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Development of Circulating Electrocoagulation as a Novel Technique for the Treatment of Raw Vinasse Effluents of Ethanol Production Industries

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Vinasse is a well-known associate of sugar cane/beet ethanol production wastewater. In the present work, the electrocoagulation technique at a constant current intensity combined with circulation flow introduces a new and economic method for the treatment of vinasse effluent using iron and/or aluminum without changing the initial conditions of the sample solution. Spectrophotometric determination was used to quantify the COD and turbidity in effluents as the most regarded vinasse footprints. The proposed CCD method was used as an experimental design, having as independent variables, applied current, pH, electrode material, and the reaction time, evaluated for highest COD, turbidity, and color removal. The optimum conditions for removal of COD, color, and turbidity determined by response surface modeling (RSM) showed that the proposed circulating electrocoagulation (CEC) method using aluminum-graphite electrodes at a current of 167 A m^{-2}, pH 7, and a reaction time of 45 min can be used with high efficiency for the treatment of vinasse wastewater in the ethanol production industry. The proposed method is very simple, cheap, and fast and can be used as a pilot system in alcohol factories to recover the water which can, in turn, be utilized for agricultural purposes and other industries.

Keywords: Circulating electrocoagulation, Vinasse, COD removal, Color removal, Turbidity, Wastewater treatment

INTRODUCTION

Annual global ethanol production reached 103 billion liters at the end of 2021. Biofuels are the main consumers of ethanol claiming more stable prices and less environmental impact compared to fossil fuels [1]. Unexpectedly, covid19 pandemic has also triggered another increased global demand for ethanol. These translate into a massive 3.4 MCM of daily produced vinasse pushing wastewater treatment facilities to their limitations. This issue arose concerns about the preservation of water resources. Amongst the various contaminants of the wastewater effluents of alcohol production industries, the vinasse has stood out clearly. Vinasse is a colored liquid with high density and contains large amounts of various organic compounds and small amounts of minerals [2]. Vinasse remains after the distillation of alcohol at a rate of 12 liters for one liter of produced alcohol [3-5]. Evaporation ponds are the final destination of large amounts of untreated wastewater effluents [6,7]. As a result of the alcohol over-production, we noticed some reports on vinasse introduction to surface water resources due to the limited capacity of evaporation ponds with increased emerging problems to farms and aquacultures due to the high percentage of organic and mineral materials in them [8,9].

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Exposure of vinasse to the environment causes serious problems due to the high nutrient load [10]. Although very rich, the disposal of vinasse into agricultural fields would rise other concerns [11]. Vinasse, saturate the soil readily and impose the risk of water resources contamination [12,13], causing serious environmental problems such as depletion of dissolved oxygen, death of aquatic animals and plants, emerging pollutants that prevent the use of water for human daily life; in addition, it provides unpleasant odors [12]. In order to reduce the environmental impact of vinasse, it is very important to carry out a treatment step before discharging it into water sources or soil.

Biological treatment methods such as anaerobic digestion are commonly used to diminish the harmful effects of vinasse on the environment [4,14-17], however, the existence of resistant organic compounds such as humic acids, tannins, lignins, and polyphenols makes it more difficult to use these methods in the purification of vinasse [18,19]. The elimination of the stable dark brown color of vinasse is another problem of wastewater treatment units [20,21].

The authors have combined biological and physicochemical methods that are more effective for the purification of vinasse and various wastewaters containing organic compounds [22,23]. Electrocoagulation has been highlighted as an electrochemical method with many advantages for the electrochemical treatment of wastewater [24]. In electrocoagulation, a suitable sacrificial anode releases suitable ions that can be precipitated at specific pHs to remove the desired organic or inorganic contaminants from the wastewater through trapping or adsorption processes [25]. There are three stages in the electrocoagulation process. In the first step, cations such as Al^{3+} and/or Fe^{3+} are produced from the oxidation of the sacrificial metal (Fe or Al as the anode) and react with hydroxide anions arising from water reduction on the cathode surface (or hydroxide ions present at the desired pH) to create suspended particles required for coagulation process [26]. The generated hydroxide ions are adsorbed onto the colloidal particles forming the floccules, and trapping contaminants [27-32]. Simultaneously, micro bubbles of oxygen and hydrogen produced from the electrolysis of water on the surface of the anode and cathode push the coagulated materials to the surface of the wastewater under treatment and make it clear. Moreover, as a secondary oxidizing process, electrochemically generated hydroxyl radicals react quickly with organic materials using hydroxylation or dehydrogenation [33,34].

For the Al electrode as the anode, an oxidation reaction produces Al^{3+} according to the following reaction (Eq. (1)):

$$Al_{(s)} \rightarrow Al^{3+}{}_{(aq)} + 3e^{-} \qquad (1)$$

As shown in Eq. (2), the main process in the cathode is the reduction of water molecules to produce hydroxide ions and hydrogen gas in a neutral or alkaline solution.

$$2H_2O + 2e^{-} \rightarrow H_{2\,(g)} + 2OH^{-}_{(aq)} \qquad (2)$$

Finally, colloidal $Al(OH)_3$ particles can be produced from the reaction of $Al(OH)_3$ can be Al^{3+} and OH^{-} (Eq. (3):

$$Al^{3+}{}_{(aq)} + 3OH^{-} \rightarrow Al(OH)_{3\,(s)} \qquad (3)$$

Although electrocoagulation has received great prominence as an efficient wastewater treatment technique [35-37], there are still some drawbacks to vinasse electrocoagulation treatment efficiency. Many researchers used innovations to overcome these drawbacks, *e.g.*, the addition of hydrogen peroxide [38,39], dilution [40], or biological pretreatment of effluents [41,42].

Considering the current perspective, for the first time, in the present work the application of circulating electrocoagulation (CEC) (as an additive/dilution-free, simple, cheap, and fast process) using Al and graphite electrodes to reduce COD, color, and turbidity from vinasse effluent in alcohol production industry was investigated. The application of sample circulation as a novel proposed technique combined with electrocoagulation prevents the temperature increase and probable liquid evaporation during the electrocoagulation process caused by the exothermic nature of the electrocoagulation technique. The experimental design followed by response surface modeling methodology was used for the optimization of the CEC process.

EXPERIMENTAL

Reagents and Materials

Vinasse containing wastewater effluents was collected weekly from a local sugar beet-feeding ethanol production factory in Miandoab, Iran. Samples were stored in glass containers and were analyzed immediately according to the experimental planning matrix. Samples were analyzed for COD, turbidity, and color as vinasse indices before electrocoagulation (Table 1).

In preparing the solutions, 1 M NaOH solution was the only chemical pretreatment additive added in small aliquots for adjusting pH to 5, 7, or 9 according to the experimental design. The suggested setup for performing CEC is shown in Fig. 1. In this configuration, a combination of Fe and/or Al as an anode, graphite (GR), and/or stainless steel (SS) as a cathode is employed in a 1 L reservoir. Cathode and anode electrodes with dimensions of 60 mm × 50 mm × 2 mm were used. A constant current in the range of 33 to 167 A m^{-2} was applied, while keeping the distance between the electrodes fixed at 20 mm. Circulating the effluent flow made it possible to keep the applied current constant while ensuring optimized treatment efficiency. It should be noted that the preliminary tests without flow circulation, showed an intensive electrical resistance generating too much heat. Circulation prevents the temperature increase and probable liquid evaporation during the electrocoagulation process caused by the exothermic nature of the electrocoagulation technique. All experiments were performed at room temperature with a constant circulation flow rate of 10 ml s^{-1}.

Experimental Design

The Central Composite Design (CCD) was utilized in Minitab software with four independent variables including electric current, electrode type, pH, and treatment time. A complete CCD design was carried out, counting for 45 experiments with different setups and test conditions. The efficiency of CEC was based on the percentage of COD, turbidity, and color removal as the main indices of vinasse. Generally, the pollution load of vinasse is described by chemical oxygen demand (COD), turbidity, and color by which vinasse is characterized [5,12,13,43].

COD and color were measured using HACH methods 8000 and 8025 respectively on a HACH DR 6000 spectrophotometer and turbidity was measured on a HACH 2100 Turbidimeter, both for the raw effluents and the treatment remaining sludge. Samples were homogenized for turbidity and COD analysis and were centrifuged for 3 min at 7000 rpm for color analysis.

Table 1. Raw Effluent Wastewater Analysis in 12 Samples Collected Weekly for 3 Months

	COD ($mg\ l^{-1}\ O_2$)	Color (Pt-Co CU)	Turbidity (NTU)
Average	46550	15300	697
Minimum	41600	12100	487
Maximum	48400	17200	1198

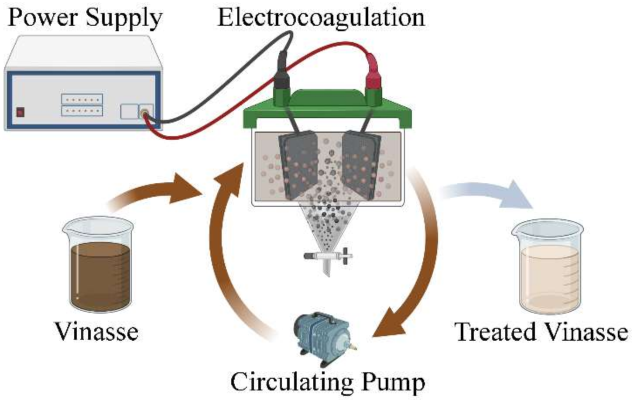

Fig. 1. Representative diagram of the circulating electrocoagulation system (CEC).

Response surface modeling and contour profiling were used to determine the optimal CEC conditions proposed for vinasse treatment based on the removal of color, turbidity, and COD. For this purpose, as well as for the statistical analysis of the results of the CCD matrix, the analysis of variance (ANOVA) generated automatically from the Minitab program was used.

The coded values of the independent parameters are summarized in Table 2. It should be noted that the results of preliminary tests of vinasse effluent were used to obtain these values. The influence of the independent parameters of the response surface modeling was determined from the results of the removal efficiency obtained from color, COD, and turbidity where optimization of the response variable was sought.

Table 2. Levels Studied for the Independent Variables of the Experimental Design Matrix

Variables	Independent levels		
	-1	0	1
Electrode type (Anode- Cathode)	Al-SS	Fe-SS	Al-GR
pH	5	7	9
Current density (A m^{-2})	33	83	167
Time (min)	15	30	45

RESULTS AND DISCUSSION

Optimization of the CEC Process

The effect of the nature of various electrodes on CEC in the electrochemical treatment efficiency of vinasse was investigated. For this purpose, the removal of COD, color, and turbidity of vinasse using a combination of different electrodes as anode and cathode were evaluated. The results obtained are apparent in Table 3. As seen, in simultaneously analyzed vinasse indices, in the 15th run, there were high removals of COD, Turbidity, and color, reaching an average removal of more than 85%. Accordingly, with a high current at a short time (3rd Run), the effluent, after treatment, showed significant color and turbidity removal while COD was not quite competing. Moreover, the removal efficiency at pH 7 is greater than the pH values of 5 and 9.

The developed model was regressed based on Eqs. (4) to (6) represented in Table 4 and according to selected factors (pH, current density, and time) to remove COD, color, and turbidity. According to the experimental model displayed for the treatment of vinasse, ANOVA was carried out to verify the results. The initial step of validation took place using the F-test and R square, shown in Table 4, where it is possible to identify the parameters and interactions that significantly influence the dependent variables, with 95% confidence. For the COD, color, as well as turbidity removal variables, the p-value was statistically satisfactory at 95% confidence. Furthermore, the regression is greater than the residuals for all variables, confirming that the suggested statistical model is valid.

Afterward, the response surface graphs for the COD, turbidity, and color removal models were obtained as presented in Fig. 2. These graphs indicate the percentage of removal of vinasse treatment indices as a function of the independent variables of applied current, electrode type, pH and the treatment time. The electrode type variable was excluded since only the Al-GR combination was able to withstand the treatment time and there was no competition in the removal of vinasse.

According to Fig. 2 and Pareto charts (Fig. S1, supplementary materials), it can be seen that adjusting the pH to around 7, operation time of 45 min, associated with a maximum applied current of 167 A m^{-2}, in a constant circulation media, shows a significant increase in vinasse treatment efficiency. Also, applying the high values of current causes greater removal of vinasse treatment indices. This fact is probably related to the increased dissolution of the anode at a higher current density which triggers greater removal of vinasse characteristics. Since the current study is the only successful attempt at the one-stage vinasse circulating electrocoagulation treatment, currently there is no direct comparison with similar studies. However, the

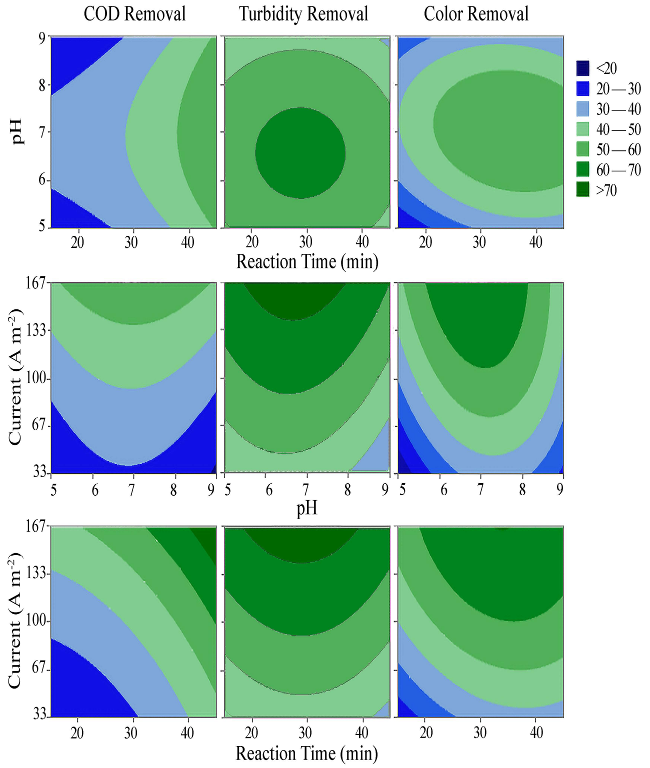

Fig. 2. Response surface contour profile of COD, turbidity, and color removal.

Table 3. Treatment of Vinasse by CEC Process on the Scale of COD, Color, and Turbidity

Run	Time (minutes)	pH	Current ($A\ m^{-2}$)	Electrode combination	COD removal %	Turbidity removal %	Color removal %
1	15	7	33	Al–SS	26	8	65
2	45	5	83	Al–GR	59	75	76
3	15	7	167	Al–SS	44	91	91
4	30	9	33	Fe–SS	6	15	67
5	30	7	83	Al–GR	49	43	85
6	15	9	83	Al–SS	7	1	62
7	30	9	167	Al–GR	47	51	86
8	45	7	33	Al–SS	48	4	72
9	15	5	83	Al–GR	22	30	26
10	45	7	33	Al–GR	56	4	76
11	45	7	33	Fe–SS	61	42	81
12	30	5	33	Al–SS	16	38	24
13	15	5	83	Al–SS	18	33	38
14	45	7	167	Al–SS	66	87	93
15	45	7	167	Al–GR	80	81	95
16	45	9	83	Al–GR	46	2	72
17	30	5	167	Al–SS	50	46	88
18	30	7	83	Fe–SS	38	72	96
19	45	9	83	Fe–SS	62	30	78
20	45	9	83	Al–SS	41	3	70
21	45	5	83	Al–SS	52	19	91
22	45	5	83	Fe–SS	49	20	36
23	15	7	167	Al–GR	40	78	87
24	15	7	33	Fe–SS	51	34	73
25	30	5	167	Al–GR	54	60	81
26	30	7	83	Fe–SS	37	77	98
27	15	7	33	Al–GR	24	22	46
28	30	5	33	Al–GR	18	37	26
29	15	9	83	Al–GR	15	5	58
30	45	7	167	Fe–SS	66	33	93
31	30	7	83	Fe–SS	43	75	96
32	15	7	167	Fe–SS	47	41	83
33	30	9	167	Fe–SS	53	43	84
34	30	5	167	Fe–SS	72	78	93
35	30	7	83	Al–SS	49	74	97
36	30	7	83	Al–SS	36	58	90
37	15	9	83	Fe–SS	32	9	43
38	30	7	83	Al–GR	38	53	92
39	30	9	167	Al–SS	63	55	87
40	30	7	83	Al–SS	43	50	89
41	30	5	33	Fe–SS	30	23	51
42	30	9	33	Al–GR	15	17	45
43	30	7	83	Al–GR	36	29	84
44	15	5	83	Fe–SS	37	28	34
45	30	9	33	Al–SS	19	2	49

Table 4. Response Surface Modeling Summary

Model	R_{sq}	R_{sq} adjusted	F-value	Error	Equation*
COD removal	80.80%	68.71%	6.68	0.97%	Eq. (4)
Turbidity removal	73.10%	56.17%	4.32	3.14%	Eq. (5)
Color removal	82.97%	72.24%	7.74	1.37%	Eq. (6)

Eq. (4) $\text{COD Removal} = -0.73 - 0.004\,t + 0.283\,p + 0.066\,a + 0.0002\,t^2 - 0.023\,p^2 + 0.117\,a^2 + 0.0003\,t\times p + 0.002\,t\times a + 0.011\,p\times a$

Eq. (5) $\text{Turbidity Removal} = -2.487 + 0.047\,t + 0.651\,p + 0.692\,a - 0.0007\,t^2 - 0.053\,p^2 - 0.271\,a^2 - 0.0001\,t\times p - 0.0004\,t\times a + 0.036\,p\times a$

Eq. (6) $\text{Color Removal} = -4.042 + 0.048\,t + 0.975\,p + 1.600\,a - 0.0004\,t^2 - 0.062\,p^2 - 0.409\,a^2 - 0.001\,t\times p - 0.005\,t\times a - 0.069\,p\times a$

*t: Time, p: pH, a: Current density.

competing enhancement in the removal rates in comparison with the two or three-stage treatment processes may have happened because of the application circulating effluent system. Applying a constant flow vinasse circulation with adjusting electrode types and distance and neutralization of the electrocoagulation system to pH 7, the current study pushed the removal rate to a higher level while keeping the energy consumption as low as possible.

Validation of the Proposed Model

After finding the optimum conditions for CEC tests *via* response surface modeling graphs, validation of the obtained results aiming to have a maximum removal rate is possible. However, the actual removal rate obtained using the optimized conditions was no higher than an average of 85% remaining considerable residuals. Figure 3 shows the Raw vinasse sample *versus* the treated vinasse at the circulating electrocoagulation optimal conditions. The reason behind the discrepancy between the actual and the estimated value can be attributed to the use of actual raw wastewater in this work. To certify the efficiency of the CEC process in vinasse treatment, a new set of samples were collected after the wastewater stabilization pond. Additionally, after applying the proposed CEC method, additional analysis was performed on the remaining sludge. The result obtained was amazingly satisfactory with an average removal rate of 93% for both COD, turbidity, and color. Although it was possible to reach higher removal rates by keeping the samples intact for 24 or 48 h to trigger the biological processes, we didn't bother since the latter doesn't define within the limits of the electrocoagulation study.

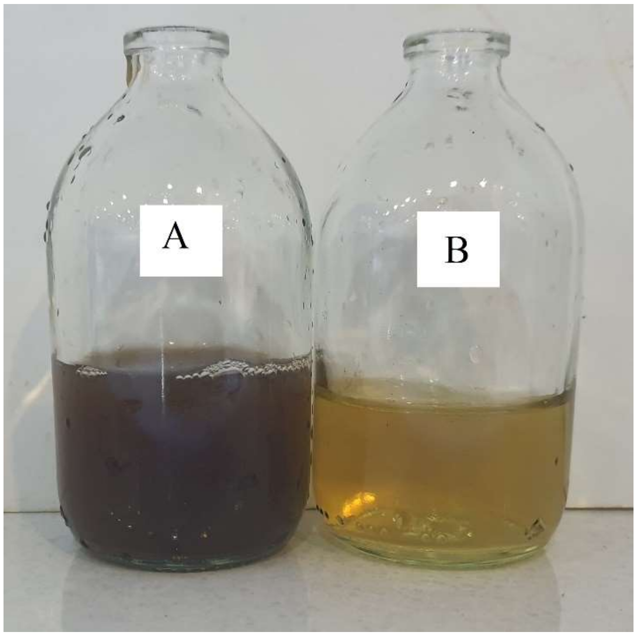

Fig. 3. Photographic images of raw vinasse (A) and after circulating electrocoagulation under optimal conditions (B).

Table 5. Comparison of Proposed ECE Results with Reported EC Studies

Waste	Applied method	Initial COD (mg l^{-1})	Electrodes	Current density (Am^{-2})	pH	Time (min)	COD removal (%)	Color removal (%)	Turbidity removal (%)	Ref.
Vinasse (Turkey)	EC-EF[a]	4750	Iron-Iron	200	5.2	180	14.3	-	-	[45]
Vinasse (Indonesia)	EC	100160	Iron-Iron	922	6.6	60	13.9	-	-	[46]
Vinasse (Malaysia)	EC	2500	Iron-Iron	13	7.0	240	72	100	-	[47]
Vinasse (Iran)	Circulating EC	46550	Al-Graphite	167	7.0	45	91	91	92	This work

[a]Electrofenton.

While several reports put forward the idea that the removal of 95% of the organic pollutants is possible by merely applying an electrochemical process [44], it must be noted that some other factors such as the characteristics of the system, the concentration of the contaminant and its structural peculiarities are also among the defining aspects of the efficiency of the process.

Comparison of Proposed CEC with Reported EC Methods

The comparison of this study with other studies on the removal of COD, color, and turbidity from vinasse wastes in different countries is shown in Table 5. As can be seen, the COD removal efficiency in this study (91%) is much higher than the electrocoagulation reported in the literature for the purification of vinasse wastewater. In addition, the short operation time of 45 min is a unique feature of our proposed method compared to the reported methods (≥ 90 min) [45-47]. Finally, high decolorization efficiency (91%), no need for pretreatment and chemical addition, as well as high turbidity reduction (92%) are other important aspects of the circulating electrocoagulation using aluminum-graphite electrodes for the treatment of raw vinasse samples.

CONCLUSIONS

Circulating electrocoagulation (CEC) using Aluminum-graphite electrodes proved to be an efficient treatment for the removal of different organic contaminates from vinasse effluents, with an average of 93%. Analysis of variance indicated that the model is valid with 95% confidence in removing vinasse indices. The analysis of the response surface modeling made it possible to determine the optimized values of the independent variables. Accordingly, the high actual removal of COD (91%), turbidity (92%), and color (91%) from raw vinasse effluent proved the efficiency of the treatment. The combination of circulation with electrocoagulation reduces the diameter of coagulant particles and increases the surface area for more effective contact with the contents of vinasse. Additionally, circulation prevents the temperature increase during the electrocoagulation process caused by the exothermic nature of the electrocoagulation technique.

It is suggested a post-treatment step to remove dissolved Aluminum that is in disagreement with the limit of some environmental regulations and to determine the intermediate compounds formed from this process.

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