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The use of multicomponent adsorption filters in water purification systems and luminescent control of ecotoxicant content

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The paper presents the results of innovative design development for multicomponent adsorption filters with sliding cartridges or with a metal-graphite electrode system. The proposed filters allow providing water purification by 98–99% simultaneously from various polluting organic substances, including polycyclic aromatic hydrocarbons. The filtering media of these filters include bentonite modified by various methods. To study the effectiveness of adsorption filters as a rapid method, qualitative spot test analysis (*sensu* Feigl) was used. To control the content of polycyclic aromatic hydrocarbons in aqueous media and to ensure effective sewage treatment of natural contaminated waters, a luminescent solid-substrate method was applied. This method permits combining the preceding sorption concentration of substances on a cellulose matrix, modified with a surfactant, and analyzing directly in the solid phase of the sorbent. Optimal characteristics of luminescent control of ecotoxicant content before and after water purification were determined. Laboratory studies on water treatment efficiency using multicomponent adsorption filter with retractable cartridges of a model solution including a complex of polluting organic substances (pyrene, chrysene, benzpyrene, resorcinol) allowed identifying optimal filtering media represented by activated silica gel; bentonite, modified with carbon nanotubes and glycerol after heat treatment at 650 °C; bentonite after heat treatment at 650 °C; bentonite, modified with carbon nanotubes after heat treatment at 550 °C. Laboratory studies of water purification efficiency by multicomponent adsorption filter with metallographic electrode system of a model solution, including pyrene, chrysene, benzpyrene, resorcinol, m-aminophenol, o-toluidine, permitted to identify the most efficient filtering media comprising of synthetic zeolite; bentonite modified with carbon nanotubes and glycerol after heat treatment at 550 °C with a gradual temperature increase; organobentonite; bentonite modified with carbon nanotubes and glycerol after heat treatment at 550 °C; activated silica gel; bentonite after heat treatment at 800 °C. The proposed effective sorption water purification systems based on a multicomponent filtering media with luminescent control of ecotoxicant content in resulting solution can be recommended for successful introduction at industrial enterprises, producing wastewaters, as well as at water treatment plants.

Keywords: water quality control, water treatment, luminescent analysis, solid-substrate luminescence, adsorption filter, filtering media.

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Использование многокомпонентных адсорбционных фильтров в системах очистки воды и люминесцентный контроль содержания экотоксикантов

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В работе представлены результаты разработок инновационных конструкций многокомпонентных адсорбционных фильтров: с выдвижными кассетами; с металлографитной электродной системой. Предложенные фильтры позволяют обеспечить очистку воды на 98–99% одновременно от различных загрязняющих органических веществ, в том числе полициклических ароматических углеводородов. В состав фильтрующих загрузок этих фильтров входит бентонит, модифицированный разными способами. Обоснован выбор наиболее эффективных фильтрующих загрузок для разработанных адсорбционных фильтров. Для оценки эффективности адсорбционных фильтров в качестве экспресс-метода использовали качественные капельные реакции по Файглю.

Для контроля содержания полициклических ароматических углеводородов в водных средах и обеспечения эффективной очистки сточных или природных загрязнённых вод использовали люминесцентный твёрдофазный метод, который позволяет сочетать предварительное сорбционное концентрирование веществ на модифицированной поверхностно-активным веществом целлюлозной матрице и анализ непосредственно в твёрдой фазе сорбента. Определены оптимальные характеристики люминесцентного контроля содержания экотоксикантов до и после очистки вод.

Предложенные эффективные системы сорбционной очистки воды на основе многокомпонентных фильтрующих загрузок с люминесцентным контролем содержания экотоксикантов в воде могут быть рекомендованы для внедрения на промышленных предприятиях, являющихся источником образования сточных вод, а также на станциях водоподготовки и очистки воды от комплекса органических загрязнителей.

Ключевые слова: контроль качества воды, водоочистка, люминесцентный анализ, твёрдофазная люминесценция, адсорбционный фильтр, фильтрующая загрузка.

At the present stage of implementing the Federal Target Program “Development of the Water Management Complex in the Russian Federation during 2012–2020”, the main strategic tool is aimed to ensure sustainable development and environmentally safe operation of the water sector. The Government of the Russian Federation assigns to the priority tasks of water sector development the construction and reconstruction of sewage treatment plants complexes in many cities, including Moscow, St. Petersburg, Rostov-on-Don, Yekaterinburg, Ufa, Lipetsk, Petrozavodsk, Nizhny Novgorod, and others. In August 2017, within the framework of the Russian Federation Strategic Development Direction “Ecology”, the priority project “Preservation and Pollution Prevention of the Volga River” was launched. In this regard, ensuring quality water purification in both water supply and sanitation systems is one of the most urgent tasks of our time [1]. However, the solution of this problem is impossible without the establishment of permissible impact on water bodies [2]. Control and management of water quality in water bodies plays an important role due to exceptional importance of water in persistence of both aquatic and terrestrial ecosystems [3].

Providing the required quality of drinking water as well as water for residential use, and compliance with sanitary and environmental quality standards for wastewaters requires monitoring and quality control of water both in water supply sources and at wastewater treatment facilities. Increasing the monitoring quality of water bodies for maintaining proper levels of public health is facilitated by creating new effective technologies for water quality control.

The article is devoted to the problems of water purification and to development of novel technologies for water quality control.

Pollution adversely affects the natural sources of fresh water, which in turn has a negative impact on human health. Water supply presumes mandatory additional water treatment, which can be conducted both at municipal wastewater treatment plants and in residential water treatment systems. Implementation of sewage treatment at various enterprises before discharging wastewaters into sewers or natural water bodies is no less important [1].

Contemporary industrial enterprises do not always provide high-quality cleaning of their wastewaters. Chemical and coke plants; fuel, energy, automotive and other industrial enterprises are potential sources of toxic liquid waste. Particular attention should be paid to aromatic compounds with carcinogenic properties in sewage waters of such enterprises (benzene, its homologs and derivatives, phenols, etc.). For example, there are many phenolic compounds in the effluent of coke plants. Groundwater and surface waters are often contaminated with polycyclic aromatic hydrocarbons (PAHs) due to sewage sludge. Phenolic compounds in general represent a large group of xenobiotics of anthropogenic origin.

Complex composition wastewater treatment is particularly problematic, because number of pollutants is large enough, and it is necessary to provide comprehensive water purification from numerous chemical compounds. It is crucial to identify the composition of all pollutants in the water body, which is not possible without using innovative methods for water quality monitoring.

Currently, there are many methods used for cleaning sewage and contaminated surface waters. All of those have their own characteristics and, depending on the stage of implementation, refer to mechanical, physicochemical, biological, or disinfection methods of waters to be treated.

In connection with circulating water systems, we should note the increases in using physical and chemical methods of sewage treatment, represented by flotation, adsorption, centrifugation, ion exchange and electrochemical treatment, along with hyper filtration, neutralization, extraction, reduction, evaporation, and crystallization.

Adsorption methods have a number of advantages over other existing methods of water purification [2]. The main advantages of these methods include simplicity of technological design, ability to extract heavy metals, PAHs, aromatics and other organic ingredients, including biologically stable substances, which cannot be removed by other methods from polluted waters. Another significant advantage of adsorption techniques is the absence of secondary contaminants.

However, the choice of cleaning method and water quality control requires conducting water analyses. The required stage of water analysis includes determination concentrations of particularly dangerous highly toxic substances, which can potentially cause considerable damage to human health. PAHs occupy one of the leading positions in the list of priority pollutants. Control of PAHs content in various environmental objects is mandatory. Most PAHs are carcinogenic and mutagenic substances that can accumulate in the organisms and pose a hazard even at trace concentrations in the environment [3, 4]. Standard procedures for determination of PAHs in environmental objects are based mainly on chromatographic methods with various detectors [5, 6]. Although the analytical effectiveness of these methods is widely recognized, the PAHs analysis in this case is characterized as laborious, relatively expensive and time-consuming process. For these reasons, it is of great interest to develop simpler and more sensitive methods for the detection and determination of PAHs. Luminescent methods are especially prospective for these purposes [7].

The goal of this study involved determining the optimum characteristics of a luminescent solid-substrate method for monitoring PAHs content in aqueous media to ensure effective purification and quality control of wastewaters and natural waters, as well as development of new technologies for adsorption water treatment from complex composition based on the use of modified bentonites.

Research Methods

Methods for Water Quality Control

We selected the luminescent assay as priority pollution control technique for ecotoxicants in the water. Luminescent methods have a number of advantages for determining the PAHs in aqueous media. First, many of the PAHs representatives are capable of fluorescing, and therefore the use of luminescent methods allows obtaining the best results of the analysis. Secondly, it is well known that these methods are among the most sensitive methods of analysis, which is especially important for identifying trace concentrations of substances in environmental objects.

However, it should be taken into account that the luminescence spectra of the solutions of most organic substances are represented by broad, blurry bands. It is possible to simplify the identification of pollutants via significant narrowing of the spectrum bands, which can be achieved either by significant decrease in temperatures [8] or by using water-micellar solutions [9].

Another effective method for improving the characteristics of radiative processes in molecules is immobilization of a luminophore on a solid substrate [10–13]. This phenomenon is based on the method of solid-substrate luminescence (SSL), which enables combining preceding sorption concentrating and analysis of substances directly in the sorbent phase. Sorption of luminophores is carried out on various luminescent matrices, the most common of which is cellulose matrix [14, 15]. It is well known that signal intensity of SSL luminophores on the cellulose matrix is dependent on the presence of various substances: alkalis, salts, and surfactants. They can significantly reduce or increase the intensity of luminescence. On the basis of luminescent methods, the development of sensors and screening systems for water quality control is conducted [12, 16–18].

Hence, we used the SSL method with analytical signal registration on modified cellulose matrix as the basis for original effective and highly sensitive technique of determining the PAHs.

We took Fluka-manufactured Purum brand pyrene, the least toxic PAHs, as a model compound for our experimental study. The fluorescence spectrum of pyrene was observed in the wavelength range 360–400 nm. As the matrix modifying agent, a cationic surfactant, cetyltrimethylammonium bromide (CTAB) was used. We used cellulose matrix of Red Tape brand for sorption concentrating (solid phase extraction) and applied luminescence research. Sorption of PAHs was carried out dynamically. With this goal in mind, the solution was filtered for 30 min through a sorbent layer in a plastic cartridge. Then the sorbent was dried for 15 minutes at a temperature of 80 °C. PAHs luminescence spectra were recorded on a Perkin-Elmer fluorescence spectrometer LS 55.

Methods for Studying the Effectiveness of Adsorption Systems for Water Purification

Use of various adsorption filters is at the heart of proposed new technologies for sewage and contaminated water treatment.

To investigate the effectiveness of adsorption filters as a rapid method, we used spot test analysis, represented by qualitative chemical reactions. Regarding organic substances, this group of analytical methods was described in detail and systematized by the Austrian chemist F. Feigl [19]. The general principle of spot test reactions is the mixing of a test solution droplet with a drop of a reagent, or several reagents.

In our study we filtered the contaminated solution of a predetermined concentration through the sorbent layer. We sampled the filtrate in small identical portions and conducted the qualitative reactions for the substance in the filtrate until the break point. As a break point, we considered the lower limit of the technique sensitivity, i.e. the amount of polluting substance, at which the reaction turned out to be positive. The most effective was the variant of a sorbent filter loading, at which the break point occurred at the largest volume of the treated solution.

We investigated the efficiencies of the following sorbents:

– No. 1: organobentonite TU 952752-2000, a product of the interaction of montmorillonite clays with quaternary ammonium salts;

– No. 2: bentonite, modified by heat treatment at 650 °C;

– No. 3: bentonite, modified by heat treatment at 800 °C;

– No. 4: bentonite, modified with carbon nanotubes, glycerol and acuteheat treatment at 650 °C;

– No. 5: bentonite, modified with carbon nanotubes, glycerol and gradualheat treatment at 650 °C;

– No. 6: bentonite, modified with glycerol and heat treatment at 650 °C;

– No. 7: bentonite, modified with carbon nanotubes, glycerol andacute heat treatment at 650 °C;

– No. 8: bentonite, modified with heat treatment at 550 °C;

– No. 9: bentonite, modified with carbon nanotubes and heat treatment at 550 °C;

– No. 10: bentonite, modified with carbon nanotubes, glycerol and gradual heat treatment at 550 °C;

– No. 11: bentonite, modified with carbon nanotubes, glycerol andacuteheat treatment at 550 °C;

– No. 12: bentonite, modified with carbon nanotubes, glycerol and heat treatment at 600 °C;

– No. 13: activated silica gel (ASKG);

– No. 14: synthetic zeolite;

– No. 15: peat.

The above sorbents were chosen as the filter loading variants of tested sorption filters (Table).

Findings

Results of Studies on Luminescent Method of Water Quality Control

In the course of our experimental studies on the development of an effective method of water

Table

Characteristics of a complex sorbent

No. of a filter loading option	Top-down sequence of components included in the filter loading option
1	No. 14, 1, 2, 6
2	No. 15, 14, 1, 2, 6
3	No. 13, 4, 2, 9
4	No. 3, 4, 8, 6
5	No. 9, 4, 8, 6
6	No. 14, 10, 1, 11, 13, 3
7	No. 8, 12, 2, 5

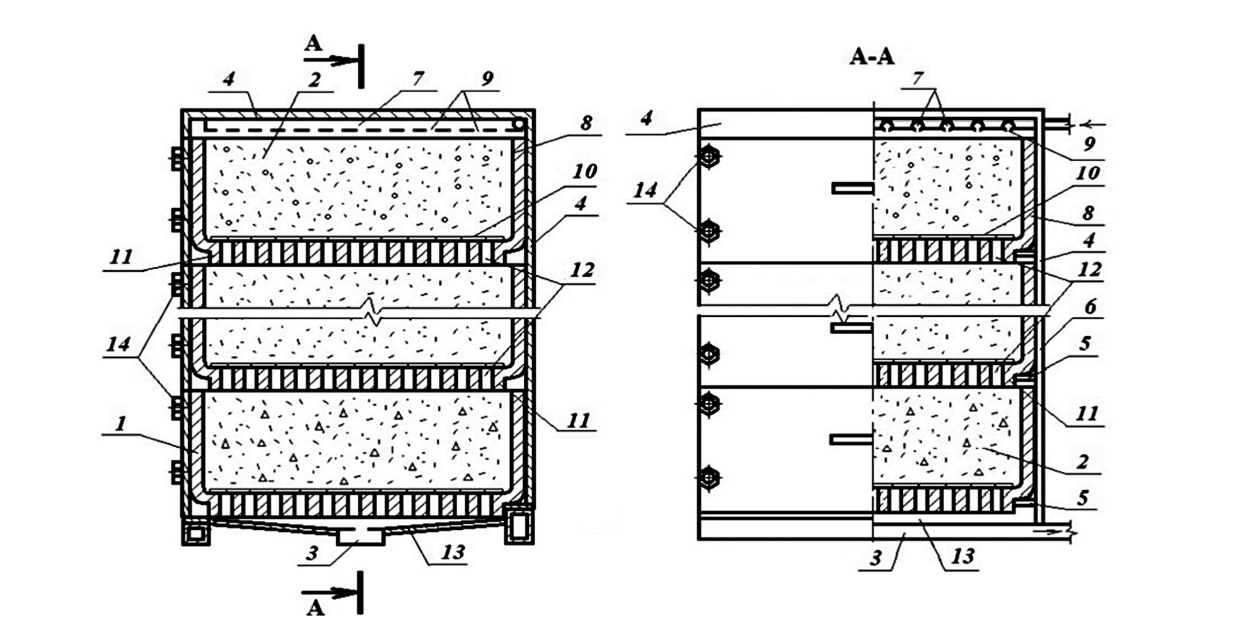

Fig. 1. Multicomponent adsorption filter with retractable cartridges: 1 – cartridges; 2 – sorbents; 3 and 12 –short pipes; 4 – filter housing; 5 – guides; 6 – walls; 7 – tubes; 8 – upper cartridge; 9 – holes; 10 – mesh; 11 – switchgear; 13 – gutter; 14 – screws

quality control for the content of ecotoxicants, we selected optimal characteristics of the luminescent determination of PAHs on a cellulose matrix.

We established that fluorescence intensity of pyrene, the PAHs representative, on the matrix is higher than in the aqueous solution, from which its sorption was conducted. We explain this finding by mobility loss of pyrene molecules during the sorption and by increase in the probability of radiative luminescent transitions (fluorescence) from the first singlet excited state to the ground state.

However, we discovered that an efficiency of sorption by a hydrophilic matrix of hydrophobic PAHs is not high enough. It can be increased by modifying the surface of the matrix with surfactants. It is known that water-micellar solutions in luminescent assay are widely used [9]. However, there are only a few studies, in which surfactants were used to observe luminescence on solid matrices [17]. We have experimentally confirmed that pyrene sorption from water-micellar solutions of surfactants, contributed to concentrating of PAHs in the surface layer of the sorbent. Such modification of the matrix surface allows improving analytical characteristics of the method.

The fluorescence spectra of pyrene on a surfactant- modified cellulose matrix were analyzed. Linear dependences of pyrene fluorescence intensity on surfactant concentrations in the solution were constructed. It was found that maximum fluorescence signal of pyrene on a solid matrix was observed at concentrations of the cationic surfactant CTAB near the critical concentration of micelle formation ($9.5 \cdot 10^{-4}$ M). Limit of pyrene fluorescent detection on the modified cellulose matrix is 4.9 ng/ml.

Novel technologies of water purification and their efficiencies

We developed the design of multicomponent adsorption filter with retractable cartridges for purification of natural polluted waters along with wastewaters with complex contamination (Fig. 1) [20]. The technological process of water treatment is as follows. Water to be treated enters evenly through the holes 9 in the tubes into the upper cartridge, which ensures full operation of the sorbents over the entire area of the cartridges. Passing through the sorbent layer in the upper cartridge, water flows through the mesh at the bottom of the upper cartridge and then through the short pipes 12 of the switchgear into the lower cartridge (Fig. 1). After passing successively through all layers of sorbents placed in the cartridges, purified water is fed to the gutter, and then is supplied to consumers. In the course of filter operation, cartridges with sorbents are inserted all the way into the housing and tightly fastened with screws, which minimizes losses of treated water and increases reliability of the structure.

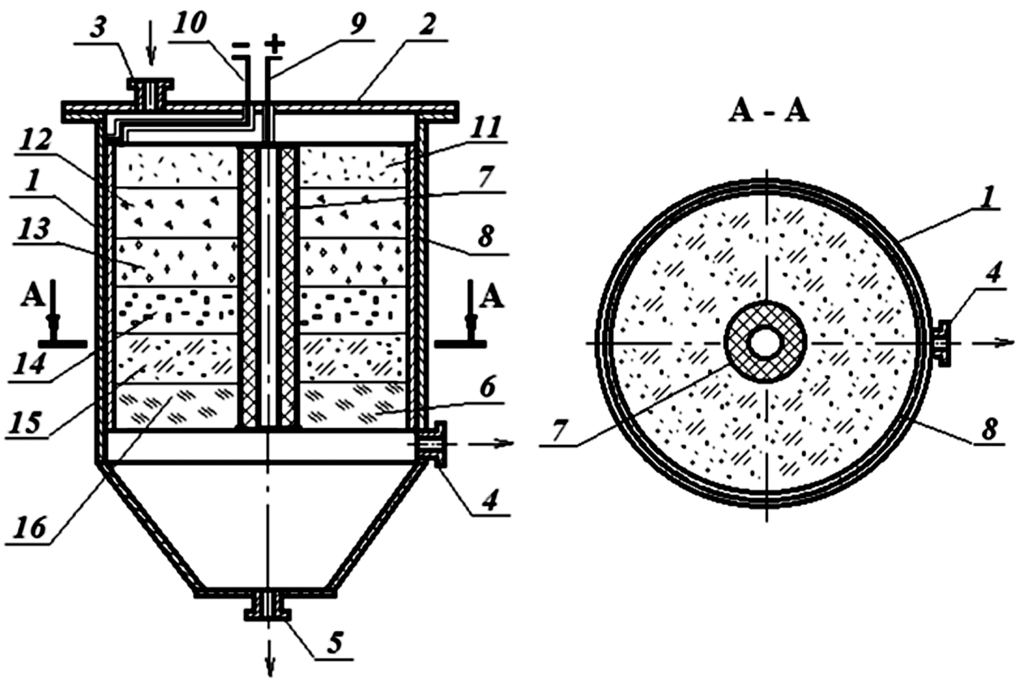

Fig. 2. Multicomponent adsorption filter with metal-graphite electrode system: 1 – filter housing; 2 – cover; 3, 4, 5 – short pipes; 6 – sorbent; 7 – metal-graphite electrode; 8 – steel electrode; 9 – cathode; 10 – anode; 11–16 – layers of a filter loading

Each filter cartridge contains a specific sorbent designed for some particular pollutant, which ensures high quality treatment of natural contaminated and sewage waters of complex composition, including various chemical compounds, such as PAHs.

To improve the adsorption effect, we developed another design of multicomponent adsorption filter, the principal feature of which was the impact of direct electric current system on the sorption (Fig. 2) [21]. The technology of water purification with a multicomponent adsorption filter is as follows.

Water under treatment enters through the auxiliary short pipe 3 into the housing to filter loading and is filtered top to bottom towards short pipe 4. The sorbent located in the electric field between electrodes 7 and 8 is polarized, resulting in polarization of colloidal particles of contaminated water to be filtered. Mutual polarization of the sorbent granules and colloidal particles contributes to high effective adsorption of pollutant ions on the surface of the sorbent granules. Sorbent regeneration is provided by reverse water current. At that time, the voltage on the electrodes is switched off, the auxiliary short pipe 4 is closed, while the auxiliary short pipe 5 is open, and then clean water is fed through the short pipe 3.

Effectiveness of multicomponent adsorption filter with four retractable cartridges was tested on the model solution containing a complex of polluting organic substances (pyrene, chrysene, benzopyrene and resorcinol), allowed identifying the most effective filter loading, providing 98.9% extraction of the pollutants from water. Based on the results of our study, we constructed a diagram presented at Figure 3. It reflects comparison of the efficiencies in five variants of a filter loading.

The adsorption filter, which showed itself most effectively for the model solution, included the loading comprising of ASKG silica gel; bentonite modified with carbon nanotubes and glycerol and heat treatment at 650 ^{o}C; bentonite modified with heat treatment at 650 ^{o}C; bentonite modified with carbon nanotubes and heat treatment at 550 ^{o}C.

Effectiveness of multicomponent adsorption filter with metal-graphite electrode system was tested on the model solution containing a complex of polluting organic substances (pyrene, chrysene, benzopyrene, resorcinol, m-aminophenol and o-toluidine), allowed identifying the most effective filter loading, providing 98.7% extraction of the pollutants from water. Based on the results of our study, we constructed a diagram presented at Figure 4. It reflects comparison of the efficiencies in seven variants of a filter loading. The adsorption filter, which showed itself most effectively for the model solution, included the loading comprising of synthetic zeolite; bentonite modified with carbon nanotubes, glycerol and gradual heat treatment at 550 °C; organobentonite TU 952752-2000; bentonite modified with carbon nanotubes, glycerol and heat treatment at 550 °C; ASKG silica gel; and bentonite modified with heat treatment at 800 °C.

Conclusion

Sorption concentrating in combination with the measurement of an analytical signal on the surface of a sorbent allows reducing the detection limit of polluting components by several orders of magnitude. Using the sorbent modified with the surfactant allows increasing the effectiveness of concentrating the pollutants on the sorbent and sensitivity of the method. It has been experimentally established that the sorption of luminophores on a cellulose matrix modified by micellar nanosystems causes a significant increase in fluorescence signal intensity. Optimal characteristics of a luminescent solid-substrate method for controlling PAHs content in aqueous media are optimally chosen to ensure purification and quality control of sewage or natural waters.

However, it should be noted that it is necessary to take into account possible composition of analyzed waters while using our original highly

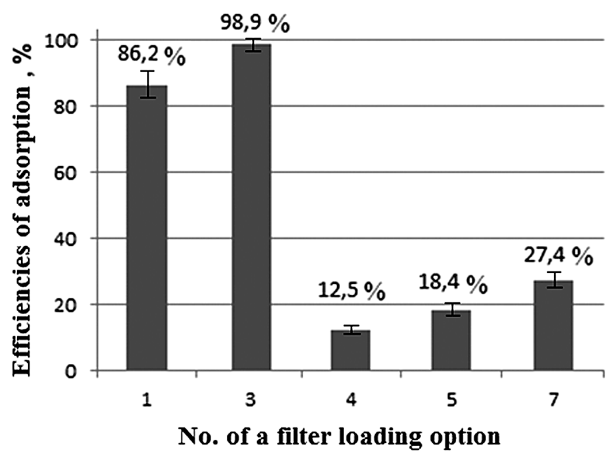

Fig. 3. Efficiencies of multicomponent adsorption filter with retractable cartridges towards the model solution

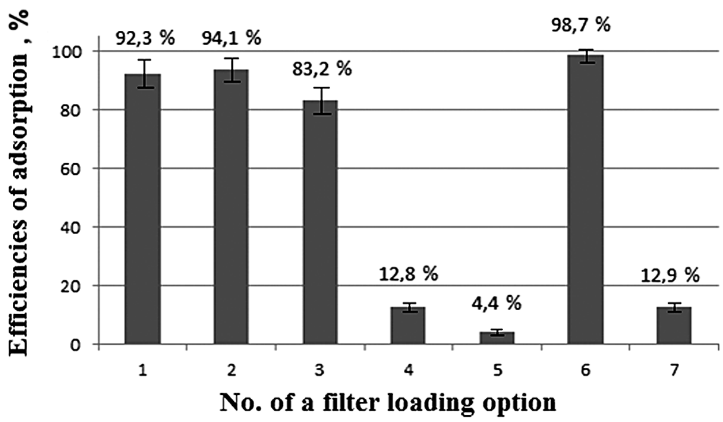

Fig. 4. Efficiencies of multicomponent adsorption filter with metal-graphite electrode system towards the model solution

sensitive luminescent assay, because presence of interfering components (for example, heavy metals) may affect the results of luminescence analysis. In some cases of analyzing highly contaminated water, it is recommended to conduct a routine stage of sample preparation, including preliminary separation of the components and cleaning of the sample. Our future study will be devoted to the development of a solid-substrate luminescent method for determination of heavy metals and their concentrations in polluted aqueous media.

We developed innovative designs of multicomponent adsorption filters for water purification from complex organic [20, 21], allowing to provide a comprehensive treatment of wastewater contaminated with complex organic compounds.

The expediency of using the proposed multicomponent adsorption filters is confirmed by the results of our laboratory studies on their adsorption efficiency towards a complex of organic compounds.

Adsorption efficiency of a multicomponent adsorption filter with retractable cartridges towards a model solution of organic compounds (pyrene, chrysene, benzopyrene and resorcinol) was 98.9% in the case of a complex filter loading comprising of ASKG silica gel; bentonite modified with carbon nanotubes, glycerol and heat treatment at 650 °C; bentonite modified with heat treatment at 650 °C; bentonite modified with carbon nanotubes and heat treatment at 550 °C.

Adsorption efficiency of a multicomponent adsorption filter with metal-graphite electrode system towards the model solution containing pyrene, chrysene, benzopyrene, resorcinol, m-aminophenol, o-toluidine was 98.7% in the case of an integrated filter media comprised of synthetic zeolite; bentonite modified with carbon nanotubes, glycerol and gradual heat treatment at 550 °C; organobentonite TU 952752-2000; bentonite modified with carbon nanotubes, glycerol and heat treatment at 550 °C; ASKG silica gel; and bentonite modified with heat treatment at 800 °C.

Therefore, we can conclude that our proposed original effective sorption system for water treatment, based on a multicomponent filter loading with luminescent control of ecotoxicant water content can be success fully recommended for implementation at industrial plants producing wastewaters, as well as at water treatment and purification stations.

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