# Remediation And Decolourization Of Distillery Spent Wash By Using Advanced Oxidation Processes – A Review

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Distillery industries are highly responsible for the tremendous generation of effluent known as distillery spent wash (DSW). Distillery spent wash is caramelized, recalcitrant toxic pollutant, comprehensive undesirable intense dark brown colour non-consumed liquid accompanied by high COD, BOD, highly acidic pH, containing organic and inorganic poisonous ingredient which depends on the raw material furnished. Distillery spent wash also contains sugar decomposition products, such as anthocyanin, tannin and xenobiotic compounds. It causes aquatic and soil pollution due to strong brown colour recalcitrant melanoidin pigment which results in the obstruction of photosynthesis, eutrophication and low pH that acidifies the soil, affecting crop growth. Thus, creating an ecological imbalance and big environmental hassle. Hence, there is the urgent need for removal of colour and pollutants from distillery effluent which has become essential for green chemistry and is approved hygienically. Novel advanced technologies have been used to reduce colour and COD. The current review paper gives an insight of electrocoagulation process and advanced oxidation processes, such as ozone, fenton, UV, hybrid treatment to treat distillery spent wash.

# **KEYWORDS**

Ozonation, Advanced oxidation, Wastewater, COD, Colour, Electrocoagulation, Distillery spent wash, Biodigested effluent

### 1. INTRODUCTION

In India, there are 319 distilleries generating 40.4 billion litre of unwanted residual liquid waste called DSW [1]. On an average 1 L of alcohol, production generates 12-15 L of wastewater [2,3]. The distillery industry generates three types of effluents - raw spent wash (SW), biodigested effluent (BDS) and lagoon effluent (LE) [4]. COD of raw spent wash is 1,10,000-1,90,000 mg/L and BOD is 50,000-60,000 mg/L [5,6]. As per the Ministry of Environment and Forest (MoEF) and Central Pollution Control Board (CPCB), distilleries are one of the topmost red industries in India, generating pollutants (distillery spent wash) having high COD, BOD, TDS, organic and inorganic toxic constitutes [7,8]. Apart from that, a pollutant has low pH, very strong odour, dark brown colour and contains dissolved impurities [9]. DSW is the highly objectionable viscous liquids which affect the surrounding area of distillery industry, primarily who furnish alcohol from the fermentation and subsequent distillation of sugar cane molasses [10]. During ethanol production, spent wash is generated and their characteristics depend on the feed of raw material [11]. The colour of molasses distillery wastewater is mainly allocated due to melanoidin alkaline degradation as a consequence of hexoses, polyphenols, caramels [12]. Melanoidin ( $C_{17-18}$  $H_{26-27}O_{10}N$ , molecular weight - 5000-40,000) is formed by Maillard reaction between the amino acid and carbonyl group [13]. Biological treatments, such as anaerobic digestion activated sludge process (ASP), anaerobic lagoon and aerobic process are found to be ineffective to degrade the melanoidin [14,15,16]. Physico-chemical technology is found to be inefficient to handle the melanoidin [17]. The present study emphasizes on remedial methods, like EC process, a combination of electrocoagulation with advanced oxidation process, such as ozonation, ozone assisted electrocoagulation, fenton, UV, the hybrid method to biodegradation and decolourization of distillery effluent.

# 2. DISTILLERY SPENT WASH GENERATION AND CHARACTERISTICS

Figure 1 and table 1 illustrates step by step process of extraction of cane juice, sugar production, molasses as a secondary product and spent wash generation from molasses. Sugarcane is extracted in sugar mill to produce sugar and bagasse as the byproduct, which is a fuel for boiler during the fermentation process. Extracted sugar is heated to evaporate water. The crystallization process is carried



Table 1. Distillery spent wash generated in different operation

Distillery operation	Parameter					Average WW	Specific WW	
	Colour	рН	TS	SD	BOD	COD	generation (KL/day)	generation (KL/alcohal)
DSW (dstillation)	Intense dark brown	4-4.5	100000	10000	45000- 60000	80000- 120000	511.4	11.9
Fermenter chilling	Colourless	6.26	1000-1300	220	100-110	500-1000	307.7	7.2
Fermenter clearout	Colourless	5-5.5	1000-1500	400-600	500-600	1200-1600	108.2	2.5
Condenser cooling	Colourless	6.8- <b>7</b> .8	700-900	180-200	70-80	200-300	34.2	0.8
Flooring wash	Faded	6	550	300	15	25	47.6	1.1
Bottling plant	Colourless	7.45	400	100	5	15	126.9	3.0

**Table 2.** Characteristics of untreated and aerobically distillery effluent in mg/L [2,8]

Parameter	Untreated distillery	Anaerobically treated effluent
рН	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 solids	1,10,000-1,90,000	70,000-75,000
Total volatile solids	80,000-1,20,000	68,000-70,000
Total suspended solids	13,000-15,000	38,000-42,000
Total dissolved solids	90,000-1,50,000	30,000-32,000
Chloride	8000-8500	7000-8000
Phenol	8000-10,000	7000-8000
Sulphate	7500-9000	3000-5000
Phosphate	2500-2700	1500-1700
Total nitrogen	5000-7000	4000-4200

out to pass the supersaturated solution to recover the sugar in crystal form. Effluent after retaining the crystallization process is called molasses, which contains a high percentage of sucrose, fructose and glucose. Molasses is diluted 1-3 fold and feed to fermentation plant for production of ethanol, rectified spirit and alcohol. During fermentation process Saccharomyces cerevisiae and Saccharomyces pombe bacteria, yeast and nutrient are added [18]. Ethanol is produced by the fermentation and distillation process. To dispose of distillery effluent, primary anaerobic treatment (biomethanated) is carried out, which reduces the COD upto 65-72% and BOD upto 80-85% means 35% COD and 20% BOD still remains in the biodigested effluent (BDE). Table 2 shows the characteristics of distillery effluent before anaerobic treatment and after anaerobic treatment. The table reveals anaerobic treatment is the primary treatment to treat the distillery effluent,

although not adequate it requires the post-treatment to safe disposal of the effluent.

# 2.1 Treatment technologies for distillery spent wash

Waste treatment aims to safely dispose of effluent into the environment. To treat the cumbersome distillery spent wash a number of advanced oxidation techniques are implemented by researchers to minimize the dark brown colour of distillery effluent and degradation of melanoidin. This review paper summarized electrocoagulation (EC) treatment, advanced oxidation process, such as ozone treatment, ozone assisted electrocoagulation, fenton process, UV and hybrid treatment to distillery wastewater.

2.1.1 Electrocoagulation: In recent years electrocoagulation (EC) is used as a powerful technique for removal of pollutants from distillery wastewater as compared to other conventional treatment. The EC has great potential to eliminate the limitations of classical methods used for the treatment of distillery effluent [19]. EC has been successfully executed by various industries, such as baker's yeast wastewater, laundry wastewater, dairy industry wastewater, electroplating rinse water, metal cutting wastewater, poultry slaughter house wastewater, textile wastewater, fertilizer plant wastewater, to remove high metals, colloidal and suspended particles, oil and greases [20]. The XRD spectrum of sludge generated in the EC indicates that sludge production in EC process is proportional to current density and operating time. The amount of sludge produced indicates a number of pollutants removed.

The EC process consists of a pair of electrodes made up of aluminium or iron immersed in distillery effluent. As per the Faraday's law during EC process, hydrogen gas is evolved at the cathode by consuming sacrificial anode [21]. In practice generally, aluminium, iron, graphite and steel electrodes are used. To achieve maximum efficiency in EC process,

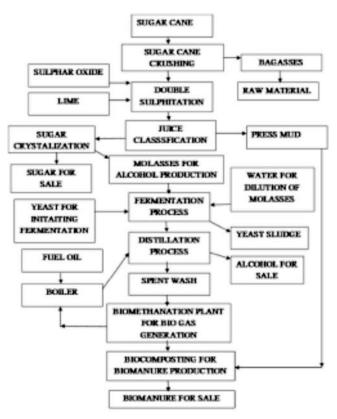


Figure 1. Sugar and alcohol processing

ohmic potential drop (IR) must be curtailed, agglomeration of H, and O, gas bubble at the exterior electrode curtailed and barrier to mass transfer through the spacing between the two electrodes must be minimum [22]. The EC process is entanglement process which consists exorbitant influence that can occur between the essential activity, such as electrochemistry, coagulation, sedimentation and flotation that arise within an EC reactor [23]. Distillery spent wash has been treated by using the Box-Behnken design of EC cell with iron electrodes and found 95% decolourization [24]. The electrochemical treatment with stainless steel plate as electrodes was used with a spacing of 2 cm between the electrodes, optimum COD removal was 58% for 120 min with a current density of 0.12 A/cm<sup>2</sup> [25]. EC process has been successfully implemented to treat the distillery spent wash by using aluminium and iron sheets as an electrode material, the ideal value of current density was established to be 71.8 mA/cm<sup>2</sup> at an initial pH of 7.2 for the effluent [26].

EC process has been executed to treat rice grain based distillery effluent where energy consumption and the effect of pH variation on the percentage of COD removal and colour removal are taken into consideration. EC treatment was unable to remove

colour 100% and to achieve 100% decolourization, EC must be followed by membrane separation [27]. RuO<sub>3</sub>-Ti and stainless steel electrodes are used to treat the distillery spent wash, 92% COD was reduced and achieve 98.1% BOD reduction [28]. Electro-Fenton (EF) and EC treatment were carried out to treat the distillery spent wash, 92.6% COD was reduced and found EF is more effective [29]. Electrochemical degradation of distillery spent wash was carried out by using stainless steel electrode and titanium electrode coated with ruthenium oxide, the decolourization efficiency increases with the chloride concentration whereas combined effects optimized the result and regression models show 83.31% colour removed and COD degradation by 39.66% [30]. Electrochemical treatment was processed on rice grain based distillery biodigester effluent (BDE) using as the sacrificial iron electrode with pH range of 3.5-9.5, current density (j) of 99.3 A/m² and electrode gap of 1.5 cm has shown 83% COD reduction and 69% colour reduction [31]. Electrocoagulation treatment implemented by using aluminium electrodes as a sacrifice electrode is favourable for remove of colour, 96.09% colour removed for pH 8 and COD removed upto 85.7% for pH 3 [32]. Electrooxidation process was carried out to treat the distillery effluent, the electrode madeup of titanium shows higher potential to reduce wastewater, the COD removed 89.62% and colour removed 92.24% [33].

2.1.2 Effect of current density, electrolysis time and pH: In an EC process, the current density is one of the important parameters which affect the operating cost and interpretation for controlling the reaction rate. Faraday's law relates the association between EC rate and current density, thus EC rate increases with an increase in current density. Electrolysis time enhance the treatment effectiveness of the EC process, as electrolysis time increase EC process highly stimulate at an optimum time. Decolourization directly depends on the concentration of hydroxyl and metal iron generated on electrodes during the EC process. Colour and COD removal of distillery effluent depend on pH variation. Acid condition stimulates the rate of degradation of distillery effluent. The rate of decolourization of distillery effluent enhances for pH 7-8. The drawback of EC process is sludge generated on large scale and secondary treatment is required. Table 3 indicates the different investigations carried out by the researcher to show the percentage degradation of colour and COD on the application of different electrodes. Figure 2 illustrates the graphical representation of EC process involving different

Table 3. COD and colour removal by electrocoagulation

		1	
Electrode used	COD removal, %	Colour removal, %	Reference
Al-Al electrodes	99.41	_	[20]
Fe-Fe electrodes	93	87	[17]
Graphite electrodes	80.6	95.6	[28]
Lead dioxide coated on titanium	90.8	98.5	[29]
Ruthenium oxide- coated titanium mesh acting as anode and stainless	39.66	83.39	[30]
Al-Al electrodes	93	76	[17]
Al-Al electrodes	85.70	96.09	[33]
Al-Al electrodes	60	89	[41]
Iron electrodes	61.4	98.4	[50]

electrodes and its percentage efficiency in removal of COD and colour.

#### 2.2 Advanced oxidation process

Advanced oxidation process includes the ozonation, ozone assisted electrocoagulation, fenton reagent, UV and hybrid treatment.

**2.2.1 Ozone treatment**: Ozone molecule  $(O_3)$  is a triatomic state of oxygen. It is an extremely versatile, powerful oxidant (oxidation potential 2.07 V), highly unstable and frequently regresses readily into oxygen (O<sub>2</sub>) [34]. Ozone can be generated in situ because it cannot be stored by compression. Ozone solubility depends upon the temperature, as temperature increases solubility decreases. Ozone is not fully soluble in water. Ozone having two modes of oxidation one is direct oxidation and the other is self-decomposition. Direct oxidation is comparatively slow and abundantly selective than selfdecomposition method. Chemical oxidation method with ozone was used to treat biologically pre-treated molasses, reaction time was influenced, 71-93% colour removed and COD reduction from 15-25% was reached after 30 min reaction time [35]. Ozone is a vital oxidizing factor which oxidizes unsaturated bonds and leads to oxidation products. But ozone dosage is less effective in eliminating organic matter as it generates organic compounds that also subscribe to COD and rarely causes complete mineralization of organic matter to carbon dioxide and water. Ozone oxidizes functional groups liable for colour, but it just changes chromospheres groups. Ozone does not break brown polymers into smaller compounds.

Ozonation is a cost-effective process for tertiary

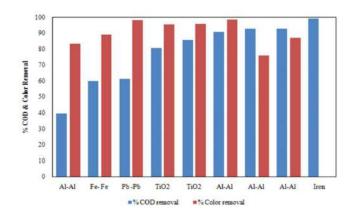


Figure 2. Electrocoagulation process involving different electrodes to remove the colour and COD

treatment of industrial wastewater as the ozonation breakdowns high atomic weight of components into lower atomic weight components but could not completely mineralize the organic matter [36]. Ozone treatment enhances the biodegradability in the absence of carbonates, the reuse of hydroxyl radicals, the ozonation of wastewater at high pH results in a coherent process, prime to a luminous removal of COD, TOC, TKN [37]. During ozone decomposition, the hydroxyl radicals produced in aqueous solutions are the sweep by bicarbonate ions in the molasses effluent [38]. Ozone in presence of tin oxide as a catalyst not only accelerates the ozone oxidation reaction but also accelerates the oxidative decolourization of molasses fermentation wastewater and the decolourization of wastewater under the influence of pH value of reactant [39]. Ozonation is highly efficient and effective disinfection agent, simple in operation and lowers the production of detrimental products or no sludge production. The only major drawback of this system is the highly expensive and mere ozonation treatment is not effective to remove the colour of distillery effluent.

2.2.2 Ozone assisted electrocoagulation: In the EC process, tremendous amounts of sludge are generated and require secondary treatment to handle the sludge. This drawback can be overcome by using ozone assisted electrocoagulation. Individual ozonation and separate electrocoagulation are less effective as compared to combine methods. Ozone reacts with an aqueous solution consisting of organic, inorganic constitute directly or indirectly by direct ozone attack or indirect free radical, involving hydroxyl radical in water by ozone decomposition [40]. Ozone assisted electrocoagulation is more effective than mere ozonation and electrocoagulation [41]. Table 4 shows the advanced oxidation process implemented by the different authors for degradation of distillery effluent.

Table 4. COD and colour removal by advanced oxidation process

Method	COD removal, %	Colour removal, %	Reference
Ruthedium dioxide coated on titanium	92.6	=	[29]
Electrooxidation in presence of NaCl with Ti/RuO <sub>2</sub> grid cathode and commercial titanium sponge anode	89	92	[33]
Ozone	15-25	80	[35]
Ozonation (O <sub>3</sub> )	13	76	[38]
$O_3 + H_2O_2$	23	89	
Ozone assisted electrocoagulation	45-92	100	[38]
H <sub>2</sub> O <sub>2</sub> + TiO <sub>2</sub>	52-58	_	[42]
H <sub>2</sub> O <sub>2</sub> + Fe + Clay	34-45		[42]
O <sub>3</sub> + H <sub>2</sub> O <sub>2</sub>	Nil	Nil	[45]
O <sub>3</sub> + UV radiation	21.5		
UV radiation + H <sub>2</sub> O <sub>2</sub>	38		[45]
FSBAR + Ozone + PAC	81	96	[45]
Flocculation + Ozone + Fenton	79	_	[47]
Electrochemical + Fenton	=	94	[48]
Catalytic-sub and supercritical	98.2	_	[49]

2.2.3 Hybrid treatment (ozone, fenton and ultraviolet treatment): Photocatalytic advanced oxidation involving use of heterogeneous combination of H<sub>2</sub>O<sub>3</sub> oxidant with photocatalyst, like titanium dioxide and clay containing iron minerals is an effective medium to reduce biodegradable organic matter as well as suspended solids present in wine industry effluents, where it was revealed that  $H_2O_2$  + clays combination requires 3 to 6 times less dosage of H<sub>2</sub>O<sub>2</sub> than H<sub>2</sub>O<sub>2</sub> + TiO<sub>2</sub> combination [42]. A number of industries are using hydrogen peroxide with a small quantity of ferrous (Fe<sup>2+</sup>) as a catalyst to treat organic pollutants. Organic compounds present in wastewater are oxidized completely by the hydroxyl radical formed by Fenton with the generation of carbon dioxide and water as a residual product. Hydrogen peroxide is preferred by many industries to increase the biodegradability and enhance the oxidation rate, massive reduction in toxicity and pretreatment to industrial effluent prior to biological treatment. Ozone treatment in presence of Fe<sub>2</sub>O<sub>3</sub> catalyst iron catalyst gave the highest efficiency in both colour and COD removal as the hydroxyl free radical generated from the catalyst is more reactive than ozone molecules [40,43].

Individual ozone treatment is less efficient as compared to hybrid treatment followed by the combination of ozone fenton, ozone-hydrogen peroxide, ozone, UV and hydrogen peroxide [44]. Ozone, UV,  $\rm H_2O_2$ ,  $\rm Fe^{2+}$  shows 100% colour and chemical oxygen demand removal with minimum energy utilization of 0.01 kWh/ m<sup>3</sup> [9,44]. Ozone in combination with UV radiation enhances the degradation of colour and COD of spent wash. The combination of ozone and hydrogen peroxide is less effective to treat spent wash [45]. Polyaluminum chloride (PAC), fungal sequencing batch aerobic reactor (FSBAR) and ozone process are carried out to treat distillery spent wash, this hybrid treatment obtains 96% decolourization and 81% COD removal [46]. During depuration of distillery wastewater by using flocculation, ozonation and Fenton's process, as BOD/COD is raised then fenton process and ozonation process has improved COD abatement, increasing in H<sub>2</sub>O<sub>2</sub> and petering out COD [47].

The hybrid method is more effective than the individual electrochemical or Fenton method for biodigesting effluent of distillery spent wash [48]. Homogeneous (MnSO<sub>4</sub>.7H<sub>2</sub>O, ZnSO<sub>4</sub>.7H<sub>2</sub>O) and heterogeneous (TiO2, CuO, MnO2) catalyst has great influence on COD and colour removal efficiency of distillery effluent, COD conversion is strongly depending upon the reaction temperature, MnO, catalyst remove COD upto 98.2% in 30 min at 400°C catalyst loading of 5% weight, pressure 25 Mpa [49]. Central composite design (CCD) and SEM analysis of SS plate electrode, shows a number of dents and scum obtained in the EC process having greater heating than sludge and can be used for making blended fuel briquettes with organic fuels [50]. Table 5 illustrates the percentage colour and COD removal by AOP.

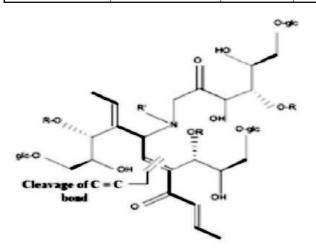
Hybrid method which consists of EC process and advanced oxidation process, such as EC, ozone-EC, peroxi–EC, photo–EC and peroxi–photo–EC, use to treat the distillery effluent which illustrates the significant interpretation, that hybrid method is a prominent technique to handle cumbersome complex distillery industry effluent [51]. Figures 3 and 4 shows percentage efficiency of advanced oxidation processes in removal of colour and chemical oxygen demand.

#### 3. CONCLUSION

From this review, it is clear that electrocoagulation process plays a key role to eliminate the colour, turbidity and chemical oxygen demand. EC rate is

Table 5. Advanced oxidation process to treat distillery effluent [44,49]

System Initial concentration COD (mg/L)		Time, hr	Operating condition		COD removed (%)	Colour removed (%)
			Catalyst	Oxidant		
Ozonation	5071	1/2	Fe <sub>2</sub> O <sub>3</sub>	Ozone	65	_
Ozone assisted electro-coagulation	2500	4	None	KI	83	_
O <sub>3</sub>	1500	4	FeSO <sub>4</sub> .7H <sub>2</sub> O	H <sub>2</sub> O <sub>2</sub>	29.17	39.62
O <sub>3</sub> /UV	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	41.67	50.62
O <sub>3</sub> /Fe <sup>2+</sup>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	48	53.95
O <sub>3</sub> /UV/Fe <sup>2+</sup>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	59	57.71
H <sub>2</sub> O <sub>2</sub> /Fe <sup>2+</sup>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	53.85	55.97
UV/H <sub>2</sub> O <sub>2</sub>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	66.67	73.72
O <sub>3</sub> / UV/H <sub>2</sub> O <sub>2</sub>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	92	81.72
UV/H <sub>2</sub> O <sub>2</sub> /Fe <sup>2+</sup>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	99.99	100
O <sub>3</sub> /UV/Fe <sup>2+</sup> /H <sub>2</sub> O <sub>2</sub>	1500	4	Sulphate heptahydrate	H <sub>2</sub> O <sub>2</sub>	100	100



| Necessaria | Nec

Figure 3. Ozone assisted electrocoagulation process cleavage carbon-carbon double bond of mela-noidin structure [10]

highly influenced by electrodes, pH, electrolysis duration, the concentration of effluent, conductivity, and current intensity. Electro-coagulation process has restrictions as this process produces sludge on a large scale, the sludge rate is proportional to the current density, operating time and concentration of effluent. The drawback of EC process can be overcome by using ozone assisted electrocoagulation process. Ozonation process is effective when hydraulic retention time is increased. Ozone assisted electrocoagulation process enhance the decolourization rate and COD degradation rate with less power consumption. Advanced oxidation process includes UV, fenton reagent, photo-fenton system and hybrid technology, which combines more than two processes and gives the best result than

Figure 4. % removal of COD and colour using advanced oxidation process

the individual process. Ozone, UV,  $Fe^{2+}$ ,  $H_2O_2$  are more efficient to treat the highly organic effluent, such as distillery spent wash. Thus there is a need to implement advanced oxidation process technology for the treatment of real distillery effluent.

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