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Purification of hot water by zeolite modified with manganese dioxide

Очистка горячей сетевой воды цеолитом, модифицированным диоксидом марганца

A.V. Chechevichkin,
N.I. Vatin,
Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia
V.V. Samonin,
St. Petersburg Institute of Technology (Technical University), St. Petersburg, Russia
M.A. Grekov,
Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia

Инженер А.В. Чечевичкин,
д-р техн. наук, директор Инженерно-строительного института Н.И. Ватин,
Санкт-Петербургский политехнический университет Петра Великого, г. Санкт-Петербург, Россия
д-р техн. наук, заведующий кафедрой В. В. Самонин,
Санкт-Петербургский государственный технологический институт (технический университет), г. Санкт-Петербург, Россия
главный инженер М.А. Греков,
Санкт-Петербургский политехнический университет Петра Великого, г. Санкт-Петербург, Россия

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Ключевые слова: горячая сетевая вода; природный цеолит; диоксид марганца; сероводород; здания; сооружения; энергетическая эффективность

Abstract. A new material is proposed for complex purification of hot water from iron and hydrogen sulphide – natural zeolite modified by manganese dioxide. It has been shown experimentally that this filtering material has high efficiency of water purification from iron and hydrogen sulphide, low mechanical degradability during operation, as well as low water consumption for washing during regeneration of the filtration media. The complex cleaning of hot water is proposed to carry out by means of two stages: the first – filtration cleaning from insoluble iron compounds with linear water velocity of 10–12 m/h, and the second – oxidation-catalytic purification from hydrogen-sulphide with velocity 1–2 m/h. Regeneration of a filtering material based on a manganese-modified natural zeolite can be carried out by back washing and chemical treatment with an oxidizer solution.

Аннотация. Для комплексной очистки горячей сетевой воды от железа и сероводорода предложен новый материал – модифицированный диоксидом марганца природный цеолит. Экспериментально показано, что данный фильтрующий материал имеет высокую эффективность очистки воды от железа и сероводорода, низкую механическую разрушаемость в процессе эксплуатации, а также малый расход воды на промывку при проведении регенерации загрузки. Предложено комплексную очистку горячей сетевой воды проводить в два этапа: первого – фильтрационной очистки от нерастворимых соединений железа с линейной скоростью движения воды 10–12 м/час, и второго – окислительно-каталитической очистки от сероводорода со скоростью 1–2 м/час. Регенерация фильтрующего материала на основе модифицированного диоксидом марганца природного цеолита может проводиться методом обратной промывки и химической обработки раствором окислителя.

Introduction

Installations and systems for preparation and transportation of hot water, as well as for distribution to use objects form a technical base of network hot water supply of buildings and structures.

Hot water systems are inextricably linked to the heat supply systems of buildings and structures. There are closed and open heat supply systems [1, 2], whose equipment for various reasons undergoes severe corrosion. For a closed system, corrosion reduction is achieved by addition various chemical reagents into the water and minimizing the entry of oxygen into the water [1, 3–6].

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Closed heat supply system provides higher energy efficiency of heat supply and hot water supply of buildings. Recently, closed heat supply systems [7, 8] have been used for the purposes of hot water supply [7, 8], in which cold drinking water is heated in heat exchangers of local heat points in buildings (including residential ones).

Both in closed and open systems of hot water supply [7, 9], due to a variety of physicochemical and microbiological processes [7, 9], there is a significant contamination of water with corrosion products [1], which can make it unsuitable for both practical use . Water contamination with corrosion products is very significant [3] and therefore complex cleaning of network hot water is currently extremely urgent.

One of the main measures to reduce corrosion in hot water and heat supply networks is to reduce dissolved oxygen in the water. This leads to the creation of anaerobic conditions in which microbiological corrosion plays an important role.

Anaerobic bacteria (mainly from Desulfovibrio and Desulfomaculum [1, 10]) intensively develop in anoxic environment at pH 5-9 when sulphates, organically substances and phosphorus are present in water (especially at elevated temperature) and hydrogen sulphide is released [1, 10].) Sulphure, in turn (in the form of hydrosulphide ions) is involved in the formation of an insoluble iron (II) sulphide corrosion product having a loose structure. This structure facilitating the development of sulfate-reducing bacteria is the basis for the formation of massive deposits in the pipelines and equipment elements. Even more anaerobic conditions are created between the surfaces of the metal and deposits, which ensure a high rate of microbiological corrosion, not only of steel, but also of aluminum and its alloys and even of brass [2, 12].

Corrosion products (mainly insoluble iron compounds) not only reduce the efficiency of the use of heating equipment, but also greatly impair the quality of the hot water consumed. This making hot water less suitable for use in domestic and industrial uses [12].

There are methods of removing iron from waters of various classes [13–20] which are not use for cleaning network hot water for various reasons:

- addition of chemical reagents (treatment with oxidants and coagulants) is unacceptable when water is disassembled by consumers;

- mesh filters, hydrocyclones and magnetic separators have low cleaning efficiency;

- fibrous filter media are difficult to regenerate.

The most suitable way, from the point of view of the ratio of cleaning efficiency, cost and ease of operation, is the filtration of hot water through the feed of fine-grained materials. These materials (quartz sand, natural zeolite, etc.) have high adhesion to suspended and insoluble iron compounds (iron III hydroxide, carbonate and iron sulphide II). Filtering through these materials allow to reduce such indicators of contamination of hot water, as turbidity, color and iron concentration, but practically does not affect the water content of substances that cause its unpleasant smell (primarily hydrogen sulphide).

To purify waters from various pollutants, natural zeolite is widely used [21–29], and filtering materials based on which have a number of advantages over traditionally used quartz sand loads: low bulk density, which helps to reduce the water consumption for regeneration by backwashing, high mud capacity (i.e., the amount of obtain suspended matter per unit volume of the layer), ion exchange capacity, etc.

Hydrogen sulphide have a high toxity to animals and humans, and high corrosive activity to plumbing equipment [1, 30]. In blood hydrogen sulphide is rapidly redused the oxidized power of haemoglobin and can act upon the central nervous system. Humans exposed to high concentration of hydrogen sulphide show simptoms of gastro-intestinal upset, anorexia, nausea, amnesia, delirium, hallucinations, low blood pressure and epileption convulsions [30, 31].

There are several techniques for removing the hydrogen sulphide from different types of water, including, aeration, ozonation, ion exchenge, reverse osmosis, biological treatment and chemical oxidation [11, 13, 14, 25, 31–34].

This methods of water purification from hydrogen sulphide and other sulfur compound (except reverse osmosis and ion exchange, witch are extremely expending), are not also use for hot water supply, since they are associated with the dosage of reagents in water and the removal of chemical reaction products, as well as with technical difficulties.

It is very promising to use filter materials based on natural manganese-containing ores (mainly pyrolusite) for this purpose [11, 34], but these materials have low mechanical strength and they are very expensive.

Recently, filtering materials modified by manganese dioxide [35–43], including those based on natural zeolites [44–46], have been developed and are being used for deionization and demanganation of natural waters. Such materials combine the advantages of manganese dioxide (high oxidation-catalytic capacity) and natural zeolite (good filtration and mechanical characteristics). The use of these materials for the complex (filtration-catalytic) purification of hot water from the compounds of iron and hydrogen sulphide is very promising.

The purpose of this work was testing of a modified by manganese dioxide natural zeolite for complex cleaning of network hot water. For its implementation the following tasks were solved:

- comparison of physical and chemical properties and performance characteristics of various granular filter materials;

- determination of parameters of hot water treatment from iron and hydrogen sulphide by various granular materials, including materials modified by manganese dioxide;

- study of the optimizing possibility of the technology of complete cleaning of hot water in real operation conditions.

Methods

Experimental studies of the processes of cleaning hot water were carried out on the installation, the scheme of which is presented in Fig.1.

The installation considered of a cylindrical stainless steel filter, having drainage slottedflow distributorsin the upper and lower parts of the filter.

Filtering granular materials were loaded inside the filter housing to a height of 750 mm, with a 250 mm height space provided for top loading for regeneration by backwashing. The filter had a water flow control system, in the forward (up-down) and reverse directions, consisting of the corresponding tapes, valves for precise adjustment of the flow rate of water during operation and during washing, as well as water sampling tapes for analysis before and after purification.

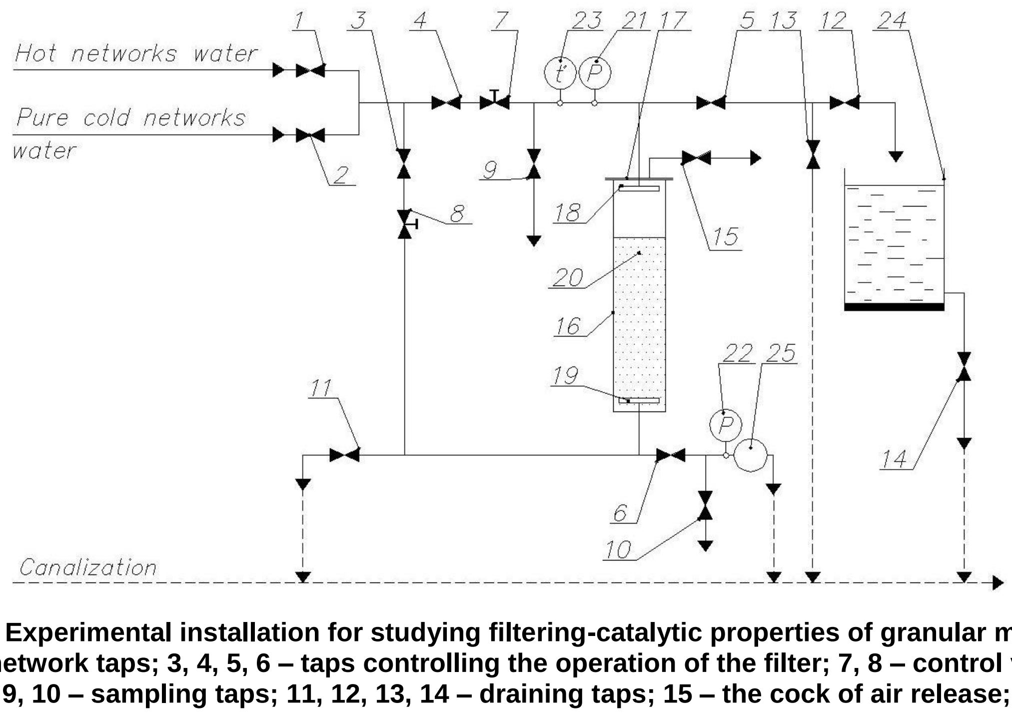

Figure 1 Experimental installation for studying filtering-catalytic properties of granular materials. 1, 2 – network taps; 3, 4, 5, 6 – taps controlling the operation of the filter; 7, 8 – control valves; 9, 10 – sampling taps; 11, 12, 13, 14 – draining taps; 15 – the cock of air release; 16 – filter housing; 17 – removable filter cover; 18, 19 – upper and lower slotted distributors; 20 – filtering material; 21, 22 – model manometers; 23 – thermometer; 24 – tank for collection of wash water; 25 – flowmeter

What is more, the unit was equipped with a thermometer to monitor the water temperature, a flowmeter and standard manometers to monitor the pressure before and after the filter. Moreover the installation had a tank 200 dm^3 volume to collect water during the backwashing of the filter. The accuracy of the determination was: the volume of the transmitted water ± 0.5 x 10^{-3} m^3, temperature ± 0.5 °C, pressure ± 0.01 bar.

The linear velocity of water flow through the filter media was the same in all experiments and was 10 ± 0.4 m/h. The linear velocity of backwash V_{bw} (m/h) for each filter material was defined as the rate of start of the entrainment of the particulate material in a separate experiment with a visual control of the light opening in the glass column.

Filtering granular materials were used in the form of identical fractions of 0.8 ÷ 1.2 mm, which were obtained with the help of a set of appropriate certified sieves and analyzer AP-20 (manufactured by "Vibrotechnik", RF).

Loss of materials during the backwashing was determined after passing through the filter loaded (in the direction from bottom to top) 120 liters (≈ 22 filter volumes) of cold purified tap water that was collected in a suitable tank (see Fig. 1) and settled in it within three days. After draining the water from the vessel, the precipitate was removed, concentrated, dried at a temperature of 70 ± 5 °C and weighed. The losses in washing P, (% mass) were determined by the formula:

$$P = \frac{m1}{M1} \cdot 100\% \quad (1)$$

where m1 – mass of air-dry sludge, g

M1 – mass of air-dry granular material in the filter, g

The dirt capacity of the burden layer of granular material GE (g / kg, mg / dm3) was determined similarly after the filter operation and washing were completed and calculated by formula (1).

The mechanical strength of the samples of granular materials was estimated as the mechanical destructibility of MD, (% mass), which was determined by the formula:

$$\mathrm{MD} = 100\% - \mathrm{MR}\,(\mathrm{s}) \quad (2)$$

where MR (s) – mechanical abrasion resistance determined according to Russian State Standard GOST 16188-70 4 [47].

The iron content in the filtration media of the granular material, C_{Fe},, (mg / g) was calculated after processing of its sample with 20% nitric acid and the determination of the total iron content in the resulting solution by the photocolorimetric method with thiocyanate ion [48] using the formula:

$$\mathrm{C_{Fe}} = \frac{m2}{M2} \quad (3)$$

where *m2* is the mass (mg) of dissolved iron in the sample *M2* (g) of the filtration media of granular material.

The residual iron content in the filtration media RC_{Fe} (mg / g) after the filter operation cycle was determined:

$$RC_{Fe}=C_{Fe}-C^0_{Fe} \quad (4)$$

where C_{Fe} – iron content in the filtration media of granular filter material after the cycle of its operation and backwashing, mg/g;

C^0_{Fe} – iron content in a pure filtration media before the filter starts to work mg/g.

The studies used hot network water of a closed heating system of an industrial facility with a water temperature of 55±5 °C, in which the concentration of iron (total) was 0.35–0.57 mg/dm^3, and hydrogen sulphide 0.18-0.23 mg/dm^3. This water had a redox potential of -18 ± 5 mV (for the cold water it was 123 ± 15 mV). Determination of hydrogen sulphide content in water was carried out by reaction with NN-dimethyl-n-phenylenediamine by photocolorimetric method [49].

The efficiency of water purification by the main polluting components (total iron and hydrogen sulphide) E_{Fe} (mass %) and E_{H2S} (mass %), respectively, was calculated by the formula:

$$E = \frac{Cin - Cpur}{Cin} \cdot 100\% \quad (5)$$

where E-efficiency of water purification by iron and hydrogen sulphide, respectively,% mass;

“Cin”, “Cpur” – concentration of pollutants in the initial (before cleaning) and purified (after purification) water, mg/dm^3.

The content of manganese dioxide in the studied materials was determined by the oxalate method [46, 50].

Quartz sand (veined crushed quartz, Karelia), zeolite sand (the breed of the Badinskoye deposit, Eastern Siberia), the same zeolite sand, but modified with manganese dioxide, as well as a number of commercial materials based on manganese dioxide were used as research objects. The latter were investigated: MnO2-coated semi-burnt dolomite (brand MZHF, RF), synthetic deferrizing material (Birm brand, USA), and manganese ore-pyrolusite (Pirolox brand, USA) which used to remove iron and manganese from groundwater.

Results and discussion

Evaluation of possibility of using for the complex treatment of hot water of various granular filtering materials was carried out according to a number of indices take a great importance in the actual operation of filters.

Table 1 compares the various characteristics of the materials studied in the work.

Table 1. Comparison of the properties of the studied granular filter materials

№	Filter material	Bulk density kg/dm^3	MD, % mass	P, % mass	RC Fe, mg/g	DC		E, % mass		V_{bw}, m/hour
						g/ kg	g/ dm^3	for iron-ion	for hydro-gen sulphide	
1	Gangue quartz (Karelia)	1.30	0.02	0.03	0.53	0.37	0.75	45.00	3.00	53.00
2	Zeolite (Badinskoye deposit)	1.00	0.31	0.18	2.87	1.09	1.09	67.00	8.00	37.00
3	Modified MnO_2 zeolite	1.07	0.07	0.03	0.36	1.04	1.11	67.00	68.00	39.00
4	MZHF	1.38	3.36	0.16	0.41	0.63	0.87	56.00	15.00	58.00
5	Birm	0.75	1.82	0.08	1.23	1.61	0.21	61.00	64.00	25.00
6	Pirolox	1.98	2.64	0.07	0.48	0.59	0.16	65.00	70.00	75.00

It can be seen from the table that the use of zeolite sand has advantages over the use of quartz sand, namely: lower bulk density and more efficient iron purification. Zeolite sand, in contrast to quartz and manganese-modified zeolite sand, has a higher mechanical destructibility. This is evidenced by higher losses during backwashing.

Modified by manganese dioxide, zeolite has high water purification efficiency on iron and hydrogen sulphide. The quartz and zeolite sands are not removed the hydrogen sulphide from hot water.

Commercial materials based on manganese dioxide (except for MZHF, for which practically all the indicators are the worst) have values of the efficiency of iron purification and mud capacity close to the modified zeolite. These materials also have higher values of mechanical breakdown rates, washing losses and backwashing rates. This makes their practical use for a real cleaning of network hot water very problematic. In addition, these materials have high cost.

Figure 2 shows dependence of change in purification efficiency on the iron and hydrogen sulphide content indicators for the three materials studied during one filter cycle, which corresponded to a transmission of 20 m^3 (3640 filter volumes) of water.

These dependences confirm the foregoing, and also indicate that at the end of the filter cycle, the efficiency of purification by iron on the manganese dioxide-modified zeolite reaches its maximum value, and by hydrogen sulphide falls to 25 %. The loss of head pressure at a linear rate of water filtration through a load of 10 ± 0.4 m/h at the end of the first filter cycle was not more than 0.1 bar. Thus, the values of filter cycle with high purification efficiency for loading with the manganese-modified zeolite is different for removing iron and hydrogen sulphide.

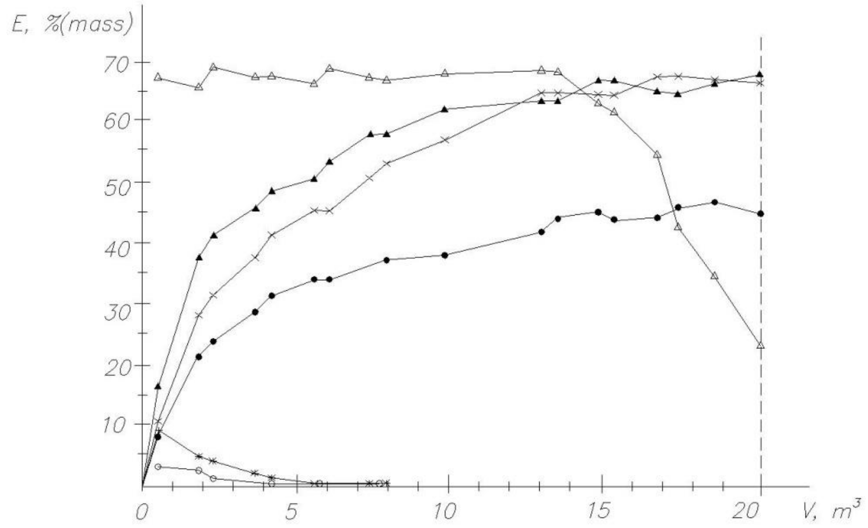

Figure 2 Dependence of iron and hydrogen sulphide purification efficiency (E) on time for one filter cycle.
- ● -, -x-, - ▲ - – by iron, -o-, - * -, -Δ- – by hydrogen sulphide, -o-, - ● - – quartz sand, -x-, - * - – natural zeolite, -Δ-, - ▲ - – manganese dioxide modified natural zeolite.
V, (m^3) – the volume of transmission water

Figure 3 shows the same dependencies as in Figure 2, but over the time of operation corresponding to 5 filter cycles, i.e. passing through the filter ≈ 100 m^3 (or 18 200 filter volumes) of water without carrying out regeneration. The efficiency of purification from iron in this case grew and amounted to 90–91 % at the end of the work, and from hydrogen sulphide it fell to 10 %. The loss of head pressure at the end of the filter operation period was no more than 0.5–0.6 barr, which is a satisfactory result.

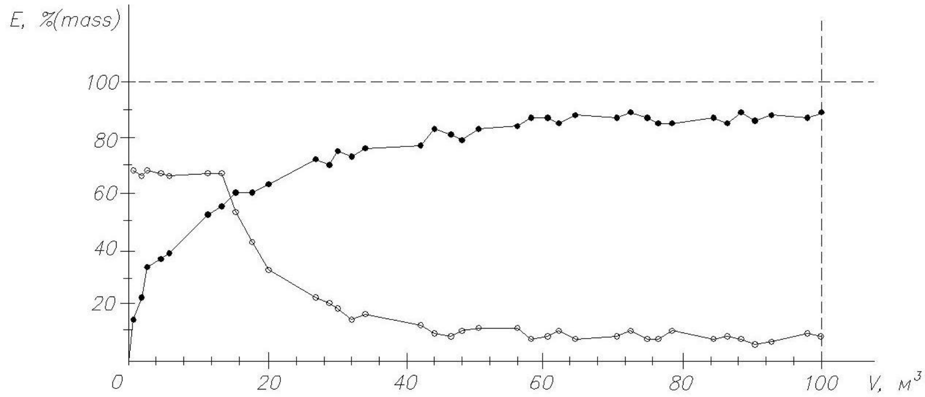

Figure 3.Dependence of iron and hydrogen sulphide purification efficiency (E) on time for five filter cycles without filter regeneration.
- ● - – by iron, -o- – by hydrogen sulphide.
Manganese-modified natural zeolite of Badinskoye deposit

Figure 4 shows the dependencies, similar to those shown in Figure 3, but for the mode of operation of the filter with periodic regeneration of the filtration media from the manganese-modified zeolite both from iron purification (backwash) and from hydrogen sulphide (chemical treatment + backwashing) for five filter cycles of 20 m^3 each. The efficiency of purification from hydrogen sulphide in this case is restored after each filter cycle by treatment of the filtration media with a solution of potassium permanganate. The average iron cleaning efficiency during each filter cycle in this case is lower due to a strong decrease in the retention of iron capacity, after washing the filtration media of the filter.

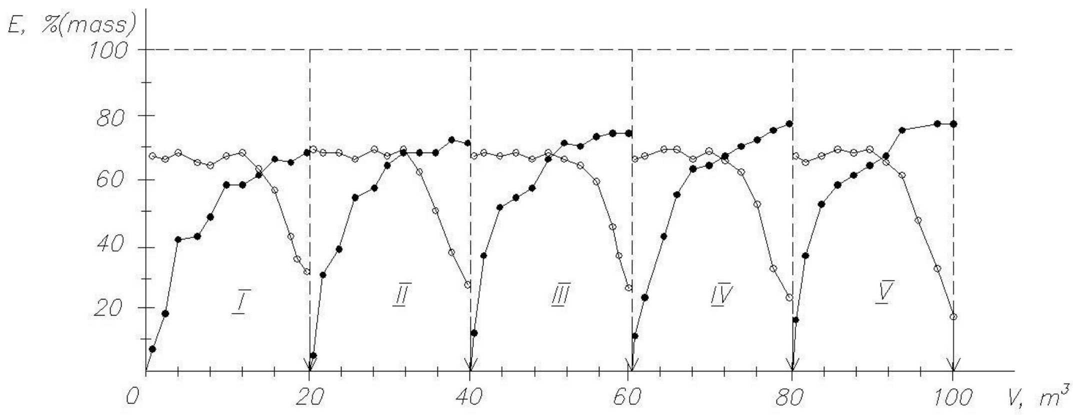

Figure 4 Dependence of iron (- ● -) and hydrogen sulphide (-o-) purification efficiency (E) on time for modified manganese dioxide natural zeolite with periodic regeneration of filter load

Thus, in the case of a real continuous operation of the filter with a modified zeolite material, the restoration of its iron cleansing ability (backwash at a linear speed of 40 ± 1 m/h) can be carried out after passing water in an amount of 18,000–15,000 filter volumes (or 17.0 m^3 of water per kilogram of loading). For hydrogen sulphide, the restoration of its cleaning capacity (chemical treatment with oxidant solution + backwashing) must be carried out after passing 3 600 filter volumes (or 3.4 m^3 water per kilogram of filtration media). Comprehensive cleaning of hot water must be carried out in two stages: the first – mechanical cleaning of iron and the second – contact oxidation of hydrogen sulphide. Regeneration of the filter load of the second stage of purification is five times more often than the first one.

Figure 5 shows the dependence of the purification efficiency on hydrogen sulphide during one filter cycle for different linear velocities of water flow through the filter loading. As a filtering material, the natural zeolite of the Badinskoye deposit, modified with manganese dioxide, was used. At low linear velocities of water movement (1–2 m/h), the efficiency of purification by hydrogen sulphide is 92–96 % mass, and will be maintained throughout the filter cycle (20 m^3 of water). At high velocities (5–10 m/h), the cleaning efficiency is lower (81–76 % by weight) and decreases significantly towards the end of the filter cycle.

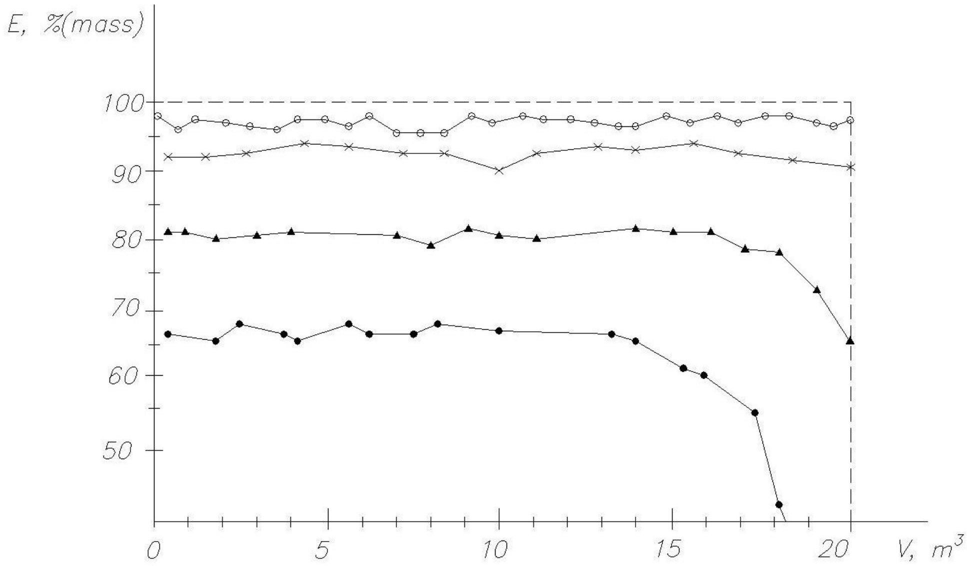

Figure 5. Influence the linear velocity of water flow in the filterefficiency of hydrogen sulphide purification efficiency (E) for loading the filter from the modified manganese dioxide of natural zeolite (Badinskoye deposit).
Linear velocitys: - ● - – 10 m/h, - ▲ - – 5 m/h, -x- – 2 m/h, -o- – 1 m/h.

The properties of materials modified by manganese dioxide based on clinoptilolit – containing rocks of various deposits in Russia were also studied. The appearance of one of these materials is presented in Figure 6.

Figure 6 Appearance of the manganese-modified filter-catalyzed materials based on the natural zeolite of the Badinskoye deposit (Russia)

Table 2 shows the results of comparative tests of these materials for cleaning hot water from iron and hydrogen sulphide. The studied materials have high values of the efficiency of purification from iron and hydrogen sulphide at a high rate of passage of water through the loading of the filter, as well as similar rates of backwashing.

Table 2. Comparison of the properties of MnO_2 – modified materials obtained on the basis of clinoptilolite-containing rocks of various deposits

№	The deposit of zeolite rocks	MnO_2, content % (mass)	MD, % (mass)	RC Fe mg/g	E, %mass		V_{50}, m^3	V_{bw}, m/h
					for iron-ion	for hydrogen sulphide		
1	Badinskoe (East Siberia)	0.36	0.07	0.36	67.00	68.00	17.50	40.00
2	Chuguevskoe (PrimorskyKrai)	0.42	0.11	0.42	61.00	70.00	23.00	46.00
3	Sokirnitskoe (Ukraine)	0.30	1.81	0.55	65.00	62.00	4.50	35.00
4	Shivirtuiskoe (East Siberia)	0.49	6.05	0.91	70.00	61.00	7.50	30.00
5	Kholinskoe (East Siberia)	0.46	4.24	0.74	69.00	69.00	8.00	34.00

From Table 2 it can also be seen that effective water purification from hydrogen sulphide for materials obtained from the rocks of the Shivirtuysky, Kholinsky and Sokirnitsky deposits is less prolonged than for materials based on the Badinskoye and Chuguevskoye deposits. The materials of the first group are characterized by a value of V50, (the volume of purified water with an efficiency of at least 50 %) is 4.5–8.0 m^3, and for the second group it is 17.5–23.0 m^3. All samples have similar values of the total content of manganese dioxide, but its spatial distribution within the material particles for these two groups is different. For samples of material based on rocks Badinskoye and Chuguyevskoye deposits, the manganese dioxide phase is located on the surface of particles of these materials [46] and is maximally available. For other samples of materials, manganese dioxide is distributed throughout the grain and its surface concentration is much lower.

Materials obtained on the basis of zeolite rocks of the Shivirtuysky, Kholinsky and Sokirnitsky deposits in addition have high values of mechanical destructibility (MD), which will lead to a faster grinding and carrying away of the load in conditions of actual operation of the filters. The same samples also have an increased ability to accumulate iron inside of filtration media particles (RCFC index, Table 2). Insoluble iron compounds that are not removed by backwashing the filter with water will greatly reduce the oxidizing ability of these materials by hydrogen sulphide due to the blocking by iron compounds of the surface of the active layer of manganese dioxide.

The high efficiency of water hydrogen sulphide purification for material on base natural piroluzite ("Pirolox") corresponds the information [11, 14] about it application for this purposes.

The process of hydrogen sulphide removing from network hot water on MnO_2-modified natural zeolite (on condition that dissolved oxygen is not present) was similar to process of chemical fixation [25, 27]. Moreover the non-catalitic oxidation process with "active oxygen" of MnO_2 and formation manganese oxides of low valency also was possible [11, 14, 23].

To summarize, in this work for the experimental conditions similar such as real hot water networks conditions [3, 5, 10] good results on purification network hot water from suspended ferrum oxides and hydrogen sulphide was obtained.

The filtering materials base on MnO_2-modified natural zeolites may be use for this purposes, as well as, materials traditionally used in groundwater purification practice [11, 13, 14]. This circumstance increases the possibilities of network hot water complex purification with use the standard filtering equipment.

Conclusion

The work has evaluated the possibilities of hot water cleaning with materials based on manganese dioxide-modified zeolite rocks in comparison with other filtering materials (including those containing manganese dioxide). Based on the results of the studies, the following conclusions are drawn:

Filtering materials based on manganese dioxide-modified zeolite rocks (from the most accessible deposits in Russia) provide good cleaning of hot water from its main pollutants – iron compounds and hydrogen sulphide.

In comparison with other granular filtering materials (quartz sand, unmodified natural zeolite), manganese dioxide-modified zeolite materials have a higher retention capacity for insoluble iron compounds and oxidative activity for hydrogen sulphide, as well as low mechanical breakdown during operation.

The advantage of manganese dioxide -modified zeolite rocks over commercial manganese dioxide-containing materials, traditionally used for deironing and demanganation of groundwater, lies in their less mechanical destructibility (MD) during operation, as well as in the lower cost of water using for washing the load in the filter during its regeneration.

The complex purification of hot network water should be carried out in the form of a two-stage process: filtration and mechanical treatment of water from iron compounds with a linear rate of movement in the apparatus of 10–12 m/h, and sorption-oxidative purification from hydrogen sulphide at a linear speed of no more than 1–2 m/hour.

To ensure the consistency of the oxidation capacity of the filtering of manganese dioxide-modified zeolite rocks by hydrogen sulphide, it must be periodically regenerated with an oxidizer solution.

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Alexey Chechevichkin,
+7(921)971-84-47; 01@6400840.ru

Nikolai Vatin,
+7(921)964-37-62; vatin@mail.ru

Vyacheslav Samonin,
+7(812)903-84-65; samonin@mail.admiral.ru

Mikhail Grekov,
+7(812)297-20-45; disgpu@spbstu.ru

Алексей Викторович Чечевичкин,
+7(921)971-84-47; эл. почта: 01@6400840.ru

Николай Иванович Ватин,
+7(921)964-37-62; эл. почта: vatin@mail.ru

Вячеслав Викторович Самонин,
+7(812)903-84-65;
эл. почта: samonin@mail.admiral.ru

Михаил Александрович Греков,
+7(812)297-20-45; эл. почта: disgpu@spbstu.ru

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