Assessment of the efficiency of innovative reagents for purification of water from the Sava River (Belgrade)

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Abstract

There is a growing focus on enhancing environmental safety in Serbia. The development of hydrosphere protection technologies is one of the priority tasks, the solution of which will considerably improve the quality of life for the population and bring the country closer to the EU standards. The Sava River is one of the largest waterways running through Belgrade. In this work it was shown that the use of complex titanium-containing coagulants allows not only for the efficient removal of dispersed particles from water and a 70 % reduction in organic compound content, but also a 66 % decrease in the level of microbiological contamination. The use of sodium ferrate, a coagulant and bactericide, allows for a significant reduction of pollutant content in water as well as complete water decontamination. It was demonstrated that the use of a complex titanium-containing reagent considerably increases the sedimentation rate of coagulation sludge by 20 to 30 % and the filtration rate by 10 to 20 %.

Keywords: Water treatment, titanium-containing coagulant, sodium ferrate.

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1. INTRODUCTION

Serbia is a beautiful, small country located in Southeast Europe, at the heart of the Balkan region. Currently, the country is preparing to accede to the European Union, which means it should fulfil several conditions (environmental, economic, political, *etc*.) imposed on all potential and current members. One of the most important requirements set for Serbia is to increase the level of environmental safety. The list of the main trends includes improving the quality of atmospheric air, constructing and operating waste treatment facilities, and organizing systems for the collection, sorting and recycling (or incineration) of waste. An equally important area is the prevention of microbiological contamination of drinking water and wastewater (The Drinking Water Directive 2020 (2020/2184) [1]. The implementation of these measures will not only allow meeting the EU requirements, but also achieving the 3, 6, 11 and 14 sustainable development goals.

Unfortunately, at present, these trends are not being implemented in practice, and the main environmental protection measures are only at the elaboration stage (except for Vojvodina). Currently, most of household and industrial wastewater flows into the rivers either after minimal treatment or without it. Large cities are provided with drinking water from ground (alluvial) sources, with 45 % spent for the needs of population, 25 % for industrial purposes, and 30 % for other uses (including agriculture) [2-5].

Industrial and sewage wastewater as well as water of surface runoff from agricultural sites is transported *via* a system of pipes into rivers downstream the flow course or is used as irrigation water for aeration fields. The major pollutants in such natural and wastewater streams nitrogen (nitrate/ammonium), phosphorus, and dissolved organic compounds that can serve as nutrients for sludge microorganisms or plants, and their concentrations and suitability for such purposes are indicated by chemical oxygen demand (COD) and by biochemical oxygen demand (BOD).

Basic physicochemical purification methods are used to remove the above pollutants from water, often in combination with filtration/sorption processes and reverse osmosis, followed by decontamination. Coagulants based on aluminium or iron salts are employed as the main consumable reagents. Although traditional reagents are highly efficient and moderately priced, they are not always suitable for treating wastewater with complex compositions [6].

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The need to use filtration/sorption arises from the presence of xenobiotics and dissolved organic compounds such as pharmaceutical substances, pesticides, and petroleum products, in the water. Reverse osmosis is necessary when concentrations of salts or heavy metals significantly exceed safe limits. To ensure bacteriological safety, chlorine-containing reagents (such as hypochlorite) are commonly used; in some cases, ozone or ultraviolet radiation is also employed.

To address these challenges, innovative reagents such as coagulants based on titanium compounds [7-10] or the coagulant-bactericide sodium ferrate [11-12] can be used. The advantages of ferrates over traditional regents are widely noted by researchers and scientists. It has been demonstrated that, due to their pronounced oxidizing effect, ferrates are less prone to form organic iron-containing complexes. Equally important is their ability to reduce the content of dissolved organic compounds such as phenols, pesticides and xenobiotics, thereby improving organoleptic quality indicators (taste, UV 254, and odour) of water. The use of ferrates requires significantly lower reagent costs for pH correction of the treated water, and the resulting coagulation sludge settles more easily and rapidly than that produced by pure aluminium or iron salts [13-15].

Titanium-containing reagents, in turn, can reduce coagulant consumption by a factor of 2 or more and significantly improve the removal efficiency of suspended solids as well as suspended and dissolved organic compounds [16-18]. These reagents are weakly bactericidal, while the resulting sludge is easier to filter and is less toxic as compared to aluminium sludge [19-20].

Summarizing the above, we can conclude that ferrate and titanium-containing coagulants are highly promising for use in the treatment of surface water sources. The main objective of this work was to assess the quality of surface water in the Sava River and to develop a process flow diagram for water treatment/purification using innovative reagents, specifically titanium-containing and ferrate coagulants.

2. MATERIALS AND METHODS

The research was conducted in the wastewater treatment laboratory at the Department of Industrial Ecology, Faculty of Biotechnology and Industrial Ecology, D. Mendeleev University of Chemical Technology of Russia (Moscow, Russia).

Water samples (5 dm^3) were taken from the "Savska Promenade" area of Belgrade between July 8 and 25, 2024. This sampling point was chosen because the low water flow rate at this location (resulting in minimal turbidity) and the absence of man-made sources of domestic wastewater discharge in the immediate vicinity (only storm drains are present).

Jar-tests were performed in 5 repetitions for each dose of reagent and were conducted on a JLT 4 laboratory flocculator (VELP, Italy) using the standard procedure [9-10,21]. The rapid coagulation phase (intensive mixing of the coagulant in water) lasted 120 s, the flocculation phase (slow coagulation) is 480 s, and the sedimentation time was 20 min.

Coagulant samples were aqueous solution of aluminum sulphate Kemira ALS – 8.0 wt.% and polyaluminium chloride Kemira PAX-XL-18 – 18 wt.% produced by Kemira (Kemira, Finland) and a sample of innovative complex titanium-containing coagulant obtained according to the previously described method (95 wt.% aluminum sulphate + 5 wt.% TiO_2) [10].

Sodium ferrate (synthesized by electrochemical method at department industrial ecology Mendeleev University of Chemical technology, Russia, Moscow), a coagulant-oxidizer (bactericide) obtained in the steam process of anodic dissolution of iron in alkalis (11.2 g dm^{-3} by Fe^{6+}), was also tested as an unconventional reagent. This reagent has appeared on the market quite recently but has proven itself well in the processes of cleaning wastewater of complex composition (for example, landfill leachate) [12, 22].

The following equipment was used to determine the quantitative and qualitative characteristics of wastewater samples.

The concentration of heavy metals was determined by atomic -emission spectroscopy with magnetic plasma by a Spectrosky device (Skygrad, Russia) and by a spectrophotometer DR 6000 (HACH, USA). To determine the particle size of coagulation sludge, a digital meter Analysette 22 NanoTec Fritsch (Germany) was used. pH was measured using a digital meter (pHHQ11B, HACH, USA). The content of suspended solids and sedimentation rate were determined using a portable turbidimeter (HANNA 98703, HACH, USA), as the suspended solids content serves as a rapid indicator of water pollution and reagent treatment effectiveness.

To determine individual parameters of the river water samples before and after the reagent treatment, the following analytical control methods were used.

Oxidisability was determined by the reference methods (Dichromate oxidizability) [23]. The content of oils (gasoline, diesel) was determined by the Soxhlet method based on extraction with carbon tetrachloride followed by IR spectrophotometry using a KN-2N device (Sibecopribor, Russia). The content of dissolved salts (mineralization) was determined gravimetrically. The degree of microbiological contamination of the water was determined using test kits [24].

The selected indicators are highly significant and enable evaluation of both source water quality and the efficiency of reagent-based purification.

The filtration rate was determined by passing a specified volume of pre-coagulated water through a 15 μm filter for 60 s and then measuring the volume of filtrate collected.

The error in the results of analyses for the main pollutants did not exceed 15 % (calculated error + measurement error of the device).

3. RESULTS AND DISCUSSION

At the first stage of research, the composition of water was determined by chemical and bacteriological indicators. Data on the content of pollutants and the maximum concentrations established by the regulatory framework for household water [1] are presented in Table 1.

Table 1. *Results of studying water quality in the Sava River*

Indicator	In water	Standard [1]
pH	7.3	6.5 to 9.5
Turbidity, NTU	49.8	1.0
Oxidisability, mg(O) dm^{-3}	14.8	5.0
Content of NO_3, mg dm^{-3}	4.4	50
Content of NH_4, mg dm^{-3}	0.2	0.5
Content of PO_4, mg dm^{-3}	0.85	0.05*
Content of Al, mg dm^{-3}	0.3	0.2
Content of Fe, mg dm^{-3}	0.45	0.2
Content of oil, mg dm^{-3}	0.12	0.05*
Mineralization (salinity), mg dm^{-3}	212	500
Number of *Klebsiella pneumoniae,* cells *per* mL	500	0
Number of *Enteroccocus faecalis* cells *per* mL	1,500	0
Number of *Escherichia coli* cells *per* mL	1,000	0
Number of *Staphylococcus aureus* cells *per* mL	5,500	0
Colony forming unit (CFU), cells *per* mL**	12,800	0

*The indicator is not standardized for water in the Republic of Serbia, but is currently used in Russia and the Asian region (Sanitarian rules 1.2.3685-21 and WHO/SDE/WSH/05.08/123, respectively).
**Not found in water samples: *Citrobacter freundii; Salmonella Enteritidis; Salmonella flexneri; Proteus mirabilis; Aspergillus niger; Saccharomces cerevisiae; Candida albicans; Pseudomonas aeruginosa*

Data in Table 1 show that water in the Sava River contains phosphate anion, nitrates and chemical oxygen demand in considerable excesses. Impurities of metals such as Zn and Cu were found in water in concentrations not exceeding the current standards (<0.2 mg dm^{-3}). Data in Table 1 also show that the selected sample is characterized by microbiological contamination with pathogenic microorganisms such as *Staphylococcus aureus* as well as a significant water contamination with *Klebsiella pnemoniae*, *Enterococcus* and *E. coli*. A high level of biological contamination can lead to outbreaks of various diseases and the active growth of biofilms on the surfaces of pipeline equipment [25].

The next stage of the research involved assessing the efficiency of reagent treatment for reducing turbidity and the content of dissolved organic compounds in the water. The results of the experiment are presented in Figures 1 and 2.

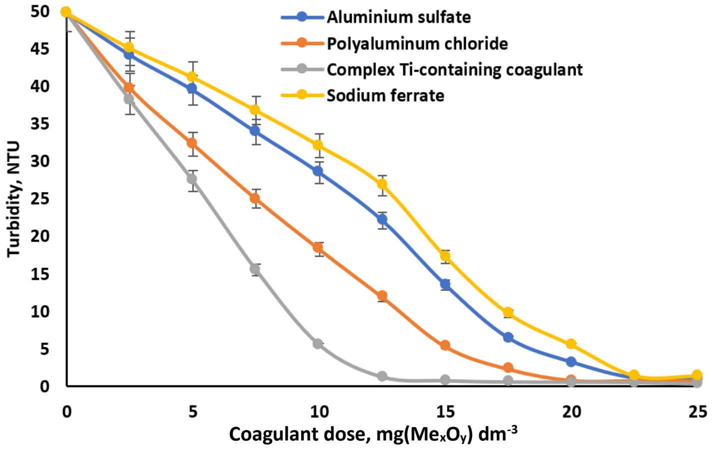

Fig. 1. *Effect of coagulant type and dose on turbidity index.* Me_XO_Y *stand for aluminium sulphate and polyaluminium chloride* Al_2O_3*, for sodium ferrate* Fe_2O_3 *and for complex Ti-containing coagulant -* Al_2O_3*+*TiO_2

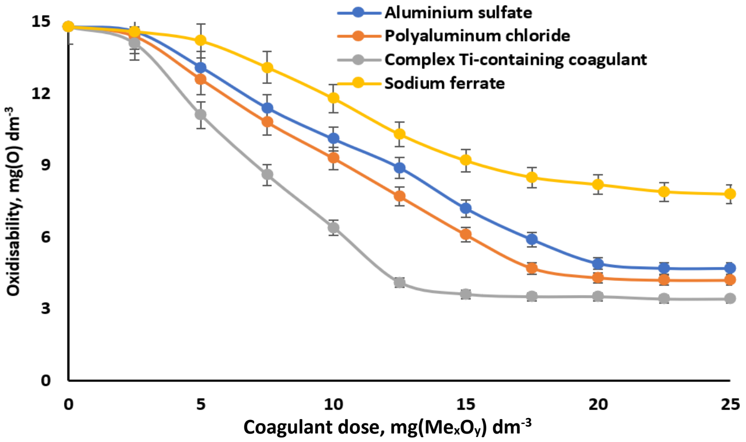

Fig. 2. *Effect of coagulant type and dose on oxidisability index.* Me_XO_Y *stand for aluminium sulphate and polyaluminium chloride* Al_2O_3*, for sodium ferrate* Fe_2O_3 *and for complex Ti-containing coagulant -* Al_2O_3*+*TiO_2*.*

Data from Figures 1 and 2 show that the complex titanium-containing coagulant was the most efficient, achieving the lowest residual turbidity and oxidizability values at a dosage of 12.5 to 15.0 mg dm^{-3}. High efficiency of the complex reagent is due to the parallel processes of primary nucleation (neutralization coagulation) of positively charged hydrolysis products of aluminium compounds and negatively charged hydrolysis products of titanium salts, as well as polycondensation/polymerization processes (flocculation) of meta- and orthotitanic acids being formed during hydrolysis [26-28]. The size of dispersed titanium particles in purified water was 0.8 to 0.9 µm, which, according to literary data, indicates the safety of the reagent for humans (absence of cytotoxicity) [29].

To achieve a similar residual turbidity for the traditional aluminium-containing reagents, dosages were 20 and 22.5 mg dm^{-3} for polyoxychloride and aluminium sulphate, respectively. These dosages are standard for purification of slightly turbid waters for drinking purposes.

Sodium ferrate demonstrated the lowest efficiency in reducing turbidity (residual value of 1.4 NTU exceeds the standard), but the effect of complete water decontamination was achieved during treatment (Table 3). Low efficiency of ferrate in reducing turbidity can be attributed to the formation of iron complexes with organic humic acids.

For all coagulant samples, a decrease in the content of dissolved organic matter (indicated by oxidisability and colour) was recorded; 70 % for the complex reagent, 40 to 50 % for the reagents containing aluminium, and 30 % for sodium ferrate.

The sediments (sludge) obtained using ferrate and the complex titanium-containing reagent differed from those formed with the traditional coagulants based on aluminium salts used in this study. Data on the technological parameters for sediment separation (precipitation/filtration) are presented in Table 2.

Table 2. *Technological parameters for sludge separation for different reagent types*

Reagent type	Particle size range, μm	Sludge sedimentation time, min	Filtration rate, mL min^{-1}
Aluminium sulphate	340 to 430	6.5	53
Polyaluminium chloride	400 to 490	5.5	59
Complex Ti-containing coagulant	480 to 550	4.0	67
Sodium ferrate	640 to700	4.5	42

Data in Table 2 clearly show that the coagulation sludge forming when the complex titanium-containing coagulant and sodium ferrate are used has an increased sedimentation and filtration rate, which will improve the efficiency of the treatment facilities. Sludge sedimentation time was decreased by 20 to 30 % and filtration rate by 10 to 20 % compared to the traditional reagents.

So, the optimal dosages of reactants were determined as: 12.5 mg dm^{-3} for Complex Ti-containing coagulant and 22.5 mg dm^{-3} for all another reagent. Residual concentrations of pollutants and microbiological contaminants obtained with optimal doses of reagents are presented in Table 3.

Table 3. *Residual concentrations of pollutants at optimal dosages of reactants:*

Indicator	Source value	Complex titanium-containing coagulant	Aluminium sulphate	Polyaluminium chloride	Sodium ferrate	Standard
pH	7.3	7.1	6.9	7.1	8.1	6.5-9.5
Turbidity, NTU	49.8	0.45	0.9	0.74	1.2	1.0
Oxidisability, mg(O) dm^{-3}	14.8	3.6	4.9	4.3	7.9	5.0
Content of NO_3, mg dm^{-3}	4.4	4.1	4.1	4.1	4.1	50
Content of NH_4, mg dm^{-3}	0.2	0.2	0.2	0.2	0.2	0.5
Content of PO_4, mg dm^{-3}	0.85	0.03	0.07	0.04	0.2	0.05
Content of Al, mg dm^{-3}	0.3	0.05	0.07	0.06	0.1	0.2
Content of Fe, mg dm^{-3}	0.45	0.1	0.12	0.1	0.28	0.2
Content of Ti, mg dm^{-3}	0	0.07	0	0	0	0.1
Content of oil, mg dm^{-3}	0.12	0.03	0.05	0.04	0.05	0.05*
Mineralization (Salinity), mg dm^{-3}	212	219	225	223	227	500
Number of *Klebsiella pneumoniae,* cells *per* mL	500	145	470	450	0	0
Number of *Enteroccocus faecalis* cells *per* mL	1,500	480	1,400	1,400	0	0
Number of *Escherichia coli* cells *per* mL	1,000	350	950	920	0	0
Number of *Staphylococcus aureus* cells *per* mL	5,500	1,250	4,800	4,500	0	0
Colony forming unit (CFU), cells *per* mL**	12,800	3,900	11,650	10,500	0	0

Table 3 shows that, despite its high efficiency, the complex titanium-containing coagulant does not meet the standard for microbiological safety. It should be noted that water treatment with the titanium-containing reagent made an almost 3-fold reduction in the level of biological contamination of water. Sodium ferrate was the least efficient in terms of removing suspended solids (turbidity) and other indicators, while providing complete water decontamination/disinfection.

The sludge produced by the coagulation treatment process is classified, based on its chemical composition, as low-hazard (non-toxic) waste and can be disposed of in solid municipal waste landfills.

The cost of titanium-containing reagents and ferrates is, on average, 10 to 15 % higher than that of aluminium sulphate, but about 50 % lower than that of PAX. However, their increased cleaning efficiency, faster sedimentation and filtration processes, and disinfectant effect are expected to improve the performance of treatment facilities and reduce operating costs.

Analysis of the obtained results indicates that reagent treatment enables efficient purification of water from the Sava River. The residual concentrations of almost all analysed pollutants are within the established standards.

The obtained results are in good agreement with the results reported for the use of pure titanium salts, while the cost of the innovative complex reagent (synthesized for the first time) was 2-3 times lower [7-8].

Physicochemical purification by coagulation/flocculation and sedimentation cannot be used as the sole treatment process but should be incorporated into a comprehensive water treatment system such as the one presented in Figure 3.

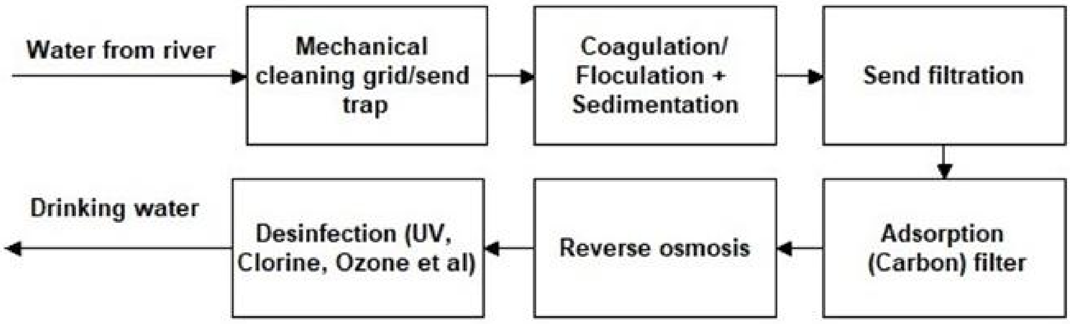

Figure 3. *Scheme of a proposed complex purification system of water from the Sava River (Belgrade)*

The proposed process flowsheet (Figure 3) enables the production of high-quality clean water from portions of the Sava river that receive primarily surface runoff. The key area of using the treated water will be to meet the needs of industrial enterprises (*e.g.* paint and varnish, petrochemical industries).

The adsorption and reverse osmosis purification stages (standard drinking water purification methods) are required because of possible fluctuations in pollutant concentrations depending on the season, while these units can be in the form of bypass lines and be connected as needed.

4. CONCLUSION

In this work, the quality of surface water from the Sava River (Belgrade, Serbia) was assessed first. The results showed that the primary pollutants in the water were suspended solids (turbidity), phosphates, dissolved organic compounds (as indicated by oxidizability and colour), and an extremely high level of microbiological contamination, including pathogenic *Staphylococcus aureus.*

In the next step, it was demonstrated that using a complex titanium-containing coagulant enables highly efficient removal of most undissolved impurities (suspend solids) from water, as well as a 70 % reduction in organic impurity content. Achieving equivalent purification efficiency with traditional aluminium-containing coagulants required an effective dose that was, on average, 1.5 to 2.0 times higher.

The use of sodium ferrate enabled complete elimination of microorganisms; however, the residual turbidity and organic matter content remained much higher compared to treatments with aluminium salts or the complex reagent.

It was demonstrated that the use of the innovative, complex titanium-containing coagulant significantly intensifies the formation and settling of coagulation sludge. Based on the obtained data, a process flow diagram was developed for purification of water from the Sava River for household use, in accordance with the Drinking Water Directive 2020 (2020/2184) [1].

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Conflicts of Interest: *The author declares no conflicts of interest.*

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Procena efikasnosti inovativnog reagensa za prečišćavanje vode iz reke Save (Beograd)

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(Naučni rad)

Izvod

U Srbiji se sve više pažnje posvećuje unapređenju ekološke bezbednosti. Razvoj tehnologija za zaštitu hidrosfere predstavlja jedan od prioritetnih zadataka, čije će rešavanje značajno poboljšati kvalitet života stanovništva i približiti zemlju standardima Evropske unije. Reka Sava je jedan od najvećih vodotokova koji protiču kroz Beograd i predstavlja važan resurs koji zahteva efikasno upravljanje kvalitetom vode. U ovom radu je pokazano da primena kompleksnih koagulanata koji sadrže titanijum omogućava ne samo efikasno uklanjanje dispergovanih čestica iz vode i smanjenje sadržaja organskih jedinjenja za 70 %, već i smanjenje nivoa mikrobiološke kontaminacije za 66 %. Upotreba natrijum-ferata, koji deluje i kao koagulant i kao baktericid, omogućava značajno smanjenje sadržaja zagađujućih materija u vodi, kao i potpunu dekontaminaciju vode. Takođe je pokazano da kompleksni koagulacioni reagens koji sadrži titanijum značajno povećava brzinu sedimentacije koagulacionog mulja za 20 do 30 % i brzinu filtracije za 10 do 20 %.

Ključne reči: Tretman vode; koagulant sa titanijumom; natrijum-ferat.