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PURIFICATION OF Cr(VI)-CONTAINING WASTEWATER BY CHEMICAL PRECIPITATION: TEST RESULTS OF AN EXPERIMENTAL-INDUSTRIAL INSTALLATION

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We present the main results of experimental and industrial study of the developed process and pilot plant for purification of the Cr(VI)-containing rinse water by chemical precipitation. This technique implies full or partial return of treated water to rinse workpieces after chromium plating and prepare working solutions (precipitators). We characterize the manufacturing scheme of the pilot plant and the mode of its performance. Saturated aqueous solutions of $Ca(OH)_2$ and $Ba(OH)_2$ were used as precipitating reagents. The tests confirmed the possibility of automatic dosing the reagents with due regard for the pH value of a medium, which can be used in both continuous and periodic rinsing. The operating conditions of rinsing the workpieces after electrochemical chromium plating were justified. We derived the equation for the calculation of the total salt content in the rinse water after its purification. We determined the duration of the rinsing without the need to renew the bath volume. The performed calculations substantiated the possibility of reducing the consumption of fresh water supplied for rinsing by the recycling of wastewater without deteriorating the quality of the rinsed workpieces. The reduction of water consumption by 35% was observed as compared with that stated by the regulations.

Keywords: Cr(VI)-containing rinse water, pH of the medium, reagent, dosing, purification;,residual concentration, water consumption.

lIntroduction

Hexavalent chromium compounds are widely used in modern industry [1]. Many chemical and electrochemical processes imply the utilization of Cr(VI) compounds: chromium plating, chemical oxidation, etching and passivation of metals and alloys, etching of polymer products before metallization, etc. These processes are the source of the formation of a significant amount of Cr(VI)-containing wastewater: both spent electrolytes and rinsing water.

All Cr(VI) compounds are toxic and carcinogenic. According to WHO recommendations (Guidelines for drinking-water quality. 4th ed. World Health Organization, Geneva, 2011), the maximum permissible limit of Cr(VI) in drinking water is 0.05 mg/dm³. According to USEPA (National Primary Drinking Water Regulations. EPA 816-F-09-004. United States Environmental Protection Agency,

2009), the maximum limit of Cr(VI) in wastewater is 0.1−1.0 mg/dm³ for wastewater discharge into inland water. Discharge of Cr(VI)-containing wastewater into the sewer without any prior treatment is prohibited in Ukraine too.

For purification of aqueous solutions from Cr(VI) compounds at a low concentration, some methods based on various physicochemical processes [2] have been proposed: electrocoagulation [3], ion exchange [4], membrane separation [5], electrodialysis [6], adsorption [7] and chemical precipitation [8]. Each of these methods has its advantages and disadvantages. The results of the SWOT-analysis [9] allow characterizing these methods in the following way:

– Ion exchange, membrane and adsorption methods require the regeneration of expensive ion exchange resins, sorbents and membranes and, ultimately, their utilization after use, which creates

environmental, technical and economic problems. Therefore, these methods should be used only for fine water treatment.

− Electrocoagulation causes the formation of a large number of flooded sludges based on $Fe(OH)_3$ with different composition. Their proper aqueous suspensions have a low stratification rate and are difficult to filter. In addition, precipitates formed during electrocoagulation cannot be easily recycled. In general, this method is not suitable for the use in the creation of a closed water loop cycle in enterprises [10].

− Chemical precipitation methods often require the use of expensive reagents. Their end products are usually sludge and wastewater with high salt content [2]. In addition, the spread of chemical precipitation methods based on the formation of poorly soluble salts ($BaCrO_4$, $PbCrO_4$, etc.) is hindered by the complexity of the manufacturing schemes. This is especially important in the treatment of large volumes of wastewater after chromium plating, chromating and passivation with a variable concentration of CrO_3, which can vary from a few units to several hundred mg/dm^3 in a relatively short period of time [2]. However, these methods allow obtaining a sufficiently low residual concentration of CrO_3 in the purified water, which makes them the most attractive for industrial implementation.

The purpose of this work was to conduct industrial tests of the developed process and pilot plant for chemical precipitation of Cr(VI) in wastewater and optimize treatment regime.

Experimental

The proposed manufacturing scheme provides a full or partial return of the purified water to rinse the workpieces after the stage of chromium plating and to prepare working solutions (precipitators).

The pilot plant for utilization of chromium-containing wastewater was mounted with the maximum use of the available operating equipment. The design capacity of the chrome plating is 5.0 m^2/h, the actual productivity at the time of testing was 3.2 m^2/h. The manufacturing scheme is presented in Fig. 1.

Acidic Cr(VI)-containing wastewater from the installation of the electrochemical chrome bath goes to a collector 1. The wastewater is fed from the plating equipment by centrifugal pump 9. Before the centrifugal pump, lime milk is dosed to the sewage from container 8. Acidic Cr(VI)-containing wastewater is mixed with the alkalizing solution and neutralized. Then, it enters the ruff mixer 2. The solution of $Ba(OH)_2$ is dosed to the ruff mixer 2 from the container 3.

At the outlet of the ruff mixer and after the pump 9 (in the pipeline), a pH meter an an automatic control device are installed that automatically adjust the medium pH by dosing the reagent.

The resulting suspension from the ruff mixer 2 goes through a perforated (in the lower part) loading pipe to the central part of the reactor-decanter 4. The growth of crystals, aggregation of $BaCrO_4$ and $BaCO_4$ particles, and their sedimentation are completed in the reactors 4. The clarified water is drained into the collector of purified water 5 through the fitting and pumped (by the centrifugal pump) to use in the production, in the preparation of lime milk and barium hydroxide solutions, and in the rinsing of workpieces after electroplating.

The sediment, which is accumulated in the reactors-decanters 4, is periodically removed. Reactors (decanters) 4 operate alternately. On the
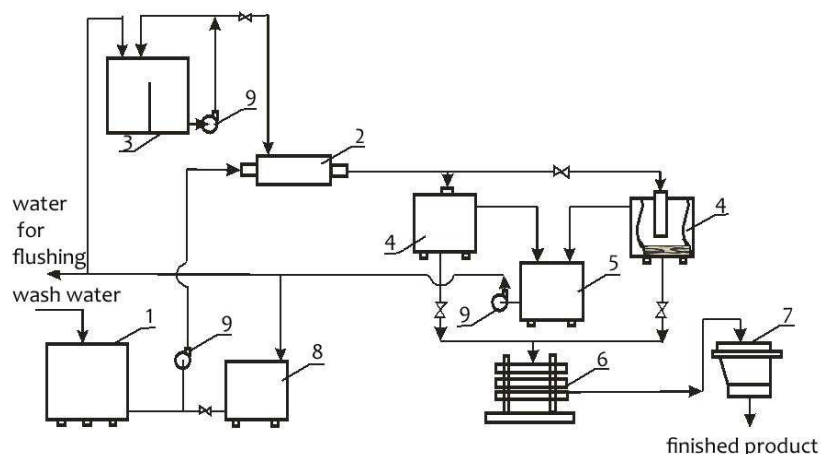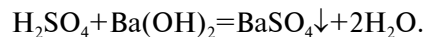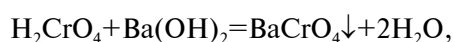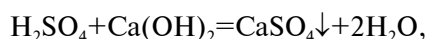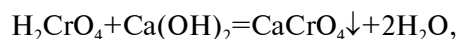

Fig. 1. Pilot plant manufacturing scheme of purification and neutralization of Cr(VI)-containing wastewater: 1 − collector of wastewater; 2 − ruff mixer; 3 − container for the preparation of $Ba(OH)_2$ solution; 4 − reactors (decanters); 5 − collector of purified water; 6 − filter-press; 7 − dryer; 8 − container for $Ca(OH)_2$ solution or aqueous extract; 9 − pump

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filter 6, the precipitate is dehydrated, partially dried, reloaded for final drying to the dryer 7 and unloaded into a container.

The test was performed in two stages. The wastewater from the rinsing of details after electrochemical chromium plating with the initial concentrations of CrO_3 and H_2SO_4 equal to 179.9 mg/dm^3 and 8.2 mg/dm^3, respectively, and pH of 4.9 was used in the first stage. The wastewater with the concentrations of CrO_3 and H_2SO_4 equal to 128.02 mg/dm^3 and 6.6 mg/dm^3, respectively, and pH of 5.2 was used in the second stage. A clarified part of the suspension of lime milk with a concentration of approximately 100 g CaO per dm^3 was used for alkalizing of the original Cr(VI)-containing water up to pH 10.1−10.3. An aqueous solution of $Ba(OH)_2$ with the mass concentration of 3.7% was used to achieve pH of 11.2 for the $BaCrO_4$ and $BaSO_4$ formation. The temperature of aqueous solutions and suspensions was 18−20^{0}C. These conditions of sedimentation were chosen based on the results of our previous laboratory studies [11].

To determine the content of the solutions components, some standard methods [12] were used. The temperature and pH of the medium were measured using a stationary pH meter (pH-101K with electrodes E528BNC) and a portable pH meter (pH/ORP-meter AZ-8601).

The main reactions that take place in the system are described by the following equations of neutralization and chemical precipitation:

$$H_2CrO_4+Ca(OH)_2=CaCrO_4\downarrow+2H_2O,$$

$$H_2SO_4+Ca(OH)_2=CaSO_4\downarrow+2H_2O,$$

$$H_2CrO_4+Ba(OH)_2=BaCrO_4\downarrow+2H_2O,$$

$$H_2SO_4+Ba(OH)_2=BaSO_4\downarrow+2H_2O.$$

Results and discussion

The main results of the conducted pilot tests are presented in Table. The actual amount of treated water was 7.6 m^3 and 17.3 m^3 in the first and second series, respectively. A salt thin film on the rinsed products after drying was not visually detected in both series of experiments. A maximum total salt content in purified water was ~395.2 mg/dm^3 and ~660.3 mg/dm^3 in the first and second series of experiments, respectively.

The results of the first series of experiments show that the content of CrO_3 in purified water is in the range of 0.43−0.47 mg/dm^3 and it increases to 0.48−0.51 mg/dm^3 (in the second series of experiments) [11].

For the first series of experiments, the total consumption of lime milk was ~0.032 m^3 and the specific consumption of lime milk was $\frac{0.032}{7.6}\approx 0.004$ m^3/m^3. The consumption of barite water was ≈ 0.05 m^3, which corresponds to the molar ratio $MC_1=\frac{n_{Ba(OH)_2}}{n_{H_2SO_4}+n_{H_2CrO_4}}\approx 1.1$. The specific consumption of barite water was $\frac{0.05}{7.6}\approx 0.007$ m^3/m^3. For the second series of experiments, the total consumption of lime milk was ≈ 0.07 m^3, the specific consumption of lime milk was $\frac{0.07}{17.3}\approx 0.004$ m^3/m^3, and the consumption of barite water was ≈ 0.052 m^3, which corresponds to the molar ratio $MC_2=\frac{n_{Ba(OH)_2}}{n_{H_2SO_4}+n_{H_2CrO_4}}\approx 1.06$. The specific consumption of barite water was $\frac{0.052}{17.3}\approx 0.003$ m^3/m^3.

The results of the pilot testing

Wastewater		The composition of purified water				Working time, hours	Water consumption, m^3/h	The composition of the sediment according to X-ray diffraction analysis, rel.%			
CrO_3 concentration, mg/dm^3	pH	Concentration, mg/dm^3			pH			$BaCrO_4$	$CaCrO_4$	$BaSO_4$	$CaSO_4$
		CrO_3	Ba^{2+}	Ca^{2+}							
179.92 (the first series of experiments)	4.9	0.46	32.2	60.13	10.9	5.0	0.6	82.1	8.8	9.1	Traces (<1%)
		0.43	33.3	62.12	11.0	1.5	1.9				
		0.47	29.3	64.13	11.1	0.5	2.1				
		0.44	31.1	68.11	11.2	0.5	1.8				
128.02 (the second series of experiments)	5.2	0.51	41.7	76.20	11.0	4.0	0.6	85.6	4.2	6.7	3.5
		0.48	48.2	88.30	10.9	10.0	1.2				
		0.50	49.3	95.20	11.0	0.5	2.8				
		0.51	51.5	108.3	11.2	0.5	2.7				

An increase in the volume of purified water due to the dosing the reagents can be neglected, since the total specific consumption of reagent solutions was ≈ 0.011 m^3/m^3 and 0.007 m^3/m^3 for the first and second series, respectively.

Our results showed that the Ca^{2+} content was in the range of 60.1−108.3 and the Ba^{2+} content was in the range of 29.3−51.5 mg/dm^3. The presence of these ions in purified water does not create any obstacles for its reuse in the rinsing of details after electrochemical chromium plating as well as for the preparation of lime milk and barium (II) hydroxide solution. The total salt content in the technical water received for the washing of the products was 210−240 mg/dm^3 (an average value was 225 mg/dm^3). After the first experiments series, the total salt content in the purified wash water was ~395.2 mg/dm^3 and it was accepted as the initial salt content for the second experiment series. The dependence of the total salt content in the purified wastewater on its consumption is shown in Fig. 2.
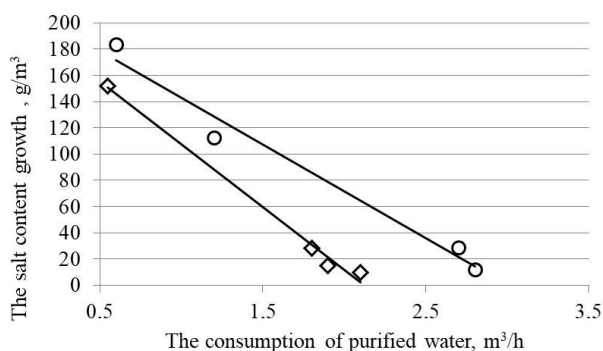

Fig. 2. Dependence of the salt content in purified water on its consumption: $\diamondsuit$ − the first series of experiments; $\bigcirc$ − the second series of experiments

The change in salt content was calculated by the formula:

$$M_t = \Delta C_t \cdot W, \qquad (1)$$

where M_t is the increase in the total salt content in purified water (g/h); ΔC_t is the difference between the total salt concentrations in the purified and initial wash water within the series (g/m^3); and W is the wash water consumption (m^3/h).

The dependence presented in Fig. 2 is satisfactorily described for the first series of experiments by the following linear equation (R^2=0.9):

$$\Delta C_t = 256.9 - 85.4W, \qquad (2)$$

where W_i is the initial consumption of wash water.

According to the selected purification scheme, the general equation of material balance for the process of wash water purification from Cr(VI) compounds can be written in the following form:

$$M_0 + M_r + M_x = M_1, \qquad (3)$$

where M_0, M_r, M_x and M_1 are the total mass of salts that enter the system with the initial technical water, the mass of recirculating water, the mass of salts that were formed by chemical reactions and remain in solution, and the mass of salts that leave the system, respectively.

In equation (3), the mass of salts can be replaced by the product of water consumption (m^3/h) and the total concentration of water-soluble salts (mg/dm^3): $M_j = W_j C_j$. Provided that the already purified water is subject to partial or complete recycling and the change in its quantity will proportionally lead to the change in the amount of fresh water, the equation of material balance will be written as follows:

$$(1-n)W_0 C_0 + n W_0 C_1 + W_x C_x = W_1 C_1, \qquad (4)$$

where W_0, W_x and W_1 are the consumption of fresh water for rinsing of products, the consumption of water with reagents for cleaning and the consumption of water that leaves the system, respectively (m^3/h); C_0, C_x and C_1 are the total salt concentration in fresh water, the increase in the total salt content after purification and clarification of water, and the total salt concentrations in purified water which left the system and recirculated in the system, respectively (mg/dm^3); and n is the proportion of water retained in circulation.

The value of n is calculated by the following equation:

$$n = \frac{W_p}{W_0 + W_p}. \qquad (5)$$

For the reuse of purified water, the numerical value of C_1 is important, it expresses the total salts concentration in the purified water that left the system. The value of C_1 affects the quality of the rinsing of workpieces. There is no standard method for assessing the quality of washing. Therefore, the value of C_1=800 mg/dm^3 was established in a specially conducted experiment; if this value is exceeded, then the presence of salt thin film can be seen on the surface of washed and dried details. We accepted this value as the maximum possible salt content in purified water.

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According to graph in Fig. 2 and the equation (2) for the first series of the experiment, an increase in the total salt content after purification and clarification of water, depending on the amount of water supplied for washing details, can be expressed in the following way:

$$C_x = \Delta C_t = q - bW_0 \cdot (1+a), \qquad (6)$$

where a is the amount of solution that enters the system with reagents for water purification with respect to the amount of fresh wash water; it is defined as:

$$a = \frac{W_x}{W_0 + W_x}. \qquad (7)$$

In equation (7), parameter a is variable and depends mainly on both the concentration of CrO_4^{2-} and SO_4^{2-} ions that come with the products for washing and the residual concentration of Ca^{2+} and Ba^{2+} ions in water after purification. Parameter a was determined by experimental measurements. The value of parameter a was $a_1 = 0.013$ and $a_2 = 0.007$ for the first and second series of the experiments.

Substitution of expressions (5)−(7) into material balance (Eq. (4)) and its further transformation, allow us to write the equation for calculating the final concentration of the total salt content in the wash water after its purification as follows:

$$C_к = \frac{C_0(1-n) + (1+a)\left(nC_0 + q - bW_0 \dfrac{1}{1-a}\right)}{1-n(1-a)}. \qquad (8)$$

Analysis of Eq. (8) shows that under the condition $W_0 = 0$ and $n = 1$, the flushing occurs only in the water that is in the flushing bath with a working volume $V = 1.5$ m³, without updating it to achieve a total salt concentration of not more than 800 mg/dm³, the «buffer capacity» of the system being 468.6 mg/dm³. Based on this, the time during which it is possible to wash the products without updating the volume of the bath is equal:

$$\tau = \frac{b}{\Delta C - C_н} V_в = \frac{95.6}{800 - 468.6 - 225} 1.5 \approx 1.4 \text{ hours.}$$

The consumption of fresh wash water, without its partial return to washing, must be at least 2.2 m³/h to avoid an increase in the concentration of salts. Using Eq. (8), we calculated a possible amount of purified water that can be returned to the washing of products (the case when $0.5 \leq W_0 \leq 3$ and $0.2 \leq n \leq 0.8$) (Fig. 3).
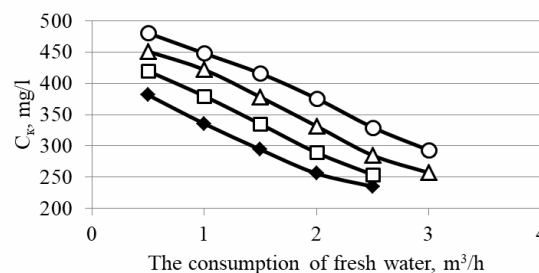

Fig. 3. Dependence of the total salt content in the wash water on the consumption of fresh water (W_0) and the fraction of purified water (n) that is returned for flushing: ◆ − n=0.2; □ − n=0.4; △ − n=0.6; ○ − n=0.8

Taking into account the obtained data, we proposed the following washing conditions: the fresh water consumption of 0.6−0.8 m³/h and the purified water consumption in recycling of 0.2−0.4 m³/h at the total consumption of not more than 1.6 m³/h (corresponding to n=0.3−0.5). In this particular case, the minimum water consumption is limited to 0.8 m³/h, which is due to the prevention of the formation of salt deposits on the washed products. In adition, the maximum total water consumption is 1.6 m³/h, which is due to the need to ensure the stratification of the created suspension in the settling reactor for complete using its clarified part.

The results of the second series of experiments with partial recycling of purified water are presented in Table. At the total consumption of water for washing of 2.7−2.8 m³/h (at n~0.45), there is an insufficient stratification of the suspension. Therefore, washing of products after electrochemical chromium plating was carried out at an average total water consumption of 1.3 m³/h, including a return to washing of 0.4−0.5 m³/h purified water, which corresponds to n=0.3−0.4. The results of tests of a pilot plant showed that the consumption of fresh water for washing products was reduced by 35%.

Conclusions

The results of the tests of a pilot plant showed the effectiveness of the chemical precipitation treatment of Cr(VI)-containing wastewater in the concentration range of 128−180 mg/dm³ with preliminary neutralization by dosing a saturated solution of calcium hydroxide up to pH 10.1−10.3 and subsequent dosage of a saturated aqueous solution of barium hydroxide as a reagent-precipitant until reaching pH 10.9−11.2. Under these conditions, the residual concentration of Cr(VI) compounds in the

clarified parts of the suspensions does not exceed 0.5 mg/dm^3. Continuous water treatment can be realized with the dosage of reagents to adjust the required pH value of the medium. We confirmed the possibility of a 35% reduction of the consumption of fresh water for washing products after the electrochemical chromium plating process due to its recycling.

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РЕАГЕНТНЕ ОЧИЩЕННЯ Cr(VI)-ВМІСНИХ ПРОМИВНИХ ВОД: РЕЗУЛЬТАТИ ВИПРОБУВАНЬ ДОСЛІДНО-ПРОМИСЛОВОЇ УСТАНОВКИ

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В роботі наведені основні результати дослідно-промислових випробувань розробленої технології та установки для реагентного очищення Cr(VI)-вмісних стічних вод, що передбачає повне або часткове повернення очищеної води на промивання виробів після стадії хромування та на приготування робочих розчинів − осаджувачів. Охарактеризована технологічна схема установки та режим її роботи. Як реагенти-осаджувачі використовувались насичені водні розчини Ca(OH)$_2$ та Ba(OH)$_2$. Підтверджена можливість автоматичного дозування реагентів в залежності від pH середовища, що може бути використано, як для безперервного, так і періодичного промивання. Запропоновано режим промивання виробів після електрохімічного хромування. Отримано рівняння для розрахунку загального солевмісту в промивній воді після її очищення. Визначено тривалість часу, протягом якого можливе промивання виробів без оновлення об'єму ванни. Розрахунками, виконаними за результатами випробувань, обґрунтована можливість скорочення витрат свіжої води, що подається на промивання, шляхом оборотного використання очищеної води без погіршення результатів промивки виробів. Практично підтверджено скорочення витрат води на промивання на 35% від регламентованих.

Ключові слова: Cr(VI)-вмісні промивні води, pH середовища, реагенти, дозування, очищення, залишкова концентрація, витрати води.

PURIFICATION OF Cr(VI)-CONTAINING WASTEWATER BY CHEMICAL PRECIPITATION: TEST RESULTS OF AN EXPERIMENTAL-INDUSTRIAL INSTALLATION

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We present the main results of experimental and industrial study of the developed process and pilot plant for purification of the Cr(VI)-containing rinse water by chemical precipitation. This technique implies full or partial return of treated water to rinse workpieces after chromium plating and prepare working solutions (precipitators). We characterize the manufacturing scheme of the pilot plant and the mode of its performance. Saturated aqueous solutions of $Ca(OH)_2$ and $Ba(OH)_2$ were used as precipitating reagents. The tests confirmed the possibility of automatic dosing the reagents with due regard for the pH value of a medium, which can be used in both continuous and periodic rinsing. The operating conditions of rinsing the workpieces after electrochemical chromium plating were justified. We derived the equation for the calculation of the total salt content in the rinse water after its purification. We determined the duration of the rinsing without the need to renew the bath volume. The performed calculations substantiated the possibility of reducing the consumption of fresh water supplied for rinsing by the recycling of wastewater without deteriorating the quality of the rinsed workpieces. The reduction of water consumption by 35% was observed as compared with that stated by the regulations.

Keywords: Cr(VI)-containing rinse water; pH of the medium; reagent; dosing; purification; residual concentration; water consumption.

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