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Comparison of treatment potential of electrocoagulation of distillery effluent with and without activated *Areca catechu* nut carbon

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Abstract

Physico-chemical characteristics of distillery effluent samples have been determined. The water quality parameters (WQPs) measured are colour, pH, electrical conductivity, turbidity, total dissolved solids (TDS), chloride, total hardness (THA), calcium, iron, bio-chemical oxygen demand (BOD) and chemical oxygen demand (COD). Almost all the values of WQPs of the distillery effluents have been found to be very high and well above the permissible limit suggested by Bureau of Indian Standards (BIS). Electrocoagulation (EC) technique is employed to treat the distillery effluents. Removal efficiency of WQPs is compared by adopting EC technique with and without the addition of indigenously prepared activated areca nut [botanical name: *Areca catechu*; kotta pakku, in (vernacular)] carbon (AAC). About 99% of turbidity has been removed in both the cases. The experimental results also revealed that the WQPs like EC, TDS, etc. of the effluents could be reduced. Loss of weight of sacrificial electrode (anode) is also ascertained. EC with AAC is found to be more effective than EC without AAC.

Keywords: Electrocoagulation; Activated areca nut carbon (Areca catechu) (AAC); Aluminium and iron electrodes; Distillery effluent

1. Introduction

There are nearly 290 distillery units present in India, of which, 17 units are located in Tamil Nadu. All these distillery industries use molasses obtained from sugar industries as raw material and generate large amount of effluent. Alcohol is separated by distillation and the residual liquor is discharged as effluent. This effluent, called as spent wash, is hot, highly acidic, dark coloured and contains high percentage of organic and inorganic matter [1], both in suspended and dissolved forms. For every litre of alcohol production, about 12–141 of wastewater is being discharged [2], which affect the soil and groundwater resources of the region. The prolonged discharge of the effluent renders toxicity to the soil on the same part of the land [3].

Thermal degradation of reducing sugars and the amino compounds is mainly responsible for the dark colour of distillery effluents. This is because of the formation of melanoidins and polyphenolic compounds or complexes. The dark colour will prevent the penetration of sunlight and will damage the aquatic eco system [4,5]. Melanoidin is not easily decomposed by the conventional biological treatment processes and so it produces high chemical oxygen demand (COD), which is a major problem for pollution control [6].

Though, the distillery effluent contains no toxic substances, it has high amount of organic matter, which can cause high amount of BOD and COD. Further, it depletes dissolved oxygen (DO) in the receiving water bodies. This depletion of DO induces the water bodies to be highly odoured due to the destruction of flora and fauna. In the long run, the water body will become a dead pool of water due to eutrophication. All these facts reveal that the distillery effluent is highly polluted and it needs treatment before discharge into the natural water stream or land.

Conventional methods dealing with the treatment of distillery effluents consist of various combinations of biological, chemical and physical methods. Several methods viz., filtration using variety of filters, coagulation by added chemicals, reverse osmosis, adsorption, ion exchange process, dissolved air flotation method

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etc., are available for treating distillery effluents [7]. Many of these methods are using more quantity of water and they are either pollutant specific, less efficient and more expensive than simple discharge without any effluent treatment. Hence, these methods become obsolete and new methods need to be sought.

Electrocoagulation (EC) in conjunction with electroflocculation is one of the methods, which is simple, but very effective for treating many turbid waters and wastewaters [8]. Various authors have proved the removal of pollutants by EC technique successfully. Mention may be made on the treatment of potable water [9,10], textile wastewater [11-13], tar, sand and oil shale wastewater [14], urban wastewater [15], tannery wastewater [16], restaurant wastewater [17] and food wastewater [18]. EC has also been used to remove dyestuff [19-22], arsenic [23] and nitrate [24] from wastewater. This process is characterised by an effective removal of pollutants [25–28], compact size of the equipment, simplicity in operation, low capital and operating cost [29]. This process is more effective than other methods because of the subsequent electroflocculation [25–28]. In electroflocculation, the pollutants are removed by the hydrogen bubbles, which are generated during EC. These bubbles uplift the coagulated/agglomerated pollutant particles to the surface, from where it could easily be removed. It is also reported that electrolytically added aluminium ions are more active than chemically added aluminium ions [30]. This means that, less aluminium is required and electrocoagulation could be used to treat a number of wastewaters, which could not be handled by chemical flocculants such as alum.

This process is used to remove suspended (fine) solids and dissolved molecules. The suspended solids include bacteria, algae, clay and spores. The dissolved matters are organic matter (humes), dye molecules, detergent molecules and other materials. We report that the addition of indigenously prepared activated areca nut carbon (AAC) during EC enhances the rate of removal of turbidity to the order of 99% in the distillery effluent. The results of EC with and without AAC by employing iron (cathode) and aluminium (anode) electrodes, in the electrochemical treatment of distillery effluent are presented and discussed here.

2. Experimental

Distillery wastewater samples for this study have been collected from the discharge stream of a distillery industry situated in Tirunelveli district of Tamil Nadu, India. During sampling, the samples have been collected in a 21 polythene can, once in 4 for 24 h and they are mixed in equal proportions to get uniform homogeneous sample [31]. Random selection procedure was adopted for the selection of both sampling unit and the sampling point in a given site [32]. The sampling of effluents and its characterisation were carried out as per the method recommended by APHA [32] and methods reported in literatures [31-33]. The values of physico-chemical characteristics of distillery industrial effluent are shown in Table 1. The effluent was first filtered using a screen filter (bar and then fine screen) to remove large suspended visible solids before carrying out further studies. The experimental set-up with laboratory prototype reactor is schematically shown in Fig. 1.

Thermostatically controlled inclined electrochemical reactor (conical flask with spout, 250 ml capacity) made up of borosilicate glass was placed inside a beaker (500 ml capacity), which was then placed on magnetic stirrer (Remi-1MLH). The electrochemical reactor consisted of two mono polar electrodes, one cathode and another anode viz., iron (stainless steel—SS) and aluminium, respectively. Both the electrodes are purchased from the local market (purity: Al=99.5%, Fe=99%). The dimen-



Fig. 1. Schematic diagram of experimental set-up.

DC POWER SUPPLY (0 - 30 V; 0 - 2 A)

Table 1

Range and average values of WQPs of distillery effluents, limit for drinking purpose and discharge of industrial effluents, prescribed by BIS

S. no.	Parameter ^a	Range (minimum –	Average	Limit prescr by IS:10500	ribed), 1983	Limits prescribed by industrial effluents d	IS for Ischarged	
		maximum)		Allowable	Maximum permissible	Into inland surface water IS:2490, 1974	Into onland for irrigation IS:3307, 1977	Into public sewers
1.	Temperature (T)	30.4–32.8	31.92	-	-	Shall not exceed 40 °C	-	45 °C at the point of discharge
2.	pН	5.71-6.86	6.22	6.5-8.5	6.5-8.5	5.5-9.0	5.5-9.0	5.5-9.0
3.	Electrical conductivity at 25°C	2800-12610	5157.50	-	_	-	3000	-
4.	Alkalinity	820-1125	952.17	_	_	_	_	_
5.	Total suspended solids	3103.3-21183.3	7168.89	_	_	100	200	600
6.	Total dissolved solids	6763.3-39903.3	15390.55	500	1500	2100	2100	2100
7.	Total hardness	1220-7460	2880	300	600	-	-	_
8.	Temporary hardness	100-560	270	_	_	-	-	_
9.	Permanent hardness	1070-6920	2610	_	_	-	-	_
10.	Chlorides	816.5-2996.2	1577.38	250	1000	1000	600	1000
11.	Sulphates	99.9-1907.8	659.48	150	400	1000	1000	1000
12.	Phosphates	9920-20160	14693.33	_	_	5 ^b	-	_
13.	Sodium	393.1-779.6	570.68	_	_	-	60	60
14.	Potassium	594.9-1004.5	823.38	_	_	-	-	-
15.	Calcium	276-972	506.67	75	200	-	-	-
16.	Magnesium	127.2-1207.2	387.20	30	100	-	-	-
17.	Iron	8.66-40.66	16.97	0.3	1.0	3	3	3
18.	BOD	5636.2-14820.4	9776.81	_	_	30	100 ^c	350 ^c
19.	COD	8032.1-28842.4	14555.12	_	_	250 ^d	-	-
20.	Total solids	9963.3-60086.7	22226.11	_	_	-	-	-
21.	Total fixed solids (inorganic solids)	3370–18566.7	7696.11	-	-	-	-	-
22.	Total volatile solids (organic solids)	6006.66-41520	14530	-	-	-	-	-
23.	WQI	3441.1-6348.3	4882.48	_	-	-	-	_

Ref. [34].

^a Unit: in mg/l or ppm, except pH, EC (µmho/cm) and WQI.

^b If all these pollutants are present at the maximum permissible concentration, the effluent may lead to ethrophication. Therefore data on ecological changes should be monitored.

^c Further relaxation may be decided by the concerned agencies.

^d For paper, dyestuff, pesticide and certain chemical and petrochemical industries, these values are relaxed; it shall be ensured that the effluent passes the test for lethal toxicity as given in IS:6582, 1972.

sion of iron (stainless steel cathode) electrode and aluminium (anode) electrode is $104 \text{ mm} \times 25 \text{ mm} \times 6 \text{ mm}$ each. The spacing between the electrodes was maintained at 28 mm. The weight of iron electrode (electronic balance, anamed make, 100 g capacity, 0.001 g accuracy), before and after EC without AAC has been noted to be 72.544 and 72.577 g (+0.033 g is gained for 30 min of EC). This is because of little electro-deposition of aluminium ions on SS cathode. Aluminium electrode looses 0.098 g from its initial weight (initial weight = 42.682 g; final weight = 42.584 g) for 30 min of EC without AAC. During EC, Al³⁺ ions from aluminium electrode are leached out and taken part in coagulation process. It is settled as sediment or floated on the surface (due to bubbles) and it could easily be removed. After 60 min of EC without AAC, the iron and aluminium electrodes weigh 72.604 and 42.512 g, respectively. Considering EC with AAC, the iron electrode gain 0.002 g from its initial weight (initial weight = 72.543 g; final weight = 72.545 g) and the aluminium electrode loose 0.072 g from its initial weight (initial weight = 42.503 g; final weight = 42.431 g) after 30 min.

After 60 min, the iron and aluminium electrode show 72.549 and 42.363 g, respectively. The electrodes are cleaned initially with acetone and then washed [20] with a solution of concentrated HCl (100 ml) and hexamethylene tetramine (2.8% w/v; 200 ml). The electrodes are connected to a DC power supply (30 V, 2 A—Sigma electronics, Bangalore, India).

About 200 ml of well-mixed, screened, homogeneous distillery effluent was taken in a conical shaped 250 ml borosilicate electrolytic cell (conical flask). The temperature of the effluent before EC was noted to be 30 °C. The temperature was maintained throughout EC (deviation ± 1 °C). The current density was adjusted to a desired value (182 A/m²). The whole set-up was placed on a magnetic stirrer and the sample under study was subjected to slow constant stirring. EC has been continued with fresh set-up and the WQPs have been determined at every interval of 10 up to 60 min.

Simultaneously, similar set-up was made with another electrolytic cell with intermittent addition of 1 g of indigenously prepared AAC powder (size: $420 \,\mu$ m) with constant, slow stir-

Characteristics of distillery industrial wastewater before and after EC with and without AAC in presence of iron and aluminium electrodes

ring to facilitate effective coagulation and agglomeration of colloidal particles. The areca nut particles have been cut into small pieces, washed, dried, carbonised in a muffle furnace (at 250 °C), digested and activated for 30 min (2N HNO₃), and sieved (Jayant sieve, India) to a constant particle size (420 μ m). Similarly, EC was continued with fresh set-up and the parameters have been determined at the interval of 10 up to 60 min.

During EC, suspended matter in both the cell was removed whenever needed. Then, the cell was allowed to stand aside for 12 h. Now, the electrolytically added insoluble aluminium ions, which is much more active than chemically added aluminium ions [30] combine readily with the toxic materials present in the effluents and settle them easily as sludge. The insolubility of aluminium ions, in the form of aluminium hydroxide and the nature of its effective coagulating power facilitate EC. Most of the aluminium ions are used in the coagulation process and hence, the level of aluminium ions are reduced to less than 0.1 mg/l after 12–24 h [25–29].

The samples present in both cells were filtered separately through G3 microfibre filter. The filtrates were analysed to estimate the various physico-chemical parameters [32,33]. The above filtrates of the both the samples with and without added AAC, were again electrocoagulated for a period of contact time, after cleaning the electrodes. At the end of each run, the electrodes were cleaned and thoroughly washed with water to remove any solid residue on the surfaces and reweighed.

3. Results and discussion

Table 1 lists the water quality parameters (WQPs) of distillery effluent before EC treatment. The observed WQPs are found to be higher than the prescribed limits by Bureau of Indian Standards (BIS) for the discharge of industrial effluents [34,35]. This indicate that the distillery effluent could be discharged only after proper effluent treatment. Hence, in the present study EC technique is employed for the effluent treatment.

Aluminium and stainless steel (iron) are chosen as electrode materials for EC. They are readily available and are very effective in EC [17]. The current density applied is 182 A/m^2 . The rate of turbidity removal by the EC technique with and without AAC is found to be high, of the order of 99%. This is because the micro bubbles and electrolytically generated aluminium ions coagulate with charged particles in the effluent and agglomerate it. They are later settled or floated on the surface from which they could easily be removed. In the case of parameters like Cl⁻, SO₄²⁻, TDS, total hardness, Ca²⁺, BOD and COD, the removal efficiency varied between 18.3% and 96.8% for the EC with and without AAC (Table 2). The overall removal efficiency of pollutants of distillery effluents by the EC with and without AAC is shown in Fig. 2. Table 2 represents the data of the WQPs noted for EC with and without AAC from 10 to 60 min. In presence of AAC, EC is found to be effective than EC without AAC.

3.1. Effect of initial pH

Initial pH is an important operating parameter in EC process. Generally, the pH of the effluent is changed during the EC [9,17].

WQPs	Before treatment	Removal af	ter EC treatme	ent									
		Without A <i>ℓ</i>	AC					With AAC ^a					
		10 min	20 min	30 min	40 min	50 min	60 min	10 min	20 min	30 min	40 min	50 min	60 min
Colour	Dark	Dark	Dark	Light	Light	Light	Almost	Dark	Light	Light	Light	Almost	Almost
	brown	brown	brown	brown	brown	brown	colour-	brown	brown	brown	brown	colourless	colourless
							less						
Turbidity (NTU)	613	36.8	18.4	5.91	4.9	4.3	3.85	27.6	12.9	3.13	3.10	2.8	2.7
Initial pH	4.78	4.89	5.01	5.22	5.47	5.71	5.96	4.99	5.26	5.59	5.91	6.18	6.41
Chloride (mg/l)	1008.2	945.7	883.2	823.6	814.6	802.5	788.1	912.4	812.6	710	695.7	684.6	667.4
Sulphate (mg/l)	214.9	191.1	166.76	141.2	106.4	66.4	20.9	184.6	153.4	121.8	89.4	50.3	6.9
Total dissolved solid (mg/l)	10206	8858.8	7389.1	6195	5643.9	5184.6	4511.1	7919.9	6001.1	4694.8	4062	3408.8	2857.7
Total hardness (mg/l)	2150	1911.4	1646.9	1400	1285.7	1223.4	1150	1864.1	1642.6	1382.7	1277.1	1081.5	006
Calcium (mg/l)	272	242	214	184	178	172	168	236	206	178	170	164	156
lron (mg/l)	11.39	9.77	8.23	6.6	5.06	3.62	2.09	9.64	8.0	6.2	4.52	2.8	0.94
BOD (mg/l)	11653.2	10464.6	9182.7	7900.9	6572.4	5115.8	3565	9812	8029.1	6351	4533.1	2855	1199.7
COD (mg/l)	18868.8	16415.9	14302.6	12019.4	9679.7	7151.3	4618.8	16189.4	13585.5	10943.9	8604.2	6094.6	3754.1

With a total addition of 2 g of AAC



Fig. 2. Comparision of the treatment efficiency of distillery effluent for 30 and 60 min of EC with and without AAC.

This variation in initial pH of the solution depends upon the electrode material, adsorbent material and on the initial pH of the effluent. At high pH, the Al^{3+} ions as $Al(OH)_3$ act as a coagulant. The wastage of Al^{3+} ion occurs at pH less than 4 and greater than 10. Hence, the pH of the effluent is adjusted by adding 1 M H₂SO₄ or NaOH solution to maintain the pH 4–10.

The initial pH of the raw distillery effluent is 4.78. During the electrocoagulation without AAC at 15 V DC, the pH increased to 5.22 and 5.96, for a period of 30 and 60 min, respectively. In a similar set-up, the distillery effluent is treated with 1 g of AAC powder ($420 \,\mu$ m) and the pH of the effluent increased to 5.59, after 30 min. Further addition of 1 g of AAC registered an increase of pH to 6.41 after 60 min. Evolution of hydrogen bubbles at cathode and subsequent reaction with anions such as Cl⁻, SO₄^{2–}, HCO₃⁻, NO₃⁻ etc., caused an increase in pH [7]. Moreover, these anions replace some of the OH⁻ ions from Al(OH)₃ and cause giant leap in the increase of pH. As a result, the pH of the distillery effluent is increased to 5.96 for 60 min of EC without AAC. However, due to the addition of AAC (adsorbent), this value is further increased to 6.41 for EC with AAC. These observations are shown in Fig. 3.



Fig. 3. Variation of initial pH in distillery effluent with time during EC treatment with and without AAC.



Fig. 4. Effect on total hardness in distillery effluent by EC with and without AAC.

3.2. Effect on total hardness and calcium

The efficiency (in percentage) to remove total hardness by EC with and without AAC is given in Fig. 4. The values of total hardness, as ppm of CaCO₃ and calcium as Ca²⁺ (in mg/l) are 2150 and 272, respectively. After 30 min of EC without AAC, at 15 V DC, the amounts of total hardness and calcium decreased, respectively, to 1400 and 184 ppm. The corresponding values after the addition of AAC are 1382.7 and 178 mg/l, respectively. After a period of 60 min, the values of hardness and calcium are noted to be 900 and 156 mg/l for EC. This is because of the evolution of hydrogen at cathode and subsequent increase in pH of the effluent. The sacrificial aluminium anode electrode leaches to generate aluminium ions which react with free OH- ions forming Al(OH)₃. The Al(OH)₃ produced at high pH, reacts with calcium and magnesium ions in the effluent and co-precipitate them, which are removed later. Hence, reduction in Ca²⁺ and Mg²⁺ ions is observed in the distillery effluents and this process is further enhanced due to adsorption by AAC.

Reduction in the level of hardness after 30 min of EC is noted. No passivation occurred.

3.3. Effect on TDS, chloride and sulphate

Due to collective effect of EC viz., electrochemical oxidation and adsorption capacity of AAC, the TDS of distillery effluent is greatly reduced (72%) by the EC with AAC, compared to that of EC without AAC (55.8%) after 60 min of EC at 15 V DC (Fig. 5).

Similarly, EC without AAC lowered the percentage removal of chloride and sulphate ions (the % of removal for Cl⁻ and $SO_4^{2-} = 21.8\%$ and 90.26%, respectively) compared to that of EC with AAC for 60 min. The same trend is observed in the case of the % of removal of Cl⁻ and SO_4^{2-} at 30 min also. The results are presented in Fig. 6.

3.4. Effect on iron

The electrochemically produced hydroxide ions combine with Al^{3+} and Fe^{3+} ions and form their hydroxides viz., $Al(OH)_3$



Fig. 5. Effect on TDS in distillery effluent by EC with and without AAC.



Fig. 6. Effect on chlorides and sulphates in distillery effluent by EC with and without AAC.

and Fe(OH)₃. The precipitated hydroxides are coagulated as flocs due to the H₂ bubble formed during electrocoagulation. This may greatly reduce the iron and aluminium content of the wastewater. Hence, it is observed that the rate of iron in distillery effluent is greatly increased by the EC carried out in presence of AAC (91.7%) than in the absence of AAC (Fig. 7). The enhanced rate of removal is due to the adsorption of these metal ions on the surface of AAC.



Fig. 7. Effect on Iron in distillery effluent by EC in presence and absence of AAC.



Fig. 8. Removal of BOD and COD in distillery effluent by EC with and without AAC.

3.5. Effect on BOD and COD

Fig. 8 shows the efficiency of the removal of BOD and COD of the distillery effluents by EC with and without AAC. The data reveal that the EC in presence of AAC is effective compared to that in the absence of AAC.

Nearly, 69.4% BOD and 75.5% COD are removed by EC in the absence of AAC in 60 min, while it increase, respectively, to 89.7% and 80.1% in the presence of AAC. This is because of the occurrence of other processes such as electroflotation, electrochemical oxidation, chemical reactions and adsorption [11], along with electrocoagulation. Nevertheless, all the processes highly depend on charge loading. That is why higher removal rates are observed for 60 than 30 min. Besides adsorption, electrochemical oxidation, charge loading and electrocoagulation time caused a high reduction in COD present in the distillery effluent.

4. Conclusion

Based on the results of the present study the following conclusions are arrived at:

- It has been found that the EC is more effective in the presence of AAC for removal of pollutants on distillery effluents, than in the absence of AAC.
- One-hour period of electrocoagulation has a higher removal efficiency than that at 30 min, both in the presence and absence of AAC.
- The colour, which is not easily degraded by most of the conventional treatment methods, is almost completely removed by EC in the presence of AAC.
- It is observed that there is no vast difference between EC with or without AAC on removal of turbidity, which is almost completely removed (error: ±1-2%). The presence of AAC is found to be more effective for all the other WQPs.
- The initial pH of the distillery effluent increases after 60 min of electrocoagulation in presence of AAC.

From the above results, it is concluded that the electrocoagulation is very effective for treating distillery effluents in the presence of AAC than in the absence of AAC.

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