Arsenic-containing compounds have a harmful effect on living organisms due to high toxicity. The median half-lethal dose (LD_{50}) of arsenic compounds is in the range of 0.014–0.185 g/kg. But chronic arsenicosis usually begins at doses thousands of times lower than half-lethal doses, acting on the body for a long period of time [1]. Inorganic arsenic-containing substances that enter the human body with drinking water are the greatest threat to human health. Long-term intake of arsenic-containing compounds with drinking water can lead to cancer of the blood, liver, lungs, skin and many other organs. Non-carcinogenic effects include genotoxic action and immunological, neurological and endocrine disorders [2].

The increased arsenic content in artesian waters of Central Europe in the Carpathian Mountains region is due to the leaching of arsenic-containing rocks cropped up from deep layers and can reach several thousand μg/l [3]. Such arsenic-containing natural waters are found in the central and eastern regions of Europe, since deposits of coal, iron ores and non-ferrous metal ores are often accompanied by the presence of arsenic-containing rocks [4]. Despite the close attention of the world community, in many countries of Central and Eastern Europe, studies of arsenic compounds removal are carried out to a very small extent [5].

A significant part of the consequences of arsenic exposure on the body are irreversible, so the main health care measure is to prevent the appearance of arsenic compounds in drinking water. Therefore, there is a need to develop effective and safe technologies for deep water purification from arsenic compounds.

Irrational water resources usage leads to a significant increase in the content of natural organic substances in surface waters, which contributes to an increase in the tap water color index and the formation of carcinogenic disinfection by-products [6, 7]. It should be noted that in natural waters arsenic compounds exist not only in the form of arsenates, arsenites or their methylated forms, but also as complexes with humic substances [8, 9]. Thus, the development of methods for highly efficient removal of As(V) and As(III) compounds from water, including in the presence of humates, is relevant for many countries.
2. Literature review and problem statement

The paper [4] gives information that arsenic is found in soils, rocks and water in oxidation states –3, 0, +3 and +5. As(III) under reducing conditions and As(V) under oxidizing conditions are the stable forms of this element in natural waters.

The leaching of arsenic compounds from minerals, rocks and soils is the main reason for the increased content of this element in natural waters [10]. The maximum permissible concentration of arsenic in drinking water according to the standards is 10 μg/l, while in many countries the arsenic content in natural waters is thousands times higher than this value [11]. Fig. 1 shows the map of the probability of geogenic arsenic contamination in groundwater.

According to Fig. 1, the probability of increased natural arsenic content in groundwater exists in many countries, which makes the arsenic compounds removal an urgent task in water treatment. In such areas, arsenic level in groundwater can often reach 40 mg/l, which is 4,000 times higher than the maximum allowable arsenic content in drinking water [5].

The paper [12] presents information that artesian waters with high arsenic concentrations and mineralization are typical for a number of areas in Central Europe. Namely: mineralization of about 2–5 g/l with an arsenic content of 0.7–5.0 mg/l, mineralization above 30 g/l with an arsenic content of about 100 mg/l, etc. Ground waters of the mining regions, especially gold deposits, are also requiring attention [1].

Methods for arsenic removal from water include: precipitation (coagulation, reagent softening), ion exchange, adsorption, baromembrane methods (microfiltration, ultrafiltration, reverse osmosis), but adsorption is the most common treatment method [13].

Different sorption materials are used for the adsorption removal of arsenic-containing substances. The adsorption efficiency is highly dependent on the type of adsorbent and pollutant, water pH, temperature, etc.

Activated carbon is one of the most used adsorbents for arsenic-containing substances removal from water [14]. But when activated carbon is used, organic functional groups can be washed off from its surface, which increases the organic content in water, and this is undesirable due to the possibility of by-products formation at the disinfection stage. According to [15], when iron oxide is introduced into the activated carbon structure, it shows significantly better results than unmodified carbons. The paper [16] shows that the use of cetylpyridinium chloride-modified activated carbon powder can successfully remove arsenate ions from water. In this case, the maximum amount of adsorbed arsenate is 0.087 mmol/g. However, cetylpyridinium chloride is a toxic organic compound, and therefore wastes after the synthesis of this sorbent require deep treatment, because they can cause significant harm to aquatic organisms.

Iron oxides and hydroxides are also common sorbents for natural water purification from arsenic-containing pollutants. These compounds demonstrate high results and do not require dosage of additional oxidizing agents [1]. However, granular iron-containing sorbents do not make it possible to fully use all adsorption material due to blocking of the reaction surface by poorly soluble iron arsenates. According to [2], maghemite (γ-Fe₂O₃) nanoparticles show high efficiency in the process of As(V) compounds adsorption removal from the aqueous medium: the maximum adsorption reaches 50 mg/g. In the case of nano-scale adsorbents usage, there is a problem of their complete removal from water [17], since the intake of solid nanoparticles via drinking water consumption can lead to numerous health problems.

The promising sorbents for water treatment include Dowex SBR-P anion exchange resin, whose matrix contains tin(IV) and iron(III) oxides [11]. This material has a better arsenic compounds adsorption than commercial arsenic removal sorbent samples. The sorbent simultaneously functions as an anion exchanger, a sorbent, and an oxidizing agent due to modifying by tin(IV) and iron(III) oxides. But during adsorption, toxic tin compounds can enter the treated water. Also, according to [18], Dowex ion exchange resins modified with group IV hydroxides have a high adsorption capacity for arsenate ions (63–82 mg/g), but the high cost of raw materials significantly increases the cost of the adsorbent.

![Fig. 1. Map of the probability of increased arsenic content in groundwater [10]](image-url)
Materials of natural origin are used for the adsorption of arsenic-containing substances from natural waters: zeolites, active aluminum oxides, etc. However, all these sorbents usually require additional dosage of oxidizing agents to convert the As(III) form into As(V) [19]. According to [15], the study of the following sorbents is promising: hydroxides, oxhydroxides and oxides of iron(III), magnetite, aluminum oxides, chitosan-containing sorbents, CuO, metallic iron, zirconium compounds, TiO₂, MnO₂, activated carbon, industrial wastes. These compounds are used not only as individual sorbents, but also as components of new combined sorbents [19]. The following systems are of scientific interest: chitosan-based TiO₂, porous silicon oxide modified by bis (3-triethoxysilylpropyl) tetrasulfide, hydrogel-carbon composite, zirconium-modified activated carbon fibers, zeolite-graphene composites [20]. A separate array of studies is devoted to iron-containing sorbents: chitosan base with double Fe-Mn oxide, CeO₂/Fe₂O₄ nanosorbent, magnetite-modified natural clinoptilolite-type zeolite, Fe-sericite composite, iron-doped multilayer carbon nanotubes [21]. New effective adsorbents are often expensive due to a complex synthesis process, expensive consumables, or multiple components (nano-dispersed metallic iron, doped carbon nanotubes, zirconium-, cerium- and yttrium-containing sorbents, etc.). The disadvantage of such adsorbents is the possibility of water contamination with heavy metals or other toxic components, which are included in these sorbents (manganese- and tin-containing adsorbents, etc.). It is also known that chitosan- and alginate-based sorbents can be hydrolyzed and pollute water with organic substances, and nano-dispersed sorbents are difficult to separate from water.

Based on the above, a combination of adsorption by a fine iron-containing sorbent with subsequent microfiltration or ultrafiltration membrane separation of spent sorption materials looks promising in the context of arsenic removal from the aqueous medium.

3. The aim and objectives of the study

The aim of the work was to develop a method for obtaining effective iron-containing sorption materials for removing arsenic compounds from the aqueous medium and a sorption-microfiltration method for removing arsenic (III and V) from natural waters. To achieve the aim, it was necessary to solve the following tasks:

- to study the removal of arsenic from the aqueous medium by synthesized powder iron-containing sorbents;
- to study sorption activated carbon-based granular material for the removal of arsenes from water;
- to determine the efficiency of removal of arsenic-containing substances from model aqueous solutions using suspension iron-containing sorption materials (ISM);
- to develop a flow sheet for using suspension iron-containing sorption materials to remove arsenic compounds from the aqueous medium.

4. Materials and methods of synthesis and study of the effectiveness of iron-containing sorption materials

4.1. Synthesis of sorption materials

Iron-containing powder sorbents were synthesized in several ways. According to the first method, sorption materials were obtained by precipitation of iron hydroxide from concentrated FeCl₃ solution with 10% NH₄OH solution at pH 10. Then the obtained precipitate was dried at 105°C for 10 hours and calcined at appropriate temperatures (from 150°C to 900°C) for 1 hour (PFA series). In the second case, the sorbent was synthesized by precipitation with 10% NH₄OH solution from concentrated FeCl₃ solution at pH 10 and subsequent drying at lower temperatures (from 18°C to 75°C) (PFU series). According to the third method, the sorbent was obtained by precipitation of iron hydroxide from FeCl₃ solution using thermal hydrolysis of urea and subsequent drying at 105°C for 10 hours (PFU series).

The synthesis of the sorbent (activated carbon/iron (III) oxyhydroxide) was carried out as follows: 50 g of urea was dissolved in 200 ml of distilled water, weighed portion of activated carbon (10 g) and FeCl₃ solution (equivalent to 3 g of salt) were added. Then, with constant stirring, it was heated to 95°C and boiled for 30 minutes. During the process, the following reaction took place:

\[
2(NH₂)₂CO+FeCl₃+4H₂O \rightarrow \rightarrow FeOOH+3NH₄Cl+NH₃+2CO₂.
\]

After the end of the synthesis, the mixture was cooled and the obtained sorption material was washed several times with distilled water until the Cl⁻ ions disappeared in the filtrate. Then the sorbent was dried at a temperature of 105°C in a drying oven for 8 hours.

The synthesis of suspension iron-containing sorption materials of SFU series was carried out according to the procedure: 150 g of urea was dissolved in 200 ml of distilled water, iron(III) chloride solution with a concentration of 0.6 mol/l was added. The obtained mixture was heated with constant stirring to a temperature of about 95°C and boiled at this temperature. The ratio of urea and FeCl₃ was varied from 5 to 15, and the heat treatment time was varied from 15 to 60 minutes.

4.2. Method of sorption removal of arsenic-containing pollutants from model solutions

For the sorption removal of arsenic-containing compounds, 0.05 g of powder, 0.1 g of activated carbon-based granular material and 0.011 g of suspension sorption material were weighed and placed in conical flasks together with 200 ml of water, which was treated. The flasks were placed on a shaker. Sorption times were varied to 5, 10, 20, 40, 90, and 120 minutes. At the end of the sorption, the water samples were filtered; the pH value was measured, and the samples were prepared for analysis.

4.3. Method of obtaining adsorption isotherms of arsenic compounds in model solutions

Conical flasks with a volume of 250 ml were filled with 200 ml of the prepared solution with different concentrations for each of arsenic-containing substances. The adsorbent was dosed into the prepared solutions (0.05 g for powder; 0.1 g for activated carbon-based granular material, 0.005 g for suspension sorbents). After achieving equilibrium, the contents of the flasks were filtered and analyzed for arsenic content by photometry using Systea EasyChem (Italy).

4.4. Determination of As(V) content in the aqueous medium using Systea EasyChem

For the determination of As(V) concentration, the standard Systea EasyChem method for the analysis of ortho-
phosphate in water was improved. Working solutions were prepared according to the following procedure: reagent 1 – a mixture of ammonium molybdate solution, antimony potassium tartrate solution, and 5 N sulfuric acid solution; reagent 2 – ascobic acid solution. Determination of As(V) in the concentration range from 2 μg/l to 200 μg/l by Systea EasyChem was carried out with the programmed volume parameters: sample volume – 550 μl, reagent 1 volume – 100 μl, reagent 2 volume – 20 μl. Time parameters also were varied: incubation time after adding reagent 1 – 34 s, incubation time after adding reagent 2 – 544 s, stabilization time – 26 s, measurement time – 3 s. The studies were carried out at a wavelength of 880 nm.

4.5. Capillary Suction Time (CST) test
The study of the ability of the adsorbent suspension to hold water was carried out using the CST test. For this, the adsorbent suspensions were diluted to a concentration of 10 g/l in terms of dry matter. The diluted suspension with a volume of 5 ml was poured into a funnel fixed on a special absorbent filter paper, which was sandwiched between two non-absorbent blocks. The period of time between the first and second contacts of the liquid in the paper with electrodes was determined using the CST timer.

5. Results of research on arsenic removal from water by powder, activated carbon-based granular material, and suspension sorbents and the development of a flow sheet

5.1. Sorption removal of arsenic-containing pollutants from model solutions using sorbents of PFU and PFA series
As a result of sorption experiments, it was found that sorbents of PFU series, obtained by heterogeneous precipitation at different heating temperatures, demonstrate similar efficiency in the removal of arsenate ions. Fig. 2 demonstrates that samples with drying temperatures from 75 °C to 150 °C have almost the same adsorption efficiency of arsenate ions as samples with higher heating temperatures. This fact testifies to the uselessness of energy consumption for calcining samples at temperatures above 150 °C.

5.2. Kinetic study of As(V) removal process
To understand the dependences of the adsorption process, it is necessary to choose the correct model that adequately describes the kinetics of this process. Thus, the obtained data on arsenate ions adsorption from model solutions were transformed into equations of various models (zero, first, second, and pseudo-second orders).

As a result of sorption experiments, it was found that the kinetics of adsorption of arsenate ions by iron-containing sorption materials is most promising sorption material, and it is advisable to choose sorbents of PFU series for further syntheses and studies.

The calculated rate constants for FeOOH change in the following order: \( k_{\text{NaCl}} > k_{\text{NaSO}} > k_{\text{NaHCO}} \) (Table 1). In all cases, the coefficients of determination are close to 1, which also confirms the pseudo-second-order model of arsenate ions adsorption by iron(III) oxyhydroxide (sorbent of PFU series).

It should be noted that for all three tested sorption materials of PFA and PFU series (oxide, hydroxide, and oxyhydroxide sorbents), the process of arsenate ions adsorption is adequately described by the pseudo-second-order model.
A number of rate constants for arsenate adsorption by amorphous hydroxide and oxide sorbents generally demonstrate similar trends. As shown in Fig. 4 and Table 1, arsenic is most efficiently removed by oxyhydroxide sorbent (FeOOH) in the model solution without additives and least efficiently in the presence of hydrocarbonate ions.

### Table 1

| Model solution | Rate constant \( k \), g/(μg·min) | Coefficient of determination \( R^2 \) |
|----------------|----------------------------------|--------------------------------------|
| Without additives | 0.101                            | 0.999                               |
| With NaCl       | 0.083                            | 0.998                               |
| With Na\(_2\)SO\(_4\) | 0.077                            | 0.996                               |
| With NaHCO\(_3\) | 0.032                            | 0.997                               |
| With CaCl\(_2\) | 0.108                            | 0.997                               |

5.2. Sorption removal of arsenic-containing pollutants from model solutions by activated carbon-based granular material

To create sorbents based on activated carbon, BAU (Russia), KAU (Russia), Filtrasorb F300 and Filtrasorb F400 (USA) were used. As a result of their modification with iron(III) oxyhydroxide, all samples showed similar adsorption efficiency toward arsenate ions (initial arsenic concentration was 500 μg/l). The sample based on BAU had an insignificant superiority in adsorption characteristics in contrast to other samples (Fig. 5), so BAU was chosen for further studies.

It is known that the oxidation of activated carbon with various oxidizing agents makes it possible to increase the content of oxygen-containing groups on its surface. This contributes to both physical and chemical fixation of iron(III) oxyhydroxide on the activated carbon surface. Oxidation was carried out with hydrogen peroxide and potassium permanganate solutions with constant heating and stirring. On the basis of oxidized BAU samples, iron-containing sorption materials were synthesized by modifying with iron(III) oxyhydroxide. The results of arsenate removal from water by oxidized BAU-based sorption materials are shown in Fig. 6.

It can be seen from Fig. 6 that all composites are effective against arsenic, among which the most effective is modified BAU-based sample, in which BAU was oxidized with potassium permanganate. The arsenic removal using this sample is 89 %, which is 51 % more than unmodified carbon, and 2 % and 3 % more than other modified samples. Nevertheless, additional oxidation has a very insignificant effect on the sorption activity of the synthesized iron-containing sorption materials relative to As(V) compounds.

ISM based on activated carbon showed approximately 2 times lower efficiency than PFA and PFU series sorbents (powder iron(III) oxide, iron(III) oxyhydroxide and amorphous iron(III) hydroxide).

Thus, we can conclude that it is reasonable to use iron-containing sorption materials not as a component of granular composite ISM, but as an individual finely dis-
persed phase. It should be noted that the finely dispersed iron-containing sorbents are difficult to separate from the aqueous phase due to their high stability (the suspension remains stable for at least 5 days). For this reason, it is advisable to separate the spent finely dispersed iron-containing sorbent from water using filtration membranes.

5.3. Suspension sorption materials of SFU series

It is known that during drying, aggregation of synthesized particles occurs. So, to use sorbents in the form of finely dispersed powder after heat treatment, a grinding stage is required. However, mechanical comminution produces sharp-angled particles that can damage the membrane surface during separation. Thus, mechanical comminution is undesirable, and therefore the drying step itself is also unwanted. Therefore, it is proposed to store and use the synthesized iron-containing sorbent in the form of a suspension.

According to the data of preliminary studies [7], the sorption materials of SFU series are characterized by particles of spherical shape and almost the same size, which is positive when used in the sorption-membrane treatment method. This homogeneity of the formed particles is explained by the fact that in the case of homogeneous precipitation, the particles are slowly formed from a solution with an equable concentration of the precipitant and are not further subjected to any destructive processes.

One of the most important characteristics in selecting a suspension sorbent for use in combination with a membrane is the ability of the sorbent to retain water, since this significantly affects the speed of the treatment process. For using sorbents in the sorption-membrane treatment method, it is advisable to choose a sorption material with a minimum water retention time. In order to determine the water retention ability for the sorbents of SFU series, the CST test was carried out.

According to the CST test, the most promising sorbents are SFU (8/30) and SFU (8/60), which differ only in heating time: 30 or 60 minutes (Table 2). Since they demonstrate almost the same result (63.7 s and 61.3 s), in order to reduce the sorption material cost, it is advisable to choose a sorbent at a lower processing temperature – 30 minutes (SFU (8/30) sample).

The efficiency of SFU series adsorbents in relation to arsenate and arsenite ions was determined on model solutions with different arsenic contents (500, 2000, 4000, 10000, 12000, and 15000 μg/l). The adsorbent dose was selected according to previous studies [7] and amounted to 50 mg/l. All SFU series samples showed similar sorption properties against arsenate and arsenite ions (Fig. 7).

5.4. Flow sheet of the use of SFU series suspension sorption materials

Proceeding from the fact that SFU (8/30) is the most rational sorption material, a water treatment flow sheet with its use is proposed. In this case, a new approach to the removal of waste sorption materials is implemented (Fig. 8). The SFU (8/30) sorbent (suspension iron(III) hydroxide), synthesized according to 4.1, is used in the flow sheet.

According to the flow sheet, the treated natural water is mixed with the sorbent suspension in mixer 2 (contact time 30 minutes). After that, the spent finely dispersed sorbent particles are separated on microfiltration membranes 3, and the treated water goes into reservoir 5. The proposed flow sheet is effective for removing both arsenic compounds and spent finely dispersed sorbent.

| Sample     | CST, s |
|------------|--------|
| SFU (15/30)| 98.4   |
| SFU (10/30)| 93.9   |
| SFU (8/30) | 63.7   |
| SFU (10/15)| 97.5   |
| SFU (10/60)| 88.2   |
| SFU (8/60) | 61.3   |

As can be seen from Fig. 8, SFU series sorbents demonstrate approximately the same high efficiency in removing arsenic from model solutions with different initial concentr
6. Discussion of the results of the studies on the effectiveness of iron-containing sorption materials

The advantage of this study is that the developed sorption materials are effective toward arsenic compounds at their relative cheapness. The disadvantages include the need to develop a method for recycling spent sorbent for further usage.

The developed sorption materials can help in solving such important problems as hygiene and health care, since arsenic compounds are one of the most dangerous inorganic pollutants of natural waters. The work considered various types of sorption materials and experimentally selected the most effective samples.

According to the studies, carbon-based adsorbents showed approximately 2 times lower efficiency (Fig. 5, 6) than PFA and PFU series sorbents (Fig. 2–4). The samples of SFU series showed a significantly higher sorption activity (Fig. 7, 8). Therefore, the SFU series is the most promising for arsenic removal from the aqueous medium.

A simple and effective way to separate finely dispersed adsorbents from water is to use microfiltration membranes. SFU series sorbents not only show high efficiency in the removal of arsenic compounds, but, according to previous studies [7], they demonstrate the ability to remove humates from water and protect the microfiltration membrane from colloidal fouling.

As a result, the finely dispersed sorption material properties were fully implemented in the developed flow sheet (Fig. 8). In mixer 2, the adsorption of arsenic compounds on sorbent particles is carried out, and mixing helps to move the adsorption process from the external diffusion region to the kinetic one. The pore size of the microfiltration membrane 3 does not allow the sorption material particles to be taken out with treated water, and they form a protective layer on the membrane surface. As a result, natural organic compounds, which are the reason for the formation of colloidal fouling, are unable to form a gel layer on the membrane surface due to the protective sorbent layer. During backwashing, the spent sorbent is removed from the membrane surface, leaving it clean even without the use of special reagents for enhanced backwash.

The proposed flow sheet is a promising way to improve water treatment systems in regions with a high content of arsenic compounds in natural waters. In the future, it is planned to study the synthesized sorbent on various real natural waters and develop methods for utilizing the spent sorbent in order to convert the technology into a “green” and waste-free one.

7. Conclusions

1. It is shown that powder sorbents based on iron-containing materials are effective in relation to arsenate ions, while iron(III) oxyhydroxide is the most active in removing arsenic compounds from water. It was determined that the kinetics of adsorption of arsenate ions on iron(III) oxides, iron(III) oxyhydroxides, and iron(III) hydroxides is described by the pseudo-second-order model.

2. It was found that iron-containing sorption materials based on activated carbon showed approximately 2 times lower efficiency than powdered iron(III) oxide, iron(III) oxyhydroxide and amorphous iron(III) hydroxide.

3. Analysis of the results of capillary suction time test and adsorption of arsenic compounds made it possible to choose rational parameters of obtaining the suspension sorbent by homogeneous precipitation, which is highly active to arsenic compounds. In this case, the rational parameters are: the ratio of urea to iron(III) chloride is 8:1; the boiling time is 30 minutes. It was experimentally found that suspension sorbents are effectively removed using microfiltration membranes. In addition, they are effective not only for removing arsenic compounds, but also humic substances, which confirm the prospects of using finely dispersed iron(III) oxyhydroxide in the context of the sorption-membrane method of natural water treatment.

4. On the basis of the results obtained, the flow sheet for the use of suspension iron-containing sorption materials

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**Fig. 8. Flow sheet of water treatment:**

1 – sorbent suspension storage tank; 2 – mixer; 3 – microfiltration membrane; 4 – tank with suspension after membrane backwash; 5 – treated water storage reservoir
for the removal of arsenic compounds from natural waters is proposed. It is shown that the removal of arsenic compounds from natural water to sanitary standards (10 μg/L) is advisable to carry out by the adsorption method using suspension oxihydroxide sorbent particles, followed by separation of the spent sorbent using microfiltration membranes.

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