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# PROBLEMS OF SOAPSTOCK TREATMENT OF VEGETABLE OIL PRODUCTIONS AND THEIR SOLUTIONS

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Wastewater generated during vegetable oil production contains various pollutants that enter it during soapstock processing: fats and fatty acids and their salts (aqueous soap solutions), glycerin, phosphoglycerates, neutral fat, phosphatides, proteins, carbohydrates, dyes, unsaponifiable and waxy substances, salts, mechanical impurities, etc.

*Aim.* The purpose of the work was to study the processes of purification of industrial wastewater from oil production and to propose an effective technology for their treatment, taking into account the regulatory requirements for the discharge of treated wastewater into the city sewage system.

*Methods.* Chemical oxygen demand (COD) was determined by the dichromate method. The concentration of suspended solids was determined by gravimetric method.

*Results.* As a result of research, calcium carbonate was chosen as an alkaline reagent. After treatment of soapstock with calcium carbonate followed by flotation, the effect of removing the suspended particles was 70–75%, and COD decreased by 60%. On the basis of the research, a technology for processing soapstock was proposed, including sequential processes of physicochemical wastewater treatment — averaging, alkalization with calcium carbonate, stage I of flotation, coagulation, stage II of flotation, oxidation with hydrogen peroxide, filtration through quartz filters and adsorption on carbon filters.

*Conclusion.* An effective technology for preliminary cleaning of the soapstocks oil production has been developed. This will significantly reduce the concentration of organic matter and other pollutants in soapstocks, which will significantly reduce the impact of such effluents on the processes of biological wastewater treatment of urban wastewater treatment plants.

***Key words:*** soapstock; vegetable oil; pollutants; technology; treatment; wastewater.

Wastewater generated during the production of vegetable oil contains various pollutants that enter it during the soapstock processing: fats and fatty acids and their salts (aqueous soap solutions), glycerin, phosphoglycerides, neutral fat, phosphatides, proteins, carbohydrates, dyes (carotene, carotenoids, chlorophyll, etc.), unsaponifiable and waxy substances, salts — sodium sulfate and chloride, mechanical impurities, etc.

Soap impurities have a complex and volatile composition, which depends on the nature and properties of their constituents, the amount of substances associated with fats and other factors. Wastewater is characterized by high concentrations of organic pollutants in terms of COD, high suspended solids, and low pH (Table 1).

In a phase-dispersed state, such wastewater is a stable emulsion with water. The presence in wastewater of phospholipids, which are emulsion stabilizers, leads to complications of phase separation. Wastewater contains suspended solids, colloidal substances and various organic and inorganic solutes as well. Soaps contained in wastewater had a high stabilizing and absorption capacity, due to which they absorbed a significant part of

*Table 1.* **Average value of indicators of wastewater from vegetable oil production**

| No | Indicator | Units of measurement | Average value |
|---|---|---|---|
| 1 | pH | – | 2 |
| 2 | Suspended solids | mg/L | 6 300 |
| 3 | Non-volatile suspended solids | mg/L | 700 |
| 4 | Dry residue | mg/L | 13 400 |
| 5 | Ignited residue | mg/L | 1 300 |
| 6 | COD | mg/L | 40 000 |

impurities such as phosphatides, proteins, mucus, dyes and others [1].

Wastewater color varies from chestnut brown to brown. Dyes can be divided into three groups: substances that are in the fat cells and turn into oil unchanged; substances that change composition and color during oil production; substances that are formed during oil production when heated.

Wastewater in terms of the content of pollutants exceeds the norms for discharge into city sewer systems. Therefore, before being discharged into city treatment facilities at local treatment facilities, they must be treated by physicochemical and biological methods.

Flotation combined with sedimentation has proven to be the most effective way to remove grease, soap, and suspended solids from water [2]. At the same time, there is a decrease in fat content by 10 or more times, soaps — by 4–5 times.

Comparison of the methods of coagulation-sedimentation and coagulation-flotation in terms of the efficiency of removing organic matter from wastewater due to COD, suspended solids showed that the use of coagulation and flotation enables to obtain a higher efficiency of removing organic matter, suspended particles due to effective adsorption of soap and fat on suspended solid particles, particles of metal hydroxides formed during coagulation, and flotation of these pollutants on the surface of the liquid and the formation of flotation sludge [3, 4].

For the treatment of soapstocks after physical and chemical treatment, it was proposed to use aerobic biological treatment [5]. The technology of such wastewater treatment using coagulation with the addition of coagulant $Al_2(SO_4)_3$, flotation and biological treatment in an aerotank is considered. When the COD of untreated wastewater is 1 800 mg/L, a decrease in the COD of 98% is achieved and the quality of treated wastewater is ensured, which is acceptable for discharge into a natural reservoir.

It was found that when using iron chloride $FeCl_3$ as a coagulant [6] in the treatment of soapstocks with an initial COD of 220,000 mg/L and high turbidity, the optimal pH value, which provides the best performance of wastewater treatment in terms of COD, is 8.5 for the pH range 2–13, the cleansing effect of COD reached 80% at a dose of coagulant $FeCl_3$ — 800 mg/L.

To clean soapstocks, absorption methods are also used, when choosing which it should be noted that when the temperature rises above 80 °C, the peroxide content of fatty acids increases significantly, and when the temperature drops below 50 °C, a mass is formed that is difficult to absorption purification [7].

Membrane methods are used for local cleaning of soapstocks. Fats are successfully separated from an aqueous solution by ultrafiltration at a pressure of up to 6 atm, since fats have practically no osmotic pressure. Low molecular weight fatty acids and other related substances with low osmotic release due to nanofiltration. Reverse osmosis is used at high osmotic pressure [2, 8].

The proposed technology includes cleaning of soapstocks by two-stage flotation in the presence of reagents under pressure and gravel filtration. The use of ultrafiltration made it possible to abandon the use of a reagent, the second stage of flotation and gravel filtration. The COD value after the first stage of flotation was 1 000 mg/L, and after ultrafiltration — 250 mg/L with the initial COD value up to 5 000 mg/L [9].

The use of ultrafiltration makes it possible to effectively use expensive ingredients contained in wastewater, as well as reuse purified water in production. The disadvantages of membrane methods are their high cost, the need for membrane regeneration and preliminary removal of substances that cause turbidity of water — suspended and colloidal, leading to clogging of the membranes. Consequently, membrane methods are difficult to operate, require complex equipment, and are very expensive [10].

The aim of the work was to study the processes of purification of industrial wastewater from oil production and to propose an effective technology for their local treatment to regulatory requirements for the treated wastewater discharge into the city's sewerage system. To achieve these goals, a study was conducted using the actual production of soapstocks from vegetable oil in 2021.

## Materials and Methods

COD was determined by the dichromate method (according to managing normative document in Ukraine — KND 211.1.4.021-95. "Method for determining the chemical oxygen demand (COD) in surface and wastewater". The concentration of suspended solids was determined by gravimetric method.

For the study, vegetable oil production soapstocks were used, which were characterized by the pollutants indicators given in the Table 1. The COD value of soapstocks was 40 000 mg/L, the pH value was 2.

For leaching, a comparison of the effect of two reagents on soapstock, namely NaOH and $CaCO_3$, was used. A 20% NaOH solution was added to the soapstock samples at a dose of 20 mL/L, which ensured an increase in pH to 7. When using $CaCO_3$, the dose was 2 g/L.

The following soapstock processing processes have been consistently studied:

1. Aeration using an aquarium compressor and aerator, installed with a capacity of 250 ml with the test soapstock, for 24 hours.

2. Chemical precipitation with calcium carbonate $CaCO_3$ at a dose of 1.8–2.0 g/L, pH 5.5, reaction time 10–15 min and flotation (stage I) with air supply through fine-porous aerators for 75 min, air flow rate was 8 $m^3/(m^2 \cdot h)$.

3. Coagulation was carried out with aluminum sulfate at a dose of 1–1.2 g/L at an optimum pH of 5.5. The soapstock sample was quickly mixed with the coagulant solution for 1–2 min and the stirring was continued for 15–20 min until the formation and compaction of flakes.

4. Flotation (stage II) of coagulated impurities was carried out for 75 min with air supply through fine-porous aerators.

5. The selection of purified water from the tank and the separation of the formed flotation sludge were carried out.

6. Oxidation of organic pollutants remaining in purified water at the outlet of the II stage flotation, hydrogen peroxide at a dose of 1.5–1.8 g/L, pH 4.5–5.5, process duration 2 hours with stirring laboratory magnetic stirrer.

7. Filtration of wastewater after oxidation through a sand filter. Sand for filtration was prepared as follows: thoroughly washed with running water to remove mechanical impurities; dried in an oven at 105 °C for 10 h; ignite in a muffle furnace at 600 °C for 2 hours to remove all residual contamination into ash; cooled and washed with distilled water. Thereafter, the sand was transferred to a filter and a filter layer was formed.

8. Filtration of wastewater after the sand filter through a carbon filter. Fine-grained activated carbon was used in the form of a filtering layer.

At each stage of the process, water samples were taken at the outlet, in which the indicators were determined: suspended particles and COD. The results of the analyses and the determined cleaning effects at each stage are shown in Table 2 and Fig. 1 and 2. The reliability of the obtained data was $P < 0.05$, i.e. statistically significant differences were 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

To alkalize the soapstock by adding 20% NaOH solution at a dose of 20 mL/L, the pH was raised to 7. The liquid became dark. There were some difficulties in setting the dose of NaOH to bring the pH of the wastewater to 5.5. With an increase in the alkali dose, the pH rose sharply above pH 7.

In the second variant, calcium carbonate $CaCO^3$ was used. To increase the pH of wastewater from 2 to 5.5, the dose of $CaCO_3$ was 2.4 g/L. In the process of alkalization, an intense formation of $CO_2$ gas was observed, the bubbles of which rose to the surface of the liquid with the formation of foam (about 10% of the volume). The amount of sediment was about 5%. The formation of gas promoted the flotation of pollutants — suspended particles, dissolved organic substances during their adsorption on the "bubble – solid particle" complexes. The use of $CaCO_3$ allowed the following flotation to be used to separate the flotation froth from the water. Therefore, $CaCO_3$ was chosen as the alkaline reagent, which made it possible to obtain the following positive results. When calcium carbonate reacted with water, insoluble calcium hydroxide was formed and carbon dioxide was liberated. The pH of the water increased, the

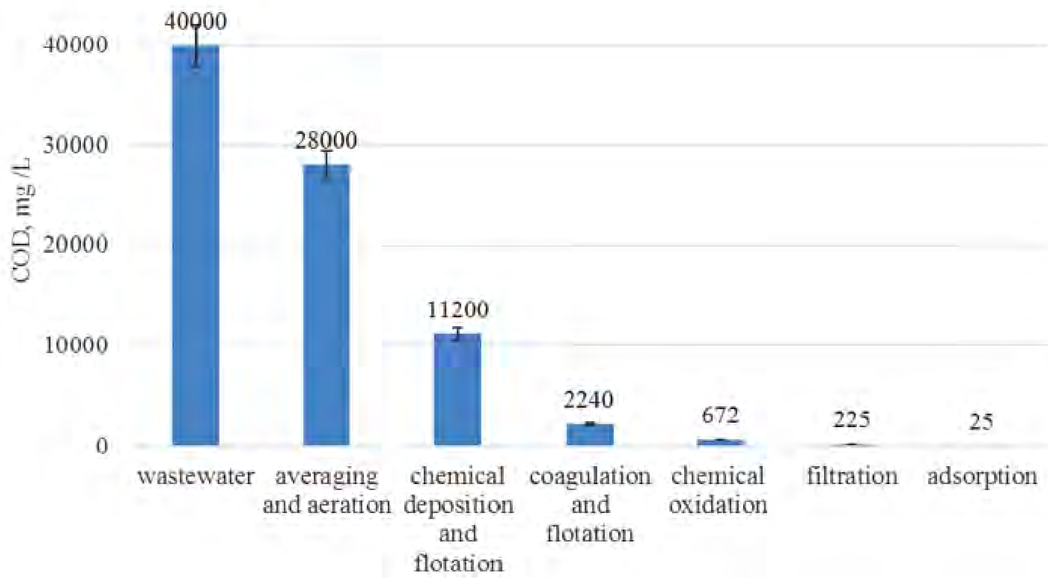


*Fig. 1*. **Change in the COD of the soap stock during treatment by technology:**
averaging and aeration — chemical deposition and flotation — coagulation and flotation — chemical oxidation — filtration — adsorption

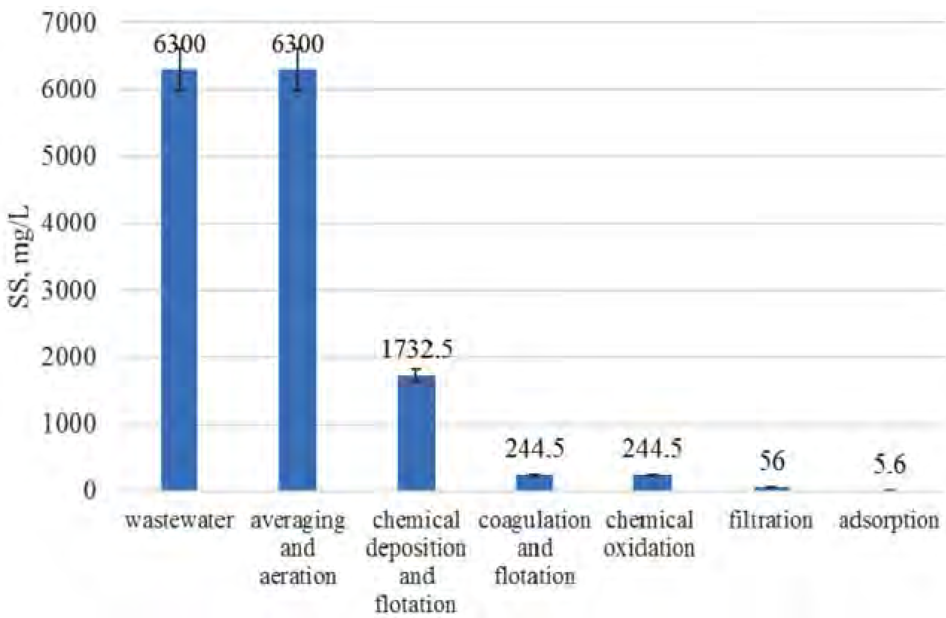


*Fig. 2*. **Change in the rate of suspended solids in soap stocks during treatment by technology:**
averaging and aeration — chemical deposition, flotation — coagulation and flotation — chemical oxidation — filtration — adsorption

emulsion bonds between organic matter and water were destroyed, and the organic solution was destabilized. Due to the formation of small particles of calcium hydroxide with a large surface area, dissolved organic substances were adsorbed on their surface and precipitated. The release of carbon dioxide contributed to the saturation of water with gas bubbles, into which surfactants were released, which were released from the solution on the surface of the bubbles and float, forming foamy sediment on the surface of the water in the float. As a result of the processing of soapstock with calcium carbonate, followed by flotation, the effect of removing suspended solids of 70–75% was obtained, and the COD was reduced by 60%.

The research results on the processing of soapstock in some physical and chemical processes of the technology given in the Table 1 showed the highest values of the effect of reducing COD — 80% with an initial COD of 40,000 mg/L, the effect of reducing the concentration of suspended solids — 70–75% with an initial 6 300 mg/L in the process of coagulation using $Al_2(SO_4)_3$ and flotation.

High effects were also observed for COD — 88–90% and suspended solids — 90% at the stage of soapstock adsorption. The use of other purification processes according to the investigated technological scheme made it possible to reduce the COD by 30% during aeration, by 60% during the chemical precipitation of $CaCO_3$ and flotation, by 70% during oxidation with hydrogen peroxide, by 65–68% during filtration on quartz filters. After all stages of sequential physical and chemical treatment according to the proposed technology, the obtained purified water was characterized by pollution indicators that did not exceed the permissible values for discharging to city treatment facilities with aeration tanks.

Based on the obtained results, the technology of local physical and chemical treatment of oil production soapstocks was developed (Fig. 3), process parameters were determined (aeration duration, reagent dose, flotation duration, oxidation, filtration rate, filtration loading height, amount of flotation sludge, and sediment formed, etc.).

The technology included the sequential processes of physical and chemical wastewater treatment — averaging, alkalization with calcium carbonate, stage I of flotation, coagulation, stage II of flotation, oxidation with hydrogen peroxide, filtration through quartz filters and absorption on carbon filters. Taking into account the uneven removal of the soap solution of oil production and fluctuations in the concentration of pollutants during a day, to equalize the quantitative and qualitative composition of wastewater when it entered the treatment plant for local treatment, it was required to use wastewater averaging, which was carried out using an aeration system.

The use of wastewater aeration in the balancing tank enabled up to 30% of organic substances contained in industrial wastewater to be oxidized with oxygen in the first stage.

At the first stage of wastewater treatment, it is recommended to use calcium carbonate $CaCO_3$ as a precipitating agent. When calcium carbonate reacts with water, poorly soluble calcium hydroxide is formed and carbon dioxide is liberated. The pH of the water increases, the emulsion bonds between organic matter and water are destroyed, and the organic solution is destabilized. Due to the formation of small particles of calcium hydroxide with a large surface area, dissolved organic substances were adsorbed on their surface and precipitated.

*Table 2*. **Change in soap stock indicators at certain stages of processing according to the proposed technology**

| No | Stages of technology, indicators | At the inlet | At the outlet | E,% |
|---|---|---|---|---|
| 1 | Averaging and aeration<br>COD, $mgO_2/L$<br>Suspended solids, mg/L | <br>40 000<br>6 300 | <br>28 000<br>6 300 | <br>30<br>0 |
| 2 | Chemical precipitation with $CaCO_3$ and flotation<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>6 300<br>28 000 | <br>1 575–1 890<br>11 200 | <br>70–75<br>60 |
| 3 | Coagulation with $Al_2(SO_4)_3$ and flotation<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>1 575–1 890<br>11 200 | <br>205–284<br>2 240 | <br>85–87<br>80 |
| 4 | Chemical oxidation with $H_2O_2$<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>205–284<br>2 240 | <br>205–284<br>672 | <br>0<br>70 |
| 5 | Filtration on quartz filters (2 stages)<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>205–284<br>672 | <br>41–71<br>215–235 | <br>75–80<br>65–68 |
| 6 | Adsorption on pressure adsorption filters (2 stages)<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>41–71<br>215–235 | <br>4.1–7.1<br>22–28 | <br>90<br>88–90 |
| 7 | Adsorption on pressure adsorption filters (2 stages)<br>Suspended solids, mg/L<br>COD, $mgO_2/L$ | <br>41–71<br>215–235 | <br>4.1–7.1<br>22–28 | <br>90<br>88–90 |

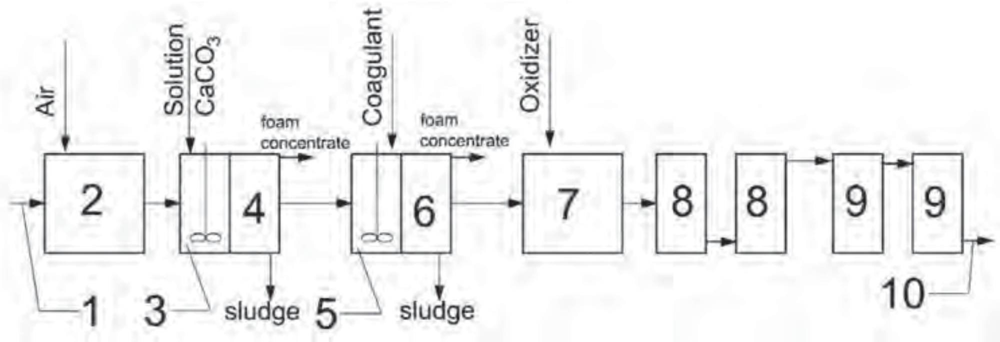


*Fig. 3.* **Technology scheme of the local physical and chemical vegetable oil production soapstock:**
*1* — wastewater from the factory; *2* — averaging; *3* — alkalizing with calcium carbonate; *4* — flotation of the first stage; *5* — coagulation; *6* — flotation of the II stage; *7* — oxidation by hydrogen peroxide; *8* — filtration through quartz filters (2 stages); *9* — adsorption on carbon filters (2 stages); *10* — treated water in the city sewer

The release of carbon dioxide contributed to the water saturation with gas bubbles, which attached to the surfactants released from the solution on the surface of the bubbles and float, forming foamy sediment on the water surface at stage I of flotation with air supply through wonderful materials. The recommended dose of calcium carbonate is 1.8–2.0 g/L, pH 5.5. The air supply to the skimmer must provide the process with gas and could be carried out in case of insufficient amount of carbon dioxide formed as a result of the reaction, for example, in case of a decrease in the reaction temperature, etc. Duration of flotation was 75 minutes.

Removal of pollutants from wastewater by flotation occurred due to the adhesion of polluting particles to the carbon dioxide bubbles formed in the reaction chamber and the air that was introduced into the water by aerators with a porous surface. Surfactants and slurry fine particles attached to the gas bubbles. The resulting complexes "bubble — solid particle — surfactant" floated to the surface of the water, forming a layer of foam (flotation sludge). Flotation was considered as a molecular adhesion process of flotation material particles at the interface between two phases — gas (air) and water. Foam and sludge were periodically removed from the float for disposal.

The next wastewater treatment process was the coagulation of pollutants with a mineral coagulant, for example, aluminum sulfate — $Al_2(SO_4)_3$ at a dose of 1–1.2 g/L at an optimum pH of 5.5. It is possible to use flocculants, for example, polyacrylamide (PAA) at a dose of 10 mg/L to form large flocs and intensify the deposition of coagulant flocs.

Due to the large specific surface area of colloidal particles, they have significant surface energy and, as a consequence, high adsorption capacity, due to which there was an effective adsorption of substances dissolved in wastewater on the surface of the resulting colloidal particles.

The use of flocculants is based on the action of flocculants, which is based on the flocculant molecules adsorption on the surface of colloidal particles, the formation of a network structure of flocculants molecules and the adhesion of colloidal particles due to van der Waals forces. Under the action of flocculants, three-dimensional structures were formed between colloidal particles, capable of faster and more complete separation from the liquid phase. Flocculation was carried out to intensify the formation of aluminum hydroxide flakes in order to increase the rate of their deposition. The use of flocculants enables to reduce the dose of coagulants, reduce the duration of coagulation and increase the of deposition rate of the resulting flocs.

Thus, with the introduction of coagulant and flocculants into industrial wastewater due to coagulation and flocculation, contaminants were removed from wastewater — impurities of varying degrees of dispersion — finely dispersed, colloidal and molecularly soluble substances due to the adsorption of these substance on the surface of mineral coagulants flocs that were formed in water, providing the necessary conditions for coagulation. Stage II flotation with aeration through porous materials was used to separate the formed flocs with adsorbed pollutants from water. The duration of the flotation was taken as 75 min. Flotation sludge was periodically removed for disposal.

Then, according to the technology (Fig. 3), the wastewater after flotation of the II stage was directed to the oxidation of pollutants remaining in the wastewater at the outlet of the flotation of the II stage, using hydrogen peroxide $H_2O_2$ as an oxidizer at a dose of 1.5–1.8 g/L, pH 4.5–5.5 for 2 hours, and contact of wastewater with an oxidizing agent under stirring conditions to carry out reactions between hydrogen peroxide and pollutants. The advantages of hydrogen peroxide using over other oxidizing reagents were the high oxidation efficiency of organic substances, the absence of residual concentrations of hydrogen peroxide in the treated wastewater due to its decomposition, the stability of the salinity of the treated wastewater, and reactions without toxic intermediate products.

The final process in the technology was wastewater filtration. For example, at first it was through loading a sand filter, the through coal. Due to filtration, small impurities were removed from the wastewater, which, after the settling stage, were removed by a stream of water: small coagulant flakes; colloidal substances that were retained on the surface of the grains of the filtering loaded due to the action of adhesion forces, mutual coagulation of colloids, adsorption on the surface of the load.

## Conclusions

Experimental studies have determined rational parameters for processing soapstocks in the production of oils: efficiency at different stages of processing, duration of aeration, dose of reagents, duration of flotation and oxidation, filtration rate, height of filtering load, amount of formed flotation sludge and sediment.

A local treatment technology was proposed. The technology includes the following processes: wastewater averaging, reagent treatment of $CaCO_3$ with flotation using aeration through porous materials, reagent treatment with coagulant $Al_2(SO_4)_3$ with flotation, similarly, chemical oxidation with hydrogen peroxide $H_2O_2$, two-stage filtration on two-stage filtration on absorption filters.

When choosing a technology, the irregularity of industrial wastewater flow, the nature, concentration and phase-dispersed composition of pollutants in oil production soapstocks, the results of sample analysis according to the following indicators as pH, suspended particles, COD, etc., and the results of experimental studies of treatment were taken into consideration. The technology ensured high-quality processing of soapstocks. Purified water according to the proposed technology could be discharged into the city sewage system and would not interfere with the operation of city treatment facilities.

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## ПРОБЛЕМИ ОЧИЩЕННЯ СОАПСТОКІВ ОЛІЙНИХ ВИРОБНИЦТВ ТА ЇХ ВИРІШЕННЯ

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Стічні води, які утворюються під час виробництва олії, містять різноманітні забруднювальні речовини, які переходять у стічні води під час перероблення соапстоків: жири й жирні кислоти та їхні солі (водні розчини мил), гліцерол, фосфогліцериди, нейтральний жир, фосфатиди, протеїни, вуглеводи, забарвлювальні речовини (каротин, каротиноїди, хлорофіл та ін.), речовини, які не омилюються, та воскоподібні, солі — сульфат і хлорид натрію, механічні домішки тощо.

*Мета.* Дослідити процеси очищення виробничих стічних вод олійного виробництва та запропонувати ефективну технологію їх локального очищення до нормативних вимог у процесі скидання очищених стічних вод у систему водовідведення міста.

*Методи.* Хімічне споживання кисню (ХСК) визначали біхроматним методом. Концентрацію завислих речовин — гравіметричним методом.

*Результати.* У процесі оброблення соапстоків карбонатом кальцію з подальшою флотацією було отримано ефект видалення завислих речовин на 70–75%, зниження ХСК — на 60%. На основі досліджень було запропоновано технологію очищення соапстоків, що включає послідовні процеси фізико-хімічного очищення стічних вод — усереднення, підлужування карбонатом кальцію, I ступінь флотації, коагуляція, II ступінь флотації, окиснення пероксидом водню, фільтрацію через кварцові фільтри та адсорбцію на вугільних фільтрах.

*Висновки.* Розроблено ефективну технологію попереднього очищення соапстоків олійного виробництва. Це дасть змогу значно знизити концентрації органічних речовин та інших забруднень у соапстоках, що суттєво знизить вплив таких стоків на процеси біологічного очищення стічних вод міських очисних станцій.

***Ключові слова***: соапстоки; рослинна олія; забруднювальні речовини; технологія очищення; стічні води.

## ПРОБЛЕМЫ ОЧИСТКИ СОАПСТОКОВ МАСЛИЧНОГО ПРОИЗВОДСТВА И ИХ РЕШЕНИЕ

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Сточные воды, образующиеся при производстве масла, содержат различные загрязняющие вещества, которые переходят в сточные воды при переработке соапстоков: жиры и жирные кислоты и их соли (водные растворы мыл), глицерол, фосфоглицериды, нейтральный жир, фосфатиды, протеины, углеводы, окрашивающие вещества (каротин, каротиноиды, хлорофилл и др.), вещества, которые не омыляются, и воскообразные, соли — сульфат и хлорид натрия, механические примеси и т. п.

*Цель.* Исследовать процессы очистки производственных сточных вод масличного производства и предложить эффективную технологию их локальной очистки до нормативных требований в процессе сброса очищенных сточных вод в систему водоотведения города.

*Методы.* Химическое потребление кислорода (ХПК) определяли бихроматным методом. Концентрацию взвешенных веществ — гравиметрическим методом.

*Результаты.* В процессе обработки соапстоков карбонатом кальция с последующей флотацией был получен эффект удаления взвешенных веществ на 70–75%, снижение ХПК — на 60%. На основе исследований была предложена технология очистки соапстоков, включающая последовательные процессы физико-химической очистки сточных вод — усреднение, подщелачивание карбонатом кальция, I ступень флотации, коагуляция, II ступень флотации, окисление пероксидом водорода, фильтрацию через кварцевые фильтры и адсорбцию на угольных фильтрах.

*Выводы.* Разработана эффективная технология предварительной очистки соапстоков масляного производства. Это позволит значительно снизить концентрации органических веществ и других загрязнений в соапстоках, что существенно снизит влияние таких стоков на процессы биологической очистки сточных вод городских очистных станций.

***Ключевые слова***: соапстоки; растительное масло; загрязняющие вещества; технология очистки; сточные воды.