

# Statistical optimization of nitrocellulose removal from industrial wastewater by electrocoagulation using response surface method

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#### ABSTRACT

Electrocoagulation (EC) method is used to remove the nitrocellulose (NC) fine particles from wastewater of NC washing section. The steel electrodes are applied as anode and cathode of the EC system. Box–Behnken design in response surface methodology is used to optimize independent variables of current density, the distance between two electrodes and initial pH of the sample solution. Removal efficiency of NC fines is calculated according to reduction of chemical oxygen demand of the samples. The statistical quantity of *P*-value indicates that two independent variables of initial pH of the sample and current density are significant (P < 0.05), whereas, variable of distance between two electrodes is insignificant (P > 0.05). On the other, analysis of variance shows that the *F*-value and *P*-value of proposed model are 37.88 and <0.001, respectively, in 95% confidence level. Also, the *P*-value of "lack-of-fit" is obtained 0.131 (>0.05) which shows the lack of fit is not significant (p of model and predicted data of model fit to actual experimental response data. The optimized conditions of current density 20 mA/cm<sup>2</sup>, distance between two electrodes 1 cm and initial pH 7 can be used to achieve maximum removal efficiency (>80%).

Keywords: Nitrocellulose; Electrocoagulation; Response surface method; Optimization

# 1. Introduction

Nitrocellulose (NC) is a single base with low-smoke powder which is used as a propellant in conventional military weapons. Its production process leads to very high water consumption. The result of this process was the production of two types of effluents. The first wastewater is contaminated with nitric and sulfuric acids (pH < 2). The second stream is at neutral pH and derives from rinse operations used to wash excess acid from newly nitrated cellulose [1,2].

The waste NC is composed of insoluble fibers, referred to as NC "fines." Because NC is insoluble in water, the suspended solids (that 50% of which are smaller than 2 microns) are present in the wastewater and are known as primarily NC fines. Therefore, development of innovative NC treatment, removal of NC micro-particles, and disposal technologies is a critical need [3,4].

Large variety of physicochemical and biological processes is developed for separation of NC fines from the waste of nitrocellulose and aged nitrocellulose in the industrial wastewater process which can be mentioned to methods of microfiltration, coagulation, sedimentation, air floatation, and alkaline or acid hydrolysis [5–7]. It is also well-established that nitrocellulose has resisted direct biodegradation under either aerobic or anaerobic conditions [8]. Among physicochemical methods, microfiltration and coagulation by addition a coagulant agent can be used for effective recycling of the removed NC. In order to effectively remove colloidal particles from a suspension, they must be converted to larger particles. However, according to the

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authors' studies, the use of electrocoagulation method to remove NC particles has not been reported. The aim of this study was to collect NC fines by electrocoagulation method without addition of coagulants.

Electrocoagulation (EC) technology is a treatment process of applying electrical current to treat and flocculate contaminants without increase coagulants. The EC process can be used for the treatment of water and wastewater. EC consists of generating coagulant species in situ by electrolytic oxidation of sacrificial anode materials triggered by electric current applied through the electrodes. The metal ions generated by electrochemical dissolution of a consumable anode spontaneously undergo hydrolysis in water, depending on the pH, forming various coagulant species including hydroxide precipitates (able to remove pollutants by adsorption/settling) and other ions metal species [9-11]. Environmental compatibility, versatility, energy efficiency, safety, selectivity, amenability to automation, costeffectiveness, and the compact and simple reactors are the advantages of electrocoagulation [12].

The efficiency of EC method in removal of microparticles depends on various factors. The design of a cell, distance between electrodes, anode material, cathode design, electrode configurations, composition of the electrolyte, optimal pH ranges, pH changes during operation, solution conductivity, power source, current density and charge loading, retention time, and flotation characterization are the effective factors [13]. In many researches, the most of these factors are used as a constant variable and a limit of the important factors are optimized by using design of experiments (DOE) methodology.

Among statistical based techniques, response surface methodology (RSM) is a powerful experimental design tool and used to optimize and understand the performance of complex systems. This method is based on the mathematical and statistical techniques and applied to develop, improve, and optimization of processes and is also used for the evaluation of relative significance of several affecting parameters in the presence of complex interactions [14,15].

### 2. Experimental

Wastewater from NC washing section was treated by electrocoagulation method. Real samples were collected from a chemical company in Iran. Samples contain NC fines were analyzed with measurement of chemical oxygen demand (5,220 COD), total suspended solids (2,540 TSS), total dissolved solids (2,540 TDS), and pH according to Standard Methods [16]. COD amount of samples was determined using DR/2000 spectrophotometer (HACH Company, USA) according to closed reflux colorimetric method [17]. The pH of the samples was 6.5–7.5. Results of analysis for the NC washing section are summarized in Table 1.

The experimental setup (Fig. 1) is an electrocoagulation cell with capacity of 1 L, blades of steel (grade 316 with >80% Fe) for anode and cathode electrodes. The electrodes dimensions were 200 mm × 80 mm × 1 mm and they placed in sample solution so that a contact surface of 100 cm<sup>2</sup> was established. A DC power supply (Irantk.ir 8083) was used to stabilization of current density. The pH of samples was controlled by using a pH meter (Metrohm 630). The time of each experiment was selected 10 min according to the preexperimental experience. Increasing this over time did not have a significant effect on removal efficiency. Temperature of the samples for EC process was controlled at 25°C by using a water bath.

The efficiency of EC process for treatment of real samples was determined with measurement of COD values before and after experiments (Eq. (1)). The terms of  $C_0$  and C are the COD values of samples (mg/L) before and after treatment, respectively.

NC fines removal efficiency 
$$\binom{\%}{=} \left[ \frac{(C_0 - C)}{C_0} \right] \times 100$$
 (1)

As mentioned in the introduction section, there are many factors that influence on the removal efficiency by electrocoagulation method. In this research, three important factors were optimized by using RSM central composite face-centered experimental design according to Box-Benhken design [18]. The effects of main independent parameters, current density, pH of the samples, and distance between two electrodes were studied on the removal efficiency of NC fines. The concentration and type of active species in EC process depend on the pH of sample. On the other hand, the charge of polluting species can also depend on the pH of sample. Current density affects the concentration of active species in electrocoagulation treatment. It is also expected that the conductivity of the solution and the accumulation of active species depend on the distance between the electrodes [19,20].

Table 1

Analysis of the NC washing section before treatment. Each experiment is repeated three times

Analysis	Amount
COD (mg/L)	$2,564 \pm 4.78$
TSS (mg/L)	$1,\!098\pm2.49$
TDS (mg/L)	$178 \pm 1.98$
рН	6.5

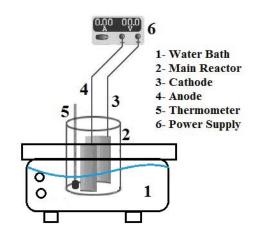


Fig. 1. Schematic of reactor design.

Fifteen experiments were employed in this design (with replicate for each experiment). The levels for the three main factors, A (initial pH of the sample), B (distance between two electrodes), C (current density), were selected from the introductory experiments. Each factor, or independent variable, is placed at one of three equally spaced values, usually coded as -1, 0, and +1 (Table 2).

# 3. Results and discussion

The reactions during the electrocoagulation process are not fully identified because they depend on the wastewater samples. The general reactions which take place in EC method are similar to the reactions chemical corrosion. In EC, an accelerated corrosion is occurred by imposing a potential more than the reversible potential of the anode. Thus, it is important that the reactions are tightly dependent on the pH values of the electrolytes. For example, when iron (and or steel with the main part of Fe, >80%) anodes are used in EC method, the following reactions are occurred in different pH values [21].

Anode:

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

$$2H_2O \rightarrow O_2(g) + 4H^+ + 4e$$
 (If the oxygen evolution  
potential is reached) (3)

Cathode:

$$2H^+ + 2e \rightarrow H_2(g) \text{ (acid medium)}$$
 (4)

 $2H_2O + 2e \rightarrow H_2(g) + 2OH^-$  (neutral or alkaline medium) (5)

The net rate of reactions depends on the mechanism of electro-dissolution of the anode, the pH and temperature of electrolyte, and agitation regime. The ferrous oxide (FeO) and or ferrous hydroxide (Fe(OH)<sub>2</sub>) are formed in the solutions but are soluble in pH less than 4.0 and thus there is no protective film on the metal surface and the metallic dissolution process is faster. The Fe<sup>2+</sup> ions can be oxidized to Fe<sup>3+</sup> in water significantly in alkaline medium and when the electrolyte is saturated with oxygen gas. The ferric hydroxides (Fe(OH)<sub>3</sub>) that are insoluble as well in a wide range of pH begin to precipitate as yellow particles [22,23].

The formed hydroxides of iron  $(Fe(OH)_n)$  remains in the solution as a gelatinous suspension and the pollutants

Table 2

Factors and levels of independent variables of Box–Behnken design in RSM

Parameter	Code	Ra	Range and level		
		-1	0	+1	
pH of sample	Α	3	5	7	
Distance between cathode and anode (cm)	В	0.5	1.25	2.0	
Current density (mA/cm <sup>2</sup> )	С	1	10.5	20	

could be removed from wastewater by complexation and or electrostatic attraction. The formed particles are then coagulated. Briefly, the electrocoagulation is made from three consecutive steps. Anodic dissolution including generation of metal cations that are produced from dissolution of sacrificial anodes due to the passage of direct electric current. Then, generation of coagulants from metallic cations reacted with hydroxyl ions (OH<sup>-</sup>) for generation hydroxylated species and ions-complexes positively charged. Finally, flotation is occurred due to small bubbles of hydrogen gas from cathodic reaction and in some cases, oxygen bubbles produced from water electrolysis at the anode [24].

Box-Benhken experimental design was used to optimize the main independent variables initial pH of the sample, distance between two electrodes and current density (Table 2). The experimental design shows the simple and combined effects of main variables on removal efficiency of NC fines. There are three major steps in DOE: (i) performing the statistically designed experiments; (ii) estimating of coefficients in the proposed model; (iii) predicting the response of procedure and control the validity of the proposed model. The response surface regression procedure was used to design of experimental conditions and the obtained data were analyzed by using of statistical analysis methods. The Minitab 18 software was used for experimental design and data analysis. The experimental conditions and obtained results (obtained and predicted data for removal efficiency of NC fines) are summarized in Table 3. Temperature of samples and retention time for EC process were 25°C and 10 min, respectively.

In the first step, Pareto-chart can be used to evaluate the overall results. Pareto-chart (Fig. 2) shows that independent variables of initial pH of sample and current density have the most influence on the removal efficiency of NC fines in EC method. The number of electrodes and the distance between them are factors that affect the efficiency of the electrocoagulation method. The distance between the two electrodes affects the conductivity of the solution. The results of preliminary experiments showed that the distance between the electrodes also affects the efficiency. However, the results in optimization process showed that in the proposed process, this parameter in comparison to two other parameters has not considerably effect.

For a more complete analysis of the results, the analysis of variance (ANOVA) of the obtained results is collected in Table 4. The statistical quantities of *P*- and *F*-value indicate that two independent variables of pH and current density are significant (P < 0.05), whereas, variable of distance between cathode and anode is insignificant (P > 0.05) [25,26].

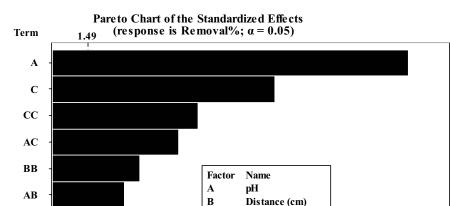
From the experimental results listed in Table 3, the response function representing the removal efficiency can be expressed as a function of pH of sample (A), distance between two electrodes (B), and current density (C). The relationship between response (%removal of NC fines) and variables was obtained as follows:

$$%R = 59.77 + 13.93A + 0.01B + 8.73C - 1.48A^{2} - 5.07B^{2} - 8.46C^{2} - 4.69AB + 7.73AC - 0.21BC$$
(6)

The coefficients obtained for parameters A and C indicate the significant effect of these parameters on the removal

No. of experiment	pH of samples (A)	Distance between cathode and anode, cm ( <i>B</i> )	Current density, mA/cm <sup>2</sup> ( <i>C</i> )	%Removal of NC fines (obtained)	%Removal of NC fines (predicted)
1	3	2.00	10.5	42.0, 39.6	43.5048
2	5	2.00	20.0	55.0, 54.3	54.0014
3	7	1.25	1.0	45.5, 45.5	48.6111
4	3	1.25	20.0	39.8, 38.8	37.9736
5	7	0.50	10.5	71.5, 75.0	72.3298
6	3	0.50	10.5	36.0, 35.4	35.6673
7	5	0.50	20.0	62.0, 41.9	54.3639
8	5	1.25	10.5	60.0, 59.4	58.9769
9	7	2.00	10.5	62.4, 61.5	63.7673
10	5	0.50	1.0	39.0, 37.0	36.5389
11	7	1.25	20.0	82.0, 80.3	80.7111
12	5	1.25	10.5	61.8, 59.5	58.9769
13	5	2.00	1.0	39.5, 40.6	36.1764
14	5	1.25	10.5	59.1, 61.2	58.9769
15	3	1.25	1.0	31.9, 32.5	34.4236

Table 3 Experimental conditions and obtained results in response surface methodology



C

6

Density (mA/cm2)

10

12

8

Standardized Effect

Fig. 2. Pareto-chart of Box-Benhken experimental design in RSM that is obtained for electrocoagulation of NC fines.

efficiency. While, the coefficient of parameter *B* indicates the insignificant effect for it. This result is consistent with the results of Pareto-chart. The quantities of *F*- and *P*-value of proposed model are 37.88 and <0.001, respectively, at 95% confidence level. The value of *P*-value < 0.05 for the model is acceptable, which indicated that the proposed model is significant. Generally, the *P*-values (Table 4) shows the importance of a variable on the response and the *P*-values > 0.1 show that the model terms are not significant [27,28]. The *P*-value of "lack-of-fit" is 0.131 (>0.05) and thus the lack of fit is not significant for the model and predicted data by model fit to actual experimental response data. Also, the value of  $R^2$  of the model is obtained 0.9446 which indicates that the quadratic equation is capable of modeling the system under the given experimental domain.

В

2

4

Residual plots for results of Box–Benhken experimental design are shown in Fig. 3. A residual plot is a graph that is used to examine the goodness-of-fit in regression and ANOVA. Examining residual plots used to determine whether the ordinary least squares assumptions are being met. If these assumptions are satisfied, then ordinary least squares regression will produce unbiased coefficient estimates with the minimum variance. Normal probability plot of residuals (Fig. 3a) applied to verify the assumption that the residuals are normally distributed. The residuals vs. order plot (Fig. 3b) to verify the assumption that the residuals are uncorrelated with each other and independent residuals show no trends or patterns when displayed in time order. Finally, the residuals vs. fits plot (Fig. 3c) to verify the assumption that the residuals have a constant variance.

14

16

Source	DF	Adj. SS	Adj. MS	<i>F</i> -value	<i>P</i> -value
Model	9	5,747.78	638.64	37.88	< 0.001
Linear	3	4,619.47	1,539.82	91.33	< 0.001
Α	1	3,214.88	3,214.88	190.68	< 0.001
В	1	0.00	0.00	0.00	0.996
С	1	1,189.56	1,189.56	70.55	< 0.001
Square	3	682.48	227.49	13.49	< 0.001
$A \times A$	1	15.48	15.48	0.92	0.349
$B \times B$	1	189.44	189.44	11.24	0.003
$C \times C$	1	526.96	526.96	31.25	< 0.001
2-Way interaction	3	772.42	257.47	15.27	< 0.001
$A \times B$	1	189.05	189.05	11.21	0.003
$A \times C$	1	512.84	512.84	30.42	< 0.001
$B \times C$	1	0.34	0.34	0.02	0.889
Error	20	337.21	16.86		
Lack-of-fit	4	115.31	28.83	2.08	0.131
Pure error	16	221.90	13.87		
Total	29	6,084.99			

Table 4 Analysis of variance for removal efficiency of NC fines

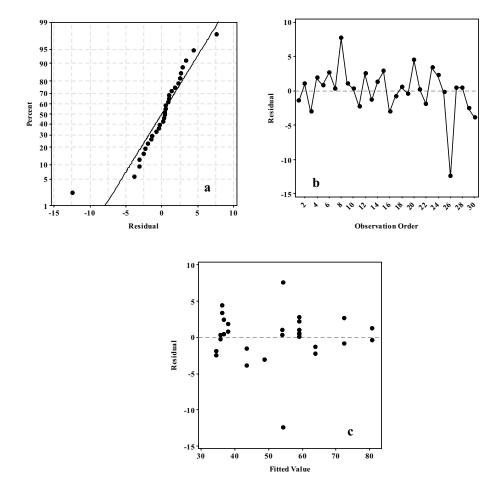


Fig. 3. Residual plots for results of Box–Benhken experimental design. (a) Normal probability plot of residuals, (b) residuals vs. order plot, and (c) residuals vs. fit plot.

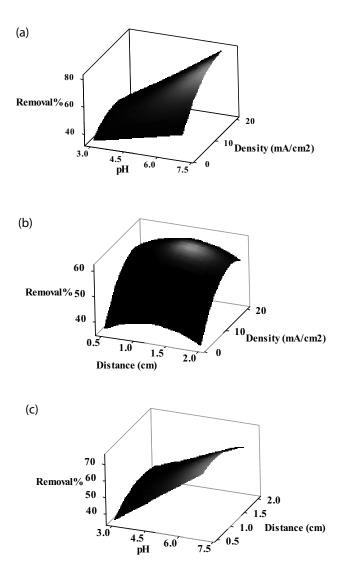


Fig. 4. Surface contour plots to estimate the removal efficiency for independent variables; pH and current density, current density and distance between anode and cathode, and pH and distance between anode and cathode.

The response surface contour plots and surface plot to estimate the removal efficiency for independent variables current density, pH of samples and distance between anode and cathode are shown in Fig. 4.

According to the statistical evaluation of the obtained results, optimized conditions for studied independent variable parameters are pH 7, current density 20 mA/cm<sup>2</sup>, and distance between anode and cathode 1 cm. The experimental and predicted removal efficiencies are obtained 80.75 ( $\pm$ 2.54) and 81.63 ( $\pm$ 2.22), respectively, in optimized conditions for electrocoagulation of NC washing section sample. Standard error for experimental value is related to three times of experiments. Increasing of removal efficiency is expected with increasing of current density. Current is applied to promote the generation of metal cations at the anode (Fe<sup>2+</sup>) and the evolution of hydrogen gas (H<sub>2</sub>) at the cathode. The Faraday's law (Eq. 7) shows the theoretical mass of metal cation generated through EC method.

$$m = \frac{Itz}{ZF}$$
(7)

where *m* is mass of metal cation generated in grams, *I* is current in amperes, *t* is elapsed time in seconds, *M* is molecular mass of the metal in grams per mole, *Z* is number of electrons transferred per metal atom, and *F* is Faraday's constant 96,485 C/mol. Therefore, the mass of Fe<sup>2+</sup> ions as coagulant species is increased with addition of current. In other words, the distance between two electrodes should be optimized so that the current paths from all points on the cathode to the anode are equivalent in distance. However, current density is not being perfectly uniform across an anode and it is highest where the anode is closest to the cathode [11–13].

Nitrocellulose micro-particles could be hydrolyzed in alkaline media (pH 7) to cellulose [2]. The amount of hydrolysis is dependent on pH of the sample. Therefore, the pH of samples must be less than of 7 to prevent the decomposition of NC molecules. Also, the ferrous ion is stable in pH values near to neutral (pH ~7).

The NC washing section samples were analyzed after treatment by using EC method in optimized parameters. The obtained results are collected in Table 5. Fig. 5 shows the image of a sample before and after treatment for the coagulation of NC fines by applied an electrical current. The electrolysis time is 10 min for EC method.

The NC fines can be removed from a wastewater sample using destabilization by the addition an electrolyte [1].

#### Table 5

Analysis of the NC washing section after treatment by using EC method. Each experiment is repeated three times

Analysis	Amount
COD (mg/L)	$493.57 \pm 4.23$
TSS (mg/L)	$98.82 \pm 2.94$
TDS (mg/L)	$18.69 \pm 2.11$
pH	6.2

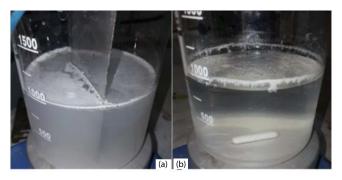


Fig. 5. Image of a sample before (a) and after (b) treatment for coagulation of NC fines by applying an electrical current at time 10 min.

An electrolyte such as NaCl salt validated particle stability for NC fines dispersed in a wastewater sample. The NC fines became unstable when the ionic strength of the sample is  $\geq$ 5 mM [1]. The Lewis acid–base interactions describe the greater attractive potential energy availability. With addition of an electrolyte, the NC fines may be easily destabilized allowing potential reuse. Production of ions such as Fe<sup>2+</sup> and OH<sup>-</sup> in EC method is due to increasing of electrolyte concentration and thus ionic strength in the sample.

Electrocoagolation of NC fine particles compared to the other used techniques for treatment NC wastewater such as microfiltration, anaerobic digestion, and chemical coagulation show the lower cost, absence of chemical and shorter time.

# 4. Conclusion

Typically, large amounts of nitrocellulose fine particles are present in industrial effluents. Therefore, the treatment of this type of effluent and the separation of NC fine particles is very important economically and environmentally. In order to separate the fine particles, floating them, and converting them to larger particles helps to separate more easily. Electrocoagulation method can be used for flocculation of NC fines. Simultaneous separation of several pollutants in one step, relatively low cost, low residual sludge volume, and ease of automation are among the advantages of this method.

In order to increase the efficiency of the process and separate the maximum fine particles, it is very important to optimize the conditions. RSM (among the design of experiments methodology) can be applied for the optimization of conditions.

For a sample contains NC fine particles with initial COD of 2,564 mg/L and in optimized conditions, current density 20 mA/cm<sup>2</sup>, pH 7, and distance 1 cm between cathode and anode, the removal efficiency of more than 80% is obtained at the time of 10 min. According to the applied potential (32 V), current (2 A), and time (0.167 h), electricity consumption is 10 kWh per m<sup>3</sup> of the sample. The obtained results of this research show that this technique can be used for the treatment of wastewater of other explosives.

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