alum is more effective than lime, alum react vigorously with oxygen as compared to lime.

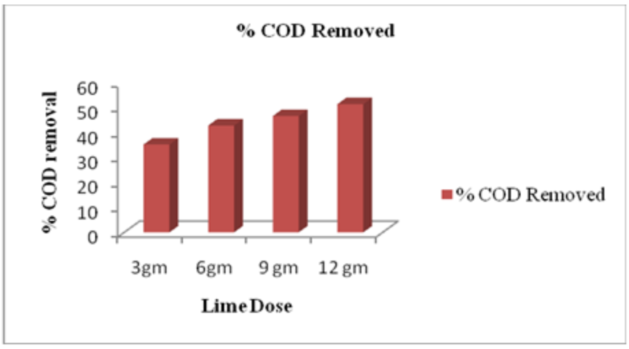

Figure 10. Effect of (Lime) coagulant dose on the COD removal Efficiency

4. Conclusion

Electro coagulation method is more effective than coagulation method. The electro –coagulation of distillery spent wash is carried out in a batch mode and optimum COD removal found to be 85.7 % for Al-Al electrodes, optimum voltage was 30 volt. Optimum decolourization of spent wash found to be 96.09% for 30 volt. So it is concluded that electro coagulation method implemented successfully to treat distillery spent wash.

List of Abbreviations

Al-Al Aluminum - Aluminum Electrodes
Fe-Fe Iron –Iron Electrodes

Pb-Pb. – Graphite – Graphite Electrodes
COD- Chemical Oxygen Demand
EEC – Electrical Energy Consumption

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