

Changes in pH during the ozonation process of surface water

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Abstract. This work considers the ozonation of surface water as an effective method for treating water to eliminate microorganisms and remove organic pollutants. However, the ozonation process changes the physicochemical properties of water, notably the pH level. Changes in pH can significantly affect the efficiency of water purification and potentially impact the condition of aquatic ecosystems when the treated water is discharged. This paper presents the results of experimental studies evaluating the dynamics of pH changes in various water bodies (distilled, ionized, tap, and river water) during the ozonation process. Special attention is given to the presence of heavy metal salts in water, which can catalyze the decomposition of ozone and intensify changes in pH. The study shows that ozonating surface water can lead to a decrease in pH, which may necessitate subsequent pH adjustments to maintain optimal levels for various uses of the treated water. The work is of practical significance for improving the efficiency of ozone use in industrial and municipal services and for water purification processes.

1. Introduction

In recent years, the intensification of global environmental pollution issues and the increase in drinking water quality standards have significantly heightened attention to water purification technologies. One of the most advanced and effective methods is ozonation, which is used for the elimination of microorganisms, oxidation of organic substances, and improvement of water taste and color characteristics. Despite its efficiency, the ozonation process accompanies changes in the chemical composition of water, including alterations in pH levels, which can significantly affect the water purification process and require additional monitoring and regulation [1,2,3].

Ozonation is a complex process that involves not only the direct oxidation of organic substances with ozone but also generates hydroxyl radicals, which can promote the further oxidation of pollutants. The efficiency of these reactions can significantly vary depending on the pH level of the environment, making pH control a critical aspect of the ozonation process [4,5].

Several factors can influence pH changes during the ozonation of natural and anthropogenically polluted waters, including the type and concentration of organic substances in the water, the initial pH level, dosage, and contact time with ozone. Increased ozone concentration may lead to a decrease in pH through the formation of acidic oxidation products, while the oxidation of certain organic compounds may result in the emergence of basic products, raising the pH level [6,7].

Understanding the mechanisms of pH changes during the ozonation of surface water and developing strategies to manage these changes are key to optimizing the water purification process. This requires a comprehensive approach that includes experimental studies of ozonation processes under various conditions and the development of mathematical models to predict pH changes and their impact on purification efficiency [8].

The aim of this scientific research is to analyze pH changes during the ozonation of surface waters, study the main factors affecting these changes, and develop recommendations for optimizing the ozonation process to enhance water purification efficiency and ensure compliance with established quality standards.

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2. Materials and Methods

The oxidation of harmful microorganisms in surface water and heavy metals in groundwater using an ozonator based on electrical discharges represents one of the new methods for purifying water composition [9,10]. Ozone is a strong oxidant that reacts with many organic and inorganic substances in water, breaking down to form oxygen. Currently, the reactions of ozone in water have been extensively studied in many foreign and domestic research works [11,12]. However, the emergence and intensive development of heterogeneous catalytic ozonation, focusing on the interaction of ozone with heterogeneous surfaces and dissolved substances in treated water, has led to a deeper investigation into this area [13].

The current state and prospects of catalytic ozonation for removing organic pollutants from water are rapidly advancing. The mechanism of degradation of organic pollutants with the participation of a catalyst, which is resistant to direct ozonation, remains unclear despite numerous studies, and there is a hypothesis that there might be a specific mechanism for each catalyst depending on the nature of the pollutant [14]. Understanding the mechanism of degradation of organic pollutants by ozone allows for the creation of systems with maximum purification efficiency and the possibility to control the process by adjusting parameters, leading to the best economic effect. In the process of developing an effective catalyst for ozonating surface water [15], we observed changes in the pH level of the treated solution or water without additional effects during ozonation.

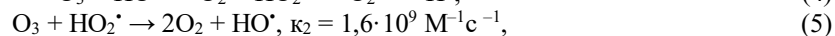
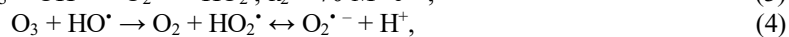
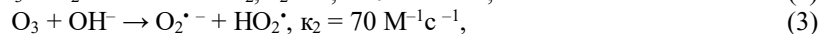
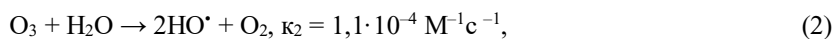
Ozone possesses a large energy reserve and relatively easily donates an oxygen atom. Its high reactivity makes ozone unstable in water. In aqueous solutions, it decomposes much faster than in the gas phase. The half-life of molecular ozone varies from a few seconds to several minutes [16].

2.1. Decomposition of Ozone in Water

The decomposition of ozone in both surface and groundwater is always described by a first-order kinetic equation.

$$-\left(\frac{d[O_3]}{dt}\right)_{pH} = k^* [O_3] \quad (1)$$

This process proceeds through the following reactions [16]:



These reactions represented by formulas (2,3,4,5,6) describe the kinetics of ozone decomposition in aqueous solutions. Each of the reactions listed demonstrates how ozone (O_3) decomposes under certain conditions, such as in an alkaline environment (with a high concentration of OH^- ions). The k values beside each reaction represent the rate constants of the reaction, describing the conductivity of the reaction at every second.

The provided chemical reactions describe the following:

(2) An ozone (O_3) molecule reacts with a water molecule (H_2O) to form hydroxyl radicals (OH^\bullet) and oxygen (O_2). The value of the k_2 constant indicates the speed of the reaction.

(3) Ozone reacts with a hydroxide ion (OH^-) to form a superoxide radical ($O_2^{\bullet-}$) and a hydroperoxyl radical (HO_2^\bullet). The speed of this reaction is given by the k_3 constant.

(4) In this reaction, ozone interacts with a hydroperoxyl radical (HO_2^\bullet) to produce oxygen (O_2) and a hydroxyl radical (OH^\bullet), and a cation H^+ is formed.

(5) Ozone reacts with a hydroperoxyl ion (HO_2^-) to produce two molecules of oxygen and a hydroxyl radical. The speed of this reaction is characterized by the k_5 constant.

(6) The interaction of two hydroperoxyl radicals (HO_2^\bullet) generates oxygen (O_2) and hydrogen peroxide (H_2O_2). Each of these reactions illustrates different pathways of ozone decomposition in water and is directly related to changes in the pH level. In an alkaline environment, the decomposition rate of ozone increases because OH^- ions engage in reactions with the ozone molecule, accelerating these reactions. Generally, the decomposition rate of ozone increases in an alkaline environment. Between $pH = 7 - 10$, the half-life of ozone is about 10 - 20 minutes. The presence of substances that absorb hydroxyl radicals significantly slows down the decomposition of ozone as a result of the following reactions [16]:



The hydroxyl radical (7) reacts with ozone to form oxygen and the hydroperoxyl radical. The high rate constant of $k_2 = 3,0 \cdot 10^9 \text{ M}^{-1}\text{c}^{-1}$ indicates its high reactivity.



The hydroxyl radical (8) reacts with the bicarbonate ion to form the hydroxyl ion and the bicarbonate radical. The average rate constant for this reaction is $k_2 = 1,5 \cdot 10^7 \text{ M}^{-1}\text{c}^{-1}$.



The hydroxyl radical (9) reacts with the carbonate ion to form the hydroxyl ion and the carbonate radical. The rate constant for this reaction is $k_2 = 4,2 \cdot 10^8 \text{ M}^{-1}\text{c}^{-1}$.



The hydroxyl radical (10) reacts with the dihydrogen phosphate ion to form the hydroxyl ion and the dihydrogen phosphate radical. This reaction has a very low-rate constant of $k_2 < 10^5 \text{ M}^{-1}\text{c}^{-1}$.



The hydroxyl radical (11) reacts with the hydrogen phosphate ion to form the hydroxyl ion and the hydrogen phosphate radical. The average rate constant for this reaction is $k_2 < 10^7 \text{ M}^{-1}\text{c}^{-1}$. These reactions demonstrate that the hydroxyl radical reacts with many substances at various rates. Some are very high, while others are low. This variation helps to understand the changes that occur in the decomposition process based on the chemical composition of the medium and the conditions of the reaction. However, these reactions represent the reactivity of each involved substance and the rate constant of the reaction in the ozone decomposition process. The OH^\bullet radical is a reactive oxidant, emerging as an intermediate product of ozone decomposition, and forms stable products when reacting with other substances.

Substances present in ozonated water can initiate, accelerate, or inhibit radical chain reactions involving ozone [17]. Initiators (OH^\bullet , $\text{H}_2\text{O}_2/\text{NO}_2^-$, Fe^{2+} , formates, humic substances) can lead to the formation of the superoxide ion ($\text{O}_2^{\bullet-}$) from an ozone molecule. Promoters ($\text{R}_2\text{-CH-OH}$, aryl-radicals, formates, humic substances, O_3) play a significant role in regenerating superoxide ions from hydroxyl radicals. Inhibitors (acidic anions, alkyl - R, carbonate - and bicarbonate ions, humic substances) can capture hydroxyl radicals without regenerating superoxide ions.

Accordingly, the stability of ozone depends on the pH level of the ozonated medium and the composition of the liquid being treated. The stability of ozone in water and aqueous solutions is crucial for its effective use. The authors [18] embarked on studying the decomposition and stabilization kinetics of ozone in water and aqueous solutions. Moreover, the work [18] considered water purity as a factor affecting the stability of ozone, evaluated through different purification methods: deionized, distilled, and tap water. The authors found that the rate constant of ozone decomposition follows the sequence tap water > distilled water > deionized water, decreasing by a factor of 9. The deviation of the kinetic curve of ozone concentration from a straight line in less purified waters suggests the involvement of impurities in the O_3 decomposition process. According to the authors, in this case, the decomposition products of ozone accumulate through a chain mechanism.

Studying the stability of ozone in the presence of hardness salts, which is used in water purification processes where these salts are present in concentrations up to 7 - 10 mM as per the established standard, is of significant interest. According to the authors' [18] data, the catalytic efficiency of ozone decomposition in water by carbonates of calcium, magnesium, and barium is significantly higher compared to the sulfates of these metals. This may be related to the interaction of ozone with carbonate and bicarbonate anions and their involvement in the radical reaction chains, as mentioned above. Therefore, despite the relative simplicity of installing an ozonation unit, it can be considered a sufficiently complex chemical process. Ozone can intensely decompose in the presence of many substances in water by engaging in radical reactions with them. However, the influence of this method's characteristics on changes in parameters such as the pH of the ozonated water has not been taken into account.

3. Results and Discussion

As objects for ozonation, model solutions of metal salts (CaCO_3 , MgCO_3 , FeSO_4 , MnSO_4), as well as deionized water, distilled water (GOST 2874-82), tap water ("Konaev Water Supply" state municipal enterprise for purification and supply), and river water (Ili River) were used. To study the stability of ozone in solutions with varying pH levels, solutions of calcium carbonate and hydrochloric acid with pH values of 11.5 and 1.1, respectively, were utilized. Ozone

was generated using the ETRO - 02 laboratory apparatus (Figure 1) [19,20]. The scientific research work was conducted at the K.I. Satpayev Kazakh National Technical Research University (Republic of Kazakhstan, Almaty, Satpayev 22). Future work considers the remote automatic control of the ozonator's operation. The accuracy of determining direction by digital spectral correlation of interferometric methods, with the reconstruction of the analytical signal for processing the incomplete spectrum of the signal, can be improved [21,22].



Fig. 1. The ETRO - 02 laboratory ozonator device based on electric corona discharge (*the ozonator device is protected by a copyright certificate "Patent No. 83218 Ministry of Justice of the Republic of Kazakhstan - 2014"*).

The volume of the liquid treated was 100 ml, and the gas was delivered through a glass filter with a porosity of 40 mesh, which was placed at the bottom of the reactor. The ozone-enriched gas used was an ozone-air mixture with an ozone concentration of 2.5 mg/l, obtained from dry air using the ETRO-02 ozonator. Experiments were conducted at a temperature of $21 \pm 1^\circ\text{C}$, with a gas flow rate of 500 ml/min. The ozone concentration in gas and liquid phases was determined using Medozon 254/5 and 254/5j ozonometers. A pH-meter/ionometer TAN-2 pH meter was used to measure the pH values of the solutions.

3.1 Changes in Hydrogen Ion Concentration (pH) During the Ozonation Process

Figure 2 below presents data on the pH changes in various types of water after ozonation. As observed, within 30 minutes, the pH for all samples decreased to values between 1.5 and 1.8. In this context, the pH change pattern for the river water sample is similar to the results obtained for distilled and deionized water samples, while a slight deviation in the initial part of the kinetic curve is noted for tap water. As mentioned earlier, according to the authors [18], the stability of the ozone decomposition rate decreases in the order of tap water > distilled water > deionized water. This indicates that the rate of ozone decomposition is highest in tap water. They suggest that this is due to the impurities in tap water, specifically the hardness salts.

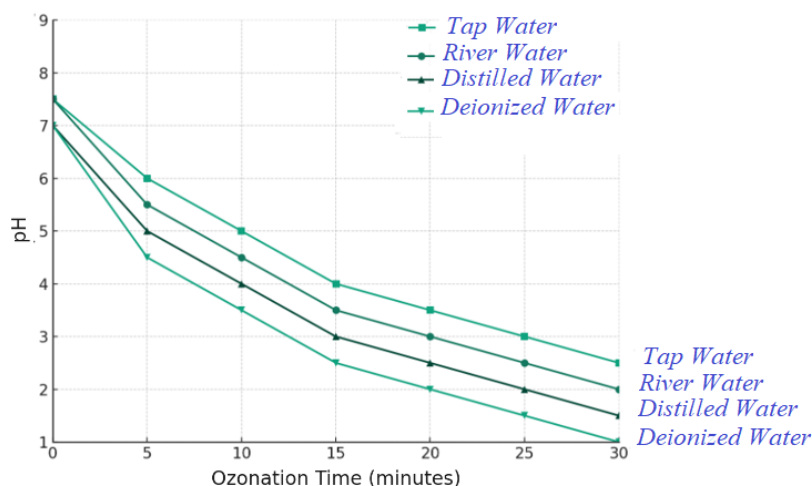


Fig. 2. Changes in the Hydrogen Ion Concentration (pH) of Various Types of Ozonated Waters

This figure 2 depicts a graph illustrating how the pH levels of different water samples change over time during the ozonation process. It shows the pH levels for four types of water samples: tap water, river water, distilled water, and deionized water from the start of the ozonation process and continuing over 30 minutes. As we can see from the graph, the pH levels of all water samples decrease, with their initial pH values around 7 - 8, dropping to between 2 - 3 after 30 minutes. The primary reason for this process is the reaction of ozone with organic and inorganic substances in the waters, resulting in a decrease in pH values. The pH decline curve for tap water appears to be somewhat gradual, which could indicate that certain impurities in the tap water (such as hardness, salts, and other compounds) may accelerate the decomposition of ozone. For river, distilled, and deionized waters, the decrease in pH levels is somewhat steeper, indicating a higher sensitivity of these water samples to ozone. However, as shown in figure 2, the initial pH value of tap water is equal to 8.0. The difference in the shapes of the pH dependency in the initial parts could be due to factors such as the ozonation time and the different initial hydrogen factor values of the water samples being ozonated. To clarify this issue, changes in ozone concentration in acidic and alkaline solutions after 20 minutes of ozonation followed by exposure to air were studied. The data obtained are presented in Figure 3.

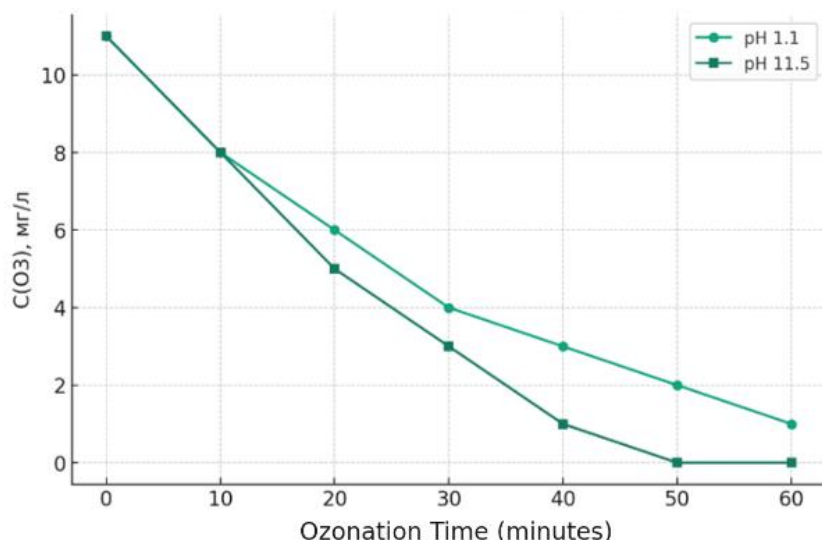


Fig. 3. Stability of Ozone in Different pH Solutions

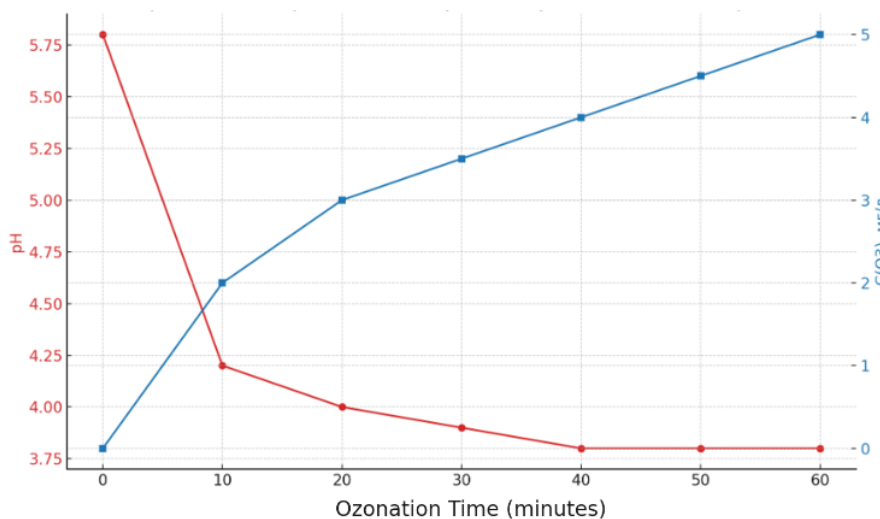


Fig. 4. Changes in pH and Dissolved Ozone Concentration During Ozonation of Distilled Water

Figure 3 presents a graph that shows the changes in ozone concentration ($C(O_3)$, mg/L) over time, measured in two solutions with different pH levels: pH 1.1 (acidic medium) and pH 11.5 (alkaline medium). As time (minutes) progresses, we observe a gradual decrease in the ozone concentration in both solutions. The initial ozone concentration was approximately 11 mg/L, which decreased to between 0 - 1 mg/L after 60 minutes. The rates of decline of ozone in solutions of different pH values vary: in the pH 1.1 solution, the decrease in ozone is relatively slow, while in the pH

11.5 solution, ozone decomposes more rapidly, indicating a higher stability of ozone in acidic conditions. This graph aids in understanding the dynamics of ozone decomposition when used in water purification, as the stability of ozone is dependent on the water's pH level and affects the efficiency of its disinfectant properties. As indicated in figure 3, after 20 minutes of ozonation, the concentration of dissolved ozone in the alkaline pH solution significantly exceeds that in the strong acid solution, which contradicts the data mentioned in the literature above. When the further supply of the ozone-air mixture is stopped, nearly 100% of the dissolved O_3 in an alkaline medium decomposes within one hour. Thus, based on the obtained data, it can be stated that under these specific experimental conditions, the solubility of ozone in an alkaline medium is several times higher than that of O_3 in an acidic pH. Additionally, the residual ozone in an alkaline medium remains stable and decomposes over a sufficiently long period. That is, the high initial pH value of tap water influences the intense dissolution and stabilization of ozone, which can explain the different nature of pH changes in samples of ozonated tap water.

To confirm this aforementioned theory, the pH changes and dissolved ozone concentration in samples of distilled and tap water during ozonation were studied. The data obtained are shown in figures 4 and 5 below.

The graph shown in Figure 4 illustrates the changes in pH and the concentration of dissolved ozone during the ozonation of distilled water samples. Over time, the pH decreases sharply but then stabilizes after approximately 10 minutes, remaining unchanged for the rest of the duration. In contrast, the concentration of dissolved ozone gradually increases as time passes. This indicates the stability of ozone in distilled water and its lower reactivity due to the absence of impurities.

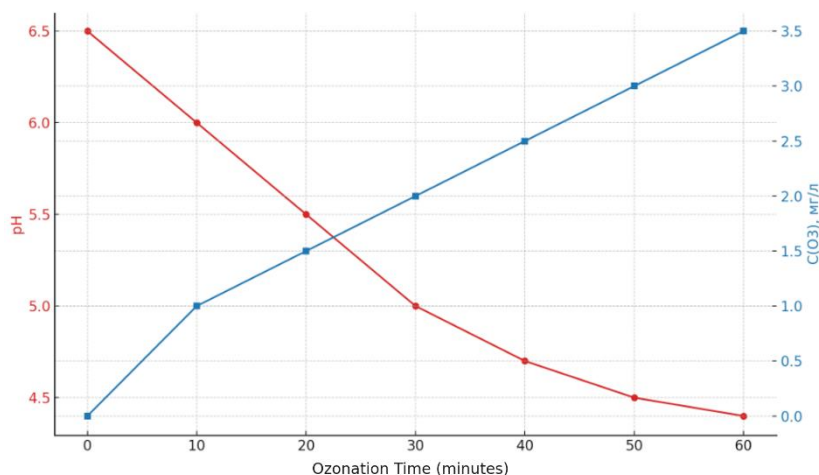


Fig. 5. Changes in pH and Dissolved Ozone Concentration During Ozonation of Tap Water

The graph in Figure 5 shows the changes in pH and the concentration of dissolved ozone during the ozonation of tap water samples. Over time, the pH gradually decreases and then stabilizes, while the concentration of dissolved ozone slowly increases. From this graph, it can be observed that ozone remains stable in tap water as well and that the concentration of dissolved ozone gradually increases over time.

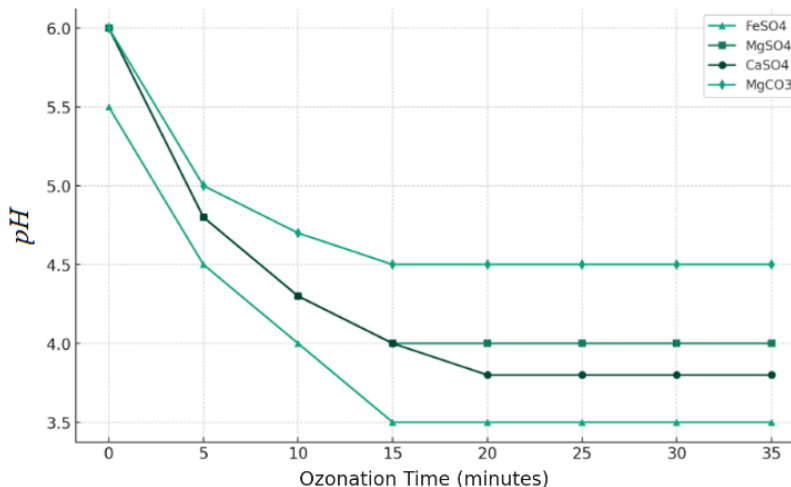


Fig. 6. The Effect of the Presence of Metal Salts (0.001 M) on pH Changes During Ozonation

The graph in Figure 6 illustrates the effect of metal salts (0.001 M) on pH levels during the ozonation process. In the first ten minutes after the start of ozonation, the pH level sharply decreases for all salts. Additionally, the pH level drops most significantly in solutions containing FeSO₄, while in those with MgCO₃, the pH decreases more slowly and remains comparatively higher. This graph demonstrates the difference in the reactivity of metal salts with ozone and the role of ozone as a stabilizer or reducer of phlegm.

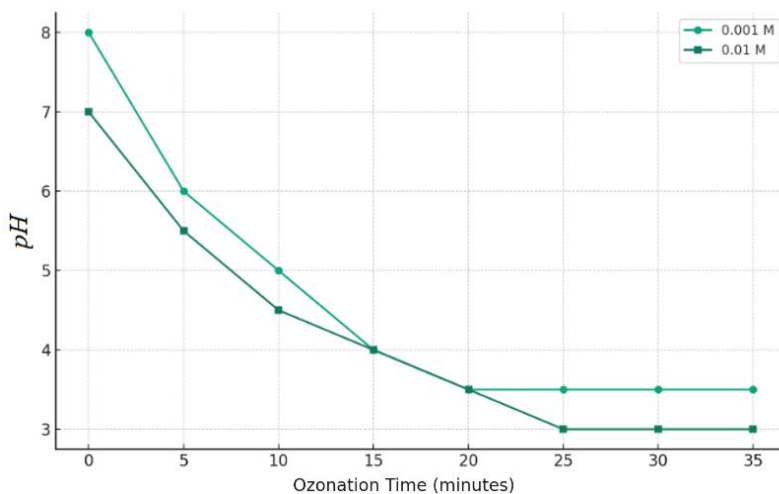


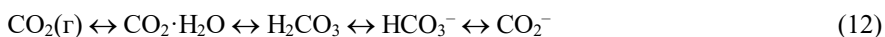
Fig. 7. The Effect of Magnesium Carbonate Concentration on pH Changes During Ozonation

Figure 7 presents a graph showing how two different concentrations of magnesium carbonate (0.001 M and 0.01 M) affect the pH level during the ozonation process. Initially, the pH value rapidly decreases and then stabilizes. The solution with a higher concentration of magnesium carbonate shows a quicker decrease in pH value, indicating a higher reactivity of magnesium carbonate with ozone and a strong effect on the pH level. It should also be noted that, despite the different natures of the pH change during the initial stage of ozonation, there is no significant difference in the change of ozone concentration in the samples being ozonated. Therefore, the differences in the pH decline curves are determined by other factors. As mentioned earlier, the presence of hardness salts affects the stability of ozone in water. Moreover, carbonate hardness salts can participate in radical reaction chains with ozone. Data on the effect of the presence of some metal salts are shown in Figure 6. As apparent, the presence of these salts at a 0.001 M concentration does not alter the characteristic of pH decline during ozonation. As demonstrated with magnesium carbonate, an increase in concentration does not affect the shape of the pH change curve (Figure 7).

Furthermore, the difference in the nature of the pH decline during the initial stage of ozonation between samples of distilled and tap water is not determined by the presence of hardness salts in the latter, nor does river water include the mentioned salts.

3.2 Discussion of the Scientific Research Work

The results of the scientific research work can be summarized with the conclusion that the pH level of surface water decreases during ozone treatment. This work considers two possible causes for the acidification of the ozonated water samples. Firstly, to increase the dissolution efficiency of ozone from the gas mixture during the ozonation process, the maximum possible contact area between the liquid and gas phases is ensured. In this case, not only the proportion of dissolved ozone increases but also the contribution of other gases in the mixture. The gas mixture provided during ozonation contains other gases besides O₃, as air is used as the initial gas, including CO₂. Carbon dioxide has a high solubility and dissolves with the formation of carbonic acid as indicated by the following formula (12) [23]:



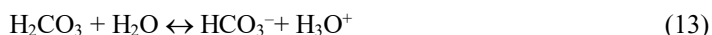
This text outlines the set of reactions that occur when carbon dioxide (CO₂) dissolves in water. The process consists of several stages, leading to the formation of carbonic acid (H₂CO₃), which in turn dissociates into bicarbonate (HCO₃⁻) and carbonate (CO₃²⁻) ions:

- CO₂(g) ↔ CO₂·H₂O - In this stage, gaseous carbon dioxide (CO₂) dissolves in water, forming hydrated carbon dioxide (CO₂·H₂O);
- CO₂·H₂O ↔ H₂CO₃ - The hydrated carbon dioxide further converts into carbonic acid (H₂CO₃);

- $\text{H}_2\text{CO}_3 \leftrightarrow \text{HCO}_3^- + \text{H}^+$ - Carbonic acid dissociates into the bicarbonate ion (HCO_3^-) and the hydronium ion (H^+);
- $\text{HCO}_3^- \leftrightarrow \text{CO}_3^{2-} + \text{H}^+$ - The bicarbonate ion further dissociates into the carbonate ion (CO_3^{2-}) and another hydronium ion (H^+).

These reactions demonstrate equilibrium conditions, meaning they do not proceed entirely in one direction but can also reverse. When CO_2 dissolves in water, its concentration affects the positioning of these equilibria. An increase in CO_2 concentration shifts the equilibrium towards the formation of carbonic acid and its ions, resulting in a decrease in the pH of the water due to the formation of hydronium ions (H^+).

Carbonic acid undergoes reverse hydrolysis (13), increasing the acidity of the environment through the following reaction:



The reaction in pattern (13) demonstrates the hydrolysis of carbonic acid (H_2CO_3) into the bicarbonate ion (HCO_3^-) and the hydronium ion (H_3O^+). This is a chemical equilibrium reaction and can be reversible. Such reactions play a significant role in explaining acid-base equilibria and affect changes in pH. When carbonic acid dissolves in water, it forms bicarbonate ions and hydronium ions, which increases the acidity of the water, i.e., decreases the pH value. This reaction depends on the concentration of carbon dioxide in water and affects the pH value. As the concentration of carbon dioxide in the environment increases, the amount of hydronium ions increases, and the pH value decreases. These acid-base reactions provide the buffering capacity of carbonic acid, protecting against abrupt changes in pH value. Therefore, an increase in dissolved carbon dioxide in the liquid can lead to a decrease in the solution's pH level. Additionally, as noted in [24-25], ozone actively reacts with nitrogen, forming dozens of toxic compounds. However, such interactions only occur at high temperatures (above 200°C). At these temperatures, the formation of higher oxides is possible, and in the presence of water or its vapor, the process continues to the production of nitrogen oxides and nitric acids [26-27]. The ozonation process of water is usually conducted at temperatures close to room temperature, which, at first glance, limits the possibility of reactions between N_2 and O_3 . There are studies [28-29] where the authors consider measurements of the mass ratio of moisture in various mixtures, liquids, as well as in bulk materials, in such cases, researchers measure free moisture, for chemically bonded water, control of the mass ratio of moisture is a special requirement. In this regard, the authors [6] also indicate that interactions between N_2 and O_3 are possible in an ozonator due to the effect of the discharge if air is used as the initial gas. No analysis of the nitrogen oxides content was conducted in the ozone-air mixture obtained during this work phase.

4. Conclusion

In concluding the scientific research, as demonstrated by the experimental results, treating water and model solutions of inorganic salts with an ozone-air mixture results in a decrease in pH values to 1 – 2. The nature of the pH change is consistent across ionized and river water samples, as well as for solutions of certain hardness salts in surface water. During the ozonation of tap water, the change in pH initially proceeds slowly but reaches the above-mentioned values after 30 minutes of the process. The significant increase in acidity of the treated liquids is suggested to be due to the dissolution of large amounts of carbon dioxide, forming carbonic acid, which lowers the pH through hydrolysis, or due to the interaction of ozone (when derived from air) with nitrogen and its oxides, forming nitric acid, which reduces the pH of the treated liquid. This issue requires further investigation, as the ozonation method using an ozone-air mixture, including for the purification of drinking water, is widely applied.

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