Removal of Lead Contaminant from Cr Surfaces by Using H₂O₂/EDTA Cleaner Solution

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Abstract

All industrial processes especially metal cleaning procedures dealing with dangerous solutions should be used as little as possible and their emission into the aquatic environment should be controlled. In this study, in order to find a proper and efficient cleaning process, lead pollution was removed from the solid matrix by using a cleaner solution containing EDTA and H₂O₂ as chelating and oxidizing agents respectively. An orthogonal array design (OAD), OA9, was employed as a chemometric method for the optimization of the procedure. The results of experiment revealed that, lead pollution (~3 g) at the predicted optimum condition can be effectively removed from the solid matrix during 4 minutes. Cleaning quality was checked by SEM/EDAX, ICP and UV spectroscopy methods. Finally, the results of this investigation showed that H₂O₂/EDTA system can be considered as a novel, safe and an efficient cleaning solution, due to its proper processing parameters.

Keywords: Surface cleaning, Lead pollution, Cleaner solution, EDTA, Safety issues, Statistical optimization.

1. Introduction

Surface cleaning is a key step during workpiece manufacturing and almost all metallic surface products need cleaning-up procedure to take out surface contaminations remaining after processing or employment.[1–3] However low cleaning-up effectiveness and damaging the surfaces restricted the employing of direct mechanical methods[4] and prevalently, the removal of surface pollutions by some solvent-based methods is better.[5–7]

These wet methods are widely used and generally efficient, but they have several disadvantages. Conventional wet cleaning processes usually consume a large number of dangerous chemicals and solvents;[8] however the use of hazardous compounds has been controlled and in some cases restricted for the reasons of health protection and environmental consideratios.[9] Besides, these traditional cleaning methods required several steps at the high temperature, 50–80 °C, to complete the removal of the heavy metal from polluted surfaces.[10] Moreover, presence of heavy metals such as lead and other pollutants in industrial wastewater due to metal finishing and metallurgy procedures remains as a serious concern, because of related health issues.[11] So, the investigation of novel safe chemical cleaning methods and overcoming these pollution issues is still attractive topic.

The results of recent investigations reveal that the combination of chelation ability of proper ligands with suitable oxidizing agents is an appropriate technique for treatment of metallic surfaces.[12] These proposed systems considerably decrease the metal adsorption on the surfaces and improve the safety issues of methods by reducing the wastewater productions.[13,14]

In industrial processes, a lot of chelating agents such as EDTA (ethylene diamine tetra-acetic acid), NTA (nitri-

lo-triacetic acid) and HEIDA (N-(2-hydroxyethyl) imino diacetic acid) are generally used in order to chelate complexation of metal ions.[15] Selecting suitable chelating agent is a critical parameter and its requirements including high stability constants of the metal complexes, fast complexation kinetics, high solubilities of the chelating agents and its metal complexes.[14] EDTA with wide range of applications is one of the most suitable chelating agents and in aqueous solutions, it can form complex in a 1:1 (EDTA-M) ratio by forming strong bonds with metal ions.[15]

Our recent research illustrated that H_2O_2/CH_3COOH cleaning system can be introduced as a low-cost and simple pickling solution in order to clean Cr surfaces from lead pollution and showed that the efficiency of method is affected by some experimental parameters.[16,17] However, these cleaning methods are usually time-consuming, especially for solids that leaching and oxidation are needed to release the metal ions before complexation. On the other hand, the procedure requires a large amount of toxic liquid which causes environmental pollution.

However, in order to improve the cleaning process, it is required to inspect the procedure with new chelating agents and exploit orthogonal array design methods for optimization of the procedure. Consequently, EDTA, well-known as a prevalent chelating agent,[12–15] was employed for the first time as an alternative chelating agent in order to omit lead pollution via a suitable, fast and convenient chemical method. Moreover, in order to determine the optimum cleaning condition, the effect of some experimental parameters including oxidizing and chelating agent concentration, stirring speed (revolutions per minute, rpm) and temperature of the cleaner solution on the cleaning quality, was evaluated by an orthogonal array design.[18,19]

The objectives of this investigation are: (1) to suggest a new method for the cleaning of Cr surfaces by using EDTA as a proper chelating agent, (2) to apply the orthogonal array design method to obtain the best cleaned surfaces by using optimal removal conditions, and (3) to confirm this method, it is compared with previous results in terms of performance, rapidity, cost and safety issues. Finally, the quality of surface treatment has been checked by Scanning electron microscopy (SEM), Energy dispersive X-ray microanalysis (EDAX) and Ultraviolet Spectrophotometric (UV) analysis.

2. Experimental

2. 1. Materials and Apparatus

ASTM B 32 standard lead bar (with 99% purity) was prepared from industrial Zanjan zinc-lead Co. (Tehran, Iran). All chemical reagents (Aqueous hydrogen peroxide solution, H_2O_2, 35% (V/V), disodium ethylene diamine tetra-acetic acid, Na_2EDTA, and lead chloride, $PbCl_2$) were purchased as analytical or certified A.C.S grades from Fluka (Tehran, Iran). All stock solutions were prepared by very high quality deionized water.

Stock EDTA solution, 0.1M was prepared by dissolving Na_2EDTA crystals in deionized water. Stock $[Pb^{2+}]$ solution, 7.2×10^{-6} M was prepared from 0.01 M $HClO_4$ and $PbCl_2$ (99.99 % purity). In order to investigate the changes in the absorbance of the *Pb(EDTA)*$^{2-}$ at 239 nm, a Milton Roy model spectronic 3000 array spectrophotometer was employed. If the room temperature is subject to variation greater than ±2 °C, a constant temperature bath is necessary. Surface cleaning quality has been evaluated using a scanning electron microscope (SEM/EDAX).[20,21] Scanning electron micrographs were recorded by means of a Philips XL30 series instrument using a gold film for loading the dried particles on the instrument. Gold films were prepared by a Sputter Coater model SCD005 made by BAL-TEC (Switzerland).[22–24] Inductively coupled plasma spectroscopy (ICP) instrument model VISTA-PRO made by varian company (Australia) was used for determination of chromium ion concentration in waste water solution.

2. 2. Cleaning Procedure

The procedure was arranged based on a wet cleaning technique which works based on the oxidation and chelation reaction principal.[16,17] However, to prepar the

Table 1. Taguchi L-9 orthogonal array design and dissolving time of 3 g lead bar.

Experiments	Process parameters				Results
	Oxidizing agent (V/V %)	Chelating agent (mM)	Stirring speed of the mixture (rpm)	Temperature (°C)	Dissolving time (minute)
1	2	7.5	25	15	29.0±0.3
2	2	15	50	20	19.0±0.2
3	2	25	75	25	13.0±0.4
4	3.5	7.5	50	25	14.0±0.2
5	3.5	15	75	15	18.0±0.1
6	3.5	25	25	20	14.0±0.1
7	5	7.5	75	20	15.0±0.2
8	5	15	25	25	5.0±0.1
9	5	25	50	15	13.0±0.2

cleaner solution, a spicefied volume stock solution of ED-TA and H_2O_2 were added in a volumetric flask and the flask was filled to the mark with deionized water. In a representative trial (run 4 Table 1), in order to dissolve 3 g metallic lead bar, a 100.0 ml of H_2O_2 (3.5% V/V with respect to total amount of solution) and a 75.0 ml of EDTA (7.5 mM with respect to total amount of solution) were added in a 1000.0 ml volumetric flask and it was filled to the mark with deionized water. subsequently, lead bar was added to the solution and the cleaner solution was stirred at the stirring speed of the mixture 50 rpm and temperature 25 °C.[16,17] After a period of mixing and ending of the disolving process, the experiment will be finished and the total dissolving time will be recorded (14 min).

2. 3. Optimization of Lead Cleaning Procedure

An experimental design method was employed to improve the efficiency of dissolving procedure.[18] The variables including percent of oxidizing agent (H_2O_2), concentration of chelating agent (EDTA), stirring speed of the mixture (revolutions per minute, rpm) and temperature of the cleaner solution were changed as shown in Table 1. Then the optimum conditions that proposed by ANOVA method were used for further investigation.

2. 4. Calibration Curve

A calibration curve was arranged by adding 1.2, 2.2, 3.2, 4.2, 5.2, 6.2 and 7.2 μM stock solution of $[Pb^{2+}]$ to the 1000 ml volumetric flasks, adding 0.2 ml EDTA solution and the volumetric flasks were then brought to a total volume with deionized water. The flasks were placed into the 25°C water bath for 4 minutes and then the absorbance of the solutions was measured at 239 nm.

2. 5. Kinetic Measurements

285 ml stock solution of H_2O_2 was placed in a 1000.0 ml volumetric flask, followed by 500 ml of the EDTA stock solution and the flask was filled to the mark with deionized water. Then, 3 g lead bar was inserted in the cleaner solution and the solution was stirred at the stirring speed of the mixture 25 rpm and temperature 25 °C. However, in order to avoid chemical error during Spectrophotometric measurement, the solution containing dissolved lead bar diluted to 1/2000 and subsequently lead oxidation was followed in a UV cell by recording the change in the absorbance of cleaner solution containing *Pb(EDTA)*$^{2-}$ as a function of the reaction time.

2. 6. Treatment of Cr Surface as Real Sample

In order to inspect the performance of the proposed cleaning method, a Cr surface sample deposited with about 3 g of metallic lead pollution was employed. However, the polluted metallic surface was picked up by novel cleaner solution at the ANOVA proposed optimum condition and the efficiency of the investigated cleaning method was accurately checked.

3. Results and discussion

3. 1. Cleaning Results and Optimization Strategy

The theory, methodology and application of OAD as a chemometric method for the optimization of the chemical procedures have been demonstrated in the literature results.[25–27] On the other hand, the analyses of the data, assuming no interaction between variables, comprise: (1) identification of the optimum condition, (2) finding of the individual influence of each factor, and (3) prediction of the result at the proposed optimum condition.[28–30] The tested factors and their levels are given in Table 1. Also, dissolving time of 3 g lead bar is presented in the last column of Table 1. The average values of the coefficients of variation of the factors at each level were calculated according to assignment of the experiment (Table 2).[18,19] For instance, to calculate the average value of dissolving time of 3 g lead bar for the effect of the temperature of the cleaner solution (T) at level 1, dissolving time of the nine trials in which temperature of the cleaner solution was set at level 1 (Trials 1, 5 and 9) were pooled and divided by the numbers of the values (three values). The average value of the three levels of a parameter displays how dissolving time will change when the level of the parameter is altered.

Table 2. Results of the main effects for each variable on the dissolving time of 3 g lead bar

Factor	level	Dissolving time (minute)
Oxidizing agent (H_2O_2) V/V%	2	20.3±0.2
Oxidizing agent (H_2O_2) V/V%	3.5	15.3±0.1
Oxidizing agent (H_2O_2) V/V%	5	11.0±0.2
Chelating agent (EDTA) mM	7.5	19.3±0.2
Chelating agent (EDTA) mM	15	14.0±0.1
Chelating agent (EDTA) mM	25	13.3±0.2
Stirring speed of the mixture (rpm)	25	16.0±0.2
Stirring speed of the mixture (rpm)	50	15.3±0.1
Stirring speed of the mixture (rpm)	75	15.3±0.1
Temperature (°C)	15	20.0±0.2
Temperature (°C)	20	16.0±0.1
Temperature (°C)	25	10.7±0.1

The analysis of variance (ANOVA) for the results (dissolving time) is shown in Table 3. In this inquiry, the influence of concentration of chelating agent (EDTA) on the dissolving time at three diverse levels (7.5, 15 and 25 mM) was

studied. It was recognized that concentration of EDTA is an important factor for the control of the dissolving time of lead bar. Also, it appeared that temperature of the cleaner solution is an important parameter that could influence the dissolving time of lead bar. Conversely, our findings confirmed that stirring speed of the mixture (revolutions per minute, rpm) during the dissolving process is not a considerable parameter for the control of dissolving time of lead bar.

Finally, the influence of percent of oxidizing agent (H_2O_2) on the dissolving time of lead bar was examined at three diverse values: 2, 3.5 and 5% V/V. The results illustrated that percent of H_2O_2 can be considered as a significant factor on the dissolving time of lead bar.

Evaluation of the ANOVA results of this investigation shows that (at 90% confidence level), with omitting stirring speed of the mixture (revolutions per minute, rpm) all variables (percent of H_2O_2, concentration of EDTA and temperature of the cleaner solution) have considerable effects on the dissolving time of lead bar during the process.

The consequences of ANOVA disclosed that the excellent optimum conditions for the producing of cleaning solution are 5% V/V of oxidizing agent (H_2O_2), 25 rpm stirring speed of the mixture, 25 mM of chelating agent (EDTA) and 25 °C temperature of the cleaner solution.

As a common rule, the maximum performance (in our study, the shortest time for dissolving of lead bar) will be obtained by means of the subsequent equation:[18]

$$Y_{opt} = \frac{t}{n} + (O - \frac{t}{n}) + (C - \frac{t}{n}) + (T - \frac{t}{n}) \qquad (1)$$

Where t/n is average dissolving time for lead bar, t and n are the grand total of dissolving time for all experiments and total number of experiments. In the equation (1), predicted dissolving time of lead bar at optimum condition is Y_{opt}. O, C and T are explained as dissolving time at optimum condition for amount of oxidizing agent, chelating agent and temperature of cleaning solution, respectively.

Also, the formula for estimation of the confidence interval of the optimum dissolving time (*CI*) results from:[19]

$$CI = Y_{opt} \pm \sqrt{\frac{F_\alpha(f_1, f_2)V_e}{n_e}} \qquad (2)$$

where $F_\alpha(f_1, f_2)$ is variance ratio for degree of freedom (DOF) f_1 and f_2 at the level of significance α (in this work, $\alpha = 90\%$) and the confidence level is $(1 - \alpha)$, f_1 = DOF of average (which constantly equals 1), f_2 = DOF of the error term, n_e = number of equivalent replications, and given by n_e = number of trials/DOF of average (always 1) + DOF of all factors results used in the estimation. Statistical calculations for determing the result and *CI* at optimum condition revealed that the dissolving time of lead bar with the shortest time will be 3.9 ± 0.4 minutes.

In the next step of our inquiry, 3 g lead bar was treated at obtained optimum condition (5% V/V of H_2O_2, 25

rpm stirring speed of the mixture, 25 mM of EDTA and 25 °C temperature of the cleaner solution). The results of experiment revealed that dissolving time of 3 g lead bar at the predicted optimum condition is 4 minutes.
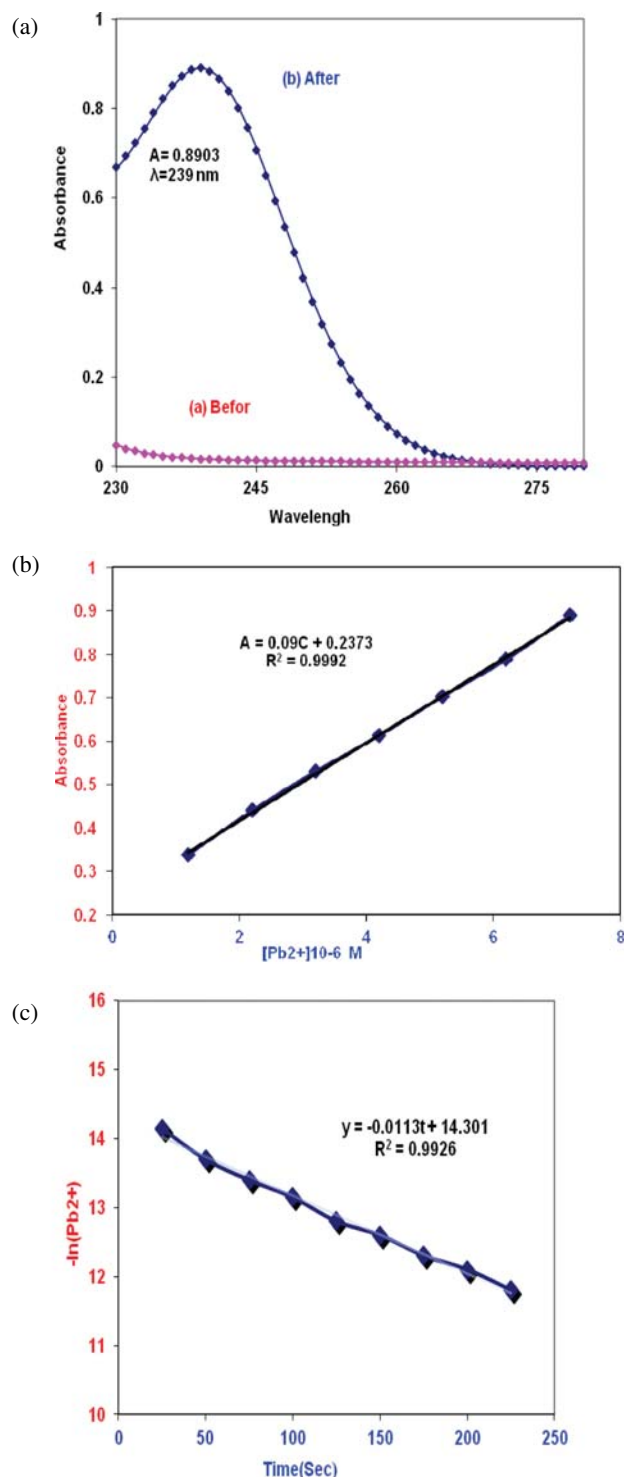

Figure 1. Experimental result of lead bar oxidation measured by UV spectrophotometric method. (a) UV spectra of the H_2O_2/EDTA aqueous solution, before and after using as cleaner solution, (b) calibration curve and (c) Values of $-\ln [Pb^{2+}]$ as a function of time.

Table 3. ANOVA table for the dissolving of lead bar with redox procedure by OA9(3⁴) matrix with the dissolving time of 3 g lead bar as the response.

Factor	Code	DOF	S	V	DOF	S'	F'	P'
							Pooled[a]	
Oxidizing agent (H_2O_2) V/V%	O	2	130.9	65.4	2	130	147.3	39.6
Chelating agent (EDTA) mM	C	2	64.9	32.5	2	64	73	19.5
Stirring speed of the mixture (rpm)	R	2	0.9	0.45	–	–	–	–
Temperature (°C)	T	2	131.6	65.8	2	130.6	148	39.8
Error	–	–	–	–	2	0.9	–	1.08

[a] The critical value was at 90% confidence level; pooled error results from pooling insignificant effect.
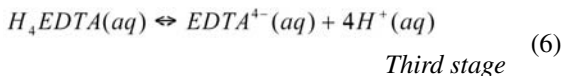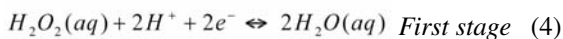
3. 2. Calibration Curve

The absorbance of cleaner solution containing *Pb(EDTA)²⁻* at the optimum conditionis demonstrated in Figure 1a. However, the absorbance against lead ion concentrations obeys the least-square line, Which [C] is expressed in moles × 10⁻⁶ (Figure 1b).

$$A = 0.09 \, [C] + 0.2373 \, ;$$
$$1.2 \times 10^{-6} \leq [C] \leq 7.2 \times 10^{-6} \, ; \tag{3}$$
$$n = 7 \, ; \qquad r = 0.9996$$

However, the results of calibration curve not only employed for the kinetic calculations but also can be used to determine the trace amounts of lead in wastewater solution by using a fast and appropriate spectrophotometric method.[31]

3. 3. Kinetic of Lead Cleaning Reaction

In this inquiry, kinetic of involved reactions were investigated in order to obtain more information from the mechanisms of the lead cleaning reactions and the Ostwald approximation method was used to determine the kinetic parameters.[32–34] However, based on the chemical data, consecutive reactions can take place in the cleaner solution:

$$H_2O_2(aq) + 2H^+ + 2e^- \Leftrightarrow 2H_2O(aq) \quad \textit{First stage} \tag{4}$$

$$Pb^{2+}(aq) + 2e^- \Leftrightarrow Pb(s) \qquad \textit{Second stage} \tag{5}$$

$$H_4EDTA(aq) \Leftrightarrow EDTA^{4-}(aq) + 4H^+(aq)$$
$$\textit{Third stage} \tag{6}$$

Subsequently, lead and EDTA ions in aqueous solution will form the stable *Pb (EDTA)²⁻ complex (eq. 7).*

$$Pb^{2+}(aq) + EDTA^{4-}(aq) \Leftrightarrow Pb(EDTA)^{2-}(aq)$$
$$\textit{Total reaction} \tag{7}$$
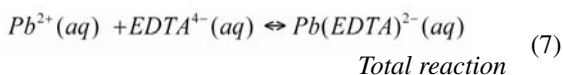
Thus, the reaction rate could be expressed by applying the following equation:

$$R = \frac{dx}{dt} = K[Pb^{2+}]^\alpha [EDTA^{4-} - x]^\beta \tag{8}$$

In this work, oxidizing and chelating agent concentrations were used at higher than stoichiometry ratio and could be assumed that, their concentrations not vary during the cleaning reaction. However by using the Ostwald approximation method,[32,35] the equation 8 can be expressed as:

$$R = K[EDTA^{4-}]^\beta [Pb^{2+}]^\alpha \tag{9}$$

If it is assumed that $K[EDTA^{4-}]^\beta \cong K'$, the reaction rate is explained as:

$$R = K'[Pb^{2+}]^\alpha \tag{10}$$

According to equation 10, the reaction rate is changed to pseudo first-order. By applying the assumption that the α (reaction order) value is equal to 1, the rate expression of reaction is simplified to:

$$-\ln[Pb^{2+}] = k't \tag{11}$$

In the pseudo first-order's approach, plot of $-\ln[Pb^{2+}]$ against of time (t) is used to calculate the reaction rate constant (k'), which $[Pb^{2+}]$ is the lead ion concentration during the reaction time (t). Therefore the rate constant (k') was calculated from the slope and the validity of estimation (α = 1) was confirmed on the data of linear regression (r).

The experimental results of $[Pb^{2+}]$, $-\ln[Pb^{2+}]$ and absorbance (A) at different time of reaction was summarized in Table 4. However, the prevalent curve follows the least-squares line (Fig. 1c):

$$Y = -0.0113t + 14.3 \, ;$$
$$25 \leq t \leq 225 \, ;$$
$$n = 9 \, ; \tag{12}$$
$$r = 0.997$$

The plot was straight lines (r = 0.997) which indicated the mechanism of cleaning reaction did not vary during the reaction time and the assumption α = 1 is acceptable. The slope of the lines is equal to rate constant (k'), 1.13 × 10⁻² (S)⁻¹.

Table 4. The experimental results of lead bar oxidation as a function of time, measured by UV spectrophotometric method.

No	Time (S)	A	$[Pb^{2+}]10^{-6}$	$-\ln [Pb^{2+}]$
1	25	0.3021	0.72	14.1
2	50	0.3399	1.14	13.7
3	75	0.3718	1.50	13.4
4	100	0.4119	1.94	13.1
5	125	0.4825	2.72	12.8
6	150	0.5343	3.30	12.6
7	175	0.6265	4.32	12.3
8	200	0.7422	5.60	12.1
9	225	0.8920	7.30	11.8

3. 4. Lead Cleaning from Cr Surface

In order to confirm the applicability of the proposed method for the cleaning of a dirty surface, a chromium plate (250×10 mm^2) that is covered with a layer (nearly 3g) of metallic lead was treated during 4 minutes with the cleaner solution at the optimum conditions (5% V/V of H$_2$O$_2$, 25 rpm stirring speed of the mixture, 25 mM of EDTA and 25 °C temperature of the cleaner solution). Visual inspections of cleaned surfaces confirm the appropriate performance of cleaner solution; however, for deep and detailed examination of the cleaning method, all surfaces were tested by UV, SEM/EDAX and ICP analysis.
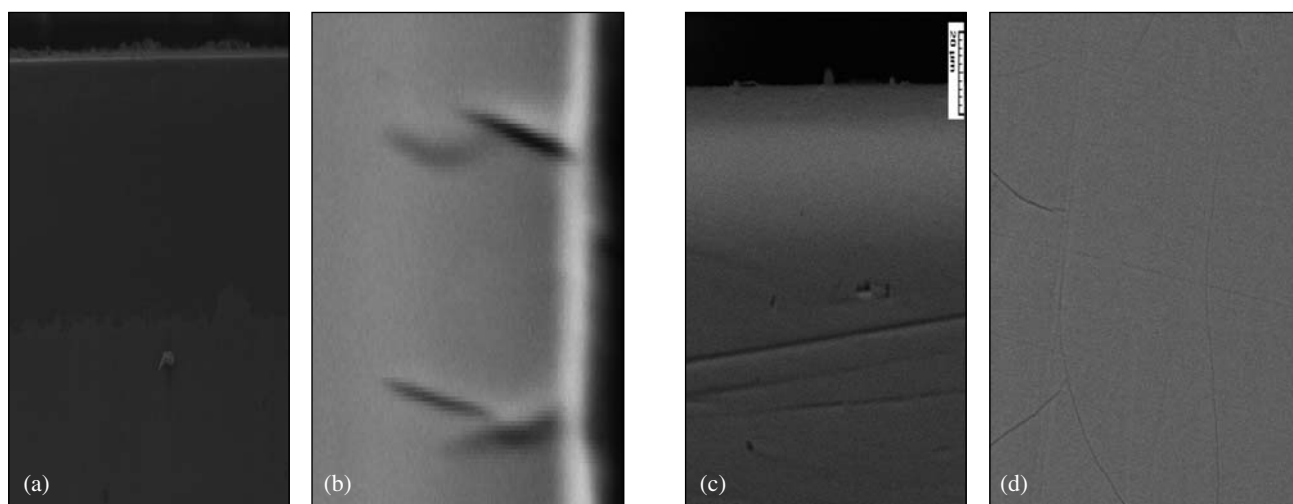
3. 5. Results of Spectroscopy Aanalysis

Figure 1a shows the UV spectra of the H$_2$O$_2$/EDTA aqueous solutions. Before its use for cleaning (spectrum a), no peaks indicating the presence of Pb–EDTA-complex ions were seen. After the solution was used for the cleaning (spectrum b), on the other hand, a peak indicating the presence of Pb–EDTA-complex ions was clearly observed at 239 nm,[4,5] showing that Pb was removed by the formation of Pb–EDTA-complex ions.

Figure 2 illustrates SEM photographs of different samples containing, dirty (Fig. 2a and 2b) and cleaned surfaces sample at optimum condition (Fig. 2c and 2d). The results approved that the uncleaned samples has lead fouling layer with about 3–4 μm average size in thickness and the most cleaned sample without any corrosion and no lead fouling streak can be obtained at the optimum cleaning condition. Furthermore, Figure 3 demonstrated the colored SEM image and map data of cross-section of real surface sample (Fig. 2b) after treatment with cleaner solution at optimum condition. Finally, according to the results of EDAX analysis (Fig. 4), lead pollution was removed completely and prefect cleaned surfaces could be successfully obtained by an experiment in optimized conditions. Also, in order to evaluate the damaging effects of cleaner solution on the metal surfaces, the Cr concentration of waste cleaner solution was checked by ICP absorption spectroscopy technique. However, the analytical results confirmed that the novel cleaning solution doesn't have any corrosion effects on the metallic surfaces.

3. 6. Comparison with Literature Results

As mentioned before, the aim of this investigation was to introduce a safe and proper alternative cleaning procedure respected to the conventional methods. However, the efficiency and safety issues of proposed method have been compared with previous results in terms of performance, cost and environmental compatibility. In this view, our literature survey showed that usual wet cleaning methods[5–8] presented numerous difficulties including: 1) adding contaminants due to insufficient cleaning and filtering of the liquid at the submicron level of cleaned surfaces,[8,9] 2) inserting of the bulk hazardous solvents[10,11] and heavy metal pollution in wastewater, and 3) consisting of several successive steps at high temperatures with

Figure 2. SEM image of (a-b) cross-section of real sample (Cr surface coated with about 3g of metallic lead) before treatment with cleaner solution, (c) cross-section of the sample after cleaning and (d) surface of the sample after cleaning at optimum condition.
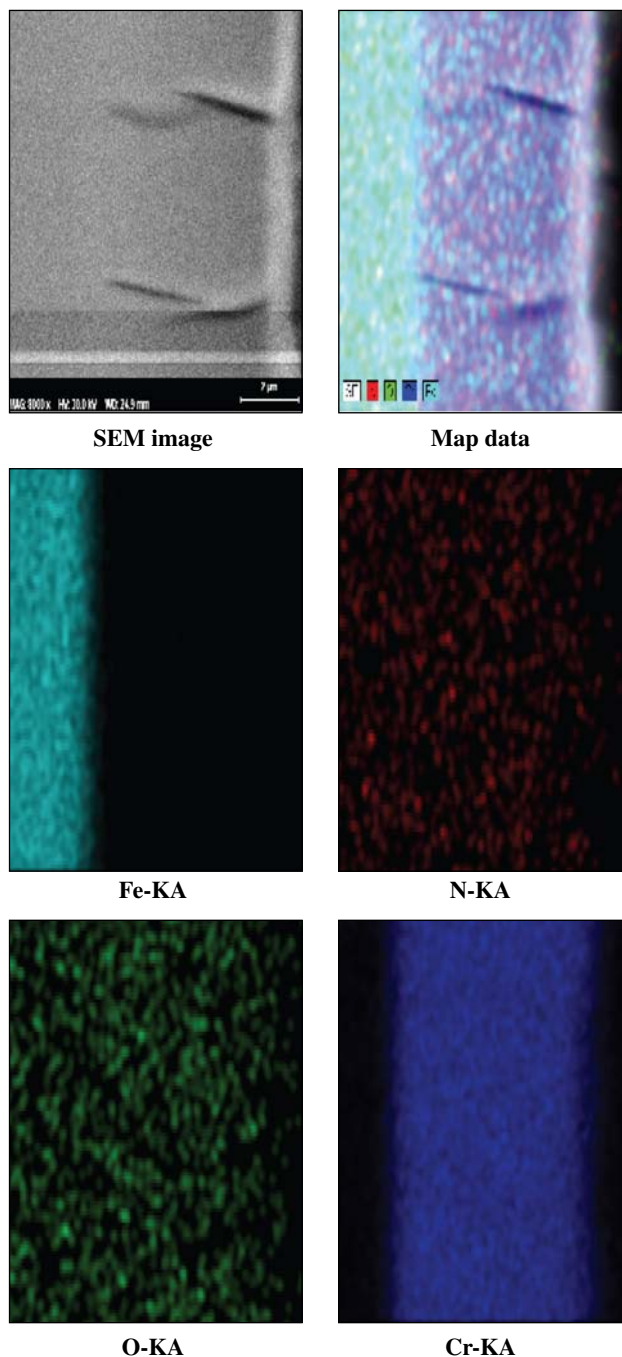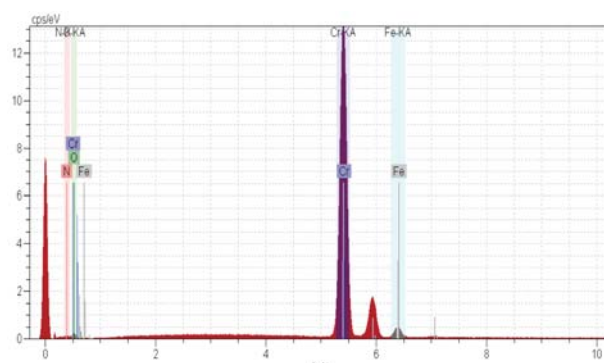
SEM image

Map data

Fe-KA

N-KA

O-KA

Cr-KA

Figure 3. Colored SEM image of Cr surface (Fig. 2b) after treatment with cleaner solution at optimum condition.

Element	Weight Percent (%)
Oxygen	0.01
Nitrogen	0.8
Chromium	95.7
Iron	2.3
Total	98.81

Figure 4. Surface EDAX spectrum of the Cr surface (Fig. 2d) after cleaning at optimum condition.

chromium surface.[16] Nevertheless, combination of chelation ability of EDTA with the statistical optimization procedure showed that under stirring speed of 25 rpm, the most cleaned sample surface was achieved by using cleaning solution with 5% V/V of H_2O_2, 25 mM of EDTA and temperature of 25 °C within 4 minutes. Also, the reaction rate constants of H_2O_2/EDTA simultaneous system obtained by UVdata revealed, the first order rate constant of the method is noticeably high and confirmed the higher performance of the proposed procedure. Also, in our novel proposed procedure, applying of H_2O_2/EDTA system as proper picking solution with a chemometric method reduces the usage of hazardous chemicals and solvents. Moreover, formation of the very stable *Pb(EDTA)*$^{2-}$ complex during the cleaning procedure, not only prevents desorption of lead ion on the Cr surfaces but also reduces the lead ion emission in the biological environment.[4] Finally, evaluation of the results confirms, combination of statistical optimization with chelation ability of proper ligand (EDTA) forced the cleaning procedure to the safe and moderate experimental conditions.

4. Conclusion

Generally, it is recommended that during the chemical procedures, the entry of heavy metal ion and hazardous chemical material into the water phase should be minimized by applying various measures. In this research, in order to minimize the environmental pollution, EDTA as a conventional chelating agent, was selected to improve the lead fouling dissolution procedure. Also, A L9 orthogonal array design method with four factors and three lev-

etching of metal surface in nm order, which makes processes very complicated.[11]

However, the simultaneous H_2O_2/EDTA cleaning methodis clearly faster and efficient than its earlier partners. For instance, the results of pervious investigation showed that for the sequential H_2O_2/CH_3COOH (HA) method, at optimum condition (HA 28% V/V, H_2O_2 8% V/V and temperature 35 °C), 20-minutes time is needed for complete removal of 3 g fouling lead layer from

els was used to optimize the cleaning process. The four factors included the stirring speed, temperature, H_2O_2 and EDTA concentrations of the cleaner solution.The variables were examined in order to increase the efficiency of the proposed method as an efficient cleaning procedure. It was found that all parameters except stirring speed of mixture have a considerable influence on the lead removal procedure. The OAD evaluation of experimental data provides optimized amount of the parameters, 5% V/V of H_2O_2, 25 rpm stirring speed of the mixture, 25 mM of ED-TA and 25 °C temperature of the cleaner solution to obtain the most cleaned sample, at which the total removal time of 3 g lead fouling layer is about 4 minutes. The results of experiments proved by using this method, Cr surfaces can be completely cleaned at proposed optimum conditions. SEM/EDAX photographs of completely cleaned samples showed that the treated surfaces were uniform without any surface corrosion. Finally, the results of this investigation confirmed the higher efficiency of the H_2O_2/EDTA solution than conventional cleaning procedures.

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Povzetek

V industrijskih procesih, še posebej pri postopkih čiščenja kovinskih površin, moramo nevarne reagente uporabljati v čim manjši meri, s čimer omejimo njihove emisije v vodno okolje. V tej študiji smo razvili učinkovit čistilni postopek za odstranjevanje svinca s trdne površine z uporabo raztopine, vsebujoč EDTA in H_2O_2 kot kompleksirajoče oziroma oksidirajoče sredstvo. Za načrtovanje in optimizacijo postopka smo uporabili kemometrično OAD metodo ortogonalne matrike (OA9). S poskusi smo potrdili, da lahko pri optimalnih pogojih v času štirih minut s površine odstranimo tri grame svinca. Učinkovitost čiščenja smo spremljali s SEM/EDAX, ICP in UV spektrofotometričnimi meritvami. Rezultati kažejo, da lahko H_2O_2/EDTA raztopino zaradi njenih ustreznih lastnosti smatramo za novo, varno in učinkovito sredstvo za čiščenje površin.