Optimization of electrocoagulation process to treat biologically pretreated bagasse effluent

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Abstract: The main objective of the present study was to investigate the efficiency of the electrocoagulation process as a post-treatment to treat biologically pretreated bagasse effluent using iron electrodes. The removal of the chemical oxygen demand (*COD*) and the total suspended solids (*TSS*) were studied under different operating conditions, such as amount of dilution, initial pH, applied current and electrolyte dose using the response surface methodology (RSM) coupled with a four-factor three-level Box–Behnken experimental design (BBD). The experimental results were analyzed by the Pareto analysis of variance (ANOVA) and second order polynomial mathematical models were developed with high correlation of efficiency (R^2) for *COD*, *TSS* removal and electrical energy consumption (*EEC*). The individual and combined effects of the variables on the responses were studied using three dimensional response surface plots. Under the optimum operating conditions, *i.e.*, amount of dilution at 30 %, initial pH of 6.5, applied current of 8 mA cm^{-2} and electrolyte dose of 740 mg L^{-1}, high removal efficiencies of *COD* (98 %) and *TSS* (93 %) were obtained with an *EEC* of 2.40 Wh, which were confirmed by validation experiments.

Keywords: electrocoagulation; iron electrode; post treatment; model development; optimization.

INTRODUCTION

The pulp and paper industry is one of the most water-dependent industries that consumes 500 m^3 of fresh water to produce a ton of paper.[1] The effluent characteristics of the pulp and paper industry vary according to the pulp process and the characteristics of raw material used in each industry. However, in general, most of the pulp and paper industries used bagasse as a raw material, due to its easy availability and eco-friendly nature. The effluents generated from bag-

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asse-based pulp and a paper industry contain high amounts of organic material and suspended solids, which are considered as major pollutants to an ecosystem. The discharge of untreated bagasse effluent to the ecosystem degrades the environmental and causes harmful effects to living organisms.[2] To overcome this problem, numerous treatment technologies have been reported for the treatment of bagasse effluent, such as bioremediation, upflow anaerobic sludge blanket (UASB) reactor treatment and fungal treatment. However, these treatment processes have drawbacks, such as long treatment time, start-up problems, maintaining environmental conditions and low removal efficiency of organic pollutants. These characteristics of a biological treatment process makes it unfit for large-scale application. Even though, biological treatment of bagasse effluent produces valuable by-products (biogas), the discharge of this effluent to ecological system is still questionable due to the presence of considerable amounts of organic matter. With environmental regulations becoming more stringent, regulatory compliance has also become a matter of increasing concern for the pulp and paper industries. Therefore, there is a critical need to install an effective post-treatment method to treat biologically pretreated bagasse effluent.[3]

In recent years, there has been increased interest in the application of electrocoagulation in the treatment of industrial wastewater. Some of the advantages of electrocoagulation are the simple equipment required, low operating cost, high removal efficiency of toxic matters at short treatment times, easily available electrode materials and easy automation of the process. In addition, it does not require any addition of chemicals, the dosing of coagulant reagents depends on the applied current.[4] Electrocoagulation is a process that generates metallic hydroxides *in situ via* electro-dissolution of a soluble sacrificial anode immersed in the wastewater. The electrochemically generated metallic ions hydrolyze near the anode to form a series of metal hydroxides that are able to destabilize the dispersed particles present in the wastewater to be treated. The destabilized particles are believed to be responsible for the aggregation and precipitation of suspended particles. Moreover, most of the electrocoagulation process use iron as the electrode material due to its higher electrical potential compared to other materials, such as aluminum and stainless steel.[5]

To the best of our knowledge, the electrocoagulation process has hitherto been applied as a pre-treatment process for various industry effluents. No investigations have been reported for the efficiency of an electrocoagulation process as a post-treatment to treat industrial effluents. Hence, in this study, it was planned to investigate and optimize the operating variables, such as amount of dilution, initial pH, applied current and electrolyte dose on the removal efficiency of *COD* and *TSS* removal in the treatment of biologically pretreated bagasse effluent using electrocoagulation (post-treatment) process *via* the response surface methodology (RSM).[6] Response surface methodology (RSM), a collection of mathe-

matical (statistical) techniques, is useful for developing, optimizing and understanding the performance of complex systems applying the minimum number of experiments. This technique conforms closely to practical results compared to theoretical models as it arises from an experimental methodology.[7]

EXPERIMENTAL

Materials

The wastewater investigated in this study was collected from a bagasse-based pulp and paper factory near Erode, Tamilnadu, India. Sample collection, preservation and characterization (pH, *COD* and *TSS*) were realized in accordance with the American Public Health Association (APHA) Standard Methods for the Examination of Wastewater.[1] Characterization was performed immediately after arrival of the samples in the laboratory. The obtained values were pH 7.04, *COD* = 1574 mg^{-1} and *TSS* = 986 mg L^{-1}. All the chemicals employed in the study were analytically pure. The electrolyte used in the study was sodium chloride, which was purchased from local suppliers, Erode, India.

Experimental procedure

The electrochemical reactor (acrylic) having a working volume of 3 L was used to treat the wastewater with iron sheets (33 cm×6 cm×0.2 cm) as the electrode. The effective surface area of each electrode was 108 cm^2. The distance between the anode and cathode was fixed at 4 cm. The assembly was connected to DC power source (Dolphin; 0–6 A and 0–30 V) to fix the desired current density. Distilled water was used to dilute the effluent and the effluent was then adjusted to the required pH using sodium hydroxide or hydrochloric acid. A schematic diagram of electrocoagulation reactor is shown in Fig. 1. In each run, 1.6 L of wastewater was placed into the reactor and all the runs were performed for a constant treatment time of 15 min under stirring at 250 rpm. The treated effluents were collected, filtered and used for the determination of the *COD* and *TSS*. All experiments were performed in triplicate and the average values were recorded. The removal efficiency (*R* in %) was calculated using the following equation:

$$R = \frac{Y_0 - Y}{Y} \times 100 \qquad (1)$$

where Y_0 and Y represent the initial and final value of *COD* or *TSS*.
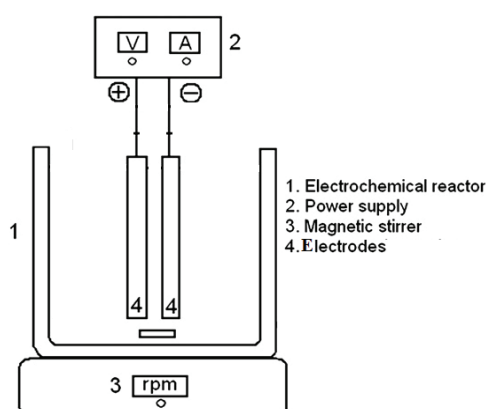

Fig. 1. Schematic diagram of the electrocoagulation unit.

Experimental design

In the present study, the response surface methodology (RSM) was used to optimize and determine the relationship between removal efficiencies of *COD*, *TSS* and *EEC* with respect to the crucial operating parameters, *i.e.*, the amount of dilution, 5–15 %, initial pH 5–9, applied current, 5–15 mA cm^{-2} and electrolyte dose, 350–750 mg L^{-1}. Box–Behnken response surface experimental design (BBD) with 29 experiments with five centre points and their results were analyzed by multiple regression analysis to evaluate the adequacy of various models (linear, interactive (2FI), quadratic and cubic) using the Stat ease Design Expert 8.0.7.1 statistical software package (Stat-Ease Inc., Minneapolis, MN, USA). The regression coefficients of the linear, quadratic and the interaction involved in the model and their effects were analyzed by the *F*-test and *P*-value. The statistical significance of the model was analyzed by pareto analysis of the variance (ANOVA). The relationship between the response and the four independent variables were correlated by a second order polynomial equation, the generalized form of which was given in elsewhere,[7] and it was used for the construction of three dimensional response surface plots to study the effect of the independent variables on the dependent variables. Finally, the numerical optimization methodology was performed to determine the effective electrocoagulation operating conditions, which was validated by conducting additional experiments.

RESULTS AND DISCUSSION

Experimental design analysis

The total number of 29 statistically designed batch experiments were performed for different combinations of the process variables in order to optimize and study the combined effect of independent variables (amount of dilution, initial pH, applied current and electrolyte concentration) on the removal efficiencies of *COD*, *TSS* and *EEC*, which are shown in Table I. The experimental data was fitted to the various mathematical models (linear, interactive (2FI), quadratic and cubic) in order to obtain regression equations. To realize this, model summary statistics (Table II) were performed to decide about the adequacy of a models among the various models, *i.e.*, the model that exhibits the highest R^2, adjusted R^2, predicted R^2 and F values and also lowest *p*-values for when compared with those of the other models.[6–8] Thus, the quadratic model was selected for further analysis to represent the electrocoagulation process. The obtained second-order polynomial equations in terms of coded factors are given below:

$$Y_1 = 95.42 + 6.11X_1 - 4.94X_2 + 11.38X_3 + 10.46X_4 - 5.64X_1X_2 -$$
$$-1.41X_1X_3 + 1.77X_1X_4 - 3.46X_2X_3 - 3.70X_2X_4 - \qquad (2)$$
$$-4.37X_3X_4 - 3.52X_1^2 - 21.46X_2^2 - 9.56X_3^2 - 11.90X_4^2$$

$$Y_2 = 91.58 + 5.04X_1 - 4.09X_2 + 12.64X_3 + 10.17X_4 - 5.87X_1X_2 -$$
$$-1.43X_1X_3 + 1.72X_1X_4 - 3.73X_2X_3 - 3.55X_2X_4 - 6.82X_3X_4 - \qquad (3)$$
$$-3.67X_1^2 - 22.06X_2^2 - 9.49X_3^2 - 12.44X_4^2$$

$$Y_3 = 3.51 + 0.19X_1 + 0.21X_2 + 3.16X_3 - 0.53X_4 + 0.14X_1X_2 +$$
$$+ 0.371.43X_1X_3 - 0.06X_1X_4 + +0.44X_2X_3 + 0.00X_2X_4 - \quad (4)$$
$$- 0.57X_3X_4 + 0.034X_1^2 + 0.67X_2^2 + 0.20X_3^2 + 0.54X_4^2$$

where, Y_1, Y_2 and Y_3 are the percentage removal of *COD*, *TSS* and *EEC*, respectively, and X_1, X_2, X_3 and X_4 are the coded values of the amount of dilution, initial pH, applied current and electrolyte concentration, respectively.

TABLE I. Box–Behnken experimental design and observed responses

Run No.	Dilution of effluent (X_1)	Initial pH (X_2)	Applied current (X_3)	Electrolyte concentration (X_4)	COD removal, %	TSS removal, %	EEC W h
1	20 (0)	5(−1)	15(+1)	600(0)	84.23	79.48	7.29
2	10(−1)	5(−1)	10(0)	600(0)	65.24	63.48	3.78
3	20(0)	7(0)	10(0)	600(0)	95.42	91.58	3.51
4	10(−1)	7(0)	5(−1)	600(0)	66.48	59.48	1.08
5	20 (0)	5(−1)	10(0)	350(−1)	55.32	48.18	5.13
6	20(0)	9(+1)	10(0)	850(+1)	63.43	58.94	4.05
7	20(0)	7(0)	10(0)	600(0)	95.42	91.58	3.78
8	20(0)	5(−1)	5(−1)	600(0)	53.16	47.98	1.08
9	20(0)	7(0)	15(+1)	350(−1)	78.64	82.45	8.91
10	30 (+1)	9(+1)	10(0)	600(0)	64.83	58.48	4.86
11	20(0)	9(+1)	10(0)	350(−1)	53.48	45.84	4.86
12	10(−1)	7(0)	15(+1)	600(0)	88.14	84.27	5.67
13	20(0)	5(−1)	10(0)	850(+1)	80.13	75.48	4.32
14	30(+1)	7(0)	10(0)	850(+1)	98.46	94.86	4.05
15	20(0)	7(0)	5(−1)	350(−1)	44.58	38.98	1.22
16	20(0)	7(0)	10(0)	600(0)	95.42	91.58	3.78
17	20(0)	7(0)	5(−1)	850(+1)	78.48	72.48	0.67
18	20(0)	7(0)	10(0)	600(0)	95.42	91.58	2.97
19	30(+1)	5(−1)	10(0)	600(0)	85.36	75.86	3.78
20	10(−1)	9(+1)	10(0)	600(0)	67.25	69.58	4.32
21	30(+1)	7(0)	15(+1)	600(0)	97.45	94.57	6.88
22	10(−1)	7(0)	10(0)	850(+1)	79.24	74.96	3.78
23	10(−1)	7(0)	10(0)	350(−1)	62.54	57.48	4.32
24	20(0)	9(+1)	15(+1)	600(0)	66.18	62.58	8.91
25	20(0)	9(+1)	5(−1)	600(0)	48.96	45.98	0.94
26	20(0)	7(0)	10(0)	600(0)	95.42	91.58	3.51
27	20(0)	7(0)	15(+1)	850(+1)	95.04	88.67	6.07
28	30(+1)	7(0)	5(−1)	600(0)	81.43	75.48	0.81
29	30(+1)	7(0)	10(0)	350(−1)	74.68	70.48	4.86

Generally, it is important to confirm that the fitted model gives an adequate estimation to predict the responses, unless the model shows poor or misleading results. Considering this phenomenon, diagnostic plots, *i.e.*, predicted *versus* actual values (Fig. 2a–c) were plotted to evaluate the model suitability and to deter-

TABLE II. Model summary statistics for *COD* and *TSS* removal

Source	SD	R^2	Adjusted R^2	Predicted R^2	PRESS	Remarks
	Model summary statistics for COD removal					
Linear	12.9022	0.4746	0.3871	0.2968	5347	–
2FI	14.2750	0.5177	0.2497	–0.0555	8026	–
Quadratic	2.7010	0.9866	0.9731	0.9226	588	Suggested
Cubic	1.9070	0.9971	0.9866	0.5868	3142	Aliased
	Model summary statistics for TSS removal					
Linear	13.5630	0.4534	0.3623	0.2624	5957	–
2FI	14.8420	0.5091	0.2363	0.2363	8793	–
Quadratic	3.7712	0.9753	0.9507	0.8580	1146	Suggested
Cubic	1.6519	0.9980	0.9905	0.7081	2357	Aliased
	Model summary statistics for EEC					
Linear	0.6713	0.9199	0.9066	0.8827	15.84	–
2FI	0.6704	0.9401	0.9068	0.8384	21.83	–
Quadratic	0.5216	0.9718	0.9436	0.8511	20.10	Suggested
Cubic	0.3697	0.9939	0.9717	0.5869	55.80	Aliased

mine the relationship between the predicted and experimental values.[9,10] The data points on these plots lie reasonably close to a straight line, which indicates that an adequate agreement between real data and the data obtained from the models.[11] The statistical significance of the regression equation was evaluated by Pareto analysis of variance (ANOVA) and the results are presented in Table III. The *F* and *p*-values of the individual and combined effects of the operating variables were found to be in the range of acceptable levels, which indicated that the model was highly statistically significant.[12] These results indicated that the developed mathematics is good enough to represent the electrocoagulation treatment process significantly.

Influence of the process variables

Four factors at five levels BBD were used in this study to investigate the influence of process variables on responses such as *COD* and *TSS* removal. To understand the interaction between the independent variables and estimate the removal efficiency of *COD*, *TSS* and *EEC* over the independent variables, three dimensional response surface plots were constructed from the developed models (Equations (2)–(4)), which are shown in Figs. 3–5.

Effect of amount of dilution

Dilution of the effluent is key parameter that strongly influenced the performance of the electrocoagulation process. The concentration of the effluent discharged after biological treatment of the bagasse effluent is not constant over time. Hence, an adopted post treatment method should be capable of treating bagasse effluent of varying concentrations. Considering this, the amount of dilution
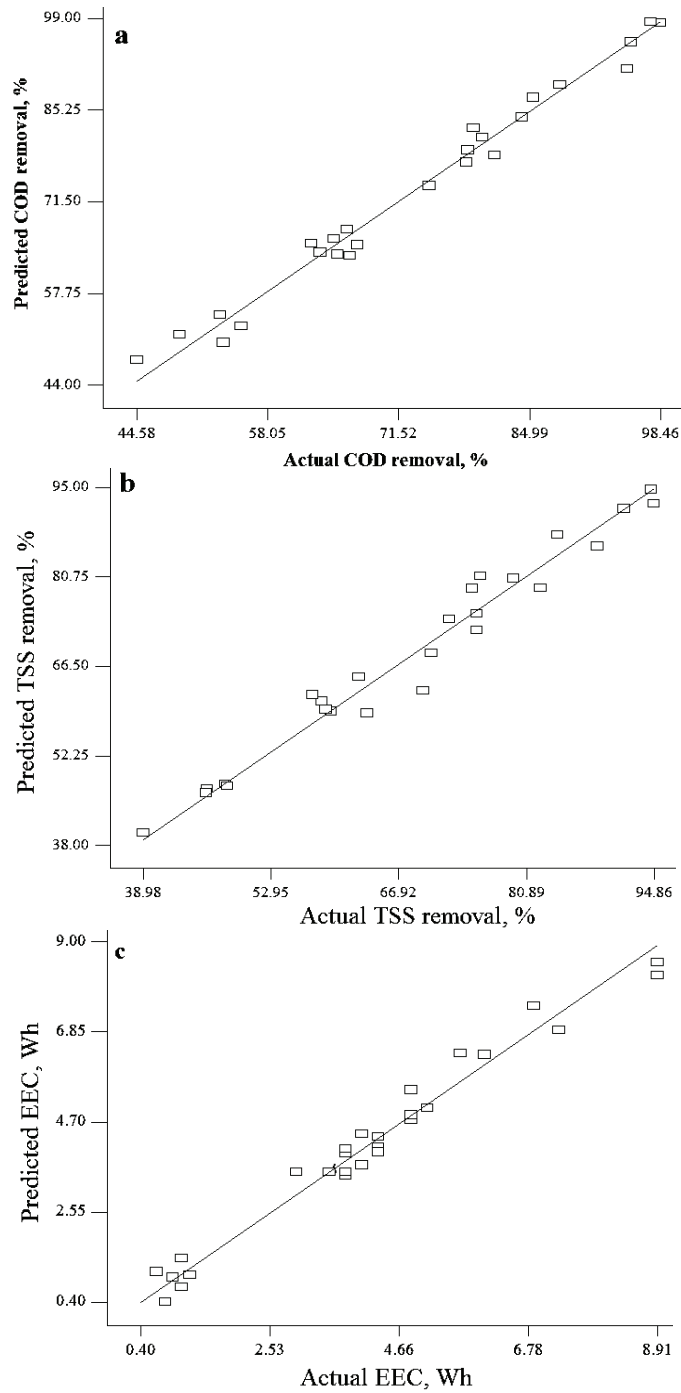
Fig. 2. Actual *vs*. predicted plot for the removal efficiency of *COD* (a), *TSS* (b) and *EEC* (c).

TABLE III. ANOVA table for responses

Response	COD removal		TSS removal		EEC	
	F-value	p-value	F-value	p-value	F-value	p-value
Model	73.45	< 0.0001	39.56	< 0.0001	34.4579	< 0.0001
X_1	61.41	< 0.0001	21.43	0.0004	1.60624	0.2257
X_2	40.18	< 0.0001	14.10	0.0021	2.00733	0.1784
X_3	213.11	< 0.0001	134.74	< 0.0001	440.661	< 0.0001
X_4	180.02	< 0.0001	87.18	< 0.0001	12.3895	0.0034
X_1X_2	17.41	0.0009	9.69	0.0076	0.26795	0.6128
X_1X_3	1.09	0.3142	0.57	0.4623	2.01272	0.1779
X_1X_4	1.72	0.2111	0.84	0.3758	0.06699	0.7995
X_2X_3	6.57	0.0225	3.90	0.0683	2.84633	0.1137
X_2X_4	7.57	0.0156	3.54	0.0807	0	1.0000
X_3X_4	10.49	0.0059	13.08	0.0028	4.81871	0.0455
X_1^2	11.01	0.0051	6.14	0.0266	0.02716	0.8715
X_2^2	409.49	< 0.0001	221.92	< 0.0001	10.8627	0.0053
X_3^2	81.27	< 0.0001	41.08	< 0.0001	0.96561	0.3425
X_4^2	126.00	< 0.0001	70.59	< 0.0001	6.95212	0.0195

was selected as a primary parameter in the electrocoagulation process. From the results (Figs. 3a and 4a), it was observed that the efficiencies for the removal of COD and TSS increased with increasing amount of dilution. This could be explained by the fact that higher amounts of dilution lead to decreasing concentrations of COD and TSS in wastewater; thus, the removal efficiencies were increased with respect to increasing dilution of the wastewater.

Effect of initial pH

In an electrocoagulation treatment process, the pH plays an important role in determining the removal efficiencies. From the obtained results (Figs. 3a and 4a), it was found that the efficiencies of *COD* and *TSS* removal increased with increasing pH up to 6.5. Thereafter, there was a drastic decrease in the efficiencies of *COD* and *TSS* removal. This is because the formation of Fe(OH)$_3$ flocks is significant in the pH range 5–7, which removes the COD and TSS *via* sweep coagulation. Above pH 6.5, monomeric anions, namely Fe(OH)$^{4-}$ species, are formed that are ineffective[13] for removal of *COD* and *TSS* from bagasse effluent. These results indicated that the initial pH value of the effluent is a primary parameter that affects the electrocoagulation process significantly.

Effect of applied current

Applied current is one of the important factors influencing the electrocoagulation process. From the obtained results (Fig. 3b and 4b), it was found that the efficiency of *COD* and *TSS* removal increased with increasing current density up to 10 mA cm^{-2}, thereafter the current density had almost negligible effects on the removal efficiencies. This could be explained by the fact that the formation of

Fe(OH)$_3$ flocks increased with increasing applied current and hence an improvement in the efficiency of *COD* and *TSS* removal was observed. However, above an applied current of 10 mA cm^{-2}, almost all the *COD* and *TSS* were removed *via* sweep coagulation and thus, the removal efficiencies were constant.[14]
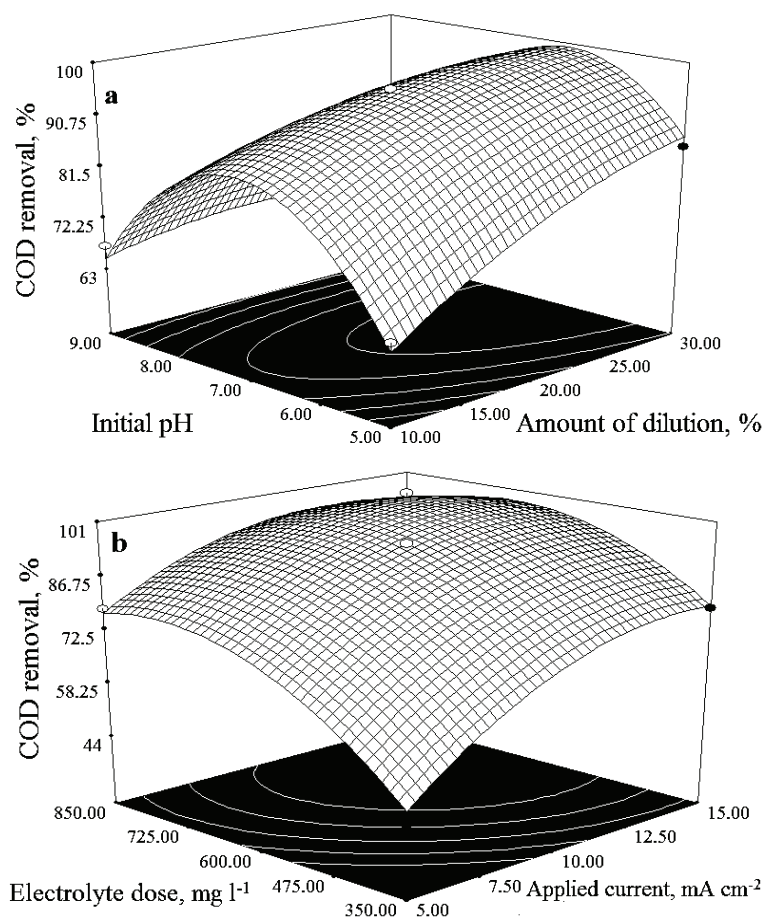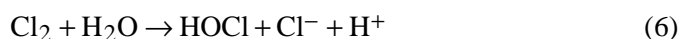

Fig. 3. Effect of operating variables on *COD* removal.

Effect of electrolyte (NaCl) dose

The electrolyte concentration is one of the prime parameter that significantly affects the performance of an electrocoagulation process. From the obtained results (Figs. 3b and 4b), it was found that efficiencies of *COD* and *TSS* removal increased with increasing electrolyte concentration up to 600 mg L^{-1}. This is due to the formation of hypochlorite (an oxidizing agent), which strongly affects the removal of the *COD* and *TSS*. The detailed mechanism is given below:[15]

$$2\text{Cl}^- \rightarrow \text{Cl}_2 + 2\text{e}^- \tag{5}$$

$$\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{Cl}^- + \text{H}^+ \tag{6}$$

$$\text{HOCl} \rightarrow \text{OCl}^- + \text{H}^+ \tag{7}$$

Further increases in the electrolyte concentration show negligible effects on the removal efficiency of *COD* and *TSS*.
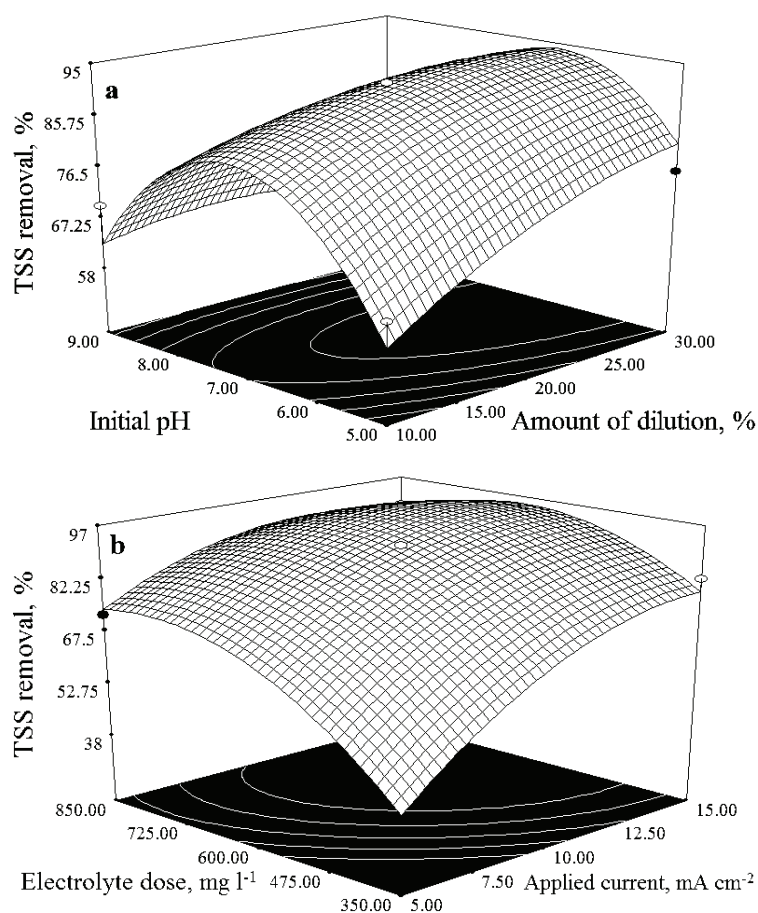

Fig. 4. Effect of operating variables on *TSS* removal.

Effect of process variables on the electrical energy consumption

Electrical energy consumption (*EEC*) is a crucial parameter in electrocoagulation process that significantly affects the economy of the process. From the obtained results (Fig. 5a and b), it was found that, the *EEC* of the electrocoagulation process is significantly affected by the initial pH (X_2), applied current (X_3)

and electrolyte concentration (X_4), whereas amount of dilution (X_1) shows a negligible effect. The EEC value of present study was calculated as follows:[16]

$$EEC \text{ (Wh)} = VAt \text{ (h)} \qquad (8)$$

The *EEC* value increased with increasing applied current (X_3) and decreased with increasing electrolyte concentration (X_4). Meanwhile, the *EEC* decreased with increasing pH up to 7, beyond that there was a drastic increase in the *EEC*. Finally, the *EEC* values of present electrocoagulation process varied in the range from 0.4 to 7.5 Wh. These results indicated that all the selected independent variables showed a considerable effect on the *EEC* value, except for X_1.
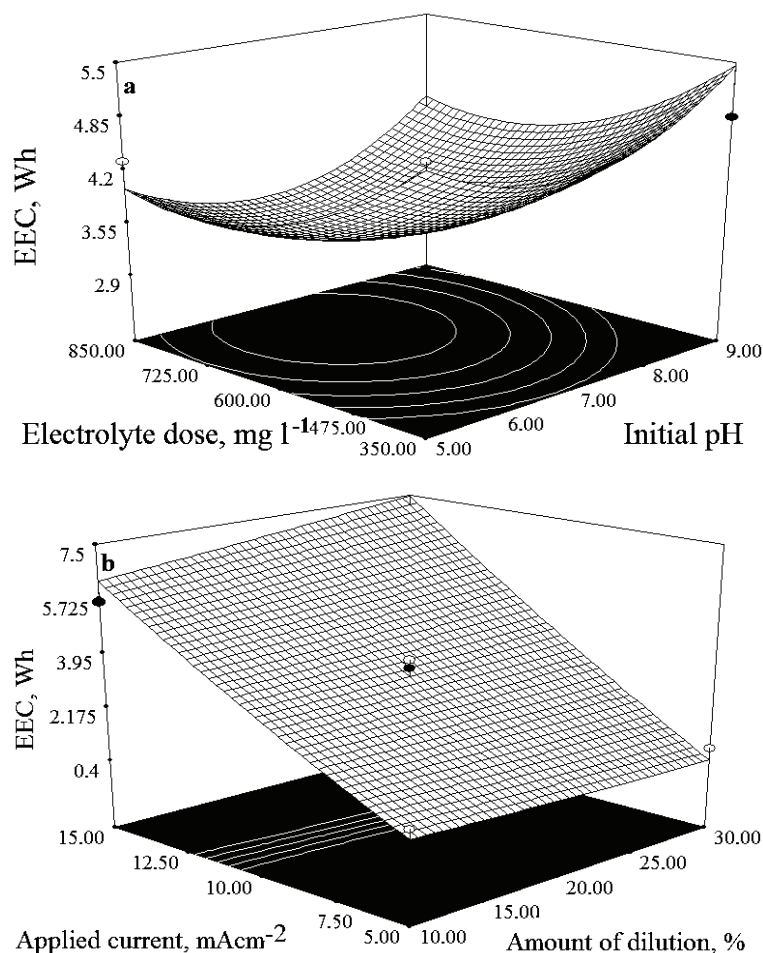

Fig.5. Effect of operating variables on EEC.

Effect of process variables on the iron concentration

In an electrocoagulation process, the quantity of produced iron is a key step. To examine the production of iron in the electrocoagulation process, the following calculations were used:[17]

$$n = \frac{It}{zF} \qquad (9)$$

where n – number of moles; I – current, A; t – time (treatment time), s; z – charge on the cation, ($z = 2$) and F – the Faraday constant, 96.5×10^4 C mol^{-1}.

The iron concentration in solution was theoretically calculated as follows:

$$c_{Fe} = \frac{nM_{Fe}}{V} \qquad (10)$$

where c_{Fe} – iron concentration in solution, g L^{-1}; M_{Fe} – molar mass of iron,; n – – number of moles; and V – volume of the cell, L. In this present study, the production of iron mainly depended on one independent parameter, namely the applied current (X_3), only. The produced iron concentration in the present study was varied between 0.89 to 2.65 g L^{-1}.

Optimization and validation

The objective of the optimization was to determine the operating conditions that gave the maximum efficiency of *COD* and *TSS* removal with a minimum EEC in the selected parameter ranges. In this study, the desirability function approach was applied to optimize the electrocoagulation process *via* a numerical optimization technique (The Derringer desired function methodology), which evaluates a point that maximizes the desirability function. The optimum operating conditions were predicted to be: amount of dilution 30 %, initial pH of 6.5, applied current of 8 mA cm^{-2} and an electrolyte dose of 740 mg L^{-1}, resulted in high efficiencies of *COD* (98 %) and *TSS* (93 %) removal with an *EEC* of 2.40 Wh, and a desirability value of 0.986. The suitability of the optimized conditions for predicting the optimum response values was tested experimentally using the same set of optimal conditions, which gave removal efficiencies of 98.06 and 92.54 % for *COD* and *TSS*, respectively, with an *EEC* of 2.34 Wh. These results clearly validated the optimized conditions.[18]

CONCLUSIONS

In this study, BBD was employed to study and optimize the process variables, *i.e.*, amount of dilution, initial pH, applied current and electrolyte concentration on the removal of *COD* and *TSS* from biologically pretreated bagasse effluent using an electrocoagulation process with iron electrodes. The results showed that all the operating variables had a significant effect on the responses and quadratic models were developed for predicting the responses. Three dimen-

sional response surface plots were used to study the combined effect of the process variables on the responses. The optimum set of operating variables was obtained using the Derringer desired function methodology. The optimal set was found to be: amount of dilution, 30 %; initial pH, 6.5; applied current, 8 mA cm^{-2} and an electrolyte dose of 740 mg L^{-1}, which gave high efficiencies for *COD* (98 %) and *TSS* (93 %) removal with an *EEC* of 2.40 Wh. The results demonstrated the technical feasibility of electrocoagulation as a possible and reliable technique for the post-treatment of biologically pretreated bagasse effluent.

ИЗВОД

ОПТИМИЗАЦИЈА ПРОЦЕСА ЕЛЕКТРОКОАГУЛАЦИЈЕ ЗА ТРЕТИРАЊЕ БИОЛОШКИ ПРЕДТРЕТИРАНОГ ЕФЛУЕНТА ОД ОТПАДА ПРЕРАДЕ ШЕЋЕРНЕ ТРСКЕ

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Главни циљ ове студије био је да се испита ефикасност процеса електрокоагулације помоћу електрода од гвожђа као накнадног третмана биолошки предтретираног ефлуента од отпада прераде шећерне трске. Уклањање хемијске потрошње кисеоника (*COD*) и укупних суспендованих честица (*TSS*) испитивани су под различитим радним условима, као што су степен разблажења, почетни pH, јачина струје и доза електролита, коришћењем анализе површине одговора (RSM) уз четворофакторијални Бокс–Бенкенов (*Box−Behnken*) експериментални дизајн са три нивоа. Експериментални резултати су анализирани Парето анализом варијансе (ANOVA) и развијени математички модели полинома другог реда са високом корелацијом за ефикасност (R^2) за *COD*, уклањање *TSS* и потрошњу електричне енергије (*EES*). Појединачни и заједнички утицаји променљивих проучавани су коришћењем тродимензионалних графика површине одговора. Под оптималним радним условима, као што је разблажење од 30 %, почетни pH од 6,5, јачина струје од 8 mA cm^{-2} и доза електролита од 740 mg L^{-1} добија се виша ефикасност уклањања *COD* (98 %) и *TSS* (93 %) са *EES* од 2,40 Wh, што је потврђено експериментима.

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