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Wastewater Treatment of Food Industry Using Electrochemically Generated Ferrates

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Abstract

The article considers a complex technology for electrochemical wastewater treatment from a meat processing plant. The process of water treatment includes a sequential processing in graphite and iron anode-based electrochemical modules, and exposure in maturation and homogenization chamber. Ferrate generation has been evidenced in the iron anode-based module, and ferrates concurrently acting as mild oxidants, coagulants, and steel corrosion inhibitors. The electrochemical potential of ferrates ranges from 1.3 to 1.5 V in aqueous solutions for the pH values between 6 and 8. Provided an increase in ferrate concentration up to 20 mg/L in the solution, the corrosion rate for capacitive equipment and pipelines is reduced by twofold. An application of iron (III) hydroxide as a product of ferrate reduction lowers wastewater clarification by 1.5-2.0 times compared to the widely used ferric iron and aluminum salts. The research findings provide for the advancement of integrated water treatment solutions and highlight the importance of implementing efficient treatment processes to minimize the environmental impact of meat processing plants.

1. Introduction

To ensure high-quality and eco-friendly water use and to reduce the anthropogenic load on water bodies are the principal governmental tasks in the field of environmental safety. These are primarily achieved through compliance with regulations and standards for wastewater treatment and its reuse in the natural water exchange chain. According to the World Health Organization survey, over 80% of the world's diseases are related to poor drinking water quality. However, an increase in human pressure on water bodies is also due to the occurrence of natural and man-made hazards [1-3].

The lack of an integrated science-based approach and relevant regulatory and methodological support, which would reduce such negative impacts depending on the dynamic changes in the internal and external environment, hinders managing of environmental conservation and restoration, and leads to unreasonable expenditure of information, material, and energy resources. In this regard, it is urgent to solve an applied research problem in the field of environmental safety, namely, to improve conceptual and theoretical foundations for managing the environmental safety of treatment technologies, and to reduce the risk of emergency situations, taking into account the resource efficiency requirements. This would contribute to compliance with standards for harmful effects on the environment [4-7].



The marketing rationale for solving this issue is that the global environmental technology market is currently valued at 235 billion dollars. Moreover, the global water purifier market grew from 11.3 to 18.8 billion dollars from 2013 to 2020, and its stable annual growth of 8-9% is expected until 2030 [8,9].

Thus, it is highly relevant to actively implement new resource-efficient technologies and engineering approaches at the nation-wide level. The aim is to reduce capital and operating costs of treatment facilities along with large-scale use of financial instruments that would allow the implementation of "green economy" projects in the field of water supply and utilities at an economically feasible level [10-12].

Physical, chemical, biological methods, or a combination thereof, are commonly used for water treatment and purification. From the technological perspective, an advancement of novel methods for purification and water treatment technologies is caused by some operational shortcomings thereof (Fig. 1).

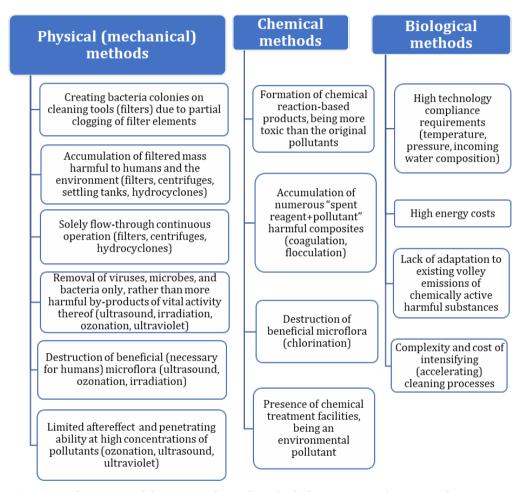


Fig. 1 Disadvantages of the commonly used methods for water purification and treatment

Here are technological disadvantages for the systems that utilize physical (mechanical) methods:

- Likelihood of creating bacteria colonies on cleaning tools (filters) due to partial clogging of filter elements
- Accumulation of filtered mass harmful to humans and the environment (filters, centrifuges, settling tanks, hydrocyclones)
- Solely flow-through continuous operation (filters, centrifuges, hydrocyclones)
- Removal of viruses, microbes, and bacteria only, rather than more harmful by-products of vital activity thereof (ultrasound, irradiation, ozonation, ultraviolet)
- Destruction of beneficial (necessary for humans) microflora (ultrasound, ozonation, irradiation)
- Limited aftereffect and penetrating ability at high concentrations of pollutants (ozonation, ultrasound, ultraviolet)

Here are technological disadvantages for the systems that utilize chemical methods:

- High probability of new chemical reaction-based compound formation, being more harmful to humans and the environment than the original pollutants (each technology)
- Accumulation of multiple "spent reagent+pollutant" harmful composites (coagulation, flocculation)
- Destruction of beneficial microflora (chlorination)



- Presence of chemical treatment facilities, being an environmental pollutant (each technology) Here are technological disadvantages for the systems that utilize biological methods:
 - Relatively high technology compliance requirements (temperature, pressure, incoming water composition) lead to high energy costs or cleaning stoppage (each technology)
 - Lack of adaptation to existing volley emissions of chemically active harmful substances (each technology)
 - High complexity and cost of intensifying (accelerating) cleaning processes (vermicomposting, biological ponds)

A cumulative disadvantage of the presented methods is the prerequisite to monitor numerous parameters of water quality and technological processes in real time. Wherein, only a few automated measuring instruments exist and operate reliably at municipal and industrial facilities (such equipment shortage is over 70%). Moreover, the feasibility of emergency natural and man-made hazards is not taken into account when designing systems, although being vital for effective and rational use of natural resources [13-18].

Having analyzed the above information, it is evident that the automated use of methods and means for processing aqueous solutions, along with experimental and analytical studies to eliminate the conceptual shortcomings of traditional approaches is the environmental safety issue. The creation of efficient treatment facilities for long-term use is challenging for technological implementation, since it requires techniques to provide the specified water quality, regardless of the revealed shortcomings. However, the advancement of effective systems is feasible only on the basis of a number of stages implemented along with pollutant removal methods. It is impossible to create an industrial water treatment system only according to the equipment catalogue, since each having its own specifics regarding input water quality, technological process parameters, drainage system modes, etc. [19-22].

Thus, the key objectives for creating combined treatment facilities based on the engineering concepts are as follows:

- Assessment of water quality and the efficiency of water use in technological processes (water technological passport)
- Development of measures to increase resource and energy efficiency of water use based on economic feasibility
- Formation of object-oriented technical specifications for the creation (modernization, reconstruction) of treatment facilities
- Calculation of various options for water treatment schemes based on life cycle indicators
- Designing equipment for a specific customer, based on the available financial tools and life cycle indicators, along with the creation of low-performance laboratory and industrial equipment for product demonstration
- Start-up (installation, assembly, commissioning, delivery to supervisory authorities, trial operation)
- Development and implementation of object-oriented technological regulations for treatment facilities
- Customer service guarantee

The first four points would reduce the cost of implementing purification (water treatment) systems by at least 20-30%. An extensive use of resource-saving technologies in the processes of wastewater and water treatment involves searching for novel complex technologies and universal reagents. The latter would operate as coagulants, oxidants, and disinfectants, being environmentally friendly without causing corrosion of technological equipment [23-25].

Aluminum sulfate is the most widely used coagulant at wastewater and water treatment plants. It has been observed to form hydrolysis compounds with advanced surface [26]. Hypochlorite and ozone are often found as disinfectants to oxidize organic compounds [27]. The drawbacks of chlorine-containing oxidants encompass high toxicity, formation of more toxic by-products than the original pollutants, high aggressiveness to equipment materials, and demand for pre-treatment of initial aqueous solutions for hypochlorite units in a separate station or module. Ozone wastewater and water treatment disadvantages include high toxicity, absence of after-effects, and demand for air pre-treatment before ozone generation [28-30].

Alkali metal ferrates can be used as reagents that integrate the above functions. These are powerful oxidants to damage microorganisms. A low-toxic ferrate reduction product in solution is Fe(OH)₃. Iron (III) hydroxide is formed as colloidal aggregates with advanced surface. These easily adsorb heavy metal ions, suspended particles and organic residues, providing additional water purification by coagulation of pollutants. Applications of ferrate to treat common pollutants and emerging contaminants such as arsenic, estrogens, pharmaceuticals, and personal-care products are being explored. Ferrate is emerging as a green chemical for organic synthesis and for treating toxins in water. Due to lower toxicity of iron compared to aluminum, sodium ferrate can be considered as a green chemical for water treatment [31-36].

However, there are problems that hinder the full-scale practical use of ferrates in the processes of industrial water and wastewater treatment [37]:



- High cost of pure sodium or potassium ferrate as a reagent for the chemical treatment of wastewater
- High energy intensity of existing technologies for electrochemical synthesis of ferrate in a flow-type or storage module, and high requirements for the composition of electrodes and electrolyte
- Obtaining toxic by-products on the surface of electrodes during electrochemical treatment of complex chemical composition wastewater
- Dependence of ferrate oxidation potential on the environmental pH
- Lack of reliable information on the effectiveness of the coagulating ability of ferrate reduction products (ferric hydroxide) in wastewater containing a large amount of suspended particles
- Lack of sufficient experimental data to conduct a comparative analysis of efficient ferrate use for specific industrial wastewater treatment in comparison with traditional technologies
- Lack of information on the effect of ferrates and reduction products thereof in wastewater on the corrosion destruction rate of technological equipment

The meat processing industry uses plenty of water to support technological processes and, therefore, is a source of large volumes of wastewater. The latter is organic substances, cleaning and disinfecting compositions, and a relatively high concentration of nitrogen and phosphorus compounds [38,39].

There is primary and, as a rule, secondary treatment before wastewater disposal. Secondary treatment involves methods of anaerobic and aerobic digestion, including chemical, physical and chemical, and electrochemical processing. Treated wastewater goes into surface water, sewers, or is used for irrigation [40,41].

Meat processing industry wastewater can be a cheap source of water and nutrients for pastures and crop production with standard excess of nitrogen and phosphorus emissions into the soil. The presence of organic substances in wastewater, affecting the operation of aeration tanks and polluting natural waters with antibiotics, hormones and other components is quite hazardous [42].

Thus, the development of an effective physical and chemical technology for treating wastewater from meat processing enterprises premised on the electrochemical ferrate generation as a coagulant, oxidizer, and corrosion inhibitor is an urgent problem. It would significantly reduce the cost of wastewater treatment from meat processing enterprises, and considerably lessen the negative impact thereof on the environment.

2. Materials and Methods

Wastewater from a meat processing plant (Pinsk, Republic of Belarus) was used as the research object. Chemical analysis of the stock solutions, and those obtained during and after processing was carried out in the accredited laboratory at Pinsk Vodokanal municipal manufacturing unitary enterprise. Initial wastewater was composed of 14 mg/L phosphates, 0.4 mg/L ammonia nitrogen, 0.3 mg/L nitrites, and 12.5 mg/L nitrates.

Amidst the experiment, the source water was sequentially pumped, using a circulation pump, through

- A graphite anode-based electrochemical module [43,44] (Fig. 2)
- A low-alloy steel chip anode-based electrochemical module to generate ferrates (Fig. 3)
- Maturation and homogenization chamber
- Closed-loop experimental installation pumped water twice (Fig. 4)

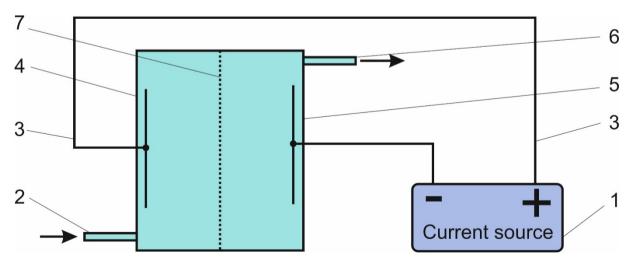


Fig. 2 Schematic for a graphite anode-based electrochemical module: (1) Electric (direct/alternating) current source; (2) Inlet pipe (purified water supply); (3) Current leads; (4) Anode (lump graphite) chamber; (5) Cathode (grade 08X18H10T steel) chamber; (6) Outlet pipe; (7) Impermeable membrane



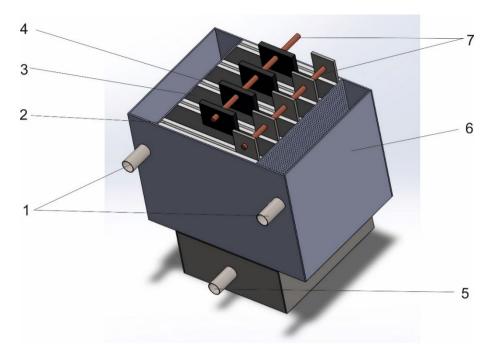


Fig. 3 Schematic for an electrochemical module to generate ferrates: (1) Outlet pipes for anolyte and catholyte; (2) Cathode (grade 08X18H10T steel); (3) Anode (low-alloy steel St3); (4) Impermeable membrane; (5) Inlet pipe; (6) Housing; (7) Current leads

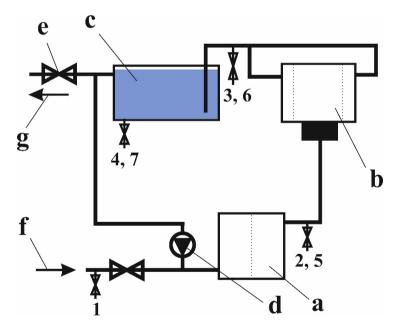


Fig. 4 Schematic for electrochemical wastewater treatment from a meat processing plant: (a) Graphite anodebased electrochemical module; (b) Iron anode-based electrochemical module (an electrochemical module to generate ferrates); (c) Maturation and homogenization chamber; (d) Circulation pump; (e) Valves; (f) Wastewater supply; (g) Removal of purified water (numbers indicate sampling sequence for the analysis)

High-frequency Smart GVI 30/60~V02 galvanic inverter was used as a current source. Current strength for the graphite anode-based electrochemical module was $70\pm8~A$, and that for iron chip anode-based electrochemical module was $60\pm6~A$.

Operating pressure in the graphite anode-based electrochemical module was $1.2\cdot105$ Pa. Water circulation rate was $35\cdot45$ m³/day. Water temperature at the beginning of the experiment was 14° C; the temperature rose to 19° C after dual transmission through the entire technological chain.

Corrosion testing was carried out using the gravimetric method [45,46], keeping samples in tap water and 3.5% sodium chloride solution with various sodium ferrate doses. Tests were carried out for 72 and 240 hours.



The mass corrosion index (g/m^2h) and corrosion rate (mm/year) were calculated premised on experimental research results.

3. Results and Discussion

An analysis of the Pourbaix diagram (Fig. 5) has evidenced that oxidation potential of hexavalent iron compounds largely depends on environmental acidity or potential of hydrogen (pH). Thus, in strongly acidic solutions (pH < 2), the system oxidation-reduction potential (ORP) reaches a value of 2.2 V, being superior to ozone, hydrogen peroxide, and permanganate anions by oxidation potential:

$$FeO_4^{2-} + 8H^+ + 3e^- \rightarrow Fe^{2+} + 4H_2O$$

An increase in pH leads to a decrease in oxidation potential of ferrate anions, and system ORP decreases to 0.57 V in a highly alkaline environment (pH \approx 14). In this case, ferrates function as relatively mild oxidants [32-34]:

$$FeO_4^{2-} + 4H_2O + 3e^- \rightarrow Fe(OH)_3 + 5OH^-$$

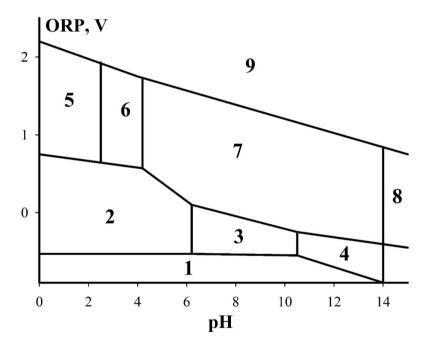


Fig. 5 Pourbaix diagram (potential-pH diagram) for Fe-H₂O system: (1) Fe;(2) Fe²⁺; (3) FeCO₃; (4) Fe(OH)₂; (5) Fe³⁺; (6) Fe(OH)₃; (7) Fe(OH)₃; (8) **Fe(OH)**₄; (9) **FeO²⁻**

Given the values of dissociation constants of ferric acid and ionic product of water, the mass fractions of various hexavalent iron compounds were calculated at different pH values. The obtained data are consistent with the research results obtained using the kinetic spectrophotometric method [47].

There are four forms of Fe (VI) compounds in aqueous solutions, depending on pH value (Fig. 6) [47]. In a highly acidic environment, the protonated form of iron acid $H_3FeO_4^+$ (1) predominates; in the pH range from 1.8 to 3.0, the solution mostly contains the molecular form of iron acid H_2FeO_4 (2); a further decrease in the acidity of the environment leads to the prevalence of hydroferrate $HFeO_4^-$ anion (3) and ferrate FeO_4^{2-} anion (4) if pH > 9. If pH=1.6, the concentrations of $H_3FeO_4^+$ and H_2FeO_4 are equal (pKa_1) . If pH=3.5, the concentrations of H_2FeO_4 are equal (pKa_2) .

Protonated and molecular forms of iron acid are not stable in aqueous solutions and are decomposed:

$$4H_3FeO_4^+ + H_2O \rightarrow 4Fe(OH)_2^+ + 3O_2$$

 $4H_2FeO_4 + 2H_2O \rightarrow 4Fe(OH)_3 + 3O_2$

The resulting poorly soluble hydroxy compounds of trivalent iron have a high specific surface area and act as an effective coagulant.

The greatest stability of hexavalent iron compounds is observed in neutral and alkaline media.



Reagents used in chemical processing and electrochemical generation thereof in solution are challenging in terms of technological equipment corrosion. This factor may diminish benefits of the proposed techniques. Therefore, it is vital to investigate the effect of electrochemically generated sodium ferrate on the change in the corrosion destruction rate of steel when justifying the possibility of introducing water purification technology and integrating electrochemical modules into the current technological scheme of the enterprise.

The conducted literature review has evidenced that sodium ferrate helps reduce the corrosion rate of structural steel, acting as a corrosion inhibitor [48-50]. This assumption has been confirmed by the experimental data (Fig. 7).

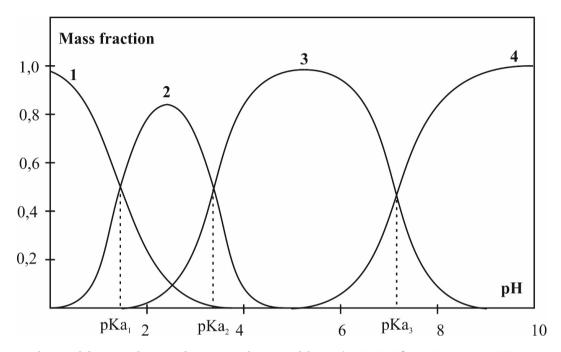


Fig. 6 Dependence of the mass fraction for various ferric acid forms $(1 - H_2 FeO_4^+, 2 - H_2 FeO_4^-, 3 - HFeO_4^-, 4 - FeO_4^{2-})$ on the pH value

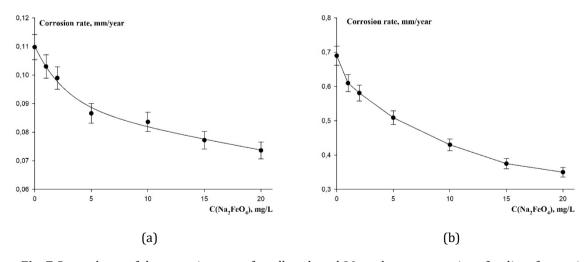


Fig. 7 Dependence of the corrosion rate of unalloyed steel 20 on the concentration of sodium ferrate in: (a) tap water (SanPiN 2.1.3684-21); (b) 3.5% sodium chloride solution (GOST 9.308-85)

An increase in pH leads to a decrease in oxidation potential of ferrate anions, and system ORP decreases to 0.57 V in a highly alkaline environment ($pH \approx 14$). In this case, ferrates function as relatively mild oxidants [25-27].

$$FeO_4^{2-} + 4H_2O + 3e^- \rightarrow Fe(OH)_3 + 5OH^-$$



Further research was aimed at investigating the effectiveness of electrochemical treatment in reducing the concentration of various contaminants in solution.

In Fig. 4, the numbers indicate sampling sequence for the analysis of the main components: (1) initial wastewater; (2) water exiting the graphite anode-based module after operating in circulation mode for 45 minutes; (3) water exiting the iron anode-based module after operating in circulation mode for 45 minutes; (4) water exiting the maturation and homogenization chamber after operating in circulation mode for 45 minutes, and chamber exposure for 20 minutes; (5) water exiting the graphite anode-based module after operating in circulation mode for 90 minutes; (6) water exiting the iron anode-based module after operating in circulation mode for 90 minutes; (7) water exiting the maturation and homogenization chamber after operating in circulation mode for 90 minutes, and chamber exposure for 20 minutes.

Considering that water, being a reagent, contains electrochemically generated components after electrochemical treatment, each water sample selected for the study was mixed in a 1:1 ratio with initial wastewater for further chemical analysis. When an electric current is passed through the electrochemical graphite anode-based module, reduction reactions of water and hydronium ions (Equations 1 and 2) occur on the surface of steel cathode. Here, nitrates and nitrites are also reduced due to denitrification (Equations 3-8).

The processes designated by Equations (3) and (6) are of utmost importance for removing nitrogencontaining components from wastewater due to the formation of gaseous nitrogen slightly soluble in water. The latter fails to participate in further electrochemical and chemical transformations and is released from the solution during the process.

Oxidation of water and hydroxide anions (Equations 9 and 10), and that of ammonium compounds (Equations 11-13) occur on the surface of bulk graphite anode in the electrochemical module:

$$\begin{array}{lll} 2H_2O-4e^-\to O_2+4H^+ & \varphi_{SHE}^0=+1.23V & (9) \\ 40H^--4e^-\to O_2+2H_2O & \varphi_{SHE}^0=0.401V & (10) \\ NH_3\cdot H_2O+30H^--3e^-\to \frac{1}{2}N_2+4H_2O & \varphi_{SHE}^0=-0.737V & (11) \\ NH_3\cdot H_2O+70H^--6e^-\to NO_2^-+6H_2O & \varphi_{SHE}^0=-0.15V & (12) \\ NH_3\cdot H_2O+90H^--8e^-\to NO_3^-+7H_2O & \varphi_{SHE}^0=+0.12V & (13) \end{array}$$

Equation (11) is of profound interest for denitrification by the reason stated above. Electrochemical generation of peroxides and ozone may also occur on the graphite anode surface. The products formed according to Equations 14-16 have strong oxidizing properties, and keep acting as oxidizing agents and disinfection reagents in wastewater after the electric current interruption:

Thus, electrochemical modules with both graphite and iron anodes operate in a complex wastewater treatment system periodically (10-20% of total time). These could considerably reduce energy costs and eliminate the major drawback of the efficient use of an electrochemical module, namely, energy intensity.

When the electrochemical iron anode-based module (low-alloy steel chip) operates, the reactions (Equations 1-8) occur on the cathode surface similar to those in the graphite anode-based module. The



processes of electrochemical ferrate generation occur on the anode surface along with the reactions designated by Equations 9-13:

$$Fe + 80H^{-} - 6e^{-} \rightarrow FeO_{4}^{2-} + 4H_{2}O$$

 $2[Fe(OH)_{4}]^{-} + 3ClO^{-} + 2OH^{-} \rightarrow 2FeO_{4}^{2-} + 3Cl^{-} + 5H_{2}O$
 $2FeO(OH) + 3ClO^{-} + 4OH^{-} \rightarrow 2FeO_{4}^{2-} + 3Cl^{-} + 3H_{2}O$

The resulting ferrates, when exposed to organic substances, are reduced and transformed into iron (III) hydroxide in the environment similar to neutral. The latter acts as a coagulant in solution:

$$FeO_4^{2-} + 4H_2O + 3e^- \rightarrow Fe(OH)_3 + 5OH^-$$

Iron (III) hydroxide interacts with fine particles and causes them to precipitate (sedimentation). This results in wastewater clarification and discoloration (Fig. 8).

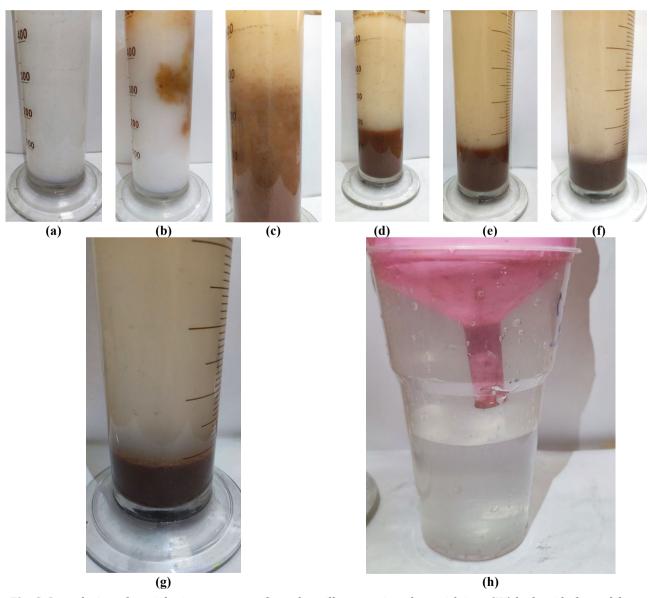


Fig. 8 Coagulation of particles in wastewater from the milk processing plant with iron (III) hydroxide formed from electrochemically generated sodium ferrate: (a) stock solution;(b) addition of Fe(OH)₃; (c) 1 min exposure; (d) 7 min exposure; (e) 8 min exposure; (f) 10 min exposure; (g) 15 min exposure; (h) filtered solution



Coagulation with iron (III) hydroxide, formed during ferrate reduction in the neutral environment, proceeds 1.5-2.0 times faster than that with hydroxy compounds of ferric iron formed during the hydrolysis of iron (III) salts, e.g., FeCl₃ [51-53]. The research results are shown in Fig. 9.

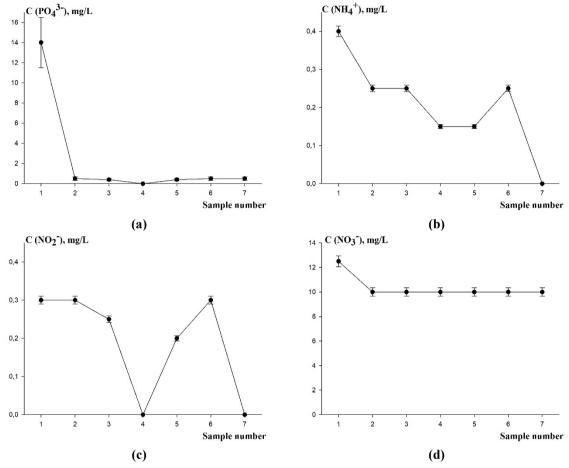


Fig. 9 Dependence of; (a) phosphates; (b) ammonia nitrogen; (c) nitrites; (d) nitrates concentration on timelocation sampling

The research results have evidenced that the low value of the solubility product of iron (III) phosphate $(SP_{(FePO_4)} = 1.29 \cdot 10^{-22})$ provides for efficient discharge of phosphate anions from purified water. High oxidation potential of electrochemical wastewater treatment products, including ferrates, facilitates reducing concentration of ammonia nitrogen and nitrites, which are oxidized predominantly to nitrates. This factor should lead to an increase in the concentration of nitrates in solution. However, a slight decrease in nitrate content is explained by reduction thereof on the cathode surface both in the graphite and the iron anode-based module.

4. Conclusion

The conducted research has confirmed the efficiency of using an electrochemical ferrate generation module in the overall wastewater treatment scheme from a meat processing plant. The following basic functions of ferrate anions in solution have been empirically validated:

- Oxidant its oxidation potential largely depends on the acidity of the medium; it acts as a mild oxidizing agent in neutral and slightly alkaline media for the pH values between 6 and 8 (E0 = 1.3-1.5 V)
- Coagulant the reduction of ferrates to hydroxy compounds of trivalent iron together with the ability thereof to form poorly soluble compounds with phosphates leads to the effective removal of the latter from wastewater
- Corrosion inhibitor an increase in the concentration of ferrate anions to 20 mg/L in solution leads to a decrease in steel corrosion rate by twofold both in tap water and in a sodium chloride solution simulating sea water, which determines the advantages of using this technology in practice

This paper renders comprehensive research into commercial feasibility of using flow-type electrochemical modules for wastewater treatment in the meat processing industry. The research findings provide for the advancement of integrated water treatment solutions and highlight the importance of implementing effective



treatment processes to minimize the environmental impact from meat processing plants. Thus, it has been experimentally proven that a complex of electrochemical graphite and iron anode-based modules contributes to the achievement of the research goal, allows for the efficient wastewater treatment from meat processing enterprises, and reduces the consumed time, resources, energy, and waste. Further research would help expand the range of practical applications of electrochemical technologies for wastewater treatment from food industry enterprises as a whole, pharmaceutical enterprises, and health facilities.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

All authors have contributed to this work.

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