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An attempt to develop low effluent and low waste technologies for industrial production facilities

The article addresses the development of low effluent and low waste technologies for the production of protective coatings and printed circuit boards at the Brest electromechanical plant (BEMP). The authors show low cost methods of multifold reduction in the consumption of "fresh" water for production purposes, and, accordingly, the amount of wastewater discharged into treatment facilities, as well as ways to substantially reduce the amount of wastewater generated in the process tanks at galvanic and painting facilities. Methods for extracting heavy metals from electrolyte collection and product rinse tanks using internal and external electrolysis and submersible electrochemical modules (SEM) are described. Ways of reducing the amounts of used process solutions (UPS) and wastewater discharged into treatment facilities are presented. The results of studies on compositions of used process solutions are described; they are used to classify UPS by their properties. This classification allows for the use of UPS instead of or in conjunction with the purchased reagents. The practice has shown a substantial reduction in the amount of purchased reagents needed for the operation of wastewater treatment plants at BEMP facilities engaged in the production of protective coatings and printed circuit boards. The rational formation of wastewater streams in the point of their generation ensures effective wastewater treatment using both traditional methods and "associated" technologies developed by the authors. The introduction of "associated" technologies allows for a manifold reduction in the secondary pollution of wastewater and, accordingly, a reduction in the amount of sludge generated in the process.

Keywords: *process tanks, rinsing operations, electrolytes, electrolysis, heavy metals, pH, reagents, used process solutions*

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INTRODUCTION

CIS has a large number of instrument and machine building enterprises. These enterprises are the main sources of environmental pollution with heavy metals [1–3]. According to the scale of stress factors, which takes into account complex negative impacts on the human body, heavy metals (135 points) leave radioactive waste (40 points) far behind [4].

In the CIS, instrument and machine building enterprises have been operating in tough economic environments, so it is a challenging task for them to find the funds that would allow building efficient and low cost treatment facilities for effluents containing heavy metals in the shortest possible time. It is possible to build such treatment facilities, only if the main production facility significantly reduces the amount of discharged effluents and pollutants.

However, putting things in order at effluent producing industries aimed at reducing water consumption and removal of toxic compounds can solve this problem to a large extent. Such approaches, implemented at minimal cost, are equivalent to expensive reconstruction and even construction of new treatment facilities. One ruble invested in the improvement of the main effluent generating production facility is equivalent to 10 rubles invested in treatment facilities. The experience of developed economies is a convincing proof [1].

Therefore, the purpose of this article is to search for and implement effective low cost technologies that can significantly reduce the amount of effluents produced by facilities engaged in production of protective coatings (PPC) and printed circuit boards (PPCB).

METHODS FOR REDUCING THE CONSUMPTION OF FRESH PROCESS WATER AND THE AMOUNT OF DISCHARGED EFFLUENTS

To create rational rinsing systems, it is first necessary to follows the effective regulations[1]. However, these are the regulations that, as a rule, are not followed by the main production process.

Process engineers, responsible for the main production facility, explain the non-compliance with these regulations by limited areas, additional costs of creating recirculation cycles inside production lines, etc. In their opinion, the implementation of these engineering solutions does not affect the quality of coatings, but creates problems and leads to an increase in the cost of their products.

Moreover, when the operation department of a treatment facility insists on reducing the amount of effluents and removing chemicals, process engineers object that this move will reduce the quality of coatings. Although, it is the non-compliance with the standards by the specialists that leads to defects in the process of the coating application.

This widely spread practice of saving several square meters of space and refusing to install additional process tanks results in the construction of huge treatment facilities. After all, the cost of designing and implementing a treatment facility depends on the effluent discharge. The higher the effluent discharge, the more expensive the treatment facility.

Process engineers will be concerned with saving water, only if the costs of effluent treatment are attributed to the cost of their products, rather than the costs of energy consumed by the enterprise.

1 GOST 9.314-90. Water for galvanic production and washing schemes.

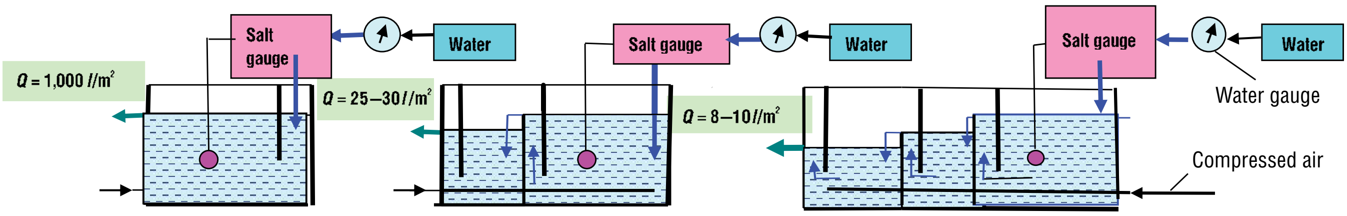


Fig. 1. Cascade flushing used to reduce the need for water per m² of coating

To minimize the effluent treatment cost, it is necessary to:

- conduct a detailed survey of the areas where the runoff producing equipment is located. The process streamlining and equipment improvement can significantly reduce process water at little cost;
- introduce rational methods of rinsing workpieces, which allow to significantly reduce both the amount of water consumed and its pollution with toxic substances [1, 2, 5].
- ensure the rational production of effluent flows at the points of their generation that allows using waste process solutions as reagents for the effluent treatment, as well as neutralization of these flows by low-cost "related" technologies [1–3].
- while improving the quality of rinsing operations, substantially reduce the removal of toxic and, at the same time, expensive chemicals[1] [4, 5] to study the composition of waste process solutions that are not regeneratable by the main production facility due to their properties to use them instead of or together with purchased reagents for effluent treatment. This will greatly reduce the need for commercial reagents and, accordingly, reduce the secondary pollution of effluents with these reagents, as well as reduce the amount of sludge to be generated [1–3].

And here rational rinsing methods become of paramount importance for the reduction of the intake of fresh water for process needs and, accordingly, the discharge of polluted effluents [13, 16].

A flushing system is considered rational, if it ensures the required flushing quality at lowest capital and operating costs, under safe working conditions and without environmental damage.

The more flushing tanks (stages) are used, the smaller the consumption of water and the discharge of toxic ingredients into effluents[1, 2] (Fig. 1, 2) [1, 3, 5]. The use of multi-staged flushing technologies makes it possible to significantly reduce water consumption without compromising the quality of coatings.

In case of single-stage flushing of products, approximately 1,000 l/m² are consumed to flush 1m² of coating, and in case of two-stage flushing, 25–30 l/m² are consumed, while three-stage flushing consumes mere 8–10 l/m². This is confirmed by the formulas used to calculate the flow rates applied to flushing technologies[2]. When four-stage flushing replaces three-stage flushing, water consumption goes down by 30–50 %. So, an increase in the number of

2 Gosstroy of the USSR, SANTEHNIIPROEKT SDI. Recommendations for the design of water supply and sewerage of plating shops. Б3-79. Moscow, 1992. (rus.).

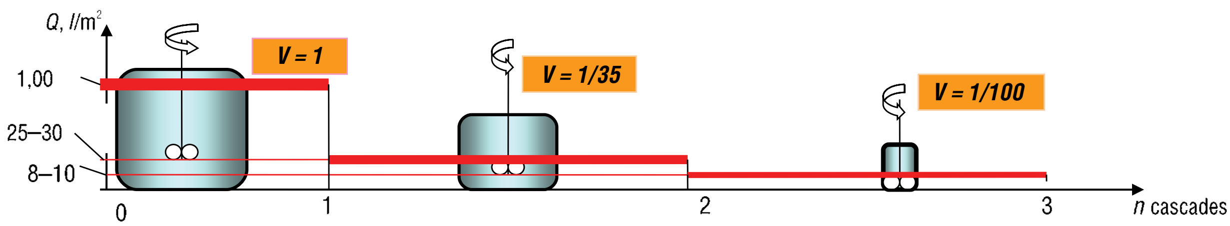
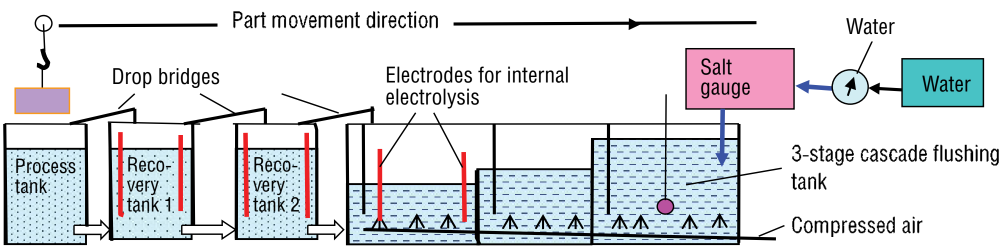


Fig. 2. Reduction in the capacity of tanks at treatment facilities



85 %	10 %	Less than 4.5 %	Traces of chemicals	% recovery of chemicals
Exposure above the bath	Recovery tank 1	Recovery tank 2	3-staged cascade rinsing tank	Description of operations

Fig. 3. Ways to reduce the amount of chemicals together with electroplating effluents

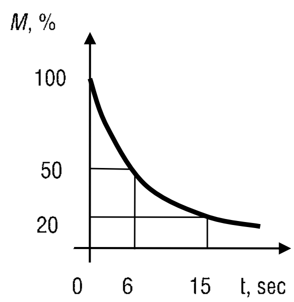


Fig. 4. The amount of electrolytes flushed away by the water M

flushing stages does not result in a significant reduction in water consumption [1–3, 10, 11, 14–17].

However, Vinogradov S.S. claims that an increase in the number of tanks periodically operating in a non-flowing flushing mode, can lead to a significant reduction in the amount of water needed for flushing and increase the duration of the non-flowing period (i.e. the number of treated surfaces, m^2) and, if we consider 8 periodically non-flowing flushing tanks, the duration of the non-flowing period reaches several years for most known cycles and the amount of contaminated flushing water is only a few cubic meters [10, 11, 17].

Hence, a small amount of water located in the tanks periodically operating in a non-flowing flushing mode is used for several years [18, 19].

In addition to the selection of appropriate flushing tanks, a number of measures can be proposed to save water; their implementation does not require any large supplementary costs, but they can significantly streamline the operation of flushing systems. Such measures include the proper organization of cascade flushings, process tanks, the installation of local water supply systems, the use of cooling water, and the intensification of flushing, which will be discussed further in the article [1, 2, 5]. Moreover, it is impossible to reduce water consumption with improperly organized multistage flushing, if it is technologically incorrect (Fig. 4). This can be easily corrected, as shown in Fig. 5, 6.

Additionally, to ensure stable conditions for the flushing of components in case of low water consumption, it is necessary to:

- organize the control and regulation of water consumption for the production of galvanic coatings separately from other production processes[2];
- install water flow limiters for water supplied to individual tanks. Taps with removed control handles, etc. can be used as water flow limiters. A control valve (with the flywheel removed) and a shut-off valve are installed at the supply pipeline to ensure stable water supply. To ensure the continuous water flow, it is necessary to have pressure regulators installed;
- install semi-submersible partitions in all flushing tanks to distribute water throughout the entire volume of the bath. Rubber tourniquets or hinged clamps are needed to fix the partitions, Fig. 5 [1, 2]. The installation of distributive water supply systems in flushing tanks is more expensive and time-consuming;
- hydraulically align the existing overflow edges of water gutters or install new ones if they are missing.
- ensure the supply of compressed air or devices for the vibration of jigs in all flushing tanks.

METHODS TO REDUCE THE REMOVAL OF THE SOLUTION FROM THE MAIN ELECTROPLATING TANKS

To calculate the specific discharge of water needed for flushing, one should take the optimal value of the specific removal of the solution q_{sp} with account taken of the solution removal by products and fixtures, as well as the standard time needed for the solution to drain from the products. The specific removal of the electrolyte solution decreases as the time needed for the solution to drain from the products increases (Fig. 4), if special devices for solution blowing or shaking off products are used.

The duration of the running-off process can be increased using the following methods:

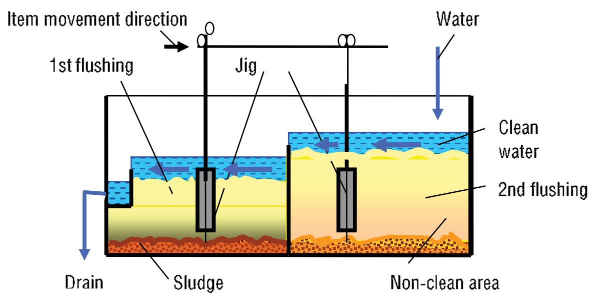


Fig. 5. Typical mistakes made during the organization of two-stage countercurrent flushings:
1 — water supply without a bottom water distributor;
2 — absence of bubbling and semi-submersible boards;
3 — incorrect organization of cascade flushings;
4 — skewed spillway; 5 — lack of salt gauges

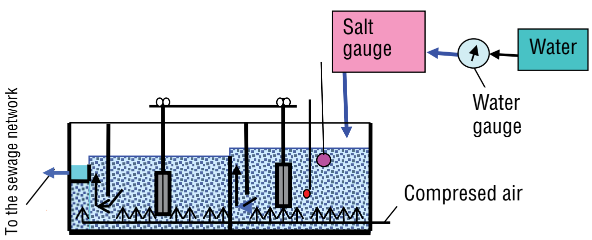


Fig. 6. Rational tank for two-stage countercurrent flushing

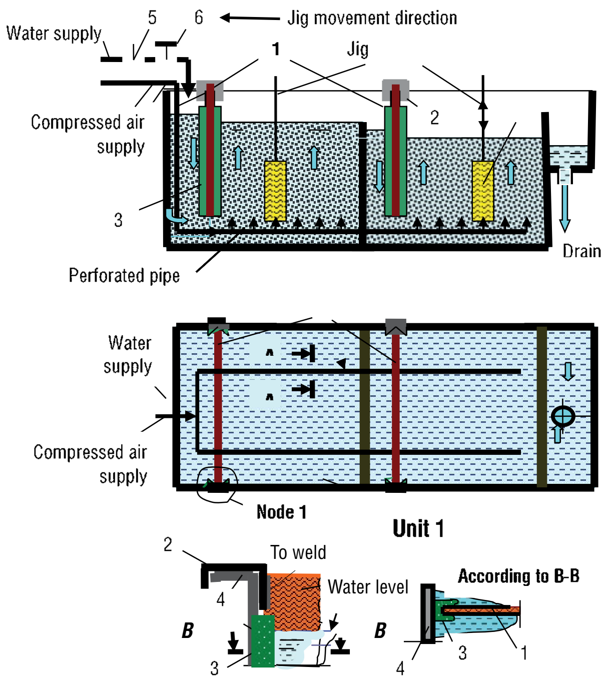


Fig. 7. Method of improving the two-stage flushing tank 1 — additional partitions; 2 — clamp for fastening the partition on the tank wall; 3 — sealing plate made of porous chemically resistant rubber; 4 — tank body; 5 — calibration valve with a reset flywheel; 6 — shut-off valve

- installation of rods above the stationary baths to hold jigs raised from process tanks above the solution level;
- slow removal of the jig from the process tank and its quick transfer to the next tank;
- choosing the optimal design of jigs, barrels and devices to ensure the flow of solution drops back into the process tank;
- keeping items above the tank surface for an optimal period of time (if automatic lines are used, it must be set by the operator), as well as the use of blowing, shaking off, etc. Only an increase in the time of delivery of items to the next tank from 4 to 16 seconds reduces the removal of the solution and the consumption of flushing water by 3 times (Fig. 6);
- it is possible to achieve a significant reduction in the amount of electrolyte "carried away" by the flushed items, if electrolytes contain surfactants, which reduce the value of the surface tension of solutions. For example, chloramine has a similar effect. By adding it in a chromium plating tank, we can reduce the surface tension of the electrolyte from 740 to 310 μ*n*/cm. As a result, the amount of chromium containing compounds that get into the flushing water is reduced by 40–45 % [7].

The specific removal of the solution is reduced, if special processing of jigs is used, followed by blowing or shaking jigs over process tanks.

In addition to the actions described above, other measures can be applied to reduce the removal of the solution, which, as a rule, do not involve significant costs. Such measures include:

- installation of gutters (drop catchers) made of anti-corrosion material and installed between the process and flushing tanks inclined towards the process tank followed by flushing;
- ensure the appropriate hanging of items that ensures minimum solution removal. If possible, flat products should be hung vertically, and the smallest side should be located along the vertical axis (Fig. 7).

Products having intricate shape should be hung in the way ensuring high quality draining. For this purpose, if possible, additional holes should be made in cup-shaped hollows.

- alignment of water drops;
- recovery tanks and the final cascade flushing tanks must have salt gauges, internal and external electrolysis devices to reduce the amount of heavy metals discharged to the treatment facilities [1, 7, 8]. The introduction of such low-cost solutions will ensure a decrease in the concentration of heavy metals in effluents discharged to treatment facilities, and they will be almost unavailable in the sludge of treatment facilities.

METHODS OF EXTRACTION OF HEAVY METALS FROM ELECTROLYTE DRAG-OUT TANKS

Electrolysis and other methods of regeneration and reclamation of used waste solutions are not considered in the article. They are considered within the framework of the basic manufacturing technology. However, let's consider methods of deposition of metals in solution recovery tanks. These methods need attention, because cations of heavy metals are of serious hazard to the environment, although post-dilution anions (SO_4^{2+}, Cl^-) are relatively harmless.

No matter how long tanks are used without any water replacement, water cannot remain there forever. However, if an interval between water replacements is several years, small local devices can be used to extract electrolyte components from effluents and return them to the production process. These devices are inefficient and not costly, although they are easy to maintain.

Such devices are:

- a series of ultra-compact submersible electrochemical modules (SEMs) developed by the Mendeleev University of Chemical Technology of Russia under the supervision of professor S.S. Kruglikov [15]. They convert process solutions or flushing water into the membrane unit. A SEM allows for the membrane electrolysis in the tanks of existing electroplating lines without any modifications;
- a membrane technology (NPP, Vladimir);
- small-sized ion-exchange modules (V.I. Zakharov, Aviapriborproekt JSC, Moscow);
- ULOS unit with a sorbent (B.N. Nechaev, NTK Regenerator, Moscow);
- simple heaters similar to tubular heating elements. They are particularly lucrative under the circumstances. As a rule, evaporation of flushing water is not economical. In the conditions under consideration, when a limited amount (several m^3) of highly concentrated effluents (several *g/l*) is generated once every few years, evaporation becomes competitive compared to other methods of extraction of components of process solutions from the flushing water due to its simplicity, universality and, most importantly, accessibility [17, 18].

To prevent heavy metal cations from getting into effluents, Ilyin offers easy-to-implement recommendations for the electrolytic extraction of metals from recovery tanks [14]. He proposes to conduct the electrolytic deposition of metals from recovery tanks using two methods: a method of internal electrolysis and using an external source to conduct electrolysis. More information about these methods is available in [14].

If it is impossible to use any local units for extracting the components of process solutions from recovery tanks and flushing water directly in flushing tanks, then the time of the non-flow period can be significantly increased or, if the non-flow period remains the same, the number of flushing tanks can be reduced. A reasonable combination of non-flow flushing in several flushing tanks and the use of inexpensive, universal and small-sized extraction units will, on the one hand, reduce the required additional area occupied by flushing tanks and, on the other hand, reduce the cost of flushing water treatment (extraction of electrolyte components and pure water) [12].

Hence, the installation of SEMs in the recovery tanks following all process operations, in which solutions containing chromic acid or its salts, as well as zinc, cadmium, nickel, etc. are used, allows small electroplating areas to operate without treatment facilities, and therefore, large-scale production facilities can drastically deload the treatment facilities in terms of the amount of effluents and toxic components removed. As a result, the cost of treatment facilities plus the additional cost of submersible modules is much lower than the cost of treatment facilities not equipped with SEMs.

The operating costs and power consumption, according to the author, are also drastically reduced when SEMs are used. Here we can refer to the experience of Uralvagonzavod OJSC, Nizhny Tagil, where the use of a submersible module at the chrome plating works had an annual economic effect of about 140,000 Russian rubles, and the payback period was 3.5 months [17].

WAYS TO REDUCE THE AMOUNT OF WASTE PROCESS SOLUTIONS AND EFFLUENTS DISCHARGED TO TREATMENT FACILITIES

To reduce the amount of waste process solutions and effluents discharged to wastewater treatment facilities, it is necessary to:

1. Implement methods of used solutions regeneration. According to[2] issued in 1992, "in accordance with the Decree of the Council of Ministers of the USSR, waste electrolytes must be regenerated and returned to production process"[2]. If there is an electrolyte regeneration unit and it operates properly, electrolytes can be used for years and only be supplemented according to the loss of components applied to coated parts[2]. Moreover, the areas occupied by the electrolyte regeneration unit and the cost of this equipment are not comparable with the cost of electrolyte neutralization at treatment facilities.

2. Prevent the contamination of process solutions with ingredients that make them inefficient due to non-compliance with the process specification, which, as a result, leads to their discharge to treatment facilities[1] [1, 4, 5, 7, 10].

The main causes of electrolyte contamination are as follows:

- items falling down from jigs, which, when dissolved, contaminate electrolytes with salts of copper, iron, zinc, lead, tin, etc.;
- lack of proper control, which leads to the fact that brass hooks to which the anodes of the items are attached are rinsed with electrolytes when their level in the tanks rises, since in this case the anodes dissolve, and copper and zinc mix with the electrolyte;
- poor degreasing of items, which leads to the contamination of electrolytes with organic substances;
- lubricating oils from monorails, telpher devices and other units of mechanized and automated production lines contaminate electrolytes.

The concept of waste process solutions is applicable only to chemical processes such as chemical nickel plating or copper plating, pickling, chromating, oxidation, etc. [7].

It is necessary to punish process engineers for each discharge. Moreover, it is preferable to extract heavy metals from disabled electrolytes using traditional technologies (for example, electrolysis) at the main production facility.

WASTE PROCESS SOLUTIONS AND THEIR USE

If we compare the composition of solutions used by the main production facility and the types of commercial reagents used for wastewater treatment, it turns out that in many cases chemicals with similar properties are used. By virtue thereof, an attempt was made to find if they can be used instead of commercial reagents by classifying these solutions by type (Table).

The classification of waste solutions not only by the pH value, but also by their properties is based on the following considerations.

Classification of waste process solutions by properties that can be used in water treatment

Properties	Description	Place of generation	Scope of application
Reducing	1) etching (steel parts) 2) activation (pickling) 3) etching in $FeCl_3$ 4) etching in $CuCl_2$ 5) undercut etching $(NH_4)_2S_2O_3$ 6) brightening in HCl 7) chemical oxidation	PCP* PCP, PCBP* PCP, PCBP PCBP PCBP PCBP PCP	Recovery of Cr(VI) and acidification of effluents containing chromium
Oxidative	Solutions containing HNO_3 1) brightening 2) etching 3) passivation 4) chrome plating	PCP	Acidification of acid-alkaline effluents
To create an acidic environment	1) acid cadmium 2) nickel plating 3) brass plating 4) tin-bismuth 5) anodizing 6) copper plating 7) ematal plating	PCP	Acidification of effluents containing chromium and acid-alkaline effluents
To create an alkaline environment	1) cathodic degreasing 2) anodic degreasing 3) electrochemical degreasing 4) chemical degreasing 5) zink plating 6) alkali cadmium plating 7) anti-corrosion treatment	PCP	Alkalinization of acid-alkaline effluents

*PCBP — printed circuit boards production;
*PCP — protective coatings production

Some acidic solutions contain oxidizing agents (nitric acid, etc.) and reducing agents (iron (II), tin (II), copper (I), etc.). It is not practical to combine them in a single tank. Such a combination leads to the oxidation of reducing agents with nitric acid.

In this case, the efficiency of using the acidic mixture as a reducing agent decreases. Moreover, excessive amounts of nitric acid, as mentioned earlier, create a number of undesirable phenomena. So, for example, they oxidize commercial reagents (iron sulfate, etc.), thereby increasing their required dose. In the process of interaction between reducing agents and nitric acid, volatile compounds dangerous for maintenance personnel are released.

Given the above, it seems reasonable to divide effluents into flushing and concentrated ones when the latter are used instead of or together with commercial reagents, as well as to divide certain types of acidic solutions into subtypes taking into account their properties. Such a division of waste acidic solutions into solutions that do not contain reducing agents and those containing them will lead to a drastic increase in the efficiency of their use.

The concentration of reducing agents in the acid waste solution isolated from the total mixture of acids will sharply increase. This will be a consequence of the lack of dilution with other acids and oxidizing agents. Toxic gaseous emissions are not available. In some cases, there will be no need for commercial reagents to reduce hexavalent chromium. And finally, the main technological problem will be solved, that is, the oxidation of ferrous iron in the process of hexavalent chromium reduction. There will be no need for alkalization of the total runoff to pH = 9.5 due to the presence of iron hydroxide (II). Dissolution of chromium hydroxide caused by high pH will be eliminated. The coagulation of suspensions and their sedimentation in settling tanks, previously hampered by the presence of iron (II), will improve.

The consumption of alkaline reagent will greatly decrease. And finally, the discharge problem of excessive pH effluent will be solved [18].

RATIONAL FORMATION OF EFFLUENT FLOWS AT THE POINTS OF THEIR GENERATION TOWARDS EFFECTIVE TREATMENT

The rational formation of effluent flows at the points of their formation allows for the effective effluent treatment using traditional methods and “related” technologies developed by the authors [1–3].

“Related” neutralization technologies (fluorine — lead — HM complex compounds, paints and varnishes, etc.) are those that are implemented within the framework of traditional process lines for the neutralization of chromium-containing effluents and all types of effluents featuring the same process parameters and reagents. The introduction of “related” technologies allows to greatly reduce the secondary pollution of effleunts and, accordingly, reduce the amount of sludge formed. The introduction of “related” technologies for the neutralization of effluents from PCP and PCBP is possible only with the rational formation of flows, namely:

- acid-alkali rinsing effleunts;
- rinsing effluents containing HM complex compounds;
- rinsing chromium-containing effluents;
- fluorine-containing rinsing effluents;
- paint-containing effluents;
- solutions containing Cr^{6+};
- solutions containing a commercial reducing agent;
- an acidic solution containing a reducing agent;
- acidic solutions without oxidizing agents;
- acidic solutions with oxidizing agents;
- alkaline WPS + CaO solution.

The expediency of such flows is described in detail in the author's monograph [1, 3]. It was used in the development of a low effluent resource-saving technology for neutralization at the BEMP treatment facilities and dozens of other enterprises in the CIS countries and abroad [1, 11, 18].

CONCLUSIONS

The introduction of efficient low-cost technologies in the operation of an enterprise allows to greatly reduce the amount of “fresh” water consumed in the process and, accordingly, reduce the discharge of effluents to be treated.

Strict compliance with process specifications during core operations ensures a multifold reduction in the amount of toxic substances in effluents discharged for treatment.

The use of methods of internal and external electrolysis, as well as the use of submersible electrochemical modules (SEMs), etc., makes it possible to extract HMs from the recovery tanks. This will allow to return up to 40 % of expensive non-ferrous metals (Zn, Ni, Cu, etc.) to the production process. These metals are in very short supply, and they are discharged in the form of WPS and flushing effluents, polluting the HM sludge.

The use of non-disposable WPS allows reducing the need for purchased reagents for effluent treatment processes and, accordingly, reduce their secondary pollution.

The rational formation of effluent flows at the places of formation creates the prerequisites for the development of a low-effluent, low-waste resource-saving technology for reagent wastewater treatment of effluents at instrument and mechanical engineering enterprises [1–3].

Methods for improving the effluent-producing PCP and PCBP are reflected in detail in the monographs and the reference manual of the authors [1–3].

The materials presented in the article will be used to further improve the effective resource-saving technology for treating effluents from PCP and PCBP.

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Опыт создания малосточных и малоотходных технологий промышленных производств

Приведен опыт создания малосточных и малоотходных технологий производств защитных покрытий и печатных плат на примере Брестского электромеханического завода (БЭМЗ). Показаны посильные службе эксплуатации предприятий малозатратные методы многократного снижения потребления «свежей» воды на технологические нужды и, соответственно, объемов сбрасываемых сточных вод на очистные сооружения, а также пути уменьшения не менее чем на порядок выноса раствора из основных технологических ванн гальванического и покрасочного производств. Описаны способы извлечения тяжелых металлов из ванн улавливания электролитов и ванн промывки изделий с помощью внутреннего и внешнего электролиза, а также погружных электрохимических модулей (ПЭМ) до их следов. Приведены пути снижения объемов сбрасываемых отработанных технологических растворов (ОТР) и сточных вод на очистные сооружения. Описаны результаты исследования составов отработанных технологических растворов, и на основании их составлена классификация ОТР по их технологическим свойствам. Такая классификация позволяет использовать ОТР вместо или совместно с покупными реагентами. Как показал опыт эксплуатации очистных сооружений сточных вод производств защитных покрытий и печатных плат БЭМЗ, использование ОТР вместо покупных реагентов уменьшило их потребность не менее, чем на порядок. Рациональное формирование потоков сточных вод у мест их образования позволяет вести эффективную очистку сточных вод не только традиционными методами, но и разработанными авторами «попутными» технологиями. Внедрение «попутных» технологий позволяет многократно снизить вторичное загрязнение сточных вод и, соответственно, уменьшить объем образующегося осадка.

Ключевые слова: *технологические ванны, промывные операции, электролиты, электролиз, тяжелые металлы, pH, реагенты, отработанные технологические растворы*

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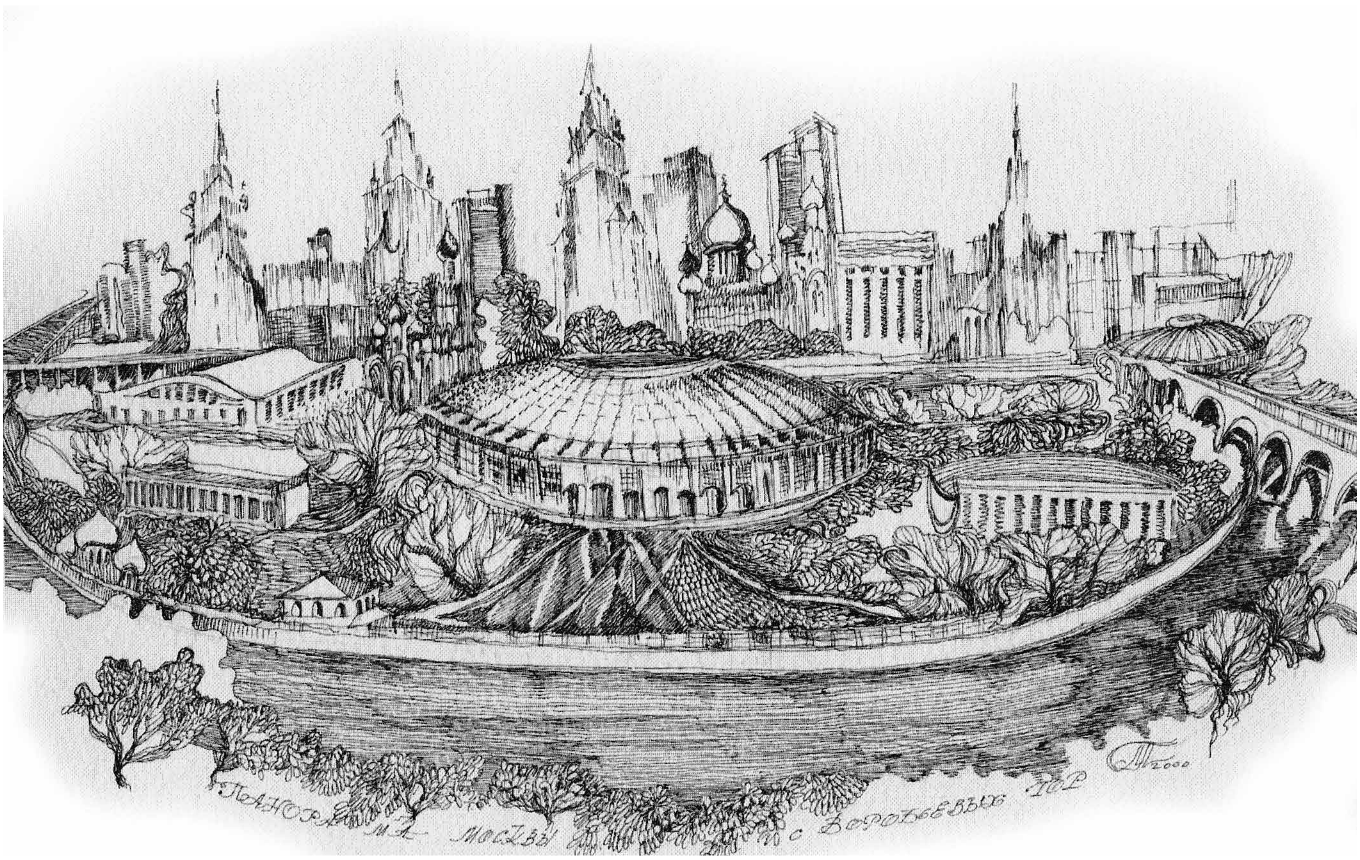

Л.И. Павлова. Москва. Панорама Москвы с Воробьевых гор. Рисунок, тушь, перо.