

Stabilization of Iron Content in Drinking Water after Sampling – Study of Tap Water in Donetsk Region, Ukraine

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ABSTRACT

Ukraine traditionally suffers from acute shortage of quality drinking water, and this problem has intensified recently due to military operations on its territory. The lack of water laboratories in the combat area made it impossible to monitor the quality of drinking water. Due to the pollution of water resources and the destruction of water supply systems, the population is forced to use water unsuitable for drinking. One of the important indicators of pollution is the high iron content in drinking water, which exceeds the maximum permissible standards in many regions of Ukraine. To analyze the iron content in water, the spectrophotometric method with 1,10-phenanthroline was chosen, which is simplest, less labor-intensive and energy-consuming. If it is impossible to carry out the analysis at the point of water sampling, the result obtained has a high error due to a decrease in the concentration of iron in the water over time. The reason for this effect is the rapid transition of iron from soluble to insoluble form. The aim of the research is to study the influence of temperature and acidity on the dynamics of iron concentration in drinking water and to develop recommendations for analyzing water for iron content in laboratory conditions. It has been proven that drinking water must be acidified to a level of pH = 1 immediately after water sampling. In the case of analyzing previously unacidified water, it should be heated to a temperature of 40 °C or higher in the presence of hydrochloric acid. It was established that the amount of total iron in the analyzed water exceeds the amount of dissolved iron by 6.4 times. The correspondence of drinking water with the requirements of the standard for iron content can only be established by determining the content of total iron in all its species.

Keywords: drinking water, iron concentration, 1,10-phenanthroline, spectrophotometric method, dynamics

INTRODUCTION

Among the many global environmental and social challenges, the problem of drinking water quality is a priority. New data from Water Resources Institute show that 25 countries – housing one-quarter of the global population – face extremely high water stress each year, regularly using up almost their entire available water supply. And at least 50% of the world's population - around 4 billion people live under highly water-stressed conditions for at least one month of the year [World Resources Institute, 2023]. According to UN data, over 785 million people still lack basic services, and 144 million people still drink untreated surface water [IHE Delft, 2020]. 1 in 3

people globally do not have access to safe drinking water [WHO, 2019].

Ukraine is one of the least well-off countries in the world in terms of reserves of usable water resources. According to the definition of the European Economic Commission of the United Nations, a state whose water resources do not exceed 1.7 thousand m³ per year per person is considered water insecure. For example, in Canada, this indicator is 94.3, in Sweden – 19.7, in the USA – 7.4, in France – 3.4, in England – 2.5, in Germany – 1.9 thousand m³. In Ukraine, this indicator is 1 thousand m³, which corresponds to the 111th place among 152 countries of the world [UN, 2023]. Ukraine is a country that is rapidly losing its drinking water resources due to military

actions on its territory. One of the most devastating consequences of military operations is their impact on the environment, in particular on the state of water supply systems and the quality of drinking water. The partial destruction and evacuation of some water laboratories from combat zones does not allow monitoring the quality of drinking water. The destruction of the water supply infrastructure during the war is used to subjugate and demoralize the population. Therefore, water supply is a critical issue for the survival of Ukrainians. According to the World Bank report, as of February 24, 2023, Ukraine's direct losses from damage to water supply and drainage facilities amounted to \$2.2 billion USA [World Bank, 2023].

During the analysis of the quality of drinking water in Ukraine, it was established that in most regions the main pollutants of tap water are iron, turbidity and hardness salts. Groundwater, which makes up more than 50% of the total water resources and lies at depths of 30–200 m, has a high iron content, which is sometimes much higher than the norm. The high iron content in drinking water gives it a metallic aftertaste, after contact with air - a different color, and also causes water distribution networks to become overgrown with deposits. Iron is an extremely important element in the human body, but its excess is very harmful to health. This substance is concentrated mainly in the blood and, as a component of hemoglobin, takes part in the transfer of oxygen, as it is a biological catalyst. But with long-term intake in the body, the liver is oversaturated with iron oxide colloids, which destroy cells and cause cancer. Excessive iron in drinking water may have negative effects and leads to arteriosclerosis [Kempf and Wollert, 2020], diabetes [Sanjeevi et al., 2018], hemochromatosis [Regan, 2009], Alzheimer's disease [Kenkhuis et al., 2021], Parkinson's disease and multiple system atrophy [Li and Reichmann, 2016]. Therefore, the iron content in drinking water should not exceed 0.2 mg/dm^3 . According to the World Health Organization, the safe daily intake of iron for humans is 0.8–1.0 mg per kilogram of body weight [FAO, 1998].

The main sources of iron in drinking water are the following: surface water and ground water sources, water treatment plants, water delivery networks to consumers. Iron is most common in ground water. Sources of iron in water are igneous and sedimentary rocks. This element is released from rocks in the process of hydrolysis and the action of carbon dioxide dissolved in water. The

low pH of the water and the presence of organic compounds in it contribute to the transition of iron into the water. Other sources of iron in ground-water may include mine waters with pollutants, effluents from industrial enterprises [Starovoit et al., 2021; Pyshyev et al., 2023; Turchanina–Rybak et al., 2021], where substances with a high concentration of iron are used or processed.

Iron is part of the coagulant and can enter the water during water purification. The amount of coagulant and, accordingly, iron entering the water depends on the level of water pollution. The use of a coagulant containing iron can increase the concentration of iron in purified water several times during the water treatment process [Pikaar et al., 2014]. Water supply systems in Ukraine are made primarily of steel and cast-iron pipes. In the case of corrosion of the water supply system, iron can appear in hot and cold-water supply facilities. The amount of iron passing into the water depends both on the quality of the pipes and on the parameters of the water quality (hardness, dissolved oxygen, pH, nitrate, residual chlorine concentrations, Larson Ratio) [Lin et al., 2021].

Iron is present in water in the form of divalent and trivalent compounds – Fe(II) and Fe(III), accordingly. In aerobic conditions, for example, in surface waters, Fe(III) compounds predominate. They undergo hydrolysis and precipitate under the influence of water. Therefore, the concentration of dissolved iron compounds is usually small. In underground, that is, anaerobic conditions, iron prevails in the form of divalent Fe(II) ions dissolved in water. In groundwater, where contact with oxygen is limited, the iron content can be significantly higher [Ityel, 2011]. Being in a liquid, iron can also take organic, colloidal (water is cloudy, but without sediment), bacterial (formed by bacteria that use metal for their vital activity) forms, and in complexes [Rich and Morel, 1990].

There are many different methods that are used to test water for iron content. The most commonly used methods are the following: spectrophotometric methods [Nurchii, 2021], fluorescence [Cha and Park, 1998], flame and electrothermal atomic absorption spectrometry [Tautkus et al., 2004; Freschi et al., 2008], inductively coupled plasma-mass spectrometry [Jeroen de Jong et al., 2008], solid-phase spectrophotometric methods [Alberti et al., 2019], chemiluminescence [Bowie et al., 1998], inductively coupled plasma optical emission spectrometry [Cui et al., 2012], flow injection analysis (FIA) [Phansi et al.,

2019], thermal lens spectrometry (TLS) coupled to FIA [Tomsič et al., 2019]. The simplest, less labor-intensive and energy-consuming method is the spectrophotometric method [Fernandes et al., 2023]. This method with 1,10-phenanthroline is declared in the Standard ISO 6332:1988 Water quality – Determination of iron – Spectrometric method using 1,10-phenanthroline.

MATERIALS AND METHOD

Study location

This study was carried out in eastern Ukraine. The object of research was tap water from the water supply system of the city Pokrovsk, Donetsk region (Figure 1). The research area is located at coordinates 48°16'42.2"N 37°10'39.6"E. Nowadays, the city of Pokrovsk is in an active combat zone. Before the start of hostilities, water supply to the Donetsk region was carried out by transporting drinking water from the main water source - the Siverskyi Donets River via the Siverskyi Donets - Donbass canal. As a result of the destruction due to shelling of the canal and pumping stations, water supply along this route was stopped. Currently, an alternative source of drinking water is used from the Karlivka

Reservoir, as well as mine water and groundwater. These events led to a significant deterioration in the quality of drinking water. The total mineralization of drinking water in the Karlivka Reservoir was three times higher than that of the water in the Siverskyi Donets – Donbass canal. As a result, the salt content exceeded the maximum permissible values from 2 to 5 times. Among the substances whose concentration was above the maximum permissible values was iron, for which the excess was up to 2 times. At the same time, the composition of tap water constantly fluctuated and required monitoring.

Despite numerous studies on the iron content in water, determining its concentration is problematic for a number of reasons. This is primarily due to the nature and characteristics of the behavior of iron in aqueous solutions, which also depends on many factors. At the same time, the main criteria for choosing a research method are focused on optimizing the time, cost of analysis and the required accuracy of the study [Tran et al., 2023].

Materials

Materials used in this study were tap water samples, sodium acetate (CAS 127-09-3), acetic acid (CAS 64-19-7), hydrochloric acid (CAS

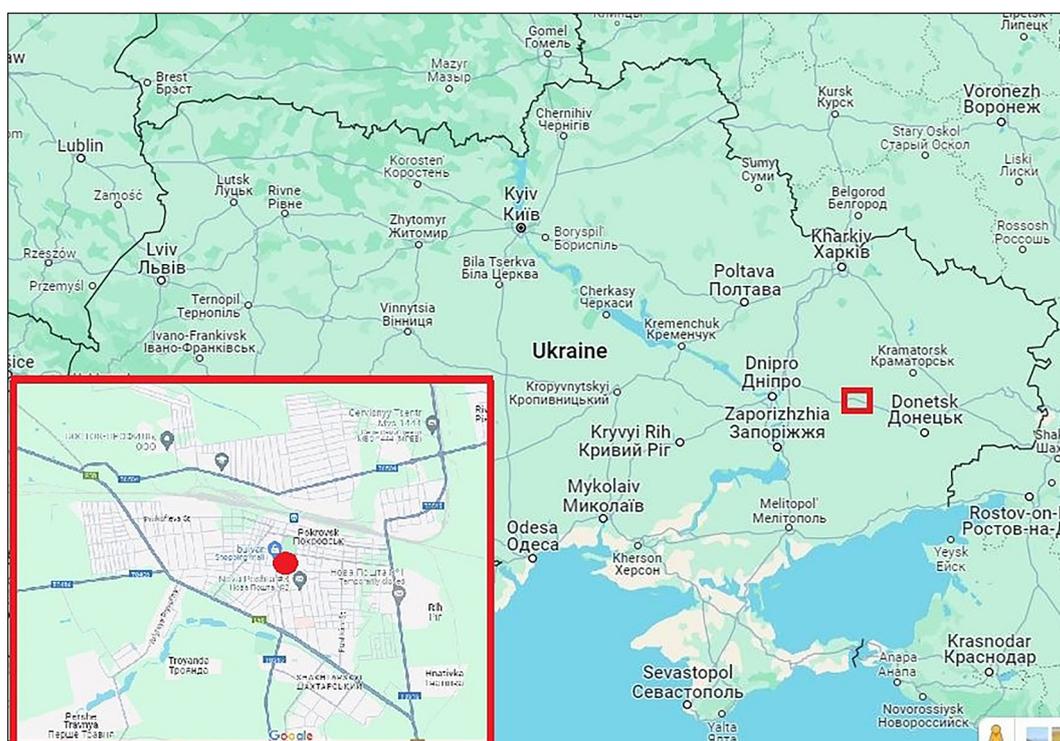


Figure 1. Geographical localization of the drinking water sampling site

7647-01-0), hydroxylamine hydrochloride (CAS 5470-11-1), 1,10-phenanthroline (CAS 66-71-7), ferroammonium alum $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (CAS 7783-83-7), distilled water. All reagents used for the analysis must be qualified as pure for analysis (PFA).

Equipment

Equipment used in this research were spectrophotometer UNICO 2150UV ($\lambda=200\text{--}1000\text{ nm}$) was used for measuring iron content in tap water samples, Mi-150 pH meter was used to evaluate the acidity level, dry-air thermostat SNOL 58/350 TermoLab was used for heating tap water samples during experiment. Additional equipment used were glass cuvettes (50 mm), analytical scales RADWAG AS 220R2, beaker glass, volumetric flasks, glass dropping pipettes, ceramic bowls.

Methodology of iron determination

To determine iron concentration, a calibration curve was constructed using the spectrophotometric method with 1,10-phenanthroline, which forms an orange-colored complex with iron. Following the procedure, a standard solution of ferroammonium alum with an iron concentration of 0.1 mg/ml was prepared. A working standard solution with an iron concentration of $5 \cdot 10^{-3}$ mg/ml was obtained by diluting the stock solution. For the series of standard solutions, specific volumes of the stock solution were accurately measured using a pipette and transferred to a set of 50 ml volumetric flasks. Hydroxylamine hydrochloride, acetate buffer, and 1,10-phenanthroline were then

added to these solutions. The prepared solutions were allowed to develop colour for 10–15 minutes and were then measured using a spectrophotometer at 510 nm. A solution containing all reagents except iron was used as a blank sample. Calibration curve is shown in Figure 2.

RESULT AND DISCUSSION

To determine the iron content in tap water, a 1-liter sample was collected in a beaker. A 25 ml portion of this sample was transferred to a 50 ml volumetric flask, and the necessary reagents were added. The solution was allowed to stand for 10–15 minutes. Then it was analyzed using a spectrophotometer. The iron content in the water was 0.130 mg/l, which is within the maximum permissible concentration (MPC) according to the Ukrainian water quality standard “Drinking water. Requirements and methods of quality control”. The standards for the content of total iron in drinking water from different sources according to the Ukrainian water quality standard is shown in Table 1.

The problem was that each subsequent analysis of the same water sample showed a result with a lower iron content. This raised doubts about the results obtained and sparked interest in studying this phenomenon. Thus, it was decided to study the dynamics of the iron concentration changes in water. For this purpose, a 1-liter sample of tap water was collected in a clean beaker. 25 ml of this water was extracted from the beaker at half-hour intervals. The analysis of the extracted water samples was performed in accordance

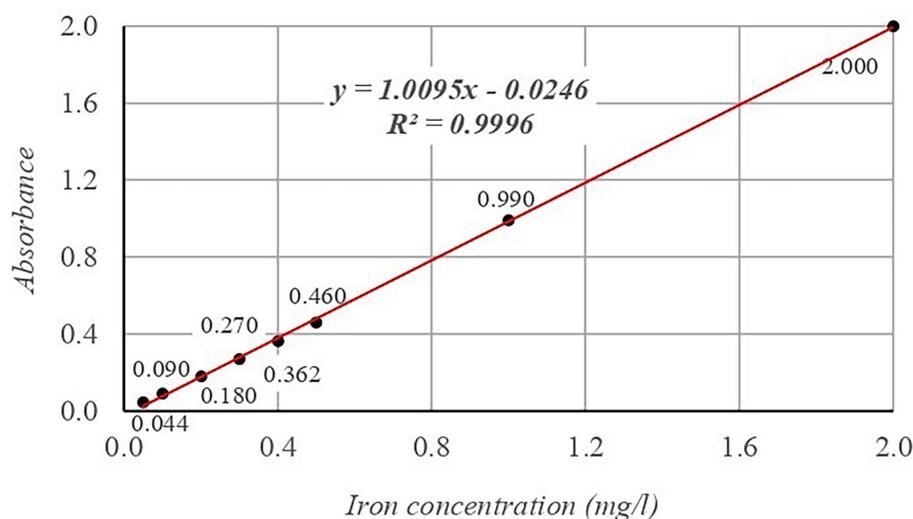


Figure 2. Calibration curve (glass cuvettes $l = 50\text{ mm}$, $t = 15\text{ }^\circ\text{C}$, $\lambda = 510\text{ nm}$)

Table 1. Standards for the content of total iron in drinking water from different sources

Name of the indicator	Tap water	Water from wells and boreholes	Bottled water from bottling points and pump rooms
Total iron, mg/dm ³	≤ 0.2	≤ 1.0	≤ 0.2

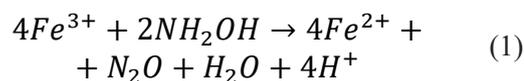
with the standardized procedure described in ISO 6332:1988. The temperature was 15 °C.

To ensure the reliability of the results, the experiment was replicated with a tap water sample collected on another day from the same sampling point. The initial iron concentration in this sample was measured at 0.080 mg/l. The different initial iron levels in the samples could potentially reflect a variation in water sources that could influence iron levels. The results of water samples studies are presented in Figure 3. These results provide insights into the dynamics of iron concentration in water samples and may help explain the main causes of the observed variations.

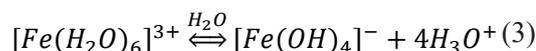
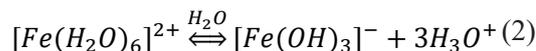
The decreasing concentration of iron in tap water over time indicates the instability of the water composition caused by ongoing chemical reactions among its components. The most substantial reduction occurs within the initial 30 minutes after water sampling. Subsequently, the rate of decrease slows down considerably, and the system approaches a quasi-equilibrium state. This may indicate that the ongoing chemical process is reversible. To fully understand this observed effect, it is critical to investigate the different species of iron present in tap water. This involves studying how each iron species interacts and transforms over time, and how these transformations affect the analysis process and contribute to the overall decrease in iron concentration.

The analysis method used is based on the reaction of ferrous ions with 1,10-phenanthroline in the pH range of 3–9 with the formation of a stable orange-red complex.

The color rapidly develops at a pH of 3.0–3.5, necessitating the addition of an acetate buffer to the solution. Hydroxylamine hydrochloride is used to convert ferric ions to ferrous ions.



For the development of a colored complex, it is essential that iron is present in a soluble form. Iron can exist in aqueous solutions in two soluble forms: Fe (II) and Fe (III). These forms are typically present as aqua complexes, specifically $[Fe(H_2O)_6]^{2+}$ and $[Fe(H_2O)_6]^{3+}$ for ferrous and ferric ions respectively. Both of these aqua complexes exhibit Lewis's acid properties. In an aqueous environment, these complexes are prone to hydrolysis reactions, leading to the formation of hydroxide species and potentially altering their solubility and reactivity [Esmailbeig et al., 2022].



The solubility of the hydrolysis products is significantly reduced, which complicates their interaction with hydroxylamine hydrochloride and suppresses the formation of a colored phenanthroline complex. This reduced solubility may be a critical factor preventing successful detection and quantification of iron. Moreover, since the investigated tap water was in contact with atmospheric air, Fe(II) underwent oxidation to Fe(III). This oxidation process results in the formation of trivalent iron, which is significantly less soluble

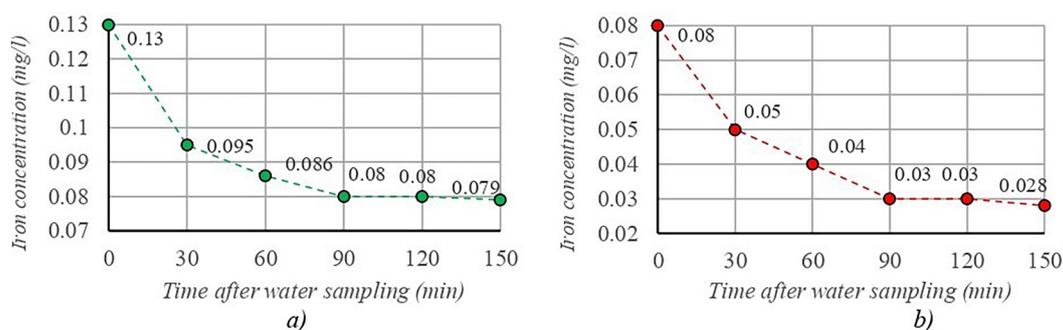


Figure 3. Iron concentration change in tap water over time: (a) at the initial concentration of iron 0.13 mg/l; (b) at the initial concentration of iron 0.08 mg/l

than divalent iron [28]. As a result, hydroxylamine is unlikely to be able to effectively reduce Fe(III) during the analytical procedure.

In addition to these factors, the tap water contains a variety of other dissolved salts, including bicarbonates. The presence of carbonate ligands plays a crucial role in this context. These ligands sharply reduce the redox potentials of high-valent transition metals, thereby facilitating their involvement in catalytic redox processes [Patra et al., 2021]. The mechanism of this process includes carbonate ligands donating electron density to the metal center, which stabilizes metals in higher oxidation states and promotes efficient electron transfer reactions. Consequently, an increase in the concentration of bicarbonates leads to a significant acceleration in the reaction rate between $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$ and O_2 , highlighting the profound impact of these anions on the dynamics of the reaction. This factor is even more significant for water from an alternative supply source, which has a notably higher salt content.

Thus, transient species such as Fe(II) create additional difficulties for analysis due to their fleeting existence and highly reactive nature. Therefore, the reduction in dissolved iron concentration in tap water over time can be attributed to the intricate parallel-sequential process of oxidation and hydrolysis of the iron (II) aqua complex [Vijay et al., 2023]. To effectively prevent iron hydrolysis reactions and increase iron solubility, it is essential to lower the pH of the solution. Therefore, when sampling water for analysis, it is strongly recommended to immediately acidify the sample. This can be done by adding hydrochloric acid or sulfuric acid to adjust the pH to 1.

According to ISO 6332:1988, if the solution contains undissolved iron, iron oxides, or iron complexes, a relevant pre-treatment is required. In particular, to dissolve these components, the solution should be boiled with acid. During the settling process, no visible signs of insoluble iron were observed. This probably happens because the oxidation and hydrolysis products form small colloidal particles, which are too small to be seen with the naked eye. Therefore, some analysts may skip sample acidification especially if the analysis is performed immediately at the sampling site. This can lead to incorrect results, as performed research has shown that iron concentration decreases rapidly immediately after sampling (Figure 3).

To investigate the impact of acidity on the iron concentration in tap water, an analysis was carried out involving the preheating of the water in the presence of hydrochloric acid. Initially, 25 ml of the tap water sample was transferred into a ceramic bowl. Following this, a one drop of hydrochloric acid, with a concentration of 36.5%, was introduced to adjust the pH of the solution to about 3. Additionally, 1 ml of hydroxylamine was added to the mixture. Then pH level of the resulting solution was measured using a laboratory Mi-150 pH meter, ensuring precise monitoring of the acidity during the experiment.

The collected samples were transferred to a dry-air thermostat and maintained at a constant temperature of 40 °C. The bowls containing the samples were left uncovered to ensure continuous exposure to air. At half-hour intervals, one bowl was carefully removed from the thermostat. After that, the contents of the removed bowl were transferred into a 50 ml volumetric flask. To this flask, 2 ml of acetate buffer and 1 ml of

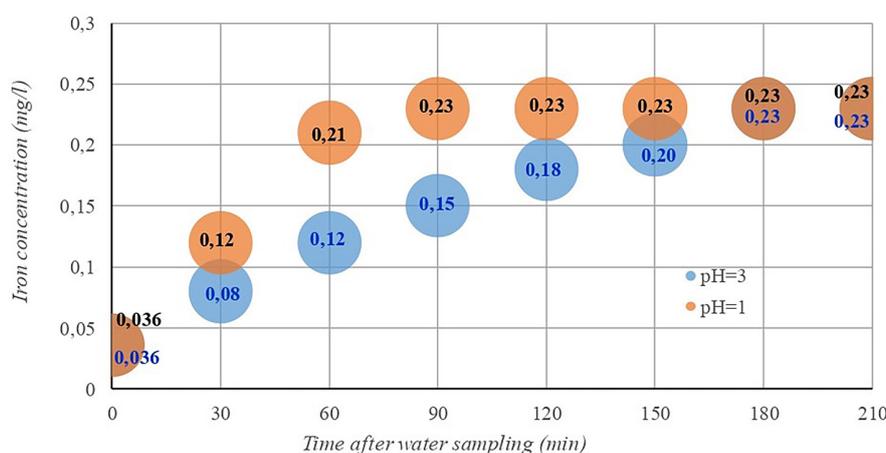


Figure 4. Dynamics of iron concentration in tap water after heating it at $t = 40\text{ }^{\circ}\text{C}$ with the addition of HCl

1,10-phenanthroline solution were added. The mixture was then diluted with distilled water until the total volume reached 50 ml. This prepared solution was allowed to sit for 10–15 minutes, during which it cooled and developed color. The solution was then analyzed using a spectrophotometer to measure the iron concentration.

A similar experimental procedure was carried out with another set of samples where the acidity of the water was reduced to pH 1.0 to observe any changes in iron concentration under the new conditions. The results of these experiments are summarized and illustrated in Figure 4. The results of the experiment demonstrated that the iron content in tap water increased when the water was preheated and treated at a lower pH level. In both experimental setups, the final iron concentration was measured at 0.230 mg/dm³. Specifically, at pH 1 this concentration was reached after 90 minutes, whereas at pH 3 it required 180 minutes.

These observations show that the acid effectively dissolved all the insoluble iron in the water in both scenarios. Notably, the dissolution process occurred twice as quickly at the lower pH level. This indicates a direct correlation between the acidity of the solution and the efficiency of iron dissolution. In contrast, an acetate buffer at pH 3.5 was ineffective in dissolving the insoluble forms of iron. Its role was limited to two key functions: first, to quantitatively bind Fe²⁺ ions into the phenanthroline complex [(C₁₂H₈N₂)₃Fe]²⁺, and second, to inhibit the oxidation of Fe²⁺ to Fe³⁺, which accelerates with increasing pH.

It is important to note that the total iron concentration in the water is 6.4 times higher than its initial concentration measured in water without acidification. This indicates that most of the iron in the water was in an insoluble state. In addition, if the water was evaluated by its initial iron concentration, it could be characterized as high quality. However, in fact, the concentration of iron in it is much higher that is a significant concern because it exceeds 0.2 mg/dm³ - the regulatory limit set for safe water quality.

The presence of high levels of insoluble iron in water can be caused by several factors. One primary cause is the oxidation of ferrous ions within the tap water. Additionally, loose deposits found in water supply pipes can also enhance to high levels of insoluble iron. These deposits consist of a mixture of iron oxides and hydroxides, such as Fe₂O₃, Fe₃O₄, and FeO(OH). They accumulate

in the metal pipes of the drinking water distribution system due to the corrosion of the pipes over time. This corrosion process results in the formation of deposits which then dissolve in the water and increase the iron content.

Both the oxidation of ferrous ions and the accumulation of corrosion-related deposits are critical factors that need to be managed to ensure water quality remains within acceptable limits. In most situations, loose deposits in water are not easily identifiable and can be difficult to eliminate with standard household water treatment methods, such as boiling. Even though these deposits might become visible when the water changes color, this visual change does not provide an accurate assessment of their potential toxicity. Smaller particles have a greater propensity to enter cells, which can lead to more pronounced cytotoxic and genotoxic effects compared to larger particles. This is because smaller particles are more likely to penetrate cellular barriers and cause damage at a molecular level, resulting in more severe health risks [Li et al., 2022].

The research has demonstrated that the concentration of insoluble iron in tap water is significantly greater than that of soluble iron. This finding carries substantial implications, particularly because insoluble iron, when consumed with drinking water, can dissolve in the highly acidic environment of the stomach (with a pH ranging from 1.5 to 2.0) and subsequently be absorbed into the bloodstream. The potential health risks associated with insoluble iron are noteworthy, as it may contribute to various health issues if not properly managed.

Based on the above, it is critical to consider the intricate behavior of iron during analysis when determining its concentration in tap water. A comprehensive assessment of iron content must account for both its soluble and insoluble forms to provide a complete and accurate picture. This detailed analysis is essential for not only evaluating the quality of the water source but also for assessing the overall condition and performance of drinking water distribution systems.

By accurately measuring both soluble and insoluble iron levels, we can gain valuable insights into the water supply's quality and the effectiveness of the distribution infrastructure. This information is crucial for ensuring the safety of drinking water and for making informed decisions regarding water treatment and distribution practices.

CONCLUSIONS

This work studied the peculiarities of determining the iron content in tap water. Throughout the analysis, it was observed that the process of iron transitioning into an insoluble species occurs relatively quickly. This may have a negative impact on the analysis result and lead to incorrect results. Therefore, the impact of temperature and acidity on the dynamics of iron concentration in tap water was investigated. It was established that acidification to pH = 1 and heating the sample to 40 °C for 90 minutes allows to determine the true initial concentration of total iron in tap water. It was found that the total iron content in the analyzed water is 6.4 times higher than the dissolved iron content.

Compliance with quality standards for iron in drinking water can only be confirmed by measuring the total iron content in all its species. For that, it is crucial to determine the iron content in drinking water immediately after sampling. If it is not feasible to conduct the analysis immediately after sampling, it is advisable to preserve the sample by acidifying it to a pH level of 1. This preservation method helps maintain the iron in its soluble form until analysis can be performed. In cases where acidification of the water sample is not possible, an alternative approach is to pre-treat the sample immediately before analysis. This involves heating the water sample to a temperature of 40 °C or higher in the presence of hydrochloric acid.

Because it is impossible to control the water intake sources, the quality of reagents, the purification process, and the condition of water supply systems during combat operations, the only feasible option is to monitor the quality of drinking water at the distribution points where the population receives it.

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