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THE INFLUENCE OF FENTON'S REAGENT ON THE RAW SLUDGE DISINFECTION

Fenton's reagent was investigated for its potential for improving the sanitary conditions of the raw mixed sludge (preliminary and surpulus) from wastewater treatment plants. Fenton's reagent doses, i.e., ferrous ions (Fe^{2+}) and hydrogen peroxide (H_2O_2), as independent agents influencing technological effects were determined. Microbial analysis is concentrated on coliform bacteria, faecal coliform bacteria, anaerobic endosporous *Clostridium perfringens* and microorganisms from the genus of *Salmonella*.

The most effective method of improving the sanitary conditions of a raw sludge was advanced oxidation process (AOP). The efficiency of the method presented depended directly on chemical reagent doses.

SYMBOLS

MPN – the most probable number,
CFU – the colony-forming unit,
d. m. – a dry mass.

1. INTRODUCTION

Wastewater treatment plant operation is closely related to the need for a proper sludge management. Sludge generated in wastewater treatment processes must be subjected to processing and afterwards reused or disposed [1]. Despite a considerable progress in past years in the technologies and methods of sludge treatment, the problem of its effective neutralization has not been solved satisfactorily. It is more and more difficult to solve it because the amount of sludge systematically increases, and its quality rarely corresponds to environmental protection requirements. The progress in sludge generation results from a high number of the new wastewater treatment plants and the quality of treated wastes [1]–[4].

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The presence of pathogens in sewage sludge is really hazardous for natural environment and one of the most relevant problems of sanitary hazard. This has to be taken into consideration while neutralizing sewage [3]–[6]. The composition of a stabilized sludge is chemically favourable, has soil-forming features and its dewatering is generally easy. However, besides these desirable features, a sludge is usually settled by the microorganisms (bacteria, viruses, parasites, fungi, protozoan, ect.). Pathogens, dangerous for human beings, and saprophytes, neutral from the sanitary point of view, can be present there [3]–[5].

The most common methods of sludge management, such as anoxic or aerobic stabilization and liming, do not allow us to obtain completely safe products in terms of sanitary conditions. Biological pollutants entering the soil disturb biocoenotic balance and are potentially hazardous for the other organisms [2]–[3], [7]–[8]. The composition of the sludge from municipal wastewater treatment plants is complex. The sludge consists of pathogens not only from hospitals and laboratories, but also from landfills, slaughterhouses and other sources such as trade, industry and agriculture. The pathogens reach wastewater treatment plants and leave them during treatment process [5].

Modifications and changes of the methods currently practiced are most advantageous, but the search for new, effective and cheap processes enabling removal of environmentally hazardous substances ought to be undertaken as well. The operations should effectively improve sludge dewatering parameters, decrease the mass of sludge, remove organic substances susceptible to putrefaction and diminish the number of pathogenic and parasitic organisms [1], [7].

In wastewater treatment, an alternative to the methods commonly applied in contaminant removal are these based on chemical reactions, mainly advanced oxidation process (AOP) [1], [9]. One of such methods is Fenton's reaction that occurs while using hydrogen peroxide (H_2O_2) and iron ions as the catalysts of the process. The reaction leads to a catalytic decomposition of hydrogen peroxide in the presence of ferrous ions Fe^{2+}, which results in the generation of free radicals ($OH^{\bullet}$) with high oxidizing potential (2.8 V) [5].

The aim of the study was to assess of the usability of AOP with Fenton's reagents for raw sludge disinfection. The results were compared with these obtained using merely sludge conditioning with ferrous ions and hydrogen peroxide.

2. MATERIALS AND METHODS

In the present study, raw mixed sludge (preliminary and surpulus) from wastewater treatment plant sedimentation tanks was used. Sludge was taken directly from sedimentation tanks. Physicochemical and microbial parameters of the sludge are given in table 1. The investigations were conducted in three phases, on laboratory-scale experimental stands at the ambient temperature ranging from 20 °C to 22 °C.

Table 1

Characteristics of the sewage sludge used in the experiment

Parameter	Unit	Min	Max	Mean
Hydration	[%]	97.525	96.92	97.08
Filtration resistivity	[kg/m]	$2.312\cdot10^7$	$3.145\cdot10^7$	$2.728\cdot10^7$
CSK	[s]	343	402	372
Dry mass	[g/dm^3]	27.490	28.840	28.265
Mineral fraction	[g/dm^3]	5.950	7.760	6.855
Volatile fraction	[g/dm^3]	19.570	20.560	20.065
COD of the filtrate	[mg O_2/dm^3]	2696.3	2949.0	2822.6
P–PO_4 of the filtrate	[mg P–PO_4/dm^3]	1250.7	1421.4	1356.3
N_{tot} of the filtrate	[mg N/dm^3]	309.5	421.7	364.3
N–NH_4 of the filtrate	[mg N–NH_4/dm^3]	193.7	231.3	212.6
Reaction	[pH]	5.78	6.49	6.13
Coliform bacteria	[MPN/g d.m.]	$7.1\cdot10^6$	$7.9\cdot10^6$	$7.4\cdot10^6$
Faecal coliform bacteria	[MPN/g d.m.]	$2.6\cdot10^6$	$3.8\cdot10^6$	$3.2\cdot10^6$
Clostridium perfringens	[CFU/g d.m.]	$8.7\cdot10^4$	$9.9\cdot10^4$	$9.3\cdot10^4$
Salmonella sp.	–	+	+	+

\+ present in the sewage sludge.

The research phases varied depending on the type of chemical reagent supplied to the technological system. The can be itemized as follows:

Phase I – ferrous ions (Fe^{2+}) in the form of $FeSO_4 \cdot 6H_2O$.

Phase II – 30% solutionn of hydrogen peroxide (H_2O_2).

Phase III – ferrous ions (Fe^{2+}) in form of $FeSO_4 \cdot 6H_2O$ and 30% solution of H_2O_2 (Fenton's reagent).

The doses of the reacting substances are collected in table 2.

Table 2

The doses of chemical reagents used in the experiment

Dose	Phase I	Phase II	Phase III	
			Fenton's reagents	
	Fe^{2+} [g/dm^3]	H_2O_2 [g/dm^3]	Fe^{2+} [g/dm^3]	H_2O_2 [g/dm^3]
1	0.25	1.00	0.25	1.00
2	0.50	2.00	0.50	2.00
3	0.75	3.00	0.75	3.00
4	1.00	4.00	1.00	4.00
5	1.50	6.00	1.50	6.00
6	2.00	8.00	2.00	8.00

Each experimental phase was divided into six technological variants characterized by different doses of the chemical substances applied to the system. The doses of reacting substances were chosen on the basis of preliminary experimental and literature data.

The research was carried out in the model laboratory reactors of 1.5 dm^3 working volume. The reactors were fitted out in magnetic stirrer (figure 1).

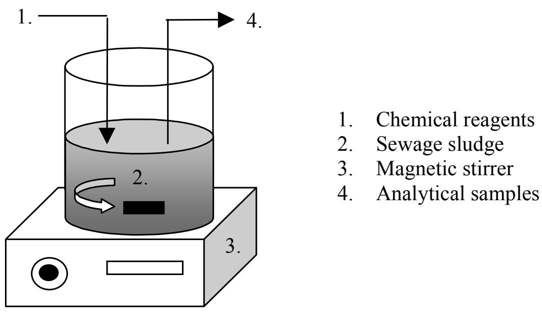

Fig. 1. Scheme of the experimental stand

At the beginning of the experimental cycle, 1 dm^3 reactor was supplied with the sewage sludge tested and then chemical reagents were dosed. In the case of the third phase, ferrous ions were first introduced to the mass of sewage, and after 10 minutes hydrogen peroxide, at a constant weight ratio of iron to hydrogen peroxide of 1:4, was supplied. During initial 30 minutes of the experiment sewage sludge was mixed by magnetic stirrer at the speed of 200 rpm in order to obtain the same concentration of chemical reagents in the whole sludge mass. After that period sludge with reacting substances was left without stirring.

Sewage sludge retention time was 24 h. The samples of sludge were taken directly from the reactors at the beginning of the cycle before reacting substances were introduced into the system and then after 24 h of the reaction time. Sewage sludge was assayed for the following microorganisms:

coliform bacteria,
faecal coliform bacteria,
Salmonella sp.,
endosporous form of *Clostridium perfringens*.

Coliform and faecal coliform bacteria from sludge samples were determined according to PN-EN-ISO 9308-1:2002(U). The samples were inoculated and incubated on lauryl-sulphate broth; their ability to produce gas was checked. Incubation of coliform bacteria and faecal coliform bacteria was carried out at the temperature of 37 °C (48 h) and 44.5 °C (24 h), respectively.

The presence of *Salmonella* sp. on the aforementioned media was determined after its previous culturing in 100 cm^3 of the broth with tetrathionate according to Müller–Kauffman (Merck) for 24 hours at 37 °C. *Salmonella* sp. bacteria were analysed for their capability to produce flagellar antigen using agglutinating serum for HM antigen (Biomed). They were finally identified with API 20E tests (bioMerieux).

In order to determine anaerobic spore-forming and sulphite-reducing bacteria of *Clostridium perfringens*, sewage sludge was analyzed according to PN-EN-ISO 2646-1:2002. Bacteria were inoculated and incubated on Wilson–Blair's broth in anaerobic jars, using AnaeroGen (OXOID) for the generation of anaerobic conditions. Bacteria surrounded by black precipitate of ferric sulphide were counted.

3. RESULTS

It was shown that in the sludge being not treated with chemical agents, the most probable number of coliform bacteria approached $7.4\cdot10^6$ bacteria/g d.m, faecal coliforms – $3.2\cdot10^6$ bacteria/g d.m; however, anaerobic endosporous form of *Clostridium perfringens* reached the level of $9.3\cdot10^4$ CFU/g d.m. Microorganisms of *Salmonella* were present (table 1).

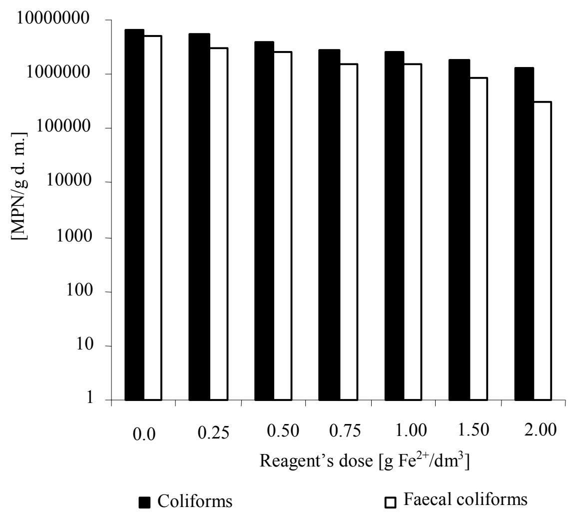

Fig. 2. Reduction of the MPN of coliforms and faecal coliforms in the excess sludge in phase I

The experiments proved that the most efficient method of raw sludge disinfection was AOP. The effectiveness of reducing the number of coliforms and *Clostridium perfringens* increased with an increase in reagent dose (figure 2). In the first variant, the 0.25 g Fe^{2+}/dm^3 and 1.0 g H_2O_2/dm^3 reduced the MPN of faecal coliforms to $7.8\cdot10^4$ bacteria/g d.m. and the MPN of coliform bacteria to $1.8\cdot10^5$ bacteria/g d.m.; however *Clostridium perfringens* reached $8.7\cdot10^4$ CFU/g d.m. (figures 4 and 7). At this dose of Fenton's reagent bacteria from the *Salmonella* sp. genus were present. *Salmonella* sp. was removed from the sludge in the variants with the reagent doses above 0.75 g Fe^{2+}/dm^3 and 3.0 g H_2O_2/dm^3 (table 3). The effectiveness of the reduction of coliforms increased with an increase in chemical reagent doses (figure 4).

The lowest number of anaerobic cells, i.e., $2.7\cdot10^4$ CFU/g d.m., was observed at Fenton's reagent doses of 2.0 g Fe^{2+}/dm^3 and 8.0 g H_2O_2/dm^3 (figure 7). Application of the highest doses of Fenton's reagent to raw sludge allows us to obtain $1.0\cdot10^2$ MPN/g d.m. of faecal coliforms and $1.5\cdot10^2$ MPN/g d. m. of coliforms (figure 4).

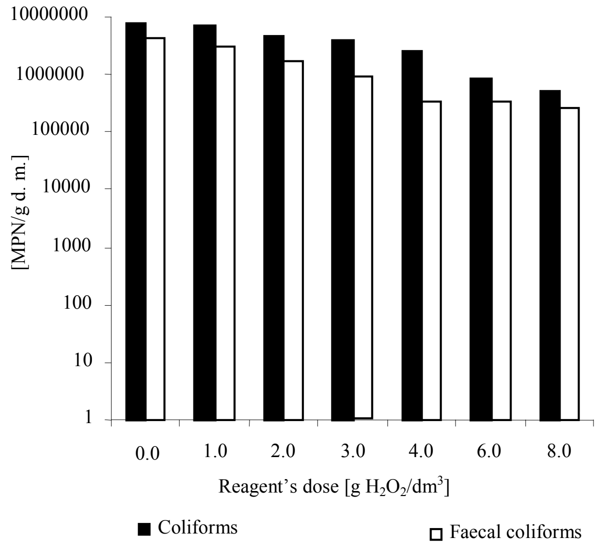

Fig. 3. Reduction of the MPN of coliforms and faecal coliforms in the excess sludge in phase II

The results of the sewage sludge disinfection by means of inorganic coagulant or hydrogen peroxide as a single disinfectant were not similarly satisfactory (figures 2–3, 5–6). The application of ferrous ions only resulted in inefficient reduction of both coliform bacteria and endosporous form of *Clostridium perfringens* (figures 2 and 5). The most effective variant allows us to achieve $2.1\cdot10^6$ MPN/g d.m. of coliforms and $5.1\cdot10^5$ MPN/g d.m. of faecal coliforms (figure 2). The results obtained differ from these obtained due to sewage sludge stabilization by AOP. They prove to be worse than the values determined for sludge untreated chemically. The MPN of coliform bacteria and of faecal coliform bacteria approached respectively $7.4\cdot10^6$ bacteria/g d.m. and $3.2\cdot10^6$ bacteria/g d.m. (table 1). The application of inorganic coagulant only to sludge mass had an insignificant effect on the change in the number of endosporous form of *Clostridium perfringens* (figure 5). None of the technological variants with inorganic coagulant allows the number of the bacteria from the genus of *Salmonella* sp. to be reduced (table 3).

Hydrogen peroxide used in phase II did not enable us to obtain the final results comparable to the results achieved with Fenton's reagent (figures 3 and 6). Disinfection of sludge was better than that observed in the first phase of the experiment. In the case of the highest dose of oxidizing agent, coliform bacteria were reduced to $8.1 \cdot 10^5$ MPN/g d.m., and anaerobic organisms – to $4.9 \cdot 10^4$ CFU/g d.m. (figures 3 and 6). In the variants with hydrogen peroxide in the amount ranging from 6.0 g H_2O_2/dm^3 to 8.0 g H_2O_2/dm^3, bacteria from the genus of *Salmonella* sp. were not present in sewage sludge (table 3).

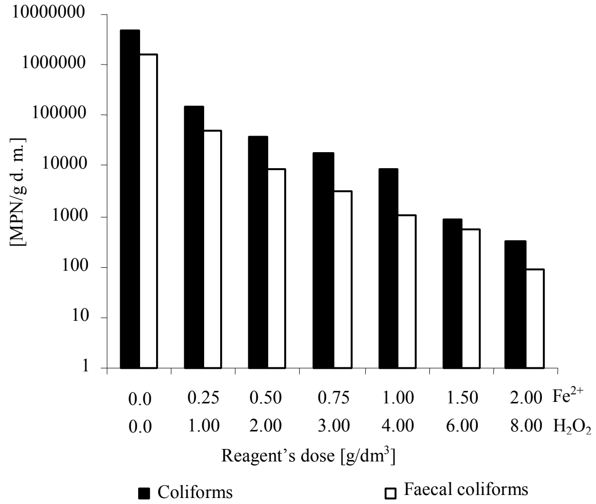

Fig. 4. Reduction of the MPN of coliforms and faecal coliforms in the excess sludge in phase III

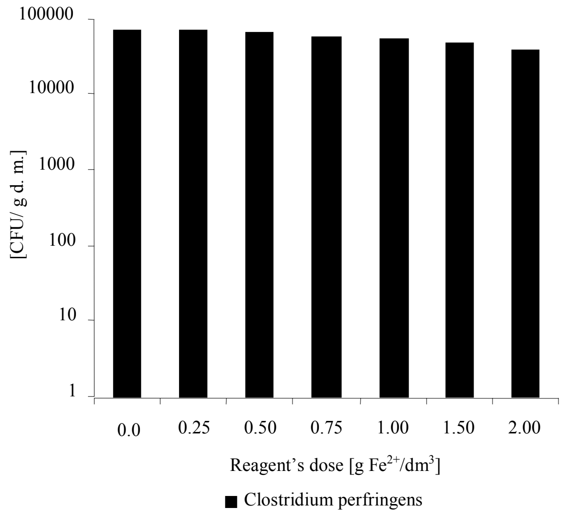

Fig. 5. Reduction of the CFU of *Clostridium perfringens* in the excess sludge in phase I

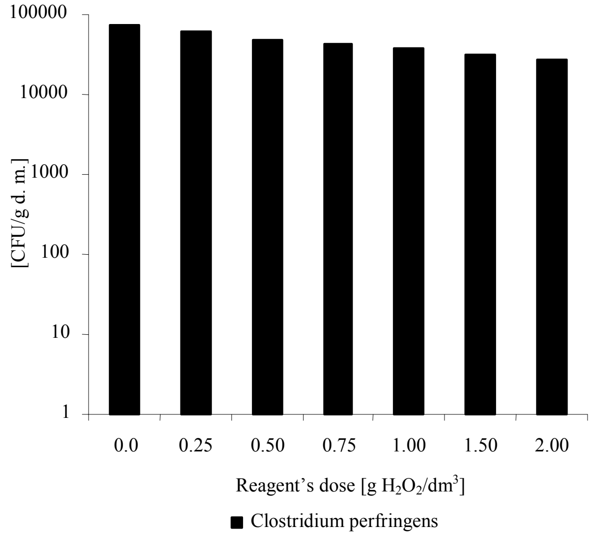

Fig. 6. Reduction of the CFU of *Clostridium perfringens* in the excess sludge in phase II

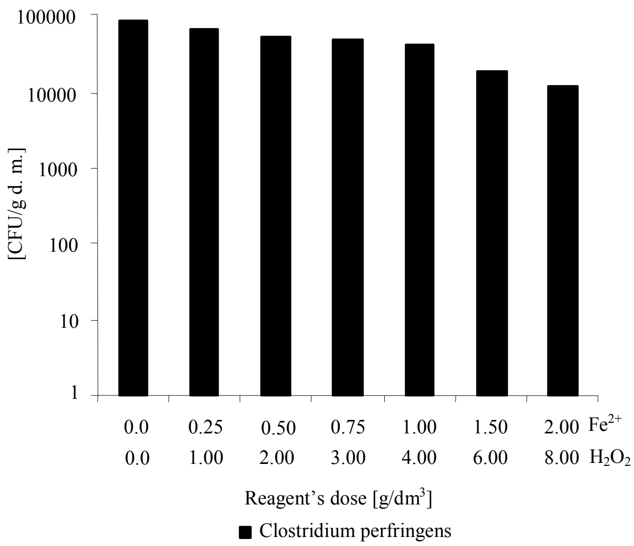

Fig. 7. Reduction of the CFU of *Clostridium perfringens* in the excess sludge in phase III

Table 3

The presence of *Salmonella* in sewage sludge, depending on the phase of the experiment and chemical reagent dose

Phase I Fe^{2+}	Dose [g Fe^{2+}/dm^3]	0.25	0.50	0.75	1.00	1.50	2.00
	Salmonella sp.	+	+	+	+	+	+
Phase II H_2O_2	Dose [g H_2O_2/dm^3]	1.00	2.00	3.00	4.00	6.00	8.00
	Salmonella sp.	+	+	+	+	–	–
Phase III Fenton's reagents	Dose [g Fe^{2+}/dm^3] [g H_2O_2/dm^3]	0.25; 1.00	0.50; 2.00	0.75; 3.00	1.00; 4.00	1.50; 6.00	2.00; 8.00
	Salmonella sp.	+	+	+	–	–	–

4. DISCUSSION

The aim of the study was to assess the usability of AOP with Fenton's reagent for raw sludge disinfection. The results were compared with these obtained by the methods using merely sludge conditioning with ferrous ions and hydrogen peroxide.

Based on the currently used technologies it seems that application of hydrogen peroxide, mostly Fenton's reagent, should lead to more effective and economical final results of sludge disinfection. Copious literature data confirms the usefulness of hydrogen peroxide to the reduction of the number of bacteria in wastewater treatment process carried out in food industry and medical centres [10]–[13].

It has been proven that disinfection of the most types of treated water is possible at the of hydrogen peroxide dose of 0.2 g H_2O_2/dm^3 applied for 30 minutes. This is also the threshold dose if organoleptic properties are taken into account. Hydrogen peroxide used for 10 days has aniseptic effect on treated water; moreover, it also reduces its colour and odour [14].

As a result of a high concentration of potentially pathogenic microorganisms and unfavourable properties and characteristics of sewage sludge, higher doses of H_2O_2 and longer sewage retention time were applied in a technological system. The oxidizer as the only agent did not allow us to obtain high technological results comparable to these obtained in the systems with advanced oxidation. This was probably caused by insufficient oxidizing potential in the whole mass of sewage sludge.

The other experiments also revealed that a direct influence of hydrogen peroxide on the strains of microorganisms rich in alimentary substances is insignificant and diverse [14]–[18]. For this reason some oxidizing substances used simultaneously in the system seem to be an effective way of water disinfection. Better results were observed during both chlorination and hydrogen peroxide use [14]. Similarly, both ozonation and hydrogen peroxide (Peroxone) addition appeared to be effective in the case of removing organic substances and in the case of disinfection. Contrary to separate ozonation or hydrogen peroxide oxidation, this method is most economical [19]–[21]. In order to intensify the final technological effect, simultaneous use of hydrogen peroxide with magnetic field, electromagnetic field or microvalves is recommended [1], [22].

In the present experiment, AOP with Fenton's reagent for pathogenic microorganisms reduction was used. The efficiency of this method is based on catalytic decomposition of hydrogen peroxide by ferrous ions Fe^{2+}, which results in free hydroxyl radicals formation [1], [9], [19]. Better disinfection effect stems from the fact that free hydroxyl radicals have higher oxidizing potential with relation to hydrogen peroxide alone. An effective interaction between hydrogen peroxide and Fe^{2+} ions affects a cellular structure of microorganism and directly leads to the reduction of their number in sewage sludge. The present experiment proved that Fenton's reagent is technologically more effective than hydrogen peroxide or inorganic coagulants is sewage sludge disinfection.

Analogous results were achieved in other studies which proved that advanced oxidation process may significantly reduce the growth of activated sludge [23].

It was shown that free hydroxyl radicals caused the damage to biological structures because of their reaction with the particles of cell structural material [16], [24]. Each particle can potentially be affected by free radicals. This reaction mostly leads to the limitation of cell's biological or biochemical activity. Proteins determining intracellular changes can serve as an example. Small modifications of enzymes structure caused by free radicals lead to their complete deactivation and uselessness. Similar phenomenon is shown for carbohydrates, fats and nucleic acid [8], [16].

Fenton's reagent used for sewage sludge stabilization gave satisfactory results because this method besides effective disinfection allows the degradation of organic compounds susceptible to putrefaction, odour removal, the improvement of dewatering parameters and the reduction of the mass and volume of the sludge [1], [25].

5. CONCLUSIONS

- The technology of raw sewage sludge disinfection based on Fenton's reagent allows us to obtain effective final results with relation to all of the analysed groups of microorganisms.
- The efficiency of the method presented depends directly on Fenton's reagent doses. An increase in the efficiency is correlated with an increase in chemical reagent doses; however, in the range from 1.5 g Fe^{2+}/dm^3, 6.0 g H_2O_2/dm^3 to 2.0 g Fe^{2+}/dm^3, 8.0 g H_2O_2/dm^3, the changes in the number of bacteria are more significant.
- The application of AOP proved to be more effective method than another methods presented in the experiment. Final results of sewage sludge disinfection several times surpassed the results obtained when hydrogen peroxide or ferrous ions were used alone.

• The method of disinfection because of its easiness, the accessibility of chemical reagents and economical reasons can become alternative technology of sewage sludge stabilization.

• The search for the agent that intensifies final technological results of sludge treatment at economical use of chemical reagents is urgently needed.

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WPŁYW ODCZYNNIKA FENTONA NA STOPIEŃ HIGIENIZACJI SUROWYCH OSADÓW ŚCIEKOWYCH

W eksperymencie analizowano możliwość zastosowania odczynnika Fentona do higienizacji mieszanych osadów surowych (wstępnych i nadmiernych). Określono wpływ dawki reagentów chemicznych (jonów Fe^{2+}, H_2O_2, układu Fe^{2+}/H_2O_2) na ostateczny efekt technologiczny. Analizy mikrobiologiczne dotyczyły bakterii z grupy coli, bakterii coli typu kałowego, przetrwalnikowych form *Clostridium perfringens* oraz bakterii z rodzaju *Salmonella*.

Najskuteczniejszą metodą higienizacji analizowanych osadów ściekowych okazała się technika pogłębionego utleniania. Skuteczność prezentowanej technologii rosła wraz z kolejnymi dawkami reagentów chemicznych wprowadzanych do układu.