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Treatment of vinasse liquid from sugarcane industry using electro-coagulation/flocculation followed by ultra filtration

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In this present work, vinasse, a by-product of sugarcane industry, was examined using combined treatment methods to purify it. Electrocoagulation/flocculation, ultrafiltration were applied as pre-treatment and post-treatment, respectively. The effectiveness of combined process was evaluated based on colour, turbidity and chemical oxygen demand (COD) removal. The efficiency of electrochemical reactor was investigated according to process variables such as retention time, electrode distance and electrolyte dose. From the results, the price to treat unit vinasse is found to be 2.5 US$/m^3 under optimum conditions. FT-IR analysis of sludge obtained shows the results of electrocoagulation process. Ultrafiltration as post treatment experiments showed the enhanced removal efficiency of colour (91%), turbidity (88%) and COD (85%). The results showed that electrocoagulation followed by ultrafiltration is a suitable combined technique to reduce the colour, turbidity and COD from vinasse liquid.

Keywords: Vinasse, Sugar industry, Electro coagulation, FT-IR analysis. Ultra-filtration, Zero Discharge.

INTRODUCTION

Ethanol is an alcohol with wide applications as a solvent, scent, flavoring and medicine and has long been used as fuel too[1]. In this sector, Brazil occupies a privileged position, leading the production of ethanol from sugarcane. At the end of the 2013/2014, Brazilian sugarcane harvesting season, approximately 27 billion liters of ethanol was produced[2]. However, in spite of the contribution of ethanol production to the Brazilian economy, the development of this sector has not been followed by environmental control measures, especially with respect to the most abundant effluent called vinasse or stillage, originating from the distillation of ethanol. The industrial production of ethanol by fermentation results in the discharge of large quantities of high strength liquid wastes generally called vinasse[3]. The production of vinasses in a sugarcane industry is in the range of 9–14 L per liter of ethanol obtained. Although, the production (Fig. 1) and the characteristics of the vinasse are highly variable depending on the raw material and the process applied to itself. These dark brown wastes are acidic (pH: 4–5) and have a high organic content (COD in the range of 50–100 g/L). Further, its dark color hinders photosynthesis by blocking sunlight and is therefore[4] deleterious to aquaticlife, when it discharged into environment without prior/partial treatment.

Generally, the vinasse is applied directly to soil, because of its organic matter and nutrient content (especially potassium, but also nitrogen and phosphorus), which makes it a good organic fertilizer for sugarcane regardless of whether it is used at ideal proportions[5]. Through, the soil application of vinasse represents the simplest and cheapest solution, using vinasse is that the large volume[6]. But, the continuous application of vinasse to the soil has the environmental impacts such as soil and groundwater contamination, salinization and seed germination inhibition. To avoid these environmental effects, changes in the national wide technical norm are required in Brazil[7].

To find out the suitable treatment procedure for managing vinasse for both environmental and economic reasons, several processes have been developed to treat vinasse wastewater. For example, by biological process such as aerobic and anaerobic methods have been traditionally used for the treatment of vinasse[8]. Aerobic treatment processes are limited by their high energy consumption needed for aeration and high sludge production. The produced sludge in aerobic process needs further technique to degrade it. The anaerobic treatment of vinasse is often slowed or impaired due to the accumulation of suspended solids in the reactor, which lead to a reduction in the methanogenic activity and biomass wash-out[9]. Both, biological processes require long hydraulic retention time and large reactor volumes, high biomass concentration and controlling of sludge loss, to avoid the wash-out of the sludge. Therefore, biological

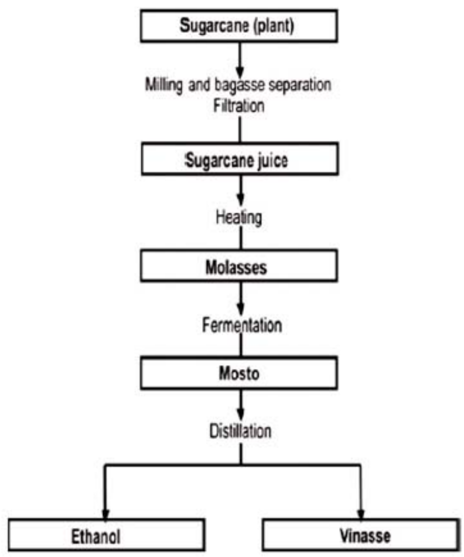

Figure 1. Production of vinasse liquid in sugarcane industry

process to treat vinasse is still questionable, due to its lack of ability[10]. Consequently, sugarcane industries have been forced to seek effective treatment technologies that are not only beneficial to the environment but also cost effective in order to fulfill the strict quality standards regarding environmental protection that are currently being developed to treat vinasse[11].

Last few decades, various physicochemical methods such as adsorption, coagulation–flocculation, and oxidation processes like Fenton's oxidation, ozonation, electrochemical oxida-tion using various electrodes and electrolytes, nanofiltration, reverse osmosis, ultrasound have also been practiced for the treatment of various industrial wastewaters[12]. But, these technologies failed to achieve the maximum COD and colour removals. Still now, single technique for the effective treatment of industrial wastewater is not yet give the optimal solution[13]. By applying, different combinations treatment methods, it is possible to achieve the maximum efficiency.

Electrocoagulation is the best pretreatment process of destabilizing suspended, emulsified, or dissolved contaminants in an aqueous medium by introducing an electric current into the medium[14]. Ultrafiltration is the effective post treatment process due to its selective contaminants removal[15]. Whereas, usage of ultrafiltration as pretreatment is maximum avoided due to its fouling problem. Therefore, the aim of this research was to perform the combined process using electrocoagulation (pre-treatment) followed by ultrafiltration (post-treatment) to treat vinasse liquid from sugarcane industry. Efficiency of combined process was investigated based on colour, turbidity and COD removal. FT-IR was used to find out the results of electrocoagulation process to treat vinasse. Finally, the cost of process to treat unit vinasse was also investigated in order to examine its economical viability.

MATERIALS AND METHODS

Materials

The wastewater used in this study consisted of vinasse derived from the ethanol manufacturing industry (Maringa, Parana, Brazil) and were stored at 4°C prior to the experiments. The characteristics of vinasse was determined using american public health association (APHA) standard methods and were shown in Table 1. Sodium chloride (NaCl) was used as an electrolyte in the form of analytical grade.

Table 1. Characteristics of vinasse wastewater

Vinasse Wastewater	
Characteristics	Value
pH	4.65
Colour [pt-co unit]	54600
Turbidity [FAU]	10400
Chemical Oxygen Demand [mg/L]	65000

Electrocoagulation experimental setup

A laboratory batch monopolar electrocoagulation reactor (ECR) was designed and constructed as shown in Fig. 2. In the ECR, two aluminum (purity of Al 95–97%) plate namely anode and cathode (dimension 400×400 mm) were used as electrodes. These were dipped into an vinasse (0.2 L) in a 0.3 L beaker. In the ECR reactor, stirring (50 rpm) was achieved using a magnetic bar placed between the bottom of the electrodes and the bottom of the beaker. A gelatinous deposition layer on the anode plate was cleaned for each run by hydrochloric acid (HCl) solution. Direct current from a DC power supply (0–30 V, 0–2.5 A, ISO-TECH, IPS--1820D) was passed through the vinasse via anode and cathode. The ECR reactor operated in a galvanostatic mode which means that the current was held constant while the cell potential varied to maintain the required current. Electrocoagulation experiments were performed (in triplicate) by varing retention time, electrode distance and electrolyte dose. The samples were taken from the upper surface for color, turbidity and COD measurement.

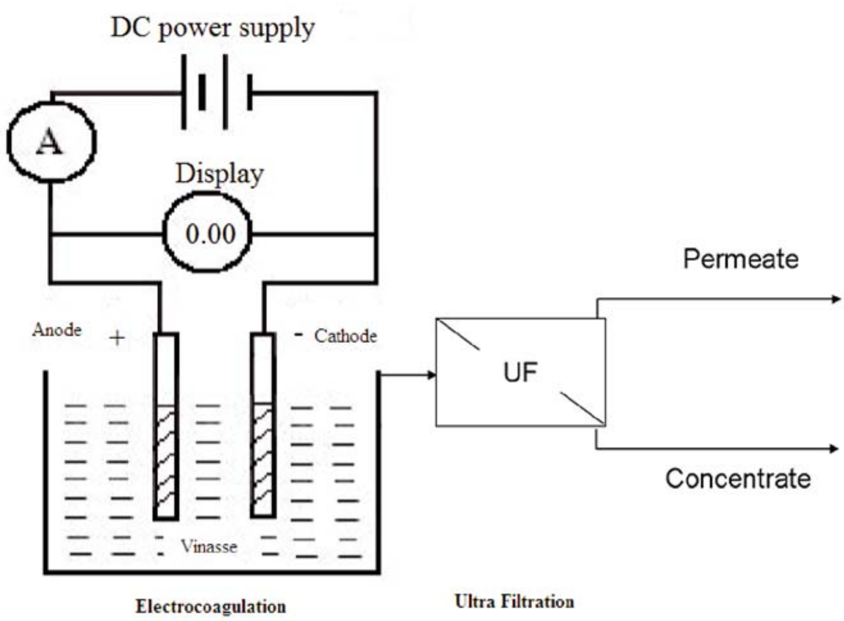

Figure 2. Schematic diagram of combined process experimental setup

Ultrafiltration experimental setup

In ultrafiltration experiments, the tubular ceramic membranes namely Al_2O_3/ZrO_2 made by isostatic pressing[16] with a length of 175.7 mm, diameter of 6.8 mm and a filtration area 0.0034 m^2 are used. The experimental equipment is a micro pilot plant and ultrafiltration (UF) NETZSCH027.06-1C1/07-0005/AI model. The feeding system of the experimental module comprises a reservoir 5-liter, stainless steel, double jacketed and a pump positive displacement with frequency converter, which allows operation at different flow, that is, it enables to vary the tangential speed of filtration. The instrumentation is made up of two pressure gauges and a flow meter for flow indication of pump feeding. Safety features present in the unit are a pressure switch, which limits the pump working pressure, and a device working against dry pump. At the membranes are installed in a stainless steel module, fixed to the pipe by means of flanges. In each experiment, approximately three liter of pretreated vinasse were placed in the feed tank and it was pumped to the pressure pipe and adjusted to commence a permeate flow. All experiments were performed with a flow rate of 700 L/h, corresponding to a tangential speed approximately 8 m/s. The permeate was collected and concentrated totally recirculated to the tank feeding. The permeate samples were taken for colour, turbidity and COD measurement in the UF.

Calculations

American public health association (APHA) standard methods were used to determine the wastewater characteristics such as initial pH, colour, turbidity and COD. The removal efficiency of colour, turbidity and COD were calculated using the following equation

$$R = \frac{C_0 - C_t}{C_0} \times 100 \tag{1}$$

where C_0 – initial concentration, C_t – concentration after ECR treatment at time t. Electrical energy consumption (E) in ECR was calculated using equation[17]

$$E = \frac{UIt_{ECR}}{V} \tag{2}$$

where E is KWh/m^3, U is the applied potential (V), I is the operating current in ampere (A), t_{ECR} is the ECR treatment time (h) and V is the volume of the vinasse (L). The amount of Al (III) released into the treated vinasse during ECR treatment was estimated manually and it confirmed by applying Faraday's law as follows[18]

$$\nabla m_t = \frac{IMt_{ECR}}{nF} \tag{3}$$

where m_t the dissolved aluminum (g), n is the number of electrons in the redox reaction, F is Faraday's constant and M is the molecular weight of Al.

Statistical experimental design

Normally, in wastewater treatment, optimization of process variables is carried out by varying a single factor while keeping all other factors fixed at a constant level. It is not only time-consuming, but also usually incapable of reaching the true optimum, due to ignoring the interactions among variables. Response surface methodology (RSM) coupled with Box-Behnken design (BBD) is a statistical technique for designing experiments, building models, evaluating the effects of several factors and searching optimum conditions for desirable responses. In this work, BBD employed for electrocoagulation process with three important operating parameters such as retention time (F), electrode diatnace (G) and electrolyte dose (H) were considered as the independent variables, while responses are colour removal (Y_1), turbidity removal (Y_2) and COD removal (Y_3). The low, middle and high levels of each variable were designated as −1, 0, and +1, respectively. The coded and actual values of the three independent variables together with their ranges are shown in Table 2. Based on BBD design empirical second-order polynomial equation was fitted to correlate the relationship between independent variables and responses. The general mathematical form of second-order polynomial equation is given as follows[19]

$$Y = \beta_0 + \sum_{j=1}^{k}\beta_j X_j + \sum_{j=1}^{k}\beta_{jj} X_j^2 + \sum_{i}\sum_{<j=2}^{k}\beta_{ij} X_i X_j + e_i \tag{4}$$

where, Y is the response; X_i and X_j are variables (i and j range from 1 to k); β_0 is the model intercept coefficient; β_j, β_{jj} and β_{ij} are interaction coefficients of linear, quadratic and the second-order terms, respectively; k is the number of independent parameters (k = 3 in this study); and e_i is the error. Further, adequacy of the mathematical model is examined using the diagnostic plots and analysis of variance (ANOVA) to find out its capacity to describe the process. The quality of the fit polynomial models were expressed by the coefficient of determination R^2. Finally, the optimal conditions were obtained by analyzing the counter plots and Derringer's desired function methodology[20]. All the statistical experiments and model equation development were performed using Design Expert software.

Table 2. Ranges of independent variables and their levels

Variable [unit]	Factors	Level		
	X	–1	0	1
Retetion time [min]	A	15	45	75
Electrode distance [cm]	B	0.5	1.5	2.5
Electrolyte dose [g/L]	C	0.25	0.75	1.25

RESULTS AND DISCUSSIONS

Mathematical model development

Understanding the interactive effects of ECR operating parameters such as retention time (F), electrode distance (G) and electrolyte dose (H) to treat vinasse, RSM coupled with BBD is used. Based on preliminary studies, BBD experiments consist of 17 runs is carried out (Table 3). To decide about the adequacy of the[21] models to represent the present electrocoagulation process to treat vinasse, sequential model sum of squares was used. Cubic model was not suggested, due to insufficient F-value and the p value. Sequential model sum of squares indicated that the quadratic model provided the best fit to BBD experimental data with the higher F-value and the lowest p value. Therefore, the quadratic model is chosen and the responses with the determined coefficients are presented as follows,

$$Y_1 = 71.90 + 2.48\ F + 9.08\ G + 20.56\ H + 6.56\ FG + 4.50\ FH + 3.06\ GH - 22.85\ F2 - 11.78\ G2 - 26.01\ H2 \tag{5}$$

Table 3. Box-Behnken experimental design and observed responses

Run	F	G	H	Colour removal [%]	Turbidity removal [%]	COD removal [%]
1	75	1.5	0.25	3.58	7.54	2.56
2	15	2.5	0.75	40.58	50.84	22.38
3	15	1.5	0.25	4.15	11.58	8.54
4	45	0.5	1.25	45.55	70.54	24.55
5	75	1.5	1.25	50.98	67.86	29.75
6	45	2.5	1.25	66.51	84.54	45.87
7	45	1.5	0.75	71.92	80.54	52.88
8	45	1.5	0.75	71.92	80.54	52.88
9	45	1.5	0.75	71.92	80.54	52.88
10	75	0.5	0.75	20.85	29.58	8.54
11	45	2.5	0.25	16.54	24.54	15.42
12	75	2.5	0.75	55.14	64.47	35.14
13	15	1.5	1.25	33.54	50.48	13.52
14	15	0.5	0.75	32.54	40.54	11.75
15	45	0.5	0.25	7.84	15.84	6.55
16	45	1.5	0.75	71.92	80.54	52.88
17	45	1.5	0.75	71.92	80.54	52.88

$$Y_2 = 80.54 + 2\,F + 8.49\,G + 26.74\,H + 6.15\,FG + 5.36\,FH + 1.33\,GH - 24.34\,F2 - 9.84\,G2 - 21.83\,H2 \quad (6)$$

$$Y_3 = 52.88 + 2.47\,F + 8.38\,G + 10.14\,H + 4\,FG + 5.57\,FH + 3.22\,GH - 21.43\,F2 - 12.01\,G2 - 17.88\,H2 \quad (7)$$

Where, Y_1, Y_2 and Y_3 are colour, turbidity and COD removal, respectively. The statistical significance of the response functions (Y_1-Y_3) are verified by ANOVA results, which are summarized in Table 4. The sufficient fit of the models are evaluated by co-efficient of variance (CV%) and adequate precision (AP)[22]. The lower CV% values revealed that, the developed models are capable to predict the experimental data. The value of AP values are in reasonable agreement with experimental data. The adequacy of developed mathematical models are also evaluated by R^2 by plotting actual versus predicted plots (Fig. 3). The high value of R^2 (>0.95) for the models indicated a high dependence and correlation between the observed and the predicted values of response[23].

Effect of process variables on electrocoagulation process

Contour plots (Fig. 4) are constructed from the developed models in order to study the individual and interactive effect of the process variables on the responses and also used to locate the optimal conditions for the maximum removal efficiencies of colour, turbidity and COD.

Effect of retention time

In ECR to treat vinasse, retention time is the significant parameter which is directly involved with production of coagulants. In order to examine the effect of retention time on the maximum removal efficiency of colour, turbidity and COD, experiments are carried out in diverse retention time and results are depicted in Fig. 4 (a, c and e). From the results, it is found that the removal

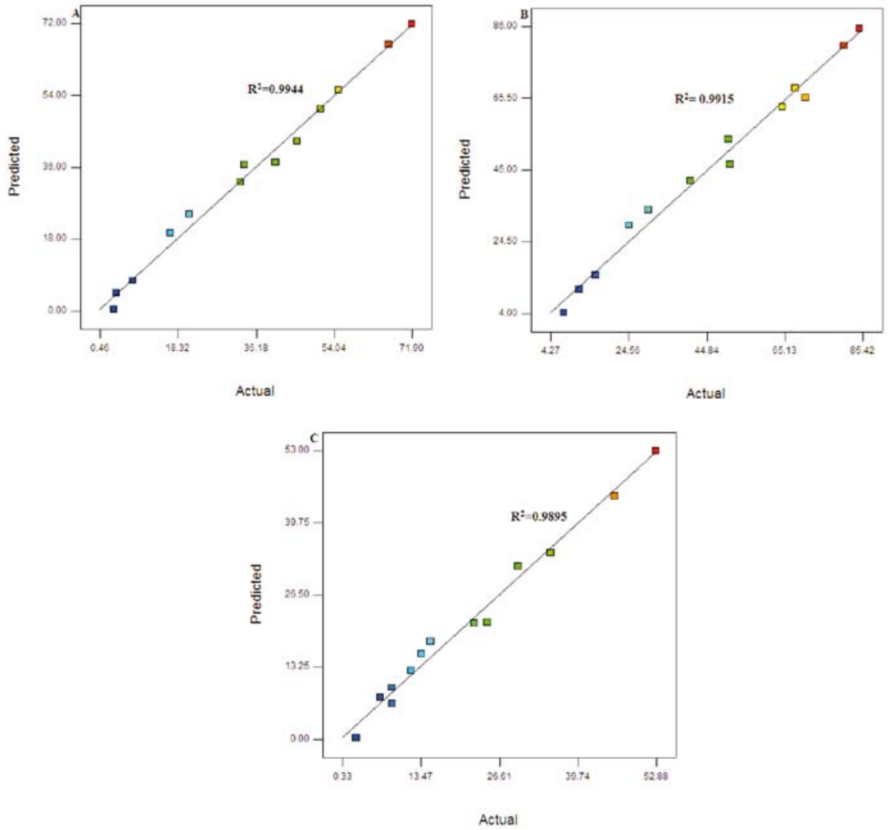

Figure 3. Actual versus predicted plot for responses a) colour removal b) turbidity removal c) COD removal

Table 4. ANOVA table for responses

Source	Y_1		Y_2		Y_3	
	F value	*p* value	F value	*p* value	F value	*p* value
Model	137.94	< 0.0001	86.92	< 0.0001	111.19	< 0.0001
A	5.77	0.0474	2.09	0.1917	8.14	0.0246
B	75.99	< 0.0001	37.55	0.0005	94.11	< 0.0001
C	396.66	< 0.0001	372.79	< 0.0001	137.66	< 0.0001
AB	20.21	0.0028	9.85	0.0164	10.73	0.0136
AC	9.50	0.0178	7.48	0.0292	20.81	0.0026
BC	4.41	0.0739	0.46	0.5204	6.94	0.0337
A^2	257.78	< 0.0001	162.58	< 0.0001	323.88	< 0.0001
B^2	68.49	< 0.0001	26.58	0.0013	101.72	< 0.0001
C^2	334.21	< 0.0001	130.81	< 0.0001	225.46	< 0.0001
CV %	6.73		7.23		8.50	
AP	31.00		27.00		28.00	

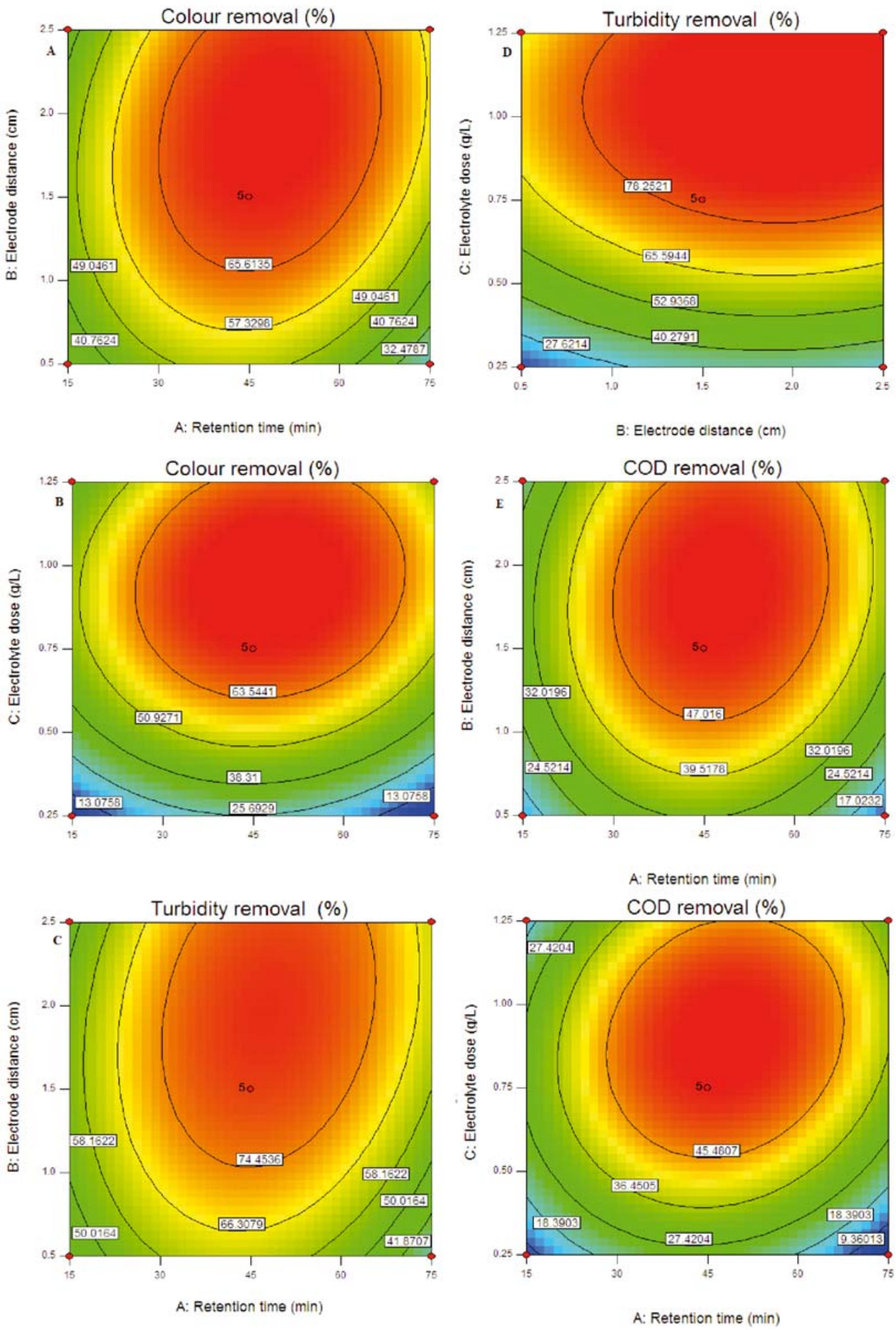

Figure 4. Effect of process variables (A, B, C and D) on EC treatment efficiency

efficiency of colour, turbidity and COD are increased with increasing retention upto 50 min. This can be due to the formation of higher amount of monomeric species in the ECR with respect to retention time as following the electrochemical reactions[24]

At the anode: $M_{(s)} \rightarrow M^{3+}{}_{(aq)} + 3e^{-}$ (8)

At the cathode: $3H_2O_{(l)} + 3e^{-} \rightarrow 3/2\ H_2 + 3OH^{-}$ (9)

In vinasse: $M^{3+}{}_{(aq)} + 3H_2O \rightarrow M(OH)_{3\ (s)} + 3H^{+}{}_{(aq)}$ (10)

M^{3+} (aq) and OH^{-} ions are generated by the electrochemical reactions to form various monomeric species, which change finally into $M(OH)_3$ according to complex precipitation kinetics. This amorphous $M(OH)_3$ flocs with large surface areas are favorable for a rapid adsorption of organic compounds and colloidal particles in vinasse. This can also explained by the fact that when high retention time was applied to the ECR, a large amount of monomeric and polymeric metal species are produced according to Faraday's law, leading to the reduction of colour, turbidity and COD in vinasse to be treated[25]. Whereas, ECR treatment efficiency is decresed beyond the electrolysis time of 50 min.

Effect of electrode distance

An electrode distance is a crucial parameter to determine the removal of colour, turbidity and COD in vinasse wastewater using electrocoagulation process. In order to evaluate its effect, experiments were performed at various electrode distance (0.5–2.5 cm) and the results are illustrated in Fig. 4 (a, c and e). From the results, it is found that the removal efficiencies of colour, turbidity and COD are increased with increasing electrode distance upto 1.5 cm. Thereafter, there is a drastic decrease in removal efficiencies of colour, turbidity and COD. This can explained by the fact that, electrical conductivity is directly proportional to the distance between the two electrodes. As the distance between the anode and the cathode (*g*) increases, resistance (*R*) offered by the cell increases by the following relation[26]

$$R = \frac{g}{KA} \quad (11)$$

where *K* is the cell specific conductance and *A* is electrode surface area. And, therefore, the current in the cell decreases at constant voltage by the relation: current = voltage/resistance. Thus production of $M(OH)_3$ (metal hydroxide) is decreased, which reduced the removal efficiencies, significantly.

Effect of electrolyte dose

Electrolyte (NaCl) dose is one of the essential parameter in electrocoagulation process to treat vinasse in ECR. In order to inspect the effect of electrolyte dose on the ECR treatment efficiency, various electrolyte doses are examined and results are shown in Fig. 4 (b, d and f). It can be seen from results that the percentage of removal efficiencies of colour, turbidity and COD are increased with an increase in the electrolyte dose upto 1 g/L. This phenomena can be attributed due to the fact that the addition of electrolyte to the vinasse involved the following reactions[27]

$$2Cl^{-} \rightarrow Cl_2 + 2e^{-} \quad (12)$$

$$Cl_2 + H_2O \rightarrow HOCl + Cl^{-} + H^{+} \quad (13)$$

$$HOCl \rightarrow OCl^{-} + H^{+} \quad (14)$$

As shown in reactions (12–14), the electrochemically generated Cl_2 and OCl^{-} served as a strong oxidant species that could oxidize organic compounds in the vinasse. Beyond 1 g/L of electrolyte dose shows the negligible colour, turbidity and COD removal in electrocoagulation process. However, it is believed that NaCl dose resulted in the reduction of cell voltages, due to increase of conductivity, which could cause a decrease in electrical energy consumption indirectly operating cost.

Optimization

In order to find out the optimum operating conditions to treat vinasse using electrocoagulation process in ECR is carried out using Derringer's desired function methodology[28]. Here, process parameters are selected as within range and responses ($Y_1 - Y_3$) fixed as a maximize. Optimal operating conditions found to be as follows: retention time of 50 min, electrode distance of 1.5 cm and electrolyte dose of 1 g/L. Under these conditions, 72% of colour, 86% of turbidity and 59% of COD removal are achieved. In order to verify the accuracy of optimal operating condition, the experiments are carried out in predicted optimum condition. The results (real experiments) showed the close agreement with predicted values and hence model is validated.

Cost evaluation

The operating cost is very important economical parameter in electrocoagulation process. The operating cost was calculated using the following equation[29]

$$\text{Operating cost (US \$/m}^3) = aC_{energy} + bC_{electrode} + cC_{chemicals} \quad (15)$$

Where C_{energy} is the energy consumption (kWh/m^3), $C_{electrode}$ is the electrode consumption (kg/m^3) and $C_{chemicals}$ is the chemical consumption (kg/m^3). Unit prices a, b and c given for the price at Brazil is as follows: (a) electrical energy price 0.2 US \$/kWh, (b) electrode material (Al) price 1.6 US \$/kg, (c) electrolyte (NaCl) price 0.008 US \$/kg. From the results, the price to treat unit vinasse is found to be 2.5 US \$/m^3 under optimum conditions to treat vinasse by ECR.

FT-IR analysis of sludge

Sludge from electrocoagulation process was investigated using Fourier transform infrared (FT-IR) analysis and the result is shown in Fig. 5. The broad band around 1090 cm^{-1} is attributed to hydrogen bonding from –OH of the precipitate. The band at 1640 cm^{-1} corresponds to the O–H deformation. Peaks at 1760 and 1420 cm^{-1} corresponding to H–O–H bond stretching at 1640 cm^{-1} to hydroxyl bending and γ'(OH) water bending vibration or overtones of hydroxyl bending. The peaks in the 400–1300 cm^{-1} region corresponded to the stretching and bending modes of Al – O. The peaks in the 500–800 cm^{-1} region corresponded to the stretching and bending modes of Al – O. Peaks at 612 cm^{-1} corresponded to vibrations of Al–O[30]. Hence, it is belived that there is formation of hydroxide species, which is responsible for coagulation process.

Combined treatment of electrocoagulation followed by ultra filtration

In post-treatment technique, the vinasse wastewater, pretreated by the electrocoagulation process is investiga-

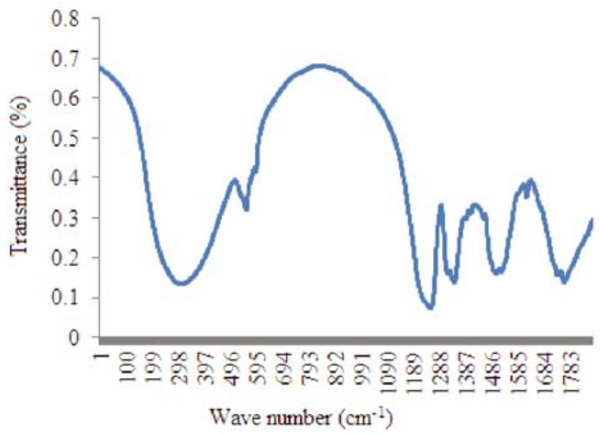

Figure 5. FT-IR analysis of sludge

ted using the ultrafiltration process. The experiments are conducted in the range as follows: temperature of 60ºC and pressure of 3.5 bar and results are shown in Table 5. From the results, it is found that remarkable increase in the removal of colour, turbidity and COD compared to electrocoagulation process, which is probably due to the high concentration of organic and/or suspended particles in the vinasse are effectively removed[31]. As shown in Table 5, electrocoagulation followed by the ultrafiltration process demonstrates more than 85% of removal in colour, turbidity and COD. The EC pretreatment strategies successfully reduce the contaminantes and it's product is suitable for discharge (Fig. 6). It is concluded that under developed conditions, EC pretreatment would improve ultrafiltration performance in water quality.

Table 5. Characteristic of vinasse after electrocoagulation (EC) and after EC followed by ultrafiltration (EC+UF)

Characteristics	Value	After EC [%]	After EC+ UF [%]
pH	4.78	5.9	5.8
Colour	54600	72	91
Turbidity	10400	86	88
COD	70800	59	85

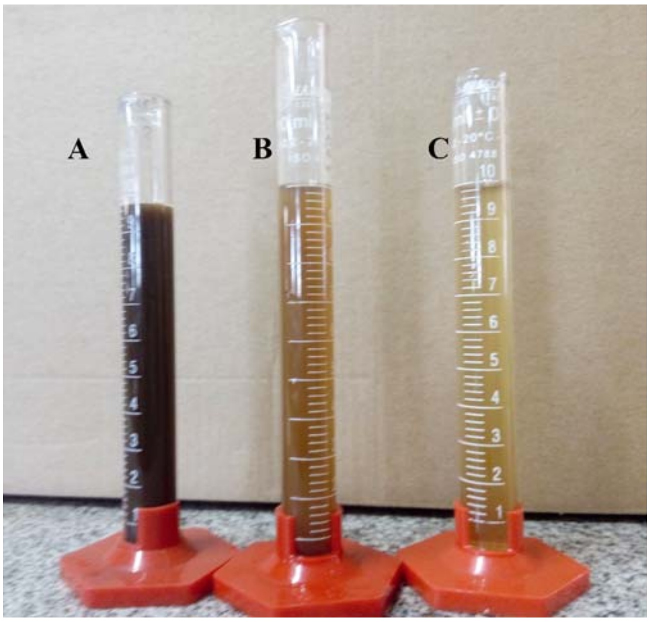

Figure 6. Raw vinasse (a) after EC (b) and after EC+UF (c)

Comparision with literature report

In order to compare the efficiency of proposed treatment technique, the achieved results are compared with existing literature report. Treatment of vinasse by using electro-dissolution and chemical flocculation study reported that only 54% of COD are removed in optimum conditions[32]. In another study, vinasse treatment by coupling of electro-dissolution, hetero-coagulation and anaerobic digestion showed the 83% COD removal in optimum conditions[33]. From the results, it is found that current proposed method showed the 85% COD removal. Hence, it is proved that the proposed method has the more removal efficiency that exisiting ones.

CONCLUSION

Electrocoagulation process followed by ultrafiltration is addressed to treat vinasse for the maximum removal of colour, turbidity and COD. Electrocoagulation process is examined based on operating parameters such as such as retention time, electrode distance and electrolyte dose using aluminium electrodes. Three factors three levels Box-Behnken design (BBD) was used to develop the second order polynomial mathematical models. Optimum operating conditions was found to be as follows: retention time of 50 min, electrode distance of 1.5 cm and electrolyte dose of 1 g/L. Under these conditions, 72% of colour, 86% of turbidity and 59% of COD removal are achieved with cost of 2.5 US $/m^3. FT-IR analysis of sludge obtained shows the results of electrocoagulation process. Ultrafiltration as post treatment experiments showes the enhanced removal efficiency of colour, turbidity and COD.

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