

# EFFECTIVE TECHNOLOGY OF PHARMACEUTICAL ENTERPRISES WASTEWATER LOCAL TREATMENT FROM ANTIBIOTICS

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The work was aimed to study the processes of industrial wastewater purification of a pharmaceutical company treatment and to recommend an effective technology for local treatment of such wastewater in accordance with the requirements of normative documents on the treated wastewater disposal into the urban sewerage system.

Model solutions of cefuroxime, a cephalosporin antibiotic, in distilled water with a concentration of 25 and 35 mg/dm<sup>3</sup> were used for the study. COD model solutions, which is, respectively, 90 and 120 mg/dm<sup>3</sup>. Chemical oxygen demand was determined by the dichromate method (according to the guiding normative document (GND) 211.1.4.021-95. “Methods for Chemical Oxygen Demand (COD) Determining in Surface and Wastewater”).

The proposed technology included the following successive processes of physical and chemical treatment of wastewater: aeration — equalization of quantitative and qualitative composition of wastewater and oxidation up to 30% of organic matter; coagulation — removing of antibiotics from wastewater by their adsorption on a highly developed surface of mineral coagulants flakes; sedimentation — separating of the formed flakes; oxidation of antibiotics destruction products by hydrogen peroxide; filtration — removal of fine impurities. Effect of COD reduction rate in the coagulation and settling of wastewater in the case of ferrous sulphate III was 79.2% and 75%, which is higher by 4,2–6,7% than when using aluminum sulphate. The COD index changing of the pharmaceutical enterprise wastewater by the stages of its purification was found as follows: “aeration — coagulation with iron sulfate III — sedimentation — oxidation — filtration”. After filtering effect of reducing COD index was 95,8–100% at the initial value of 120 and 90 mg/dm<sup>3</sup> respectively. The technology of local wastewater treatment from antibiotics was developed, which was based on the consistent use of physico-chemical methods of treatment and enabled to remove antibiotics and related substances from wastewater in accordance with the requirements of normative documents and disposal of treated wastewater into the urban wastewater system.

Application of the developed technology of local wastewater treatment from antibiotics and related substances by the pharmaceutical companies would lead to meet the requirements of industrial wastewater disposal into the city drainage system, to a significant risk reduction of the antibiotics influence on the microorganisms of active sludge of the urban wastewater biological treatment facilities, to operating costs reduction to achieve maximum allowable discharges (MAD) of wastewater into a natural reservoir.

**Key words:** antibiotics, wastewater, treatment, pharmaceutical enterprises.

Environmental protection should be a priority of state policy, Ukrainians should look at all spheres of life through an ecological prism, and business should gradually move to environmentally friendly production processes. Environmental standards must be integrated into all areas of the economy and everyday life of Ukrainians. Such a system works successfully in European countries and should be tested in our country. Only the integration of environmental policy and socio-economic development will make it possible to stabilize

the state of the environment in Ukraine and move to a model of sustainable development.

Ukraine has made significant progress in implementing European environmental legislation and meeting its obligations to implement environmental requirements such as those operating in EU countries. In particular, the Laws “On Environmental Impact Assessment” (which is already working successfully) and “On Strategic Environmental Assessment” were adopted, which oblige to take into account the needs of the environment

at the stage of development and decision-making.

Unfortunately, in relation to the normative documents of Ukraine on wastewater treatment from pollution, in contrast to the normative documents in the EU, USA and Canada, these documents do not take into account several important indicators of wastewater composition that directly affect the effects of wastewater treatment, active sludge properties and residual concentrations of environmentally hazardous pollutants in treated wastewater during disposal into natural reservoirs. Such indicators include the concentration of nitrogen-containing compounds in terms of total nitrogen, the concentration of phosphorus-containing compounds in terms of total phosphorus and the concentration of antibiotics and related substances.

The efficiency of municipal treatment plants (speed, treatment effects and depth of individual pollutants removal, the composition of their oxidation products, etc.) depends on many factors, among which one of the main is the physico-chemical composition of wastewater.

The most sensitive link to the negative impact of wastewater chemical components is the active sludge of biological treatment plants, namely its main technological properties such as the ability to oxidize dissolved pollutants and the ability to sediment and separate from the treated liquid during settling. An important indicator of wastewater safety for the reliability of urban treatment plants is the impact of their pollution on the sedimentation properties of active sludge and the ability to separate from the treated liquid during settling. These indicators are not always taken into account when controlling the composition of industrial wastewater supplied to the city sewer, as well as the presence of biologically active compounds like antibiotics, biocides, inhibitors, etc. in wastewater. Neglecting these characteristics causes a number of extremely negative consequences for water utilities: reducing the efficiency of urban wastewater treatment and increasing operating costs to achieve the required indicators, exceeding the MAD when discharging treated wastewater into natural reservoirs (indicators strictly controlled by environmental services), active sludge “swelling” with the prospect of its further loss, etc.

Although in Ukraine there are companies that provide local wastewater treatment after the production of liquid drugs, such as Private Joint Stock Company (PJSC) “Scientific Production Center (SPC) Borschagivsky Chemical and Pharmaceutical Plant”, as well

as companies that have begun work to address the issue of local wastewater treatment from antibiotics — Lekhim-Obukhiv LLC, Obukhiv city, and Chemical Plant “Chervona Zirka” PJSC, Kharkiv city, but nowadays one of the urgent problems is the presence of antibiotics in the wastewater of pharmaceutical industries.

In most cases, antibiotics and the products of their transformation do not have the ability to biological transformation, as well as they adversely affect the microorganisms of treatment plants (active sludge aeration tanks).

Physicochemical and biological methods are used to treat pharmaceutical wastewater from antibiotics.

Among the physico-chemical methods, the methods aimed at antibiotic molecules structure destroying should be noted, such as oxidation by various oxidizing agents: ozone [1, 2]; hydrogen peroxide [3, 4]; potassium permanganate [2, 5] and others.

Another group of physico-chemical methods is focused on antibiotics removal using the following methods: adsorption on coagulant or flocculant flakes [6, 7], on activated carbon [8, 9], on zeolites, etc.; membrane separation (ultrafiltration) [10, 11].

The use of biological methods of wastewater treatment from antibiotics [1, 12, 13, 14, 15] has shown good results with long-term aeration of wastewater. Thus, when aerated for 1–3 days, the purification effect by biological oxygen demand (BOD<sub>5</sub>) reached 90–96%. To increase the treatment effect of pharmaceutical companies wastewater, in the literature the following measures are proposed:

- to dilute the concentrated wastewater of the pharmaceutical company with municipal wastewater 4–5 times;
- to use two- or three-stage biological treatment technologies with a total efficiency of 90–95%;
- to dose the compounds of inorganic phosphorus in aeration tanks in the treatment of wastewater from antibiotics, because of its very small amount in the wastewater of pharmaceutical industries;
- to increase the temperature of wastewater up to 35 °C, which allows to increase the treatment efficiency up to more than 90%, etc.

In Igor Sikorsky Kyiv Polytechnic Institute, the scientists of Ecobiotechnology and Bioenergy Department conducted the research (with practical implementation) to develop a technology for local treatment of industrial wastewater from antibiotics.

The purpose of the work is to investigate the processes of industrial wastewater treatment of a pharmaceutical enterprise and to propose

an effective technology of local wastewater treatment in accordance with the requirements of normative documents on the treated wastewater discharge into the urban sewage.

### Materials and Methods

Model solutions of cefuroxime, that is cephalosporin row antibiotic, were prepared for the study in distilled water with a concentration of 25 and 35 mg/dm<sup>3</sup>. For the preparation of solutions, a pharmaceutical form of cefuroxime was used, namely tablets (cefuroxime Sandoz). 1 tablet contains 250 mg of cefuroxime and excipients. The tablets were crushed and solutions of the above concentration were prepared.

The chemical oxygen demand indexes (COD) of model solutions were determined, which were, respectively, 90 and 120 mg/dm<sup>3</sup>. Chemical oxygen demand (COD) was determined by the dichromate method (according to the guiding normative document (GND) 211.1.4.021-95. "Methods for Chemical Oxygen Demand (COD) Determining in Surface and Wastewater").

The following procedures of model solutions processing were investigated consistently:

1. Model solutions aeration by means of the aquarium compressor with air productivity of 20–40 dm<sup>3</sup>/h) and the aerator installed in the vessel of 250 ml with the investigated model solution, within 16 hours;

2. Coagulation with mineral coagulants:

- ferrum III sulfate at a dose of 50 mg/dm<sup>3</sup> by Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·7H<sub>2</sub>O was used as a reagent;

- aluminium sulfate at a dose of 50 mg/dm<sup>3</sup> by Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O was used as a reagent.

To achieve complete coagulation of mineral salts, the pH was adjusted to 7.0–7.5 with a solution of alkali NaOH.

The model solutions were quickly mixed with the coagulant solution for 1–2 min and continued to stir slowly for 15–20 min till flakes formation and agglomeration.

3. Sedimentation the coagulated impurities for 30 minutes.

4. Clarified water selection from the vessel and formed precipitate separation.

5. Oxidation of clarified water contaminants with hydrogen peroxide at a dose of 30 mg/dm<sup>3</sup> using a solution of 30% concentration for 30 min with stirring using a laboratory magnetic stirrer.

6. Solution filtration through a sand filter.

Sand for filtration (fraction of 0.5–0.8 mm) was prepared as follows: thoroughly

washed with running water from mechanical impurities; dried in an oven at 105 °C for 10 hours; fired in a muffle furnace at 600 °C for 2 hours to ash all residual contaminants; cooled and washed with distilled water. After that, the sand was transferred to the filter and filter layer of a 10 cm high was formed, filtration rate was 2 cm/min.

At each stage of the process, water samples were taken to determine the COD value. The analysis results and the determined purification effects at each stage are given in the Table and in the Fig. 1 and 2. The reliability of the obtained data is  $P < 0.05$ , i.e. statistically significant differences are found. To assess the significance of the difference between the averages of the two groups,  $t$ -test (Student's test) was used.

### Results and Discussion

The results of studies using aluminium sulfate (Table) showed lower values of COD reducing effect — 80 and 75% at the initial COD 90 and 120 mg/dm<sup>3</sup>, respectively. Therefore, a coagulant based on ferrum III sulfate has a certain advantage over aluminium sulfate.

As it is shown in Fig. 1, the greatest decrease in the COD index was observed in the coagulation processes of pollutants contained in wastewater, and in settling. For coagulation, mineral coagulants based on ferrum III sulfate and aluminium sulfate were used with pH adjustment to achieve the isoelectric region for the most complete formation of iron III and aluminium hydroxides, their coagulation and wastewater treatment from organic pollutants. The effect of COD reduction in coagulation and sedimentation of wastewater in the case of ferrum III sulfate usage was 79.2% and 75.0% in the initial COD of untreated wastewater, respectively, 90 and 120 mg/dm<sup>3</sup>.

Increasing the initial COD index value in untreated wastewater reduces the effect of organic pollutants removal by COD when using coagulation and sedimentation by 4.0–6.5%.

The use of other purification processes according to the studied technological scheme allowed COD reduction by 25.0–33.3% during aeration, by 91.7–94.4% during oxidation with hydrogen peroxide, by up to 95.8–100% during filtration.

Based on the obtained results, the technology was developed (Fig. 3), process parameters were established (aeration duration, reagent dose, sedimentation duration, filtration rate, filter loading height, amount of precipitate formed, etc.).

The results of physico-chemical treatment technology research of a pharmaceutical company industrial wastewater according to the scheme “aeration — coagulation — sedimentation — oxidation — filtration”

No. of model solutions and cleaning effect, %	COD index value. mg/dm <sup>3</sup>				
	before cleaning	after aeration	after settling	after oxidation	after filtration
Option with ferrum III sulfate coagulation					
1	90	60	12	5	0
Effect, %	–	33.3	86.7	94.4	100
2	120	90	25	10	5
Effect, %	–	25	79.2	91.7	95.8
Option with aluminium sulfate coagulation					
3	90	64	18	7	0
Effect, %	–	28.9	80	92.2	100
4	120	87	30	12	5
Effect, %	–	27.5	75	90	95.8

Note. The effects of COD index reduction were determined in each purification process with  $P < 0.05$  relative to the initial COD values before purification.

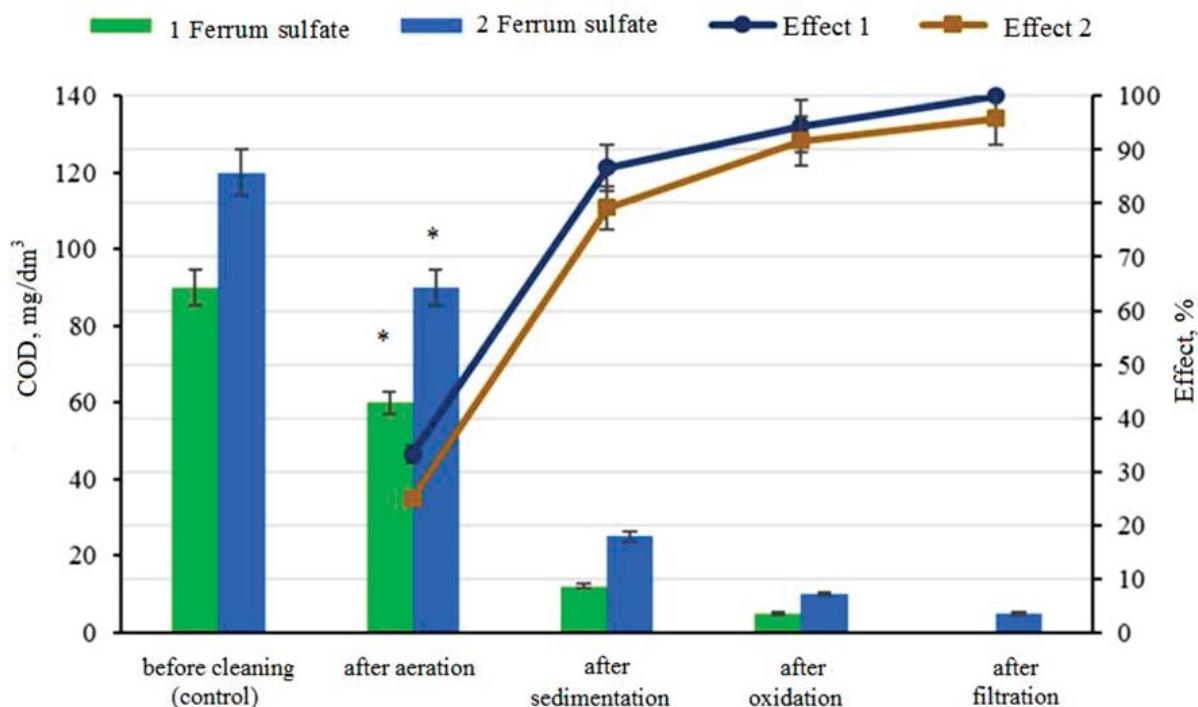


Fig. 1. Change in the COD index of a pharmaceutical company industrial wastewater and the effect of purification by COD in purification by the technology “aeration — coagulation with ferrum III sulfate — sedimentation — oxidation — filtration”:

effect 1 was determined at a COD value in untreated wastewater of 90 mg/dm<sup>3</sup> in each process relative to this value; effect 2 — at a COD value in untreated wastewater — 120 mg/dm<sup>3</sup>

\* —  $P < 0.05$  compared to the control without ferrum sulfate addition

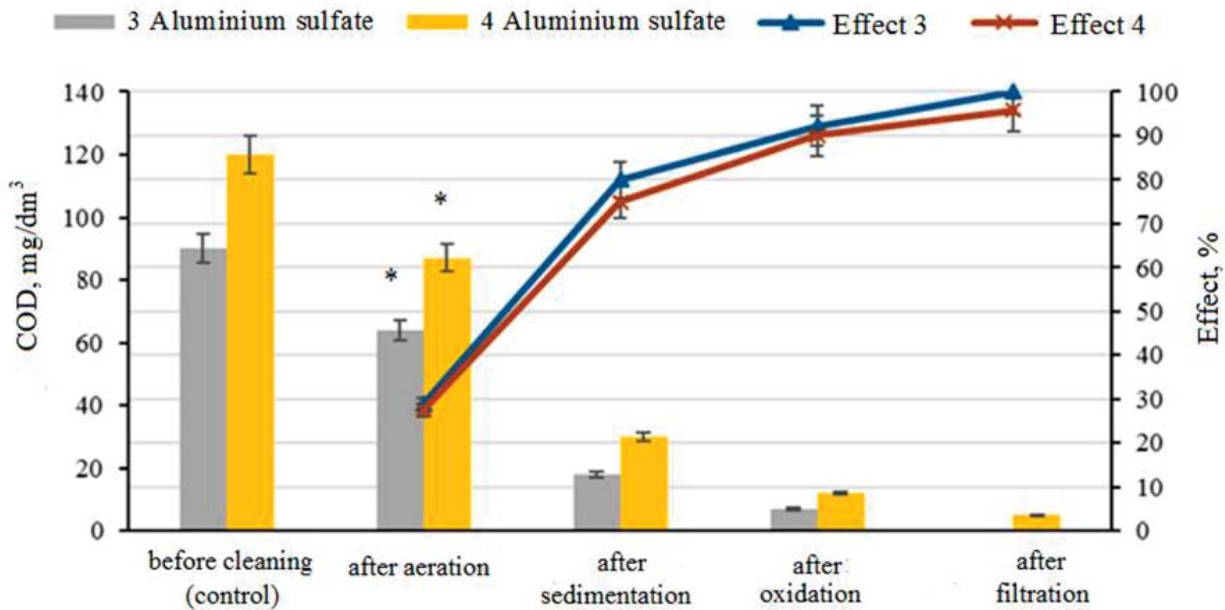


Fig. 2. Change in the COD index of a pharmaceutical company industrial wastewater and the effect of purification by COD in purification by the technology “aeration — coagulation with aluminium sulfate — sedimentation — oxidation — filtration”: effect 3 was determined at a COD value in untreated wastewater of 90 mg/dm<sup>3</sup> in each process relative to this value; effect 4 — at a COD value in untreated wastewater — 120 mg/dm<sup>3</sup>  
 \* —  $P < 0.05$  compared to the control without aluminium sulfate addition

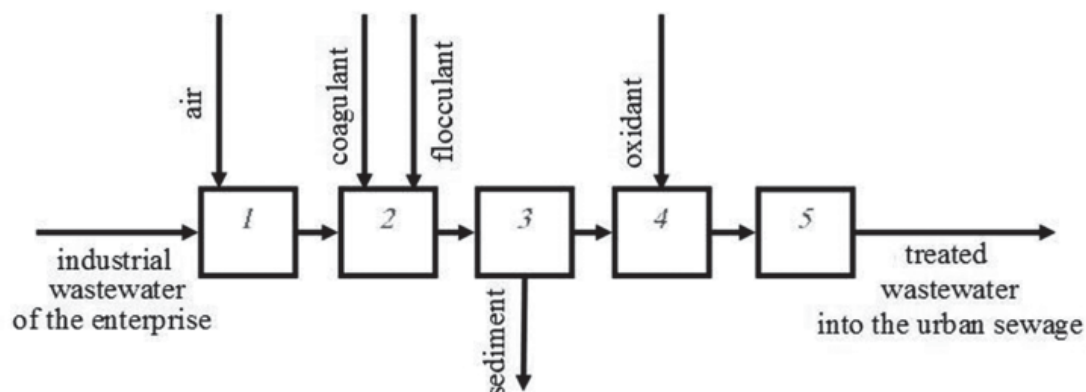


Fig. 3. Scheme of pharmaceutical company industrial wastewater treatment technology from cefuroxime: 1 — averaging; 2 — coagulation with mineral coagulants; 3 — settling; 4 — oxidation with hydrogen peroxide; 5 — filtration

The technology includes sequential processes of physico-chemical wastewater treatment, namely averaging, coagulation, settling, oxidation with hydrogen peroxide, and filtration.

Given the uneven drainage of industrial wastewater from pharmaceutical companies and fluctuations in pollutants concentrations during the day, to equalize the quantitative and qualitative composition of wastewater when it enters the treatment plant for local

treatment, wastewater averaging is required which is carried out using an aeration system.

The use of air for wastewater aeration in the averaging unit allows in the first stage to oxidize up to 30% of organic substances contained in industrial wastewater with the help of oxygen.

The next process of wastewater treatment is coagulation of pollutants with a mineral coagulant, for example, with ferrum III sulfate —  $Fe_2(SO_4)_3$  at a dose of 50–100 mg/dm<sup>3</sup>

with alkalization of wastewater with NaOH solution up to pH 7.0–7.5. It is possible to use a flocculant, for example, Magnafloc at a dose of 1–2 mg/dm<sup>3</sup> to form large flocks and intensify the process of coagulant flakes sedimentation. For coagulation, aluminium sulfate Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> can also be used instead of Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> in combination with alkali and flocculant. However, it should be noted that a more economical option is Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.

Due to the large specific surface area of colloidal particles, they have a significant surface energy and, consequently, a high adsorption capacity, due to which the adsorption of dissolved in wastewater substances takes place on the surface of the formed colloidal particles.

The flocculation process application is based on the mechanism of flocculants action, which is based on the phenomenon of flocculant molecules adsorption on the surface of colloidal particles, formation of mesh structure of flocculant molecules, adhesion of colloidal particles due to Van der Waals forces. Under the action of flocculants, three-dimensional structures are formed between colloidal particles, capable of faster and more complete separation from the liquid phase.

Flocculation is carried out to intensify the process of iron hydroxide flakes formation in order to increase the rate of their sedimentation. Flocculants usage allows reducing the coagulants dose and coagulation process duration, and increasing the rate of the formed flakes sedimentation.

Thus, when introducing coagulant and flocculant into industrial wastewater, due to the processes of coagulation and flocculation, there is removal of contaminants from wastewater, which are impurities of varying degrees of dispersion (fine, colloidal and molecular-soluble substances) due to adsorption of these substances on the highly developed surface of mineral coagulants flakes which are formed in water at maintenance of conditions necessary for coagulation.

To separate the formed flakes with adsorbed contaminants from water, the sedimentation process is used. The taken duration of sedimentation is 1.5 hours. To increase the efficiency of sedimentation (by 25–30%) and reduce the process duration (up to 15–20 minutes), you can use a thin-layer sedimentation tank with inclined shelves of parallel working tiers of the sedimentation tank.

The sludge formed in the sedimentation tank (10–12% of the wastewater volume) must be periodically taken away for consolidations. You can use filter bags to dehydrate the sludge from the initial humidity of 95.0–99.7% to the moisture content of the compacted sludge of 70–80% for further disposal.

Further, according to the technology (Fig. 3), wastewater after sedimentation is directed to the oxidation of contaminants remaining in wastewater after coagulation, flocculation and separation of sludge, using hydrogen peroxide H<sub>2</sub>O<sub>2</sub> as oxidant at a dose of 30–60 mg/dm<sup>3</sup> for 30 min of wastewater contact with the oxidant under stirring conditions to undergo reactions between hydrogen peroxide and contaminants. The advantages of hydrogen peroxide usage compared to other oxidizing reagents are high oxidation efficiency of organic substances, absence of residual concentrations of hydrogen peroxide in treated wastewater due to its decomposition, stability of salt content of treated wastewater, and the course of reactions without toxic intermediates formation.

The final process in the technology is the process of wastewater filtration, for example, through sand filtration loading. Due to filtration, fine impurities are removed from wastewater, which are carried away by the water flow after the sedimentation stage: small flakes of coagulant and colloidal substances that are retained on the surface of the grains of the filter loading due to the action of adhesion forces, mutual colloids coagulation, and adsorption on the loading surface.

## Conclusions

Wastewater treated using the developed technology can be discharged into the urban drainage system and will not interfere with the operation of municipal treatment facilities.

1. On the basis of experimental research on wastewater treatment of pharmaceutical companies from antibiotics and related substances the local treatment technology has been developed, which is based on the consistent use of physico-chemical treatment methods, can be used for local treatment of these wastewater and for antibiotics and related substances removal from wastewater in accordance with the requirements of normative documents, and disposal of treated wastewater to the urban sewerage system.

2. The performed experimental researches allowed establishing the rational parameters

of technological processes of pharmaceutical enterprises wastewater treatment from antibiotics and related substances and recommending the offered technical solutions for introduction at the enterprises of pharmaceutical branch.

3. The developed technology of local wastewater treatment from antibiotics and related substances application by pharmaceutical

companies will significantly reduce the risk of MAD exceeding on the disposal of wastewater treated in urban biological treatment plants, reduce operating costs to achieve MAD, reduce the risk of active sludge “swelling” and related operating and other economic costs.

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## ЕФЕКТИВНА ТЕХНОЛОГІЯ ЛОКАЛЬНОГО ОЧИЩЕННЯ СТІЧНИХ ВОД ФАРМАЦЕВТИЧНИХ ПІДПРИЄМСТВ ВІД АНТИБІОТИКІВ

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Метою роботи було дослідити процеси очищення виробничих стічних вод фармацевтичного підприємства та запропонувати ефективну технологію локального очищення таких стічних вод відповідно до вимог нормативних документів щодо скидання очищених стічних вод у систему водовідведення міста.

Для дослідження було використано модельні розчини цефуроксиму – антибіотика цефалоспоринового ряду, у дистильованій воді з концентрацією 25 і 35 мг/дм<sup>3</sup>. Хімічне споживання кисню модельних розчинів, становило, відповідно, 90 і 120 мг/дм<sup>3</sup>. ХСК визначали за біхроматним методом (згідно з КНД 211.1.4.021-95. «Методика визначення хімічного споживання кисню (ХСК) в поверхневих і стічних водах»).

Ефект зниження показника ХСК при коагуляції і відстоюванні стічних вод у випадку використання сульфату заліза ІІІ становив 79,2% і 75%, що вище на 4,2–6,7% ніж при застосування сульфата алюмінію. Встановлено зміну показника ХСК стічних вод фармпідприємства за етапами їх очищення: «аерація — коагуляція сульфатом заліза ІІІ — відстоювання — окиснення — фільтрування». Після фільтрації ефект зниження показника ХСК становив 95,8–100% при початкових значення 120 і 90 мг/дм<sup>3</sup> відповідно. Розроблено технологію локального очищення стічних вод від антибіотиків, яка ґрунтується на послідовному використанні фізико-хімічних методів очищення та дозволяє видалити із стічних вод антибіотики і супутні їм речовини до вимог нормативних документів та відвести очищені стічні води в міську систему водовідведення.

Застосування фармацевтичними підприємствами розробленої технології локального очищення стічних вод від антибіотиків та супутніх їм речовин призведе до забезпечення вимог скиду виробничих стічних вод у міську систему водовідведення, до суттєвого зниження небезпеки впливу антибіотиків на мікроорганізми активного мулу біологічних очисних споруд міста, до зменшення експлуатаційних витрат на досягнення гранично-допустимих скидів стічних вод у природну водойму.

**Ключові слова:** антибіотики, стічні води, очистка, фармацевтичні підприємства.

## ЭФФЕКТИВНАЯ ТЕХНОЛОГИЯ ЛОКАЛЬНОЙ ОЧИСТКИ СТОЧНЫХ ВОД ФАРМАЦЕВТИЧЕСКИХ ПРЕДПРИЯТИЙ ОТ АНТИБИОТИКОВ

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Целью работы было исследование процессов очистки производственных сточных вод фармацевтического предприятия и предложение эффективной технологии локальной очистки таких сточных вод в соответствии с требованиями нормативных документов по сбросу очищенных сточных вод в систему водоотведения города.

Для исследования были использованы модельные растворы цефуроксима - антибиотика цефалоспоринового ряда, в дистиллированной воде с концентрацией 25 и 35 мг/дм<sup>3</sup>. Химическое потребление кислорода модельных растворов составляло 90 и 120 мг/дм<sup>3</sup>, соответственно. ХПК определяли по бихроматному методу (по КНД 211.1.4.021-95. «Методика определения химического потребления кислорода (ХПК) в поверхностных и сточных водах»).

Эффект снижения показателя ХПК при коагуляции и отстаивании сточных вод в случае использования сульфата железа ІІІ составил 79,2% и 75%, что выше на 4,2–6,7% чем при применении сульфата алюминия. Установлено изменение показателя ХПК сточных вод фармпредприятия по этапам их очистки: «аэрация — коагуляция сульфатом железа ІІІ — отстаивание — окисление — фильтрование». После фильтрации эффект снижения показателя ХПК составлял 95,8–100% при начальных значения 120 и 90 мг/дм<sup>3</sup> соответственно. Разработана технология локальной очистки сточных вод от антибиотиков, основанной на последовательном использовании физико-химических методов очистки и позволяет удалить из сточных вод антибиотики и сопутствующие им вещества с требованиями нормативных документов и отвести очищенные сточные воды в городскую систему водоотведения.

Применение фармацевтическими предприятиями разработанной технологии локальной очистки сточных вод от антибиотиков и сопутствующих им веществ приведет к обеспечению требований сброса производственных сточных вод в городскую систему водоотведения, к существенному снижению опасности воздействия антибиотиков на микроорганизмы активного ила биологических очистных сооружений города, к уменьшению эксплуатационных расходов на достижение гранично-допустимых сбросов сточных вод в естественный водоем.

**Ключевые слова:** антибиотики, сточные воды, очистка, фармацевтические предприятия.