

## Improvement of Technological Modes of Electrodialysis Apparatus for Treatment of Chromium-Containing Waste Waters

Nino Mkheidze<sup>1\*</sup>, Raul Gotsiridze<sup>1</sup>, Lamzira Kontselidze<sup>1</sup>,  
Svetlana Mkheidze<sup>1</sup>, Ruslan Davitadze<sup>1</sup>

<sup>1</sup> Agrarian and Membrane Technologies Scientific Research Institute of Shota Rustaveli State University, 5, Grishashvili st., 6010, Batumi, Georgia

\* Corresponding author's e-mail: [ninomxeidze@bk.ru](mailto:ninomxeidze@bk.ru)

### ABSTRACT

The wastewaters of galvanizing plants contain toxic heavy metals, which causes pollution of the environment and endangers it. The aim of the present study includes concentration of chromium (VI) ions from chromium plating wastewaters, purification of wastewater and its reuse for development of waste-free technological cycle. This study was carried out with the pilot electrodialysis apparatus designed by the authors and produced in the Institute engineering workshop using real plating rinse water of the plating shop. The authors obtained desalinated water which may be recirculated in rinsing bath and chromium salts concentrate that can be applied in chromium plating bath after correction. As a result of the conducted experiment, the ion-exchange membranes resistant to “poisoning” with chromium (VI) ions and stable in the process of regeneration were selected and the technological modes for stable operation of apparatus matched.

**Keywords:** chromium (VI) removal, electrodialysis, ion-exchange membranes, reuse of wastewater.

### INTRODUCTION

The amount of rinse waters of plating plants in the engineering works makes 30-50% of the total amount of wastewaters (Fu & Wang, 2011). Besides, significant part of the chemical agents used for the plating purposes being washed off the plated parts flows into the sewage system. Such chemical agents are not only toxic, but deficient. For example, rinse water contains about 1% of the plating bath concentration (Gayathri & Senthil Kumar, 2010).

There are many methods for the removal of nonferrous metals from such waters, including reagent method (Mirbagheri & Hosseini, 2005), biological (Mary Mangaiyarkarasi et al., 2011, Hirpara et al., 2011), electrocoagulation (Adhoum et al., 2004, Verma et al. 2013), baro-membrane (Korus & Loska, 2009, Kozłowski & Walkowiak, 2002, Wang et al., 2007), electro-dialytic (Marder et al., 2004, Benvenuti et al.,

2014), sorption (Arslan & Pehlivan, 2007, Gode et al., 2006), ion-exchange methods (Xing et al., 2007) and combined ones (ex. ion-exchange resins and sorbents (Alekseeva et al., 2007), electrodialysis (ED) and ion-exchange (Alvarado et al., 2009), electroflotation and electrocoagulation (Gao et al., 2005), complexation and ultrafiltration (Petrov & Nenov, 2004), liquid membranes and ED (Sadyrbaeva, 2016), ED after sedimentation under the action of chemical agents (Peng et al., 2005). Some toxic metals may be sedimented from soluble compounds by means of correction of pH, but not for chromium, as chromium (IV) is soluble in the wide range of pH (Barakat, 2011). The analysis of the scientific writings shows that in spite of the diversity of methods, none may be considered universal, although the ED method has some prospects for chromium-containing water treatment (demineralization). Previously, ED was used mainly for desalination (sea water, mine waters) (Scott, 1995), but further this method

became important in minimization of impact of toxic productions on the environment (Judd & Jefferson, 2003). The ED method is a membrane process of separation of substances. It is based on the phenomenon of transfer of electrolyte ions through ion-selective membrane under influence of electric field (Strathmann, 2004). Treatment of electrolyte solutions provided in the electro dialysis apparatuses (EDA) (Figure 1).

The ED method has some advantages: no reagent requirements, lack of phase transitions of solution and low operative costs, simplicity of device design, recycling of about 60% of water, ability to utilize valuable components, ability to treat up to maximum permissible concentration. Moreover, the costs of power used in treatment of solution are lower than those connected with use of the chemical methods (Gurreri et al., 2020).

The main element of the EDA includes ion-exchange membranes (IEM). They are made by mixing of ion-exchange resin powder and polyethylene powder as a binder followed by hot-roll-forging into sheets with further reinforcing thereof or without reinforcement. Mainly cation-exchange membranes have sulfonate groups, whereas anion-exchange membranes have quaternary ammonium bases (Gurreri et al., 2020).

IEM are selected for use in concrete process taking into consideration their properties. The physicochemical properties of the membranes depend to a large extent on their composition, nature and production technology.

An ideal membrane should have the following properties: low moisture content, weak swelling

characteristic, high conductivity and selectiveness, chemical stability and mechanic solidity which provide long-term operation of membranes. Exploration of the new methods and materials, improvement of the existing methods aiming to produce high quality membranes goes on continuously (Sardohan et al., 2010, Santiago et al., 2010, Melnikov & Shkirskaya, 2019). Leading companies operating in this sphere include Astom. Corp. JP. (NEOSEPTA, Japan) (San Román et al., 2012), Du Pont (Nafion, USA ) (Mauritz & Moore, 2004), Mega Cz. (Ralex, Czech Republic) (Noubli et al., 2020). The whole range of currently produced membranes allows providing demineralization and concentration of electrolyte solutions (Barakat et al., 2011, Serdiuk et al., 2018). The studies for creation of new polymer matrixes for both homogeneous and heterogeneous IEM are being increasingly improved. Since 1962, Du Pont has held the patent for production of the sulfonic cation membrane Nafion based on fluorocarbon matrix. From 1970s perfluorated matrices have been used in the chlor-alkali media.

In the research by Dalla Costa et al. (2002) the membranes Nafion 450 (Du Pont de Nemours and Co.) and Selemion AMP (Asahi Glass Co.) were used in the process of electro dialytic removal of metal ions (Zn, Ni, Cu, Al) from the industrial waters of metal processing. The anion-exchange membranes produced from the mixture of polystyrene and polyaniline were used for purification of the chromium ions-containing industrial waters of processing of leather and metals (Moura et al., 2012). The process of removal of chromium ions

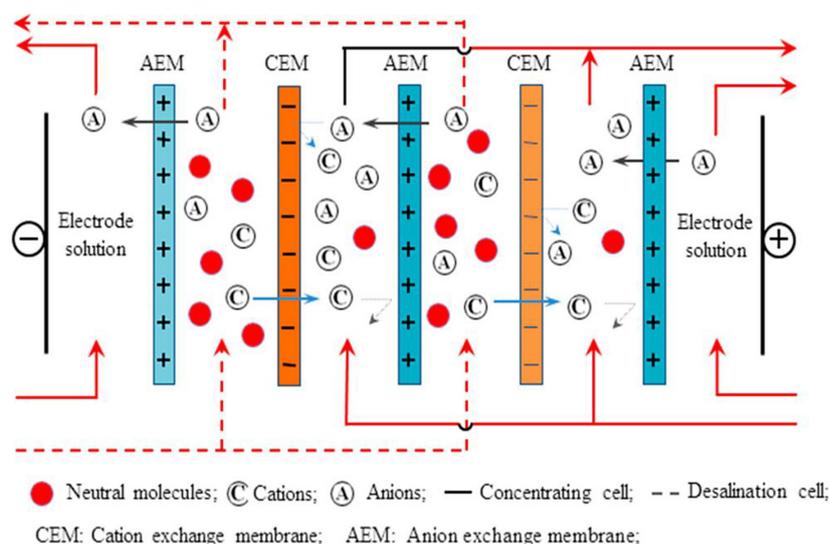


Figure 1. Scheme of electro dialysis (Khan et al., 2016)

and selective transportation of Cr (VI) through the membranes containing 5-(4-phenoxyphenyl)-6H-1,3,4-thiadiazin-2-amine was researched by the authors (Saf et al., 2011). According to the article (Nataraj et al., 2007) they used the experimental EDA in the research regarding the dependence of chromium ions removal effectiveness on such parameters, as the applied potential, pH, initial concentration of solution, and flow rate. The important data were obtained for the low initial concentration (10 ppm). The removal of Cr(VI) mainly becomes problematic due to the instability of parameters of anion-exchange membranes during the electro dialysis process, namely, increase of membrane resistance and concentration polarization, i.e. concentration of concentrate may limit electro dialysis process.

In the apparatus designed by the authors, the heterogeneous membranes produced by Ltd. “Innovative Enterprise Shchekinoazot” (Shchekino, Russia) were used. This company produces various types of heterogeneous membranes by small parcels to orders. It is the only manufacturer of monopolar heterogeneous ion exchange membranes of MK-40, MK-40L, MA-41, MA-41P brands in Russia. Previously, these membranes were actively used in EDA for water desalination, but in the last years, the apparatuses produced in Shchekino enterprise have operated in the milk processing plants. MK-40 and MA-40 differ from the Japanese and American counterparts due to thickness (0.45–0.60 mm), although in ED of dissolved solutions, the share of voltage drop caused with thickness of the material is insignificant. Due to sufficiently high selectiveness, high

mechanic stability and ability of restoration of the initial parameters after drying, they are used for EDA in desalination processes (Sarapulova et al., 2019, Gubari et al., 2021).

Taking into consideration that the membranes used in ED are easily “poisoned” in the corrosive media, the main aim of the conducted study included selection of the ion-exchange membranes used in desalination and concentration of chromium-containing solutions with the ED method, for extension of their life.

## RESEARCH OBJECTS AND METHODS

They were processed with the electro dialysis apparatus (EDA) designed by the authors and supplied with the heterogeneous membranes produced by Ltd. «Innovative Enterprise Shchekinoazot” (Shchekino, Russia). Initially, EDA was supplied with the MK-40 and MA-40 membranes, then they were replaced with MK-40L and MA-41L. The main properties of commercial membranes MA-40 and MK-40 are collected in Table 1.

MK-40 is a composite of polyethylene and cation-exchange resin received by copolymerization of styrene and divinylbenzene with further introduction of sulfonic group. MA-40 is a composite based on the EDE-10P resin received by polycondensation of epichlorohydrin and polyethylene polyamine. MA-41 is a composite of polyethylene and anion-exchange resin which is obtained by amination of the polymer received by copolymerization of styrene and divinylbenzene. The membranes are reinforced with nylon fabric

**Table 1.** Certain properties of commercial membranes

Properties of membranes	MK-40	MK-40 L	MA-40	MA-41L
Ion.exchange group	– SO <sub>3</sub> H	– SO <sub>3</sub> H	=NH; ≡N; –N <sup>+</sup> (R) <sub>3</sub>	–N <sup>+</sup> (CH <sub>3</sub> ) <sub>3</sub>
Grade of ion exchanger	KU-2	KU-2	EDE-10P	AB-17
Content of ion exchanger	65%	65%	55%	65%
Basic binder on base	polyethylene			
Fitting fabrics	nylon	lavsan	nylon	lavsan
Length x Width, mm	1420 x 450			
Thickness, mm	0.3-0.4	0.3-0.4	0.3-0.4	0.3-0.4
Transparency at fracture, MPa, not less than	11.9	10.6	12.7	11.0
Surface resistance, Ohm·cm <sup>2</sup> , not more than	10.0	14.0	12.0	20.0
Transfer number, fractions, not less than	0.98	0.96	0.94	0.96

or lavsan fabric. The membranes reinforced with lavsan fabric (MK-40L and MA-41 L) are notable for their chemical stability. For the purpose of determination of the technological parameters of ED process of desalination and concentration of the chromate-containing solution, the experimental device produced in the Institute engineering workshop was used (Figure 2).

The chromate-containing solution was pumped from the circulation reservoir into the desalination chambers of the apparatus, it was not delivered to the concentration chambers. The concentrate originated in these chambers with the hydrated ions and the water molecules transited by means of electroosmosis and aggregated in the separate

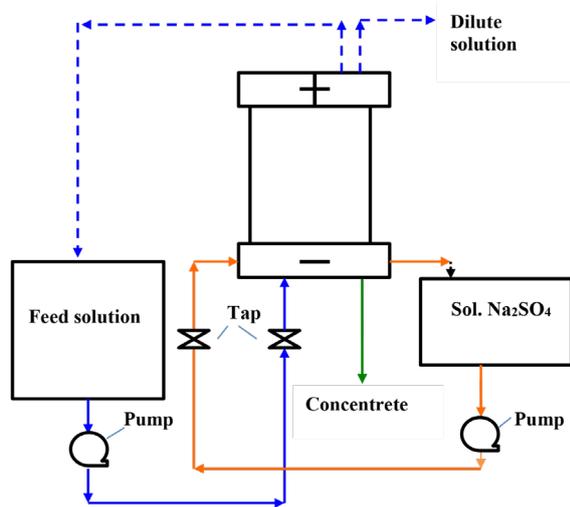


Figure 2. Technological scheme of the electro dialysis device

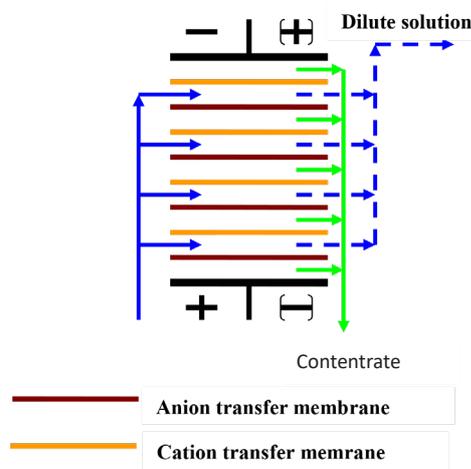


Figure 3. Scheme of distribution of the solution in the working packages of the electro dialysis device operated in the circulation mode

concentration reservoir. The electro dialysis device was operated in the circulation mode, i.e. after coming out of the apparatus, the dialyzate passed to the circulation reservoir and circulated there up to desalination (Figure 2). For the experiment purposes, the real effluents of the washing bath of plating shop of the local plant were used.

After each cycle of desalination, so-called re-polarization of the hydraulic flows and electrodes was carried out. In the case of direct polarity, the lower electrodes of the apparatus were the cathodes, while the upper ones – the anodes. After re-polarization, the lower electrodes were the anodes, whereas the upper ones – the cathodes. Under these conditions, the dialyzate chambers became the concentration ones and vice versa (Figure 3).

Operation package of EDA was equipped with MK-40 and MA-40 IEM (25 pairs of membranes in the apparatus, total area of one kind of membrane was 1.3 m<sup>2</sup>) with the liquid flow separation frames (0.9 mm height), with the corrugated mash-turbulators and platinized titan electrodes. The membrane package was assembled in the chambers as per the liquid flow distribution parallel circuit. Electrode chambers operate as the working chambers, i.e. in one polarity they operate as dialyzate chambers, but in the other one – as concentration chambers. In the case of necessity, the electrode chambers were washed with sodium sulfate solution.

### Determination of the hydraulic properties of the device

The amount of the solution passing through the electro dialysis device was determined under conditions of the direct (lower electrode is a cathode, the upper one is an anode) and reverse polarity. For this purpose, the solution consumption for each polarity was measured at the outlet from the dialyzate chambers –  $Q_d$ , providing that the pump was on and the valve located at the inlet of the solution pipe leading to the dialyzate chambers was completely open; simultaneously, the mechanic overflow of the solution from the dialyzate pipe to the concentrate chambers was measured –  $Q_c$ . Maximum capacity of the electro dialysis device was calculated with the following formula:

$$V = \frac{Q}{s \cdot n} \tag{1}$$

where:  $S$  – area of cress section of the dialyzate chamber;  $n$  – quantity of the dialyzate chambers ( $n = 25$ )

## Determination of the technological parameters of the process

The process implemented in the electro dialysis device operating in the mode of circulation and direct current. During the process, the change of concentration in the reservoir was measured. Balancing of the membranes lasted up to achievement of the constant concentration in the reservoir.

The experiment was carried out in the above hydraulic mode, under the conditions of chromate ions-contained solution ( $C_{\text{initial}} = 50 \text{ mg/L}$ ) and voltage ( $U = 250\text{V}$ ). The following parameters were determined:

- desalination cycle time, min;
- temperature of the solution in the reservoir,  $C^0$ ;
- consumption of the solution in the dialyzate ( $Q_d$ , L/h) and in the concentrate ( $Q_c$ , L/h);
- pH of dialyzate, concentrate and initial solution;
- volumes of the received concentrate ( $V_c$ ) and desalinated solution ( $V_d$ );
- direct current supplied to the device, I, mA.

After completion of each cycle, the samples from the dialysate and concentrate solutions for determination of chromate-ions content were taken (UV-1800 spectrophotometer, SHIMADZU). In the fixed time points, in the course of the process, the electroconductivity (S/m) of the dialysate and concentrate solutions was measured (HI 98194, HANNA).

## RESULTS AND DISCUSSIONS

The process of chrome-contained waters desalination and concentration in the electro dialysis device, which initially was equipped with MK-40 and MA-40 ion-exchange membranes, was studied. The MK-40 and MA-40 membranes are produced on the basis of the KU-2 and EDE-10P ionites, respectively. The initial physicochemical properties of these membranes met the technical requirements *TU* (Technical Specification 6-06-205-91) for such membranes.

The process of desalination and concentration of the chrome-containing solutions by the ED method, was performed with the solution containing chromate-ions of 50 mg/L in condition of applied voltage,  $U = 250\text{V}$ . The experiment proved that in the case of the MK-40 and MA-40 membranes, the desalination process fails due to

“poisoning” (loss of selectivity) of the MA-40 anion exchange membrane with chromate-ions, due to the presence of partial sorption of ions.

Thus, all further experiments were performed by means of the device equipped with the MK-40L and MA-41L ion-exchange membranes. The afore-mentioned membranes were reinforced with lavsan fabric for rising of their stability in the aggressive mediums. Unlike the MA-40 membrane, the MA-41L membrane is produced on the basis of strong-base anionite AB-17. Their compliance with the technical demands *TU* (Technical Specification 6-06-205-91) was established.

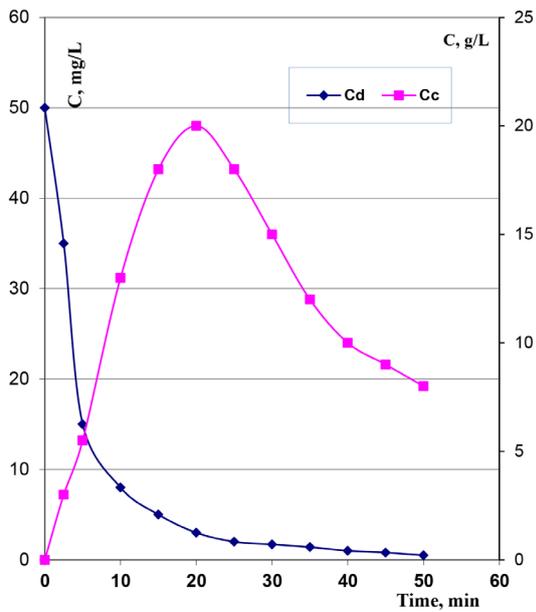
Due to liability of the anion-exchange membranes to “poisoning”, the stability of MA-41L to chromate-ions was researched and the optimal parameters of the ED process were established.

The research of the stabilization process under the conditions of circulation of chrome-containing solutions in the desalination and concentration chambers of the device without supply of current proved that the superficial layer of the MA-41L membrane sorbed chromate-ions and these ions do not practically diffuse into the membrane in spite of great gradient of concentration.

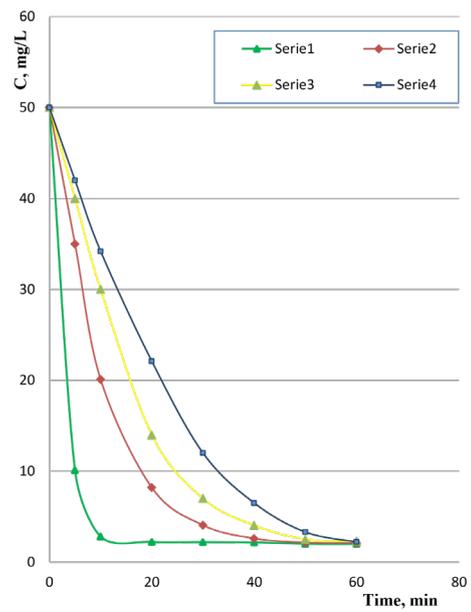
In the case of supply of electric current concentration of  $\text{Cr}^{+6}$  in the desalination chamber first dropped ( $C_{\text{initial}} = 50 \text{ mg/L}$ ), but after achievement of 6-8 mg/L, this process slowed down (Figure 4, curve 1). In the concentration chamber, the change of  $\text{Cr}^{+6}$  ions concentration achieved its maximum (Fig. 4, curve 2). This fact proves that at low concentration and corresponding low indices of current in the desalination chamber, electromigration of  $\text{Cr}(\text{VI})$  ions first equals to, but later – is lower than adsorption flow of the ions.

The process of concentration of chromate-ions in the solution was studied. First, the content of chromate-ions in the concentrate grew unevenly, then – by the equal values (25–30 mg/L). The residual concentration of  $\text{Cr}^{+6}$  ions in the desalination chamber was 2–4 mg/L in all the experiments. Such condition of the system may be called quasi-stationary, as under these conditions, the value  $dc/dt V_d$  approximately equaled to  $dc/dt V_c$ . On the basis of the results, it can be concluded that the transfer of chromate-ions to the anion-exchange membranes makes the key stage of electromembrane process.

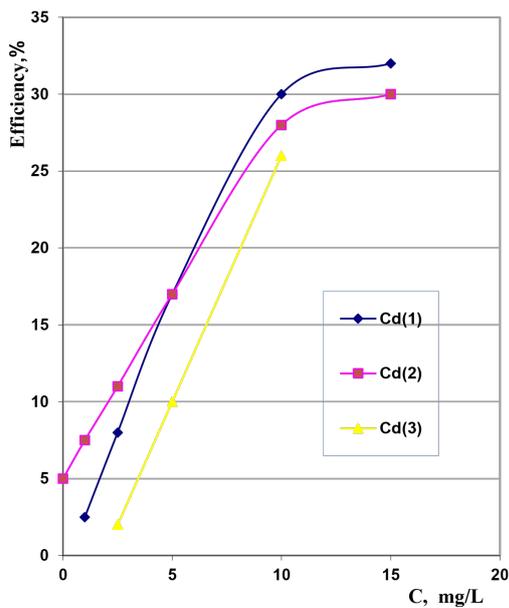
The efficiency of using current in the process of electro dialysis desalination and concentration of chrome-containing solutions with the yield calculated in the various experiments was assessed.



**Figure 4.** Change of  $\text{Cr}^{+6}$  ions concentration in the concentrate (g/L) and dialysate (mg/L) in the process electro dialysis



**Figure 6.** Time dependence of  $\text{Cr}^{+6}$  ions concentration (mg/L) in the dialysate (Series 1.  $C_{\text{conc}} = 4$  mg/L; Series 2.  $C_{\text{conc}} = 50$  mg/L; Series 3.  $C_{\text{conc}} = 100$  mg/L; Series 4.  $C_{\text{conc}} = 150$  mg/L)



**Figure 5.** Dependence of efficiency (%) on concentration of  $\text{Cr}^{+6}$  ions (mg/L) in the dialysate

Figure 5 shows the curves of change of yield depending on the concentration of chromate-ions in the dialysate. As it can be seen, deep desalination of chrome-containing waters is ineffective and it is reasonable to carry out the process up to 8–10 mg/L residual concentration of chromate-ions.

Aiming to establish the possibility of return not only water, but also concentrate to the technological cycle, the possibility of maximum

concentration of chromate-ions in the concentration chamber without considerable slowing of the desalination process down was studied. For this purpose, the demineralization process in the concentration chamber was carried out under the same conditions at the various concentrations of the initial solution.

The analysis of the curves given in Fig. 6 clearly shows slowing of the desalination process down. The obtained result gives the basis to provide return into the technological cycle of high salt-containing electrolyte – 20 g/L chrome anhydride after electro dialysis treatment of the wastewater of the chroming process in the galvanic workshop.

After desalination and concentration of chrome-containing solutions by using the electrolysis method, the physicochemical properties of the MK-41L and MA-41L membranes were studied, namely, static exchange capacity and specific conductivity of the initial membrane in the form  $\text{Na}^+$  and  $\text{CrO}_4^{2-}$  after removal from the device and after their regeneration under the static conditions. The received result pointed to the satisfactory condition of the membranes and possibility of their regeneration in case of working with chrome-containing solutions.

The experiments proved that in the case of electro dialysis treatment of chrome-containing solutions, the process of achievement of the stationary

conditions in the system is slow, which may be explained with partial sorption of the chromate-ions to the MA-41L anionite membrane. It was proven that the kinetic properties of the desalination process of chrome-containing solutions lightly changes after sufficient number of cycles (Figure 5), which means achievement of a quasi-stable state.

On the basis of the obtained results, it may be concluded that the electrodialysis device equipped with the MK-40L and MA-41L membranes may be used locally in the closed system of water supply of chroming.

## CONCLUSIONS

The single-step process of chromate-ions removal from chrome-containing solutions and their concentration was studied. It was found that:

- it is reasonable to run the process with the device equipped with the MK-40L and MA-41L membranes instead of the MK-40 and MA-40 membranes, due to “poisoning” of these latter by chromate-ions;
- passing of the chromate-ions through the MA-41L anion exchange membranes is a key stage of the electromembrane process;
- kinetic properties of the desalination process of chrome-containing solutions lightly change after completion of the certain quantity of the cycles, which points to achievement of a quasi-stable state in the system;
- deep desalination of chrome-containing waters is ineffective; it is reasonable to carry out this process up to residual concentration of the chromate-ions in the dialyzate 8-10 mg/L;
- maximum concentration of chromate-ions (~20 g/L) is achieved without considerable reduction of the desalination process rate;
- electrodialysis method allows purification of the chrome-containing waters as well as return of the obtained waters and concentrate into the technological cycle.

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