COMBINATION OF ELECTROCOAGULATION AND FLOTATION TECHNOLOGIES IN APPARATUS FOR TREATMENT OF ELECTROPLATING WASTEWATER

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ABSTRACT

Despite global problems that can affect the world economy (COVID-19, military conflicts, migration, etc.), the world's manufacturing capacity continues to grow. Inefficient water usage production schemes, an unacceptable state of water treatment facilities, outdated material-hungry wastewater processing methods lead to the worsening of Ukraine's ecological situation. Electroplating manufacturing is among the biggest industrial water consumers. One of the most dangerous contaminant is chromium electroplating line. Hexavalent chromium compounds are classified as Category 1 health hazard. Among numerous methods for treatment of chromium-containing electroplating waters the electrochemical method was chosen as the most appropriate. A fundamentally new technological scheme for the treatment of wastewater of chromium electroplating manufacturing line, with the underlying principle of creating closed-circuit resource circulation was developed. The design of the electrocoagulator was improved by equipping with special floatation device that helps to prevent the formation of channels and cavities in the near anode. The ectrofloatocoagulator, which combines galvanic and electrochemical dissolution of anode and floatation was tested. The generalized schematic of the advanced electrochemical process was showed. The proposed scheme includes electrocoagulation and flotation technologies and allows for water treatment down to regulated values with minimal power consumption, while output mud sludge with stable structural characteristics. Laboratory analysis of obtained precipitate samples, allowed to conclude that based on chemical inertness of layered double hide oxides, they can be used as landfill material, secondary raw materials: pellets in metallurgy, ceramic tiles, bricks, etc.

Keywords: Water Usage, treatment, electrocoagulator

INTRODUCTION

According to the Global industry analytics [1] over the past year alone, the electroplating industry increased its manufacturing capacity by 4%. And by estimations, the electroplating market is going to reach 17.4 bil. USD by 2024. Today, eco-friendliness is one of 5 key trends in the field.

Regrettably, due to the state of wastewater treatment, Ukraine occupies 58th places out 100 in the rating of future-ready countries, according to the international economic forum [2]. While industrially developed regions of the country suffer from lack of clean water, the electroplating industry consumes substantial amounts of water, - over 50 mils. m³ per year [3]. In electroplating facilities, water is used for making electrolytes, degreasing and etching solution, washing parts, cooling baths, and current rectifiers. It is calculated that, in electroplating 30-80% of metals, only 5-20% of acid and 2-3% is utilized effectively, the goes into wastewater which becomes the main source of environmental contamination [4].

One of the most dangerous wastewaters is those from chromium electroplating lines. Transforming activity of hexavalent chromium results in its high toxicity and significant migration potential in the environment. Toxicity of Cr^{6+} in living organisms is expressed by growth retardation, slow metabolism, genetic, gonadotrophic, embryotrophic changes, chromium compounds are classified as carcinogenic [5].

Solving the contamination problem of Ukraine's surface waters requires the rationalization of industrial water usage schemes, disposal, and treatment of industrial wastewaters. One of the priority tasks is the introduction of modern purification technologies that would allow for lowering contaminant concentrations to safe levels.

When choosing the technological solution of wastewater treatment is also important to consider conditions for the processing of solid waste, because, in addition to direct dumping of processed wastewaters into surface waters, there is also the problem of groundwater contamination. The mud storage site is especially dangerous, as storage contaminants can seep into groundwater layers.

Among numerous methods for treatment of chromium-containing electroplating waters - reagent, ion-exchange, sorption, membrane, biochemical, electrochemical, - the reagent method is the most common among domestic manufacturers. Hexavalent chromium is commonly reduced using iron sulfide, calcium sulfite, hydrogen peroxide, formaldehyde, naphtha sludge, powder aluminum, hydrazine, barium salts, lead compounds, metalworking sludge, etc. The reduction of one weight part of Cr^{6+} requires up to 16 parts of the reducing agent [6]. The reagent method results in the formation of large



amount sludge (10.0 - 12.5 kt/year), which contains large amounts of hydroxides, carbonates, and salts of heavy metals [7].

Membrane wastewater treatment methods include reverse osmosis and ultrafiltration. For removal of hexaand trivalent chromium it is recommended to employ reverse osmosis membrane, ultra, and nanofiltration, namely, Kocurek P. et al. [8] propose pressure-driven membrane separation processes, in particular, membrane for reverse osmosis typed RO98pHt. Kiril M. et al. [9] used membrane for reverse osmosis typed RO (ACM2), ultrafiltration [UF1 (20 kDa), UF2 (50 kDa), UF3 (150 kDa)] and nanofiltration NF (XN45). Keerthi V. et al. [10] propose using a hybrid membrane bioreactor (HMBR). In our opinion, membrane method can provide a rather high degree of purification, however, low chemical stability in aggressive media and high cost, limit use of membranes to non-aggressive media with the concentration of metal ions isn't much above GDK. Thus membrane technology is yet to find widespread use for local treatment of chromiumcontaining wastewater.

Sorption methods for treating electroplating wastewater include adsorption and ion-exchange. Sorbents for chrome removal include synthetic and natural materials: vesicular basalt [11], carbon slurry [12], chemical modification of silica gel [13], sulfonated peat [14], etc. Authors [15] propose using if bituminous, subbituminous, and brown coal as the absorbent load for the treatment of chromium-containing wastewater. Work is also conducted on chromium neutralization using a homogeneous mixture of volatile tar and wollastonite. It was also possible to absurd chromium with sawdust. Chromium removal method using hematite, powder titanium dioxide, chitosan sorbents are interesting. Lucerne, clover, bean seed are used as natural vegetable chromium sorbent. Industrial use of sorption methods is limited due to difficulties with sorbent regeneration and reuse.

A widespread method for removal and recovery of chromium (VI) is ion-exchange using different types of ambulates, IRA 743, IR-120, etc., which have high chemical stability and can be easily regenerated [16, 17]. But, the use of such amberlites requires preliminary removal of mechanical admixtures, oils, fats from water. However, the use of the ion-exchange method results in the issue of processing eluates and additional treatment of washing waters, due to ion-exchange leads to salt dump being higher by 2,5 - 3, in comparison to reagent method.

Lately, in Ukraine and outside, there has been an increase in research on the development of the biotechnological method for the removal of heavy metals from wastewaters, by using aerobic and anaerobic bacteria and iron and sulfate-reducing bacteria [18]. Dagang S. et al. [19] Removal of chromium by using wheat straw and eupatorium adenophorum. Tian Ai *et al.* used Phyllostachys pubescens biomass loaded Cu-S nanospheres [20] in their study. The disadvantage of the biological treatment method is the sensitivity of biomass to the composition of wastewater.

The removal of chromium (VI) can also be realized using the electrochemical method, which is based electrolysis of aqueous electrolytes. on the Electrochemical methods have a number of advantages Physico-chemical ones. Namely, thev are over ecologically clean i.e. they avoid inclusion of secondary contaminants such as anionic and cationic remains, characteristic for reagent methods.

Electrocoagulation is considered the most accessible method to be introduced for the treatment of chromium-containing wastewater. For over century long development history and practical application, it went through a series of equipment and technological changes, demonstrating high effectiveness and reliability in treating chromium-containing wastewater.

In studying the electrocoagulation process, significant work has been done by American scientists F. Stewart and S. Bonilla, Japanese scientists Lu and Chen, Soviet scientist S.V. Yakovliev, I.G. Krasnoborodko, M.M. Nazarian, P.P. Strokach, L.A. Kulskyi. Scientific developments have allowed to significantly simplify control over electrocoagulation equipment and decrease its size while maintaining high purification effectiveness [21]. Electrocoagulation can be used to remove mixtures of metal cations, or individual metal cations individually (such as nickel [21]). The soluble anode is most commonly used in electrocoagulation. In this case, electrocoagulation allows for reductive destruction of hexavalent chromium and its precipitation in for of hydroxides. Precipitated mud has a stable structure and low solubility, which prevents chromium from leaching into the environment during prolonged storage of such mud on the territory of the facility or use as secondary material for construction or metallurgical industry. Among major reason that limits the introduction of electrochemical wastewater treatment technologies; most notable are technological complexity and relatively high cost.

As such relevant direction for scientific study is the development of electrocoagulation design for electroplating wastewater treatment, which allows for the creation of low-waste closed-loops water circuits on production sites, minimize the creation of additional contamination sources and risk of disturbing ecological balance, be ecologically sage and economically viable.

MATERIALS AND DEVICES

Authors [23] have developed small electrocoagulation assembly, with the phase contact surface, that provides necessary degree of wastewater purification. Electrocoagulator consists of cylindrical, made of dielectric - polypropylene. Cathode consists of two steel plates (St.3). Plates are bent to vessel shape and fixed in electrocoagulation with two steel fittings, and a ring located outside of the vessel. Anode made form perforated titanium mesh with 2.5 mm and free crosssection 60%, which is coated ruthenium dioxide, filled with anodically-soluble material. Mesh material was chosen due to special properties titanium - weak adhesion to it of metal and non-metal deposit, and stability in the



majority of media. Titanium mesh is fixed with fittings in the central part of the cylindrical electrolyzer. Electrocoagulation was designed in a way at the formation of electrocoagulant from anodically-soluble powder would occur in electrolyzer's volume, which omits the need for a separate electrolytic bath. The current collector is composed of rods and clamps connected to electrodes with bolts. Voltage and current in electrolyzer were measured using universal tools: ammeter AP 111B (AP 171) and voltammeter V 7-30. Coulometric measurements were taken using impulse potentiostat PI-50-1.

The main disadvantage of proposed technology was the quick buildup of slime in inter-electrode space,

resulting in over current at the anode, which is accompanied by passivation of titanium anode and uneven current distribution. This also leads to the slowed-down formation of hydroxo complexes along the central axis of the anodic chamber [23].

We have set the goal to lower electrode resistance, namely, by reducing the passivation of titanium mesh.

Studies regarding the improvement of parameters and the operating regime of water treatment equipment were conducted using laboratory setup shown in Figure-1.



Figure-1. Schematic of the laboratory setup for studying electrocoagulation: 1 - a vessel for making chemical mixtures; 2 - pH - meter N_{2} ; 3 - dosing pump; 4 - wastewater inlet; 5 - electrocoagulator; 6 - wastewater outlet; 7 - power supply; 8 - rheostat; 9 - ammeter; 10 - voltammeter; 11 - potentiostat; 12 - compressed air inlet; 13 - air compressor; 14 - pressure gauge; 15 - precipitate outlet; 16 - sludge collector; 17 - pH - meter N_{2} ; 18 - pH correction reactor; 19 - graduated vessel; 20 - settling tank.



The previously prepared working solution from the vessel for making chemical mixtures (1) (with set initial chromium (VI) concentration, mineralization degree, and initial pH, which was measured with pH-meter (2)) was pumped with acid-resistant laboratory dosing pump (3), with the closed outlet (4) into electrocoagulation (5). After the level of the treated solution has risen above inlet (6), the outlet (4) was open, and power supply (7) was turned on. The resistance of the electrical circuit was controlled with a rheostat (8).

The preliminary calculated current was corrected with help of ammeter (9). Simultaneously with current supply, voltammeter (10) was turned on, in order to measure the total voltage on electrode mesh and current loads in the anodic region of electrocoagulator (5). The electrode potential of the anodically-soluble load was measured using impulse potentiostat (11).

Wastewater flow was calculated based on the time required for the complete coagulation of metals. Wastewater inlet (6) was situated in such a way, that water would fall directly into the perforated mesh of electrocoagulator (5), with part of the flow encountering resistance from anode load, receiving horizontal movement direction. The horizontal part of the flow was fooling into between electrodes, and partaking in electrocoagulation. Vertical flow is additionally felted through metal shavings of electrocoagulator (5).

Additional series of experiments were conducted employing pressure flotation. Using compressor (13), regulating pressure with a pressure gauge (14) through distributions nozzles, compressed air was fed into electrocoagulator (5). Thus, aside from chemical and electrochemical reactions, there were also diffusion and migration flows of all components, due to flow filtration through loaded shavings and agitation with compressed air, occurring inside the reactor. Wastewater saturated with iron hydroxide was directed through outlet (4) into settling tank (20). Part of the solution was taken for analysis. precipitated Excess of was removed from electrocoagulator (5) through outlet (15) and directed into sludge collector (16).

The pH of the treated solution in settling tank (20) was measured using pH-meter (17). On the next stage, the solution entered into the pH-correction reactor (18), and after acid-base treatment, a sample was taken using a graduated vessel (19). After that samples were studied to evaluate precipitation rate. At certain interval during 24h, chromium content was measured in the taken samples.

During the treatment process, the operation of all apparatuses was controlled and corrected. The duration of each experiment was set to 60 min, which was sufficient to generate a portion of a solution required for analysis while allowing disregarding the influence of non-stationary effects that occurs during launch and prior achieving stationary operation regime. After a sample of the treated solution was taken, technological regimes and process parameters were adjusted according to previously developed procure of experimental conduct.

Conducted experiments resulted in data on completeness of chromium reduction to trivalent state, removal of trivalent chromium. pH increase, anodic current yield, the power consumed per 1 m3 or processed waster, the chemical composition of the precipitate. The concentration of chromium (VI) and (III) was measured photocolorimeter PEC-M and atomic -absorption S-600 spectrophotometer according standard to procedures. pH was measured using pH-meter pH-261. Chemical composition of precipitate was studied using atomic emission spectroscopy, using «DFS-8», and chemical dissolution method. The microstructure of precipitates was studied using the petrographic method and raster electron microscope «REM-100 U».

EXPERIMENTAL PART

Throughout the laboratory study aimed to address the flaws of the prototype [22], we have improved the design of the electrocoagulator. Namely, to prevent the formation of channels and cavities in the near anode, the electrocoagulator was equipped with a pressure floatation device. Air nozzles were installed into the lower part of the apparatus, and were used to saturate wastewater with air and mix savings. Air was pumped using a special compressor at 0.6 atm. This allowed for better utilization of coagulation and flotation properties of electrogenerated coagulant and increase its sorption activity. Apparatus also did not require an additional saturation chamber, water saturated with air directly inside of the electrocoagulator. Treated wastewater was reticulated simultaneously. Flotation allowed to avoid the increase of mesh holes and possible increase of voltage. Surfacing sludge passed through the side hole of the mesh, and sunk to the bottom of the sludge collector.

Figure-2 shows the schematic of electrofloatocoagulator, which combines galvanic and electrochemical dissolution of anode and floatation.



Figure-2. Electrofloatcoagulator: a) cross-section view; b) top view; 1- frame; 2 - anode mesh; 3 - cathode; 4 - shavings; 5 - wastewater inlet; 6 - wastewater outlet; 7 - sludge collector; 8 - cathode clamp; 9 - currently leads 10,11- fitting for fixating anode and cathode; 12- sludge outlet, 13 - compressed air inlet; 14 - compressed air nozzles.

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The proposed construction bulk electrode with metals shavings load is characters by the developed surface of phase contact, which intensifies the treatment process. Due to the supply of rarefied air from bottom to top, the turbulent flow of electrolytes formed, which decreased the thickness of the diffusion layer, increasing the mass-transfer coefficient. A generalized schematic of the process is as follows (Figure-3).

In the first stage of electrocoagulation, the dissolution of anode occurs due to the flow of current in closed circuit. Anode dissolution is determined by supplied current and chemical dissolution, mainly due to short-circuited galvanic iron-titanium pair. The current yield of metal removal at minimum current loads $(1.5 - 3.0 \text{ A/m}^2)$, is 120 - 130 % of the theoretic value. Key factors

that affect anode dissolution are anode material, liquid flow, anodic current density, cell voltage, and pH. To depassivate anode, the solution of NaCl (250 - 300 mg/L) was used. The positive effect of anionic admixtures significantly intensifies anode dissolution. The supply of rarefied air into anodic mesh allows have a de-passivating effect and prevents precipitated buildup in mesh holes.

When the iron anode is used for electrocoagulation, divalent iron cations are released into solution. After the accumulation of a sufficient amount of Fe^{+2} cations, they partially react with hexavalent chromium, reducing it to the trivalent state. This also decreases the stability of negatively charged colloids, because diffusion layer thickness decreases and so zeta-potential that prevents particle agglomeration.



Figure-3. Schematic of the electroflotocoagulation process.

Formation of gaseous hydrogen at cathode (Equation 1), which also plays the role of flotation gas, basification of solution occurs.

$$2H_2O + 2e \rightarrow H_2 \uparrow + 2OH^- \tag{1}$$

When solution pH reaches that of metal hydroxide formation (5 -7.2 from experimental data), the formation of metal hydroxide occurs. At this stage, reduction of hexavalent chromium occurs predominantly due to reaction iron hydroxide - a product of the reaction between divalent iron and OH⁻, which result into formation of hydroxides - $Cr(OH)_3$, $Fe(OH)_3$ (Equation 2).

$$3Fe(OH)_2 + CrO_4^{2-} + 4H_2O \rightarrow 3Fe(OH)_3 + Cr(OH)_3 \downarrow + 2OH^-$$
(2)

 $Fe(OH)_3$ and $Cr(OH)_3$, precipitated out and absorb chromium oxyanions ($Cr_2O_7^{2-} a CrO_4^{2-}$), which are present in small amounts due to incomplete reduction of Cr^{6+} .

The second stage of coagulation boils down to the chemical formation of insoluble metal hydroxides, their crystallization and formation flakes able to sediment large fractions of solids. The obtained flakes are good collectors for precipitating chromium. Based on experimental data, the amount of dissolved anode is about 7-8 moles Fe per 1 mole Cr(IV), which is 2.3-2.6 times higher than what is required for the reduction of hexavalent chromium. In this case, the formed precipitate isn't just hydroxide mixture but is layered double hydroxide (LDH). LDH consists of a-form of "host" metal hydroxide, with part of its cation substituted with "guest" metal ions. For instance, Ni²⁺ cations can be substituted with Al³⁺. This results in the formation of excess change within the crystal lattice, which can be compensated



through the inclusion of additional anions. A general formula for LDH based on divalent metal cations $isMe1_xMe2_{1-x}^{n+}(OH)_2A_{\frac{n-2}{m}}^{m-} \cdot 0.66H_2O$, where $Me1=Ca^{2+}$, Mg^{2+} , Ni^{2+} [25], Zn^{2+} and etc., $Me2=Al^{3+}$, Fe^{3+} , Cr^{3+} , Ti^{4+} [26] etc. A - various anions. Generally, anions are those of salt precursors. In case of electrocoagulation Fe(+2)-Fe(+3) and Fe(+2)-Cr(3+) LDH are formed. IT should be mentioned, that solubility of LDH is significantly lower than that of "host' and "guest" individually. As such, LDH is almost not subjected to leaching. The respective processes occur in inter-electrode space and include one, or sometimes more stages.

Flake formation in electrocoagulator if more intense in comparison to flake formation in the chamber for reagent coagulation, which is explained by the positive effect of electrophoresis on agglomeration and hydroxide particle and mixing with air bubbles. Improved purification for electrocoagulation indicates in addition to the formation of hydroxides and their precipitation, a significant role is played by the sorption capability of electrochemically generated hydroxide, which differs in structure from that formed in reagent treatment. In the case of electroprecipitation, formed hydroxide particles are smaller than those precipitated by reagents. It should be noted that LDH also has ion-exchange capabilities and is widely used for wastewater treatment [27]. In the case of electrocoagulation formed LDH, they are capable of exchanging their anions for chromate ions. Thus the formation of LDH allows for better removal of hexavalent chromium for wastewater.

Conducted experiments resulted in data on the reduction of hexavalent chromium to trivalent form, degree of trivalent chromium removal, pH increase, anodic current yield, power consumption per 1 m^3 of wastewater (Table-1), the chemical composition of precipitates (Table-2).

 Table-1. Results comparing laboratory-industrial experiments of electrocoagulator and electrofloatocoagulator (EC- electrocoagulator, EFC - electrofloatocoagulator).

Main technological parameters				Concentration after treatment, mg/L				pH (ofter		Power	
Wastewater flow, m ³ /s		Current density, A/m ²		Cr ³⁺		Cr ⁶⁺		treatment)		kW·h/m ³	
EC	EFC	EC	EFC	EC	EFC	EC	EFC	EC	EFC	EC	EFC
$1.85 \cdot 10^{-4}$	$2.22 \cdot 10^{-4}$	5.4	5.4	0.04	0.03	-	-	8.3	8.5	2.6	1.86

Laboratory analysis of obtained precipitate samples (Table-2) revealed that precipitates had stable structural characteristics. An assumption was made that chromium ions are strongly incorporated into the crystal structure of iron hydroxide due to the formation of ferrites. Unlike hydroxides of heavy metals, their ferrites at room temperature are insoluble not only in water but also in dilute strong mineral acids, caustic alkalis which due to special (spinel) structure [7]. However, chemical composition and formation conditions indicate the formation of layered double or even triple hydroxides, solubility product of which can be as low as 10^{-90} .

Table-2. Chemical compositions of precipitate samples.

Fe, %	Cr,%	Na,%	Ca,%	Ni,%	Cu,%	Al,%	Zn,%
79.23-85.53	10.13-16.30	1.86-3.61	0.91-2.32	0.015-0.10	<0.035	<0.04	<0.025

It should be noted that the realization of the proposed setup for wastewater treatment has certain disadvantages. The closed-loop water system is assumed for facilities with water usage higher than 50 m³/day. At wastewater flow, less than 50 m³/day, recycling of treated water for internal technological need is not economically viable. For small volumes, the designed electrocoagulation setup can be used for local treatment before disposal into the municipal sewer.

CONCLUSIONS

Effectiveness of wastewater treatment in designed electrofloatocoagulator reaches 99.97%, which meets requirements for dumping wastewater into the municipal sewers. Treated water can also be recycled by the facility. The use of pressure flotation in electrocoagulator design resulted in a decrease of required

treatment time by 1.2 times, the passivation rate of anode also decreased resulting in power consumption decrease by 1.4 times.

Experimental study of formed precipitates, allowed to conclude that based on chemical inertness of layered double hide oxides, they can be used as landfill material, secondary raw materials: pellets in metallurgy, ceramic tiles, bricks, etc.

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