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TREATMENT OF CORN STALK PULPING EFFLUENT BY ELECTRO-COAGULATION

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Abstract

In this work, the possibility of treatment corn stalk pulping wastewater by electro-coagulation process was investigated in relationship with efficiency of chemical oxygen demand removal. Diluted black liquor (resulted from laboratory soda pulping of corn stalks) was used to create a simulated effluent. The electrochemical treatments were performed by using two types of sacrificial anodes: aluminum (Al-SA) and stainless steel (FeC-SA). The results of the initial stage of investigation indicated that the efficiency of the process in terms of COD_{Cr} removal strongly depend on several factors including pH, current density and the material chosen as anode. The optimization study showed the best parameters for both of the chosen materials for the sacrificial anode and also the optimal parameter for operating towards maximum efficiency. From economical perspective a steel sacrificial anode is more suitable for performing the electro-coagulation treatment of corn stalks pulping effluent. Under the conditions of the study, the optimal parameters to achieve a 67% COD_{Cr} removal efficiency for the FeC-SA were: current density of 11.2 mA/cm^2, 92 minutes of electrocoagulation treatment at a COD_{Cr} load of 2415 g/L.

Key words: corn stalks, electrocoagulation, optimization, pulping effluent, sacrificial anode

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1. Introduction

Pulp and paper industry is well known for using large amounts of processing water and for generation of high volumes of wastewater (Barjoveanu and Teodosiu, 2006). Both wood and non-wood pulping generate high amounts of spent (black) liquor with a high load of organics (mostly lignin and polysaccharides derivatives). Black liquor is evaporated and burned in pulp mills for energy and chemical recovery but some minor volumes of diluted black liquor may end up as spills in total effluent of the pulp mill. The potential damages to aquatic ecosystems list includes: prevention of the transmission of light and affects the metabolism of aquatic plant populations; the high pH value disturbs both phytoplankton and zooplankton; lignin and lignin derivatives are considered of low biodegradability, toxicity, bioaccumulation and genotoxicity; the presence of sodium compounds reduces the permeability of clay soils and inhibits the growth potential of plant roots (Ksibi et al., 2003; Muna and Sreekrishnan, 2001; Niemelä et al., 2007; Pokhrel and Viraraghavan, 2004; Rodrigues et al., 2008). Currently the pulping effluents are treated by combination of various treatment methods which are chosen depending on load and efficiency.

The configuration of wastewater treatment plants in such cases could include physicochemical stages: i.e. screening, filtration, sedimentation,

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coagulation, chemical precipitation, oxidation, adsorption and membrane filtration) and also biological stages either aerobic or anaerobic: activated sludge, aerated lagoons or anaerobic digestion (Bhargava, 2016; Wagle et al., 2020).

The coagulation-flocculation process is an intensely used process for water and wastewater treatment. The wide range of its applications include pulp and paper effluent treatment on the basis that pollutants (mostly lignin and polysaccharides degradation derivatives) contained by these wastewaters have a colloidal system behavior (Nyman et al., 1986). The main goal of the process is the establishment of premises for efficiency improvement of subsequent treatment stages. The main pollutant categories which are to be removed by coagulation-flocculation treatment process are: nutrients, organic compounds and heavy metals. The most common coagulation agents are aluminum and iron salts (chlorides, sulphates) which when dissolved in water release corresponding metallic ions and form hydroxides which destabilize the pollutant colloidal system leading to creation of flocks which may be further filtered, decanted or floated (Matusiak and Grządka, 2017; Moşneag and Popescu, 2011).

Chemical coagulation and electrocoagulation (EC) have similar mechanisms. As already mentioned in chemical coagulation the agents are brought on the scene as salts, while in the case of EC it is obvious that the ions are generated by electrochemical means at anode of an electrochemical cell which is often denoted as sacrificial anode (Vepsäläinen, 2012). Moreover, at both electrodes other competing electrochemical reactions occur: water reduction, dissolution of metal electrode, modification of acid-base equilibrium, precipitation of hydroxides and complex formation; sorption and desorption on the surfaces of electrodes. The high number of physicochemical processes which compete or succeed each other in EC gives the complexity of the treatment (Hakizimana et al., 2017). In case of utilizing of a steel sacrificial anode (FeC-SA), the occurring reactions are described by Eqs. (1-5) (Sasson et al., 2009; Lakshmanan et al., 2009; Jiménez et al., 2012):

$$Fe \rightarrow Fe^{2+} + 2e^{-} \quad (1)$$

$$Fe \rightarrow Fe^{3+} + 3e^{-} \quad (2)$$

$$Fe^{2+} + O_2 + H_2O \rightarrow Fe^{2+} + 4HO^{-} \quad (3)$$

$$Fe^{2+} + 2HO^{-} \rightarrow Fe(OH)_2 \quad (4)$$

$$4Fe(OH)_2 + O_2 + 2H_2O \rightarrow 4Fe(OH)_3 \quad (5)$$

Simultaneously with Fe-SA Eq. (6) (at anode) and Eq. (7) (at cathode) also occur:

$$4OH^{-} \rightarrow O_2 + 2H_2O + 4e^{-} \quad (6)$$

$$2H_2O + 2e^{-} \rightarrow H_2 + 2OH^{-} \quad (7)$$

In the case of using of an aluminum sacrificial anode (Al-SA) the reaction is given by Eqs. (8-10) (Mollah et al., 2004):

$$Al \rightarrow Al^{3+} + 3e^{-} \quad (8)$$

$$2Al + 6H_2O + 2HO^{-} \rightarrow 2Al(OH)^{4-} + 3H_2 \quad (9)$$

$$Al^{3+} + 3H_2O \rightarrow 2Al(OH)_3^{-} + 3H^{+} \quad (10)$$

Compared to the conventional coagulation the EC process has the following advantages: by being a nonspecific method it can be used to treat a wide range of effluents (An et al., 2017); it may combines processes of oxidation, coagulation and precipitation; reduced operational costs by elimination of the use of reagents, the amounts of sludge generated are smaller; the energy consumption may be reduced by using alternative sources to provide energy to electrocoagulation cells (García-García et al., 2015; Hakizimana et al., 2017). The most important disadvantages are: the necessity of direct current for the process; a high conductivity of the effluent is necessary and there is a lack of data for large scale construction (Garcia-Segura et al., 2017).

The efficiency of the electrocoagulation process is influenced by a number of factors such as: the sacrificial anode material and design, the applied density of current, the pH and conductivity of effluent, it's pollutant load and process time (Camcioglu et al., 2017; Espinoza-Quiñones et al., 2009; Olanipekun-Giwa et al., 2013; Sridhar et al., 2011).

The material chosen *for sacrificial anode* dictates the nature of the chemical reactions taking place. The choosing of the material to be used as sacrificial anode may be a result of the analysis regarding price and effluent to be treated most of the existing studies indicate that aluminum and iron are the most common materials with Al-SA having superior efficiencies and iron or most commonly steel having better prices (Mahmad et al., 2016; Zaied and Bellakhal, 2009). The configuration and shape of SA determines the working surface of electrode. By combining different material, the so-called hybrid SA can be obtained and by thus an increase efficiency and non-specific usage (Chen, 2004; Terrazas et al., 2010).

The *current density* (J) defined as ratio between current and active surface of electrodes plays an important role in EC process in agreement with Faradays law defined by Eq. (11). While a reduced current density is to be translated in a low amount of metallic ions for the EC, a high value of the current density might lead to secondary reactions with also reduced efficiency (Harif and Adin, 2011). The optimal value of this factor is specific and needs to be determined. Faraday's law also explains the influence of process time in constant current density – galvanostatic operation mode:

$$m = \frac{I \cdot t}{F} \cdot \frac{M}{z} \quad (11)$$

in which: m is the mass of substance liberated at anode, in grams; I is current intensity, in amperes; t is electrochemical process time, in seconds; M is molecular weight, g/mol; F is Faraday's constant, F = 96500 C/mol; z is number of transferred electrons.

The *pH-value* of effluent to be treated by EC influences the process by modifying the equilibrium between the ionic species which participate to coagulation process. The solubility of metal hydroxides, the conductivity of effluent, the dimension and structure of the colloidal particle are influenced by pH value (Garcia-Segura et al., 2017).

An increased *conductivity* of effluent leads to a reduced total electrical resistance of the electrolysis cell and improves the electrical charges transfer. Moreover, a lower voltage is necessary for the powering of the EC cell which under constant current density is translated into lower energy consumption. Achieving the necessary conductivity may be realized by introducing supplementary electrolytes. Scope of the present study is to establish the possibly of using EC as method of corn stalks pulping effluent treatment and also to establish the influence of some process factors and optimal operating conditions.

2. Material and methods

Black liquor produced during corn stalk laboratory soda pulping has been collected at the end of experiments performed under the following conditions: 12% active alkali charge (NaOH units); solid to liquid ratio 1:5; pulping time 60 minutes at 160 °C. After cooling and filtering of the liquor, the parameters displayed in Table 1 have been determined. In order to simulate a possible effluent stream, several dilutions were performed to achieve different chemical oxygen demand values.

The *electrocoagulation cell* (shown in Fig. 1) was constituted from a beaker with volume of 600 cm^3 with a stainless-steel cathode. The SA were constructed from a sheet of common steel (FeC-SA) and aluminum (Al-SA).

The electrodes had the same dimensions and an active surface of 36 cm^2. The powering of the electrolytic cell was achieved by means of stabilized adjustable direct power supply. Mixing trough, the EC process experiments was performed by means of magnetic stirrer. The effect of pH has been demonstrated by tests of electro-coagulation at different pH values (3.4, 6.4 and 9.4) different process time values and at constant current density and initial COD_{Cr} load (J = 7 mA/cm^2; COD_{Cr} = 1610 g/L). The effect of current density was studied at constant pH value and initial COD_{Cr} value dilution ratio (pH = 9.4; COD_{Cr} = 1610 g/L). The efficiency - E_{CODCr} (%) - of the EC process was determined as ratio of COD_{Cr} removal using Eq. (12).

The chemical oxygen demand was determined by colorimetry according to the method described in standard ISO 15705:2002 (Water quality - Determination of the chemical oxygen demand index (ST-COD_{Cr}) - Small-scale sealed-tube method).

$$E_{COD_{Cr}}(\%)=\frac{COD_{Cr0}-COD_{Cri}}{COD_{Cr0}}\cdot 100 \tag{12}$$

in which: COD_{Cr0} is the initial value of the chemical oxygen demand before the EC treatment according to the dilution ration; is the COD_{Cr} value at certain time i (during EC or after EC process)

Table 1. Corn stalk pulping black liquor characteristics

Parameter	*Units*	*Method/ instrument*
pH	11.2	Hanna pH-meter
Conductivity	36.0 mS/cm	(SR EN 27888:1997) VWR conductometer
Inorganic substances	15.1 g/L	Calcination at 550 °C
Chemical oxygen demand (COD_{Cr}) – potassium dichromate method	99.98 g/L O_2	ISO 15705:2002 Colorimetric Jasco UV-VIS 550
Appearance	Dark brownish liquid	

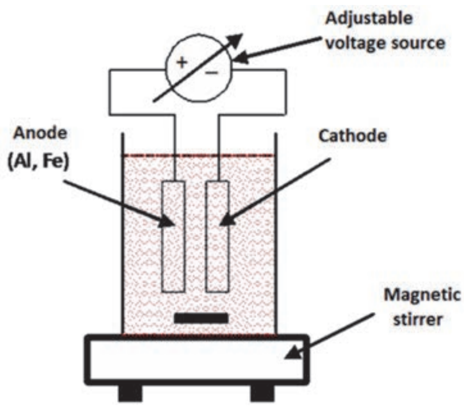


Fig. 1. Representation of the experimental set-up used for corn stalk pulping effluent electrocoagulation process study

The study of the influence factors on the efficiency of the EC process has been achieved by means of response surface methodology. A quadratic central composite experimental design, displayed in Table 2 was employed in which the following factors were considered as independent variables: X_1 - current density J (2.8 - 11.2 mA/cm^2); X_2 the initial COD_{Cr} (is changed in the interval 805 - 2415 mg/L); X_3 - process time (60 - 120 minutes). The experimental design and the subsequent data processing were performed by means of adequate software (Stat Ease Design Expert) which was also used to establish the coefficient of the polynomial Eq. (13) which relates the independent variables to system response (Y_{Al} and Y_{FeC}), in our case the EC process COD_{Cr} removal efficiency - E_{CODCr} (%) for the two types of chosen sacrificial anode materials.

The normalization of the independent variables was performed by Eq. (14), in which X_n is the normalized value of the independent variable X, X_m is the middle of the studied variation interval, while X_{min} and X_{max} are the lower and upper limit of the studied interval.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i<j} \beta_{ij} X_i X_j + \varepsilon \quad (13)$$

$$X_n = 2\frac{(X - X_m)}{(X_{max} - X_{min})} \quad (14)$$

3. Results and discussion

Fig. 2a and b display the variation of COD_{Cr} removal efficiency as a function of EC process time for different values of current density and constant initial pH. The variation curves show the increase of efficiency as a function of current density for both of the electrode types. In the first phase of the process (40 - 60 minutes) the removal of the COD_{Cr} seems faster for both Al and FeC anode types. In case of FeC-SA the effect seems to be more pronounced, as a result of increased number of chemical transformations of iron given by the two oxidation states.

Fig. 3a and b show the variation of COD_{Cr} removal efficiency versus process time at different pH values. The pH value affects the equilibrium of ionic species participating to coagulation process. Other pH influences on EC process efficiency are related to metal hydroxides stability, electric conductivity, dimensions and stability of colloidal particles. Simulated wastewater was treated at dilution resulting pH value which was 9.4 and at 6.4 pH value. The lowering of the pH has a positive influence on the EC process COD_{Cr} removal efficiency with a higher influence in case of aluminum sacrificial anode.

Although operation of EC cell at a close to neutral pH value seemed more attractive due to the perspectives of achieving of higher efficiency of COD_{Cr} removal, the RSM optimization study experiment was performed at natural pH values (as resulted after the dilution process: 8.8 - 9.4). The correction of the pH values to close neutral values generates supplementary chemical reagent consumption. The experimental design software was also used for data processing, establishment of regression Eq. coefficients, generates the ANOVA tables of the model and permits testing of model adequacy (Prob > F" = p < 0.05). Eqs. (15-16) show the dependency between the COD_{Cr} removal efficiency (Y) and chosen independent variables for steel and aluminum electrodes respectively.

In Fig. 4a and b, the response surfaces of COD_{Cr} removal efficiency for the studied electrode materials. The generated response surface by using Eqs. (15-16), show the dependence of the efficiency on process parameters.

For the steel sacrificial anode, at high chemical oxygen demand values in the range of 1200 - 2415 mg/L) and EC duration of 90 minutes the increment of the current density augments the effectiveness of organics removal. In the range of lower values of COD_{Cr} (800 - 1200 g/L), the positive variation of the current density has a less important effect on the efficiency of COD_{Cr} removal. In case of aluminum sacrificial anode, the response surface for the efficiency of COD_{Cr} removal is different comparative to the response surface obtained in case of steel electrode.

At low organic load of COD_{Cr} in the range 800 - 1200 g/L) the increase of current density leads to a minor, increase of efficiency while at high organic load (COD_{Cr} values in the range 1200 - 2415 mg/L) the positive modification of J is accompanied only by a minor increase of chemical oxygen demand removal.

Current density seems the most important factor of influence for bot of the electrode types, but the influence is different for different organic load values. This aspect was revealed by the analysis of and scoring of the contribution of independent variables on EC COD_{Cr} removal efficiency for the two types of sacrificial anodes presented in Fig. 5 in which it may be observed that current density plays the most important role in the variation of EC efficiency for both tipes of materials chosens as sacrificial anode, In the particular case of Fe-C SA is played by the mixture of factors such as current density and COD load. In the case of Al SA the most important combination of factors influence seems to be current density and time.

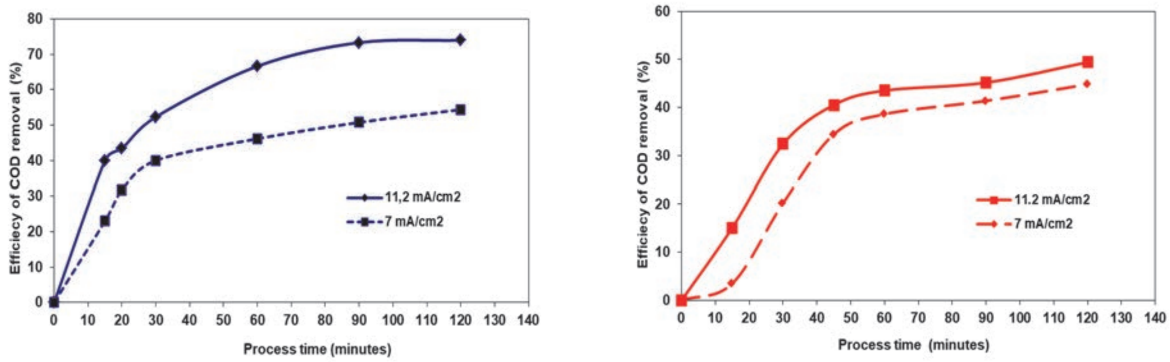
$$Y_{Fe} = -3.79 - 0.81X_1 + 33.22X_2 + 0.88X_3 + 5.78X_1X_2 - \\ - 0.12X_1^2 - 43.17X_2^2 - 4{,}37 \cdot 10^{-3} X_3^2 \quad (15)$$

$R^2 = 0.94$; $R^2_{adj} = 0.90$; $p < 0.05$

$$Y_{Al} = -103.25 + 20.06X_1 - 119.37X_2 + 2.84X_3 - \\ - 0.6X_1X_2 - 0.79X_1^2 + 60.22X_2^2 - 0.01X_3^2 \quad (16)$$

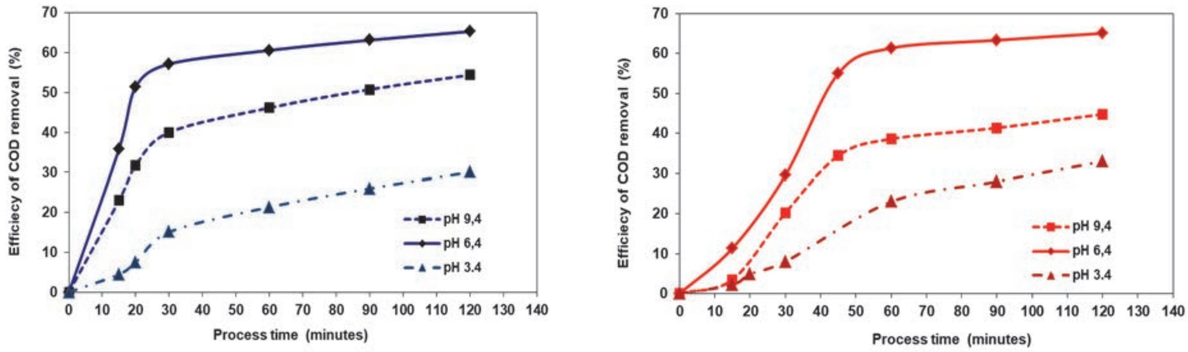
$R^2 = 0.91$; $R^2_{adj} = 0.87$; $p < 0.05$

In Table 3 the results regarding the optimal conditions of each sacrificial anode type are presented. The first two lines represent the optimal condition to achieve a maximum value for the process efficiency. A scenario in which an efficiency of 60% is required is also displayed in Table 3. Calculated and experimentally obtained values of efficiency are also given in the Table 3.



(a) (b)

Fig. 2. The effect of current density on the EC process COD_{Cr} removal efficiency in case of: FeC-SA (a) and Al-SA (b)

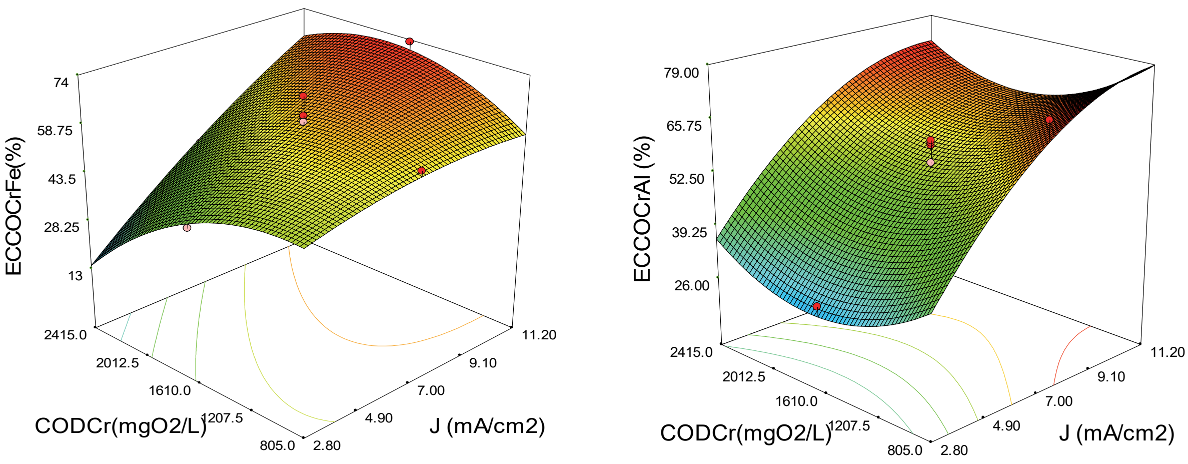


(a) (b)

Fig. 3. The effect of pH on the efficiency of the COD_{Cr} removal trough EC: FeC-SA (a) and Al-SA (b)

Table 2. Experimental design conditions and obtained results used for the mathematical modeling of EC process efficiency in case of Y_{FeC} = E $_{CODCr}$ (%) steel sacrificial anode; Y_{Al} = E $_{CODCr}$ (%) aluminum sacrificial anode

No.	X_1	X_2	X_3	Y_{FeC}	Y_{Al}
	Current density - J (mA/cm²)	*Initial COD_{Cr} (mg O_2/L)*	*Process time (minutes)*	*COD_{Cr} removal efficiency (%)*	
1	11.2	1610	90	73.3	59.16
2	2.8	805	60	42.8	15.6
3	11.2	2415	120	62.3	66.5
4	11.2	2415	60	58.3	60.7
5	7	1610	60	46.1	38.6
6	7	1610	90	50.7	55.2
7	11.2	805	60	50	70.2
8	2.8	2415	60	2.7	16.3
9	7	1610	90	61.7	60.1
10	7	2415	90	40.7	55.2
11	2.8	1610	90	40.9	29.2
12	11.2	805	120	50	63.4
13	2.8	805	120	53.6	33.3
14	7	1610	120	54.2	44.8
15	2.8	2415	120	16.2	34.2
16	7	805	90	52.9	73.2
17	7	1610	90	67.6	59.5
18	7	1610	90	61.5	49.5
19	7	1610	90	50.7	53.2
20	7	1610	90	64.7	60.7



(a) (b)

Fig. 4. The response surfaces of COD_{Cr} removal efficiency for the studied sacrificial anode types FeC-SA (a) and Al-SA (b) at constant EC process time of 90 minutes

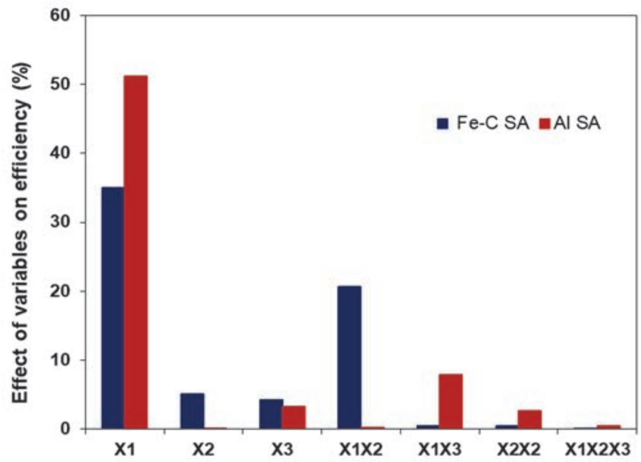


Fig. 5. Contribution of independent variables on EC COD_{Cr} removal efficiency for the two types of sacrificial anodes

Table 3. Optimal values of EC process for the proposed sacrificial electrode materials

Parameter	***X_1***	***X_2***	***X_3***	***COD_{Cr} removal efficiency (%)***	
Sacrificial electrode type	Current density, J (mA/cm^2)	Initial COD_{Cr} (mg O_2/L)	Process time (minutes)	**Y calculated**	**Y experimental**
FeC-SA	11.2	2415	92.7	65.6	67.2
Al-SA	9.3	805	80.1	73.4	70.1
FeC-SA	7.75	1610	80.8	60.0	58.2
Al-SA	9.41	1610	90.05	60.0	57.8

4. Conclusions

Alkaline pulping generates residual waste water which represents a threat to the environment due to the high organic load. In general, the black liquor is concentrated by evaporation and burned in recovery boilers. Small amounts of black liquor could end up as spills in general effluent of the pulp mill.

Our study confirms the possibility of using the electrocoagulation process for treatment of corn stalks soda pulping laboratory generated effluent. The experimental electrocoagulation process was studied in case of sacrificial anodes of steel and aluminum. Our experimental data showed that both of EC process set-ups were influenced by factors such as pH, current density, initial COD_{Cr} and time.

The RSM optimization study leaded to equations showing the dependence of efficiency of process on current density, initial COD_{Cr} load and time. For both type of electrodes it was found that current density plays the most important role. On the basis of obtained equation, optimal working conditions were established for two scenarios: a first scenario in which a maximum efficiency was established as goal. In this case an efficiency of 67.2% in COD_{Cr} removal was experimentally confirmed in case of FeC-SA while by using Al-SA an efficiency of 70.2% resulted; a second scenario in which a minimum efficiency for EC treatment of 60% was imposed.

The experimental obtained values of COD_{Cr} removal efficiency (58.2 in case of steel electrode) and the indicated parameters show that this type of sacrificial anode is much adequate in treatment of the proposed effluent.

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