World Applied Sciences Journal 20 (10): 1407-1415, 2012
ISSN 1818-4952

DOI: 10.5829/idosi.wasj.2012.20.10.268

Investigation of the Processing Parameters on the Chemical Cleaning of Fouling Lead Layer from Chromium Surfaces by Using an Orthogonal Array Design Method

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Abstract: In this research, an orthogonal array design (OAD), OA9 was exploited as a chemometric experimental method for the cleaning of chromium surfaces contamination by using an electrochemical cleaner solution that is established upon the redox reaction principle. The influence of cleaning procedure on the removal of lead fouling layer has been investigated by means of SEM/EDAX and ICP analysis. In order to estimate of the main effect of some experimental parameters, we examined the procedure with a statistical method and the results were quantitavely evaluated by the analysis of variance (ANOVA). The OAD estimation of experimental result provides optimized amount of the parameters including, H_2O_2 10% (V/V), stirring speed of the mixture 75 rpm and temperature 28°C, to achieve the most cleaned sample, at which the total removal time of 3 g fouling lead layer is predicted only 7 minutes. The prediction has a complete conformity with experimental result achieve at the similar condition that is 8 minutes. Also, the reaction rate constant of the lead bar oxidation at optimum condition was obtained from the ICP spectroscopy data that proposed by Ostwald approximation method. Finally, the results of this study illustrated that the efficiency of the proposed chemometric cleaning method is higher than that our partner sequential experimental technique.

Key words: Taguchi method · Surface cleaning · Lead pollution · Redox reaction · Cleaner solution · Statistical optimization

INTRODUCTION

Chromium coatings are applied on the surface of numerous metals such as iron, cobalt and nickel-base alloys for decorative purposes to enhance resistance and to lower the coefficient of friction. Generally, chromium is oxidized readily in the air and forming a thin nano-scale layer, adherent and transparent coating of Cr_2O_3 [1]. However for this reason some common metals such as aluminum, chromium, zinc and nickel stay bright in the clean inside atmosphere. The most important application of chromium is in the production of steel. However, high-carbon and other grades of ferrochromium alloys are added to steel to improve mechanical properties, increase hardening and enhance corrosion resistance [2-3].

Almost all metallic surfaces products need clean-up procedure to take out surface contaminations that remain after processing or employment [4-9]. However, research on the lead cleaning methods are an interesting subject since lead fouling on the chromium surfaces is a common difficulty in the metallic systems that having interaction with metallic lead in high temperatures and pressures [10-12].

Our recent investigation revealed that H_2O_2/ CH_3COOH redox system can provide a low-cost, simple, fast and effective cleaner solution to introduce a novel pickling solution in order to cleaning the lead fouling layer from chromium surfaces (Eq.1) and the quality of procedure is influenced by some experimental condition [13].

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$$Pb(s) + H_2O_2 + CH_3COOH \rightleftharpoons Pb(CH_3COO)_2(aq) + 2H_2O \quad (1)$$

Nevertheless, in order to approximate the parameters influencing on the procedure, we require using a statistical method for improvement of efficiency of the process. Consequently, an orthogonal array design (OAD) method has been used to evaluate and to optimize the most important parameters affecting the cleaning quality and increasing the efficiency of the proposed cleaning procedure [14].

Optimization is a significant step in upward a cleaning procedure. That is containing two broad organized methods containing simultaneous and sequential methods [15-16]. In second methods (e.g., simplex optimization), the response surface is consecutively followed up to a most favorable has been achieved [17]. Deficiencies of sequential methods are slow convergence on the complex response surface and difficulty in dealing with the response surface with high dimensionality. However, these methods could be suitable as a means for fine-tuning a separation [18]. On the other hand, the former optimization methods, such as mixture designs [12, 19] and factorial designs, [20-22] do not have these difficulties.

In our study, Taguchi L-9 orthogonal array design as a simultaneous method was employed to display the effect of four procedure parameters that is percent of oxidizing agent (H_2O_2), percent of chelating agent (CH_3COOH), stirring speed of the mixture (revolutions per minute, rpm) and temperature of the cleaner solution on the cleaning qualities. This plan contains nine separate experiments with particular level and a precise combination of parameters. On the other hand, the sequence in which the experiments were carried out was fortuitously to keep away from any kind of personal or subjective bias, which may be conscious or unconscious. This method ensures greater validity of test results [23-27]. For instance, in four parameters at three levels condition, the conventional full factorial design would need 3^4, or 81 experiments. But, in the present design (Taguchi L-9 orthogonal array), the necessary experiments are merely nine [28-30].

Consequently, an experimental design method was employed to examine the influences of these parameters on the removal quality of fouling lead layer and the results have been examined by Scanning electron microscopy (SEM), Energy dispersive x-ray microanalysis (EDAX) and Inductively coupled plasma spectroscopy (ICP) analysis techniques. Finally, on the basis of the ANOVA results, optimum condition for the lead cleaning of polluted surface has been proposed and the performance of investigated chemometric method has been compared with our pervious sequential experimental method.

Experimental

Materials and Apparatus: Aqueous hydrogen peroxide solution (H_2O_2) 35% (V/V) and acetic acid (CH_3COOH) 100% (V/V) were obtained from Fluka (Tehran, Iran). ASTM B 32 standard lead bar (with 99% purity) was prepared from local industrial Zanjan zinc-lead Co. (Tehran, Iran).

Scanning electron micrographs were recorded by using on a Philips XL30 series instrument and 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) [31-32]. Inductively coupled plasma spectroscopy (ICP) instrument model VISTA-PRO made by varian company (Australia) was employed for determination of lead ion concentration in waste solution.

Cleaning Procedure: The cleaning experiments were carried out using a electrochimical process which works based on the redox reaction principal [13]. In order to preparation of the cleaner solution, a known volume stock solution of CH_3COOH and H_2O_2 were placed in a volumetric flask and the flask was filled to the mark with deionized water. In a typical experiment (run 1 Table 2), in order to dissolving 3 g metallic lead bar a 14.3 ml of H_2O_2 (5% v/v with respect to total amount of solution) and a 10.0 ml of CH_3COOH (10% v/v with respect to total amount of solution) were placed in a 100.0 ml volumetric flask and the flask was filled to the mark with deionized water. After that, lead bar was inserted in the solution and the cleaner solution was stirred at the stirring speed of the mixture 25 rpm and temperature 15°C [13]. However, following a period of mixing and reducing rate of realizing gas bubbles that revealed the ending of the cleaning process, the experiment will finished and the total dissolving time will be recorded (72 min).

Optimization of Lead Dissolving Procedure: An experimental design method was employed to optimization experimental parameters for the dissolving procedure [16-17]. The variables including percent of oxidizing agent (H_2O_2), percent of chelating agent (CH_3COOH), stirring speed of the mixture (revolutions per minute, rpm) and temperature of the cleaner solution were changed as

Table 1: The various parameters selected and their respective levels in the present experimental design.

Process parameter	Symbol	Level 1	Level 2	Level 3
Oxidizing agent	O	5	7.5	10
Chelating agent	C	10	20	30
Stirring speed of the mixture (rpm)	R	25	50	75
Temperature (°C)	T	15	21	28

Table 2: Taguchi L-9 orthogonal array design and dissolving time of 3 g lead bar

	Process Parameters			Results	
Experiments	Oxidizing agent (%)	Chelating Agent (%)	Stirring speed of the mixture (rpm)	Temperature (°C)	Dissolving Time (minute)
1	5	10	25	15	72
2	5	20	50	21	66
3	5	30	75	28	41
4	7.5	10	50	28	31
5	7.5	20	75	15	39
6	7.5	30	25	21	34
7	10	10	75	21	19
8	10	20	25	28	14
9	10	30	50	15	38

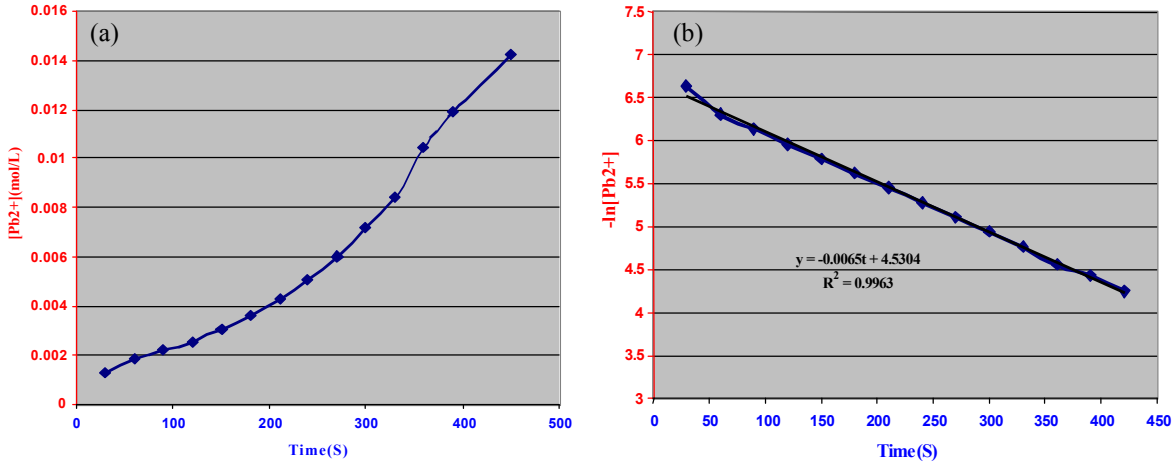


Fig. 1: The experimental result of oxidation of lead bar measured by ICP spectroscopy. (a) values of $[Pb^{2+}]$(mol/L) as a function of time, (b) values of -ln $[Pb^{2+}]$ as a function of time.

shown in Table 1 and 2. However optimum conditions which proposed by ANOVA method were employed for further lead bar oxidation studied.

Kinetic Measurements: In order to inspect the kinetic of the oxidation reaction of metallic lead bar with cleaner solution at optimum condition (proposed by ANOVA method), lead ion concentration as a function of time at aqueous solution was followed by ICP spectroscopy technique and the results were illustrated in Figure1.

Treatment of Real Sample: To estimate the usefulness of the proposed method for treatment of a polluted chromium surface a steel plate sample that electroplated with a specified thickness of chromium layer was used. Then, about 3 g of metallic lead as a pollution was deposited on the chromium surface in order to exploit as a real sample. However the dirty metallic surface was treated by cleaner solution at the ANOVA proposed optimum condition and the efficiency of the method was precisely evaluated.

RESULTS AND DISCUSSION

Dissolving Results and Optimization Strategy: The aim of this investigation was to verify how the different parameters influence on the removal of deposited lead layer from chromium surfaces by using cleaner solution. In this research the parameters be composed of percent of oxidizing agent (H_2O_2), percent of chelating agent (CH_3COOH), stirring speed of the mixture (revolutions per minute, rpm) and temperature of the cleaner solution. The tested factors and the levels are given in Table 1 and 2. Also, dissolving time of 3 g lead bar is presented in the last column of Table 2.

The average values of the coefficients of variation of the factors at each level were calculated according to assignment of the experiment (Table 3) [17-19]. For instance, to calculate the average value of dissolving time of 3 g lead bar for the effect of the percent of oxidizing agent (H_2O_2) at level 1, dissolving time of the nine trials in which percent of oxidizing agent (H_2O_2) was set at level 1 (triad 1, 2 and 3) 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.

The analyses of the data, assuming no interaction between variables, are containing: (1) identification of the optimum condition, (2) finding of the individual influence of each factor and (3) estimation of the result at the optimum condition [20-30]. The analysis of variance (ANOVA) for the results (dissolving time) is shown in Table 4.

In this inquiry, the influence of percent of oxidizing agent (H_2O_2) on the dissolving time at three diverse levels (5, 7.5 and 10% v/v) was studied. It was recognized that percent of oxidizing agent is an important factor for the control of the dissolving time of lead bar. Also, it was appear that temperature of the cleaner solution is an important parameter that could influence the dissolving time of lead bar. Conversely, our finding confirmed that percent of chelating agent (CH_3COOH) during the dissolving process is not a considerable parameter for the control of dissolving time of lead bar.

Finally, the influence of stirring speed of the mixture (revolutions per minute, rpm) on the dissolving time of lead bar was examined at three diverse values: 25, 50 and 75 rpm. The results illustrated that stirring speed of the mixture can be considered as a significant factor on the dissolving time of lead bar.

Evaluation of the ANOVA results of this investigation show that (at 90% confidence level),with omitting percent of chelating agent (CH_3COOH), all variables (percent of oxidizing agent (H_2O_2), stirring speed of the mixture (revolutions per minute, rpm) 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 10% v/v of oxidizing agent (H_2O_2), 75 rpm stirring speed of the mixture and 28°C temperature of the cleaner solution.

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

		Mean effect		
Process parameter	Symbol	Level 1	Level 2	Level 3
Oxidizing agent	O	59.7	34.7	23.7
Chelating agent	C	40.7	39.7	37.7
Stirring speed of the mixture (rpm)	R	40.0	45.0	33.0
Temperature (°C)	T	49.7	39.7	28.7

Table 4: ANOVA table for the dissolving of lead bar with redox procedure by $OA_9(3^4)$ matrix with the dissolving time of 3 g lead bar as the response

					DOF	S'	F'	P'
Factor	Code	DOF	S	V			Pooled[a]	
Oxidizing agent	O	2	2042	1021	2	2042	145.8	69
Chelating agent	C	2	14	7	-	-	-	-
Stirring speed of the mixture (rpm)	R	2	218	109	2	218	15.6	7
Temperature (°C)	T	2	662	331	2	662	47.3	22.1
Error	-	-	-	-	2	14	-	1.9

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

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 [17]:

$$Y_{opt}=\frac{T}{N}+(o-\frac{T}{N})+(r-\frac{T}{N})+(t-\frac{T}{N}) \quad (2)$$

where $\frac{T}{N}$ 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 (2), predicted dissolving time of lead bar at optimum condition is Y_{opt}. o, r and t are explained as dissolving time at optimum condition for amount of oxidizing agent, stirring speed of the mixture and temperature of cleaning solution, respectively.

Also, the formula for estimation of the confidence interval of the optimum dissolving time (CI) results from [18]:

$$CI = Y_{opt} \pm \sqrt{\frac{F_{\alpha}(f_1, f_2)V_e}{n_e}} \quad (3)$$

where F_{α} (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, α = 90%) and the confidence level is (1- α), 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 6.8±0.3 minutes.

In the next step of our inquiry, 3 g lead bar was treated at obtained optimum condition (10% of oxidizing agent, 75 rpm stirring speed of the mixture and 28°C temperature of the cleaner solution). The results of experiment revealed that dissolving time of 3 g lead bare at the predicted optimum condition is 8 minutes. The cleaning solution at this condition was employed for the kinetic studies of lead bar oxidation.

Kinetic of Lead Reaction: The reaction rate was studied to achieve more information from the mechanisms of the oxidation reaction. Our last investigations [13] illustrated that in the present method, successive reaction was occurred in the cleaner solution:

$$Pb^{2+}(aq) + 2e^{-} \rightleftharpoons \quad \textit{First stage} \quad (4)$$

$$2CH_3\,COOH(aq) \rightleftharpoons 2CH_3COO^{-}\,(aq) + 2H+\,(aq) \quad \textit{Second stage} \quad (5)$$

$$Pb^{2+}(aq) + 2CH_3COO^{-}(aq) \rightleftharpoons Pb(CH_3COO)_2(s) \quad \textit{Total reaction} \quad (6)$$

For the oxidation reaction of lead bar, equation (4) is the rate-determination step of the reaction. However, lead ions in aqueous solution will form lead acetate complexes with acetate ions (reaction 6). Consequently, the overall kinetic equation for the reaction rate could be resulting from the reaction rate of this equation (6). The reaction rate could be concluded by applying the next equation:

$$R = \frac{dx}{dt} = K(Pb^{2+})^{\alpha}(CH_3COO^{-} - x)^{\beta} \quad (7)$$

However, in this study oxidizing agent (H_2O_2) and chelating agent (CH_3COOH) were employed at high concentrations and it was presume that, their concentrations not change during the reaction. Therefore, the reaction rate (equation 7) can be simplified by using the Ostwald approximation method [33-35] and is shown as follow:

$$R = K(CH_3COO^{-})^{\beta}(Pb^{2+})^{\alpha} \quad (8)$$

If it is assumed that $K(CH_3COO^{-})^{\beta} \cong K'$, the reaction rate is defined as:

$$R = K'(Pb^{2+})^{\alpha} \quad (9)$$

The reaction rate is simplified to pseudo first-order. By using the assumption that the α(reaction order) value is equal to 1, the rate expression of reaction is simplified to:

$$-\ln(Pb^{2+}) = k't \quad (10)$$

In the pseudo first-order's approach, plot of –In(Pb^{2+}) against of time (t) is used to determine the reaction rate constant (k'), where (Pb^{2+}) is the lead ion concentration during the reaction time (t). A straight line through the data points was obtained by linear regression. Consequently the rate constant (k') was obtained from the slope and the validity of estimation (α= 1) was checked on the data of linear regression (r).

Table 5: The experimental values of -ln [Pb^{2+}] as a function of time, measured by ICP spectroscopy

No	Time (S)	[Pb^{2+}](mol/L)	-ln [Pb^{2+}]
1	30	0.0013	6.64
2	60	0.0018	6.30
3	90	0.0022	6.14
4	120	0.0026	5.96
5	150	0.0030	5.80
6	180	0.0036	5.60
7	210	0.0043	5.45
8	240	0.0050	5.30
9	270	0.0060	5.10
10	300	0.0072	4.90
11	330	0.0085	4.80
12	360	0.0105	4.60
13	390	0.0120	4.40
14	420	0.0140	4.30

However, Figure 1 and table 5 show the experimental values of (Pb^{2+}) and (Pb^{2+}) at different time of the reaction (6). The values of In(Pb^{2+}) were plotted versus the values of time (t). A straight line through the data point was achieved by linear regression. The current graph obeys the least- squares line:

$$Y = -0.0065t + 4.53 \quad (11)$$

The number of data points, n, was equal to 14, time, t, ranged from 30 to 420 seconds. The rate constant (k') was achieved from the slope of graph, 6.5×10^{-3} $(S)^{-1}$. From the linear regression, it was found that the value of r = 0.998 and hence, the assumption α= 1 is acceptable.

Lead Cleaning from Real Sample: As mentioned in the experimental section, to evaluate the efficacy of the suggested chemometric process for cleaning of a dirty surface a chromium plate with about 200 × 10 mm^2 dimension that is covered with a thin layer (nearly 3 g) of metallic lead was treated with the cleaner solution at the optimum conditions (10% of oxidizing agent (H_2O_2), 75 rpm stirring speed of the mixture and 28°C temperature). In order to lead cleaning, the dirty sample was treated according to the experimental procedure (section 2.2) and the chemical removal of lead fouling was initiated and after about 8 minutes, reducing rate of realizing gas bubbles revealed the ending of the cleaning process. Moreover, to avoid biological pollution [36], the lead acetate generated during the procedure was removed from the waste cleaner solution by a biological method [37].

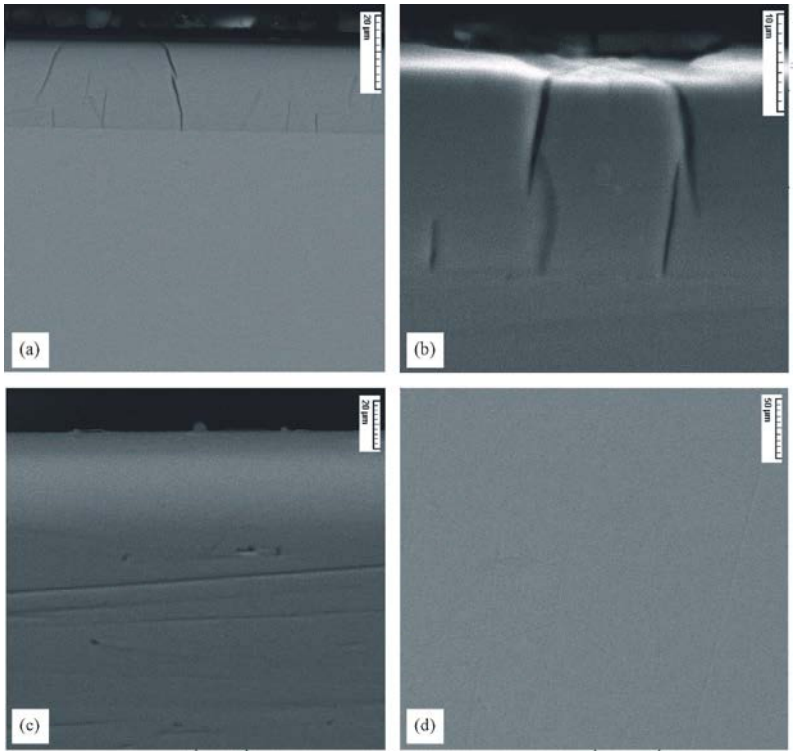
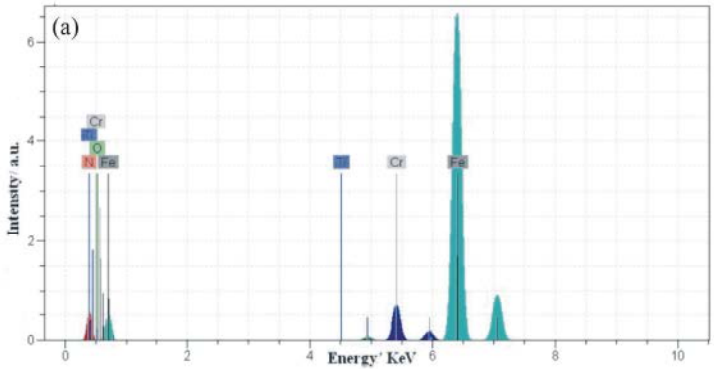
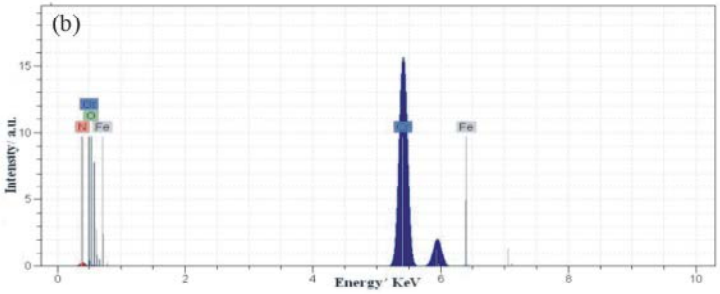


Fig. 2: SEM image of (a-b) cross-section of real sample (stainless-steel plate coated with a 20 µm layer of chromium fouling with about 3g of metallic lead) before treatment with cleaner solution, (c) cross-section of the sample after cleaning at optimum condition and (c) surface of the sample after cleaning at optimum condition.



Element	Weight Percent (%)
Titanium	0.1
Nitrogen	3.6
Chromium	4.5
Iron	91.2
Total	99.4



Element	Weight Percent (%)
Oxygen	0.03
Nitrogen	3.8
Chromium	95.5
Iron	0.3
Total	99.6

Fig. 3: EDAX spectrum of the real sample after cleaning at optimum condition, (a) cross-section analysis and (b) surface analysis

However, our primary evolutions has been shown that the procedure has proper efficiency for cleaning of a dirty surfaces, Nevertheless for more evaluation of the method all surfaces were examined by the SEM and EDAX analysis.

Results of SEM/EDAX Analysis: SEM photographs of different samples comprising, impure and cleaned chromium surfaces were illustrated in Figure 2. The photographic results demonstrated that on the dirty samples (Figure 2a-b) three layers containing lead fouling, chromium electroplated and stainless-steel body can be clearly observed. Also, (Figure 2c-d) presents that in the treated surfaces of samples, stainless-steel plate has only the chromium layer with out any corrosion and no lead fouling streak was observed on the surface of the plate. Also, according to the results of EDAX analysis (Figure 3) lead pollution was removed completely and the efficiency of the proposed chemometric method confirmed.

Comparison with Sequential Experimental Method: Recent chemometric cleaning procedure has been compared with our previous sequential experimental method for removal of fouling lead layer from chromium surfaces in terms of accuracy, rapidity, cost and performance. The present statistical cleaning method is clearly faster than its sequential partner. For example, The experimental results of our pervious sequential experimental method, showed that at the optimum condition (acetic acid concentration 28% (V/V), hydrogen peroxide 8% (V/V) and temperature 35°C), 15-minutes time is needed for complete removal of 3g fouling lead layer from chromium surface [13]. However, the statistical results showed that chromium surfaces can optimally be cleaned merely by controlling of hydrogen peroxide concentration, stirring speed of the mixture and temperature of the cleaner solution and percent of chelating agent (CH_3COOH) is not a significant parameter during the removal process. Also, the statistical results showed that under stirring speed of 75 rpm, the most cleaned sample was achieved by using cleaning solution with 10% (V/V) of hydrogen peroxide as oxidizing agent and temperature of 28°C within only 7 minutes. comparison of the results confirms that in the moderate experimental condition, lower percent of chelating agent (10 respect to 28%), lower cleaning solution temperature (28 respect to 35°C) and higher stirring speed of cleaner solution (75 respect to 50 rpm), the efficiency of the proposed statistical chemometric method is higher and the total removal time of fouling lead layer is considerably lower (7 minutes respect to 15 minutes) than our previous sequential experimental method.

CONCLUSION

In this study, we have investigated a simple, fast and controllable method for cleaning of deposited metallic lead layer as pollution from chromium surfaces with out any corrosion by using a redox reaction system. The variables were examined in order to increase the efficiency of the proposed method as potential industrial application. An OAD procedure was used for the optimization of dissolving reaction conditions and some experimental parameters, such as hydrogen peroxide concentration, stirring speed of the mixture and temperature of the cleaner solution were found to play significant role in the removal of lead fouling layer from chromium surfaces. The OAD evaluation of experimental data provides optimized amount of the parameters, H_2O_2 10% (V/V), stirring speed of 75 rpm and temperature of 28°C, to obtain the most cleaned sample, at which the total removal time of 3 g of the fouling lead layer is about 8 minutes. The experiments proved that by using this method, chromium surface could be completely cleaned at optimum conditions. SEM/EDAX photographs of completely cleaned samples showed that the treated surfaces were uniform and without any surface corrosion. Also by using ICP spectroscopy experimental data, the kinetic parameters of redox reactions of lead bar dissolving were obtained by Ostwald approximation method. The results showed that the rate constant (k') was obtained by least-squares line is equal to 6.5×10^{-3} $(S)^{-1}$, that confirmed the pseudo first-order reaction approximation. Finally; the results of this investigation confirmed that the simultaneous proposed chemometric method is considerably rapid, accurate and simple than our pervious sequential experimental methods.

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