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Comparative Effect of the Type of a Pulsed Discharge on the Ionic Speciation of Plasma-Activated Water

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Abstract: The comparison of ion concentrations, pH index, and conductivity in distilled and ground water after exposure to low-temperature plasma formed by barrier and bubble discharges is performed. It has been found that in the case of groundwater, the best performance for the production of NO_3^- anions is provided by the discharge inside the gas bubbles. For distilled water, the barrier discharge in air, followed by saturation of water with plasma products, is the most suitable from this point of view. In both treatments, the maximum energy input into the stock solution is ensured. After 10 min treatment of ground water, the pH index increases and then it decreases. The obtained numerical indicators make it possible to understand in which tasks the indicated treatment modes should be used, their comparative advantages, and disadvantages. From the point of view of energy consumption for obtaining approximately equal (in order of magnitude) amounts of NO_3^- anions, both types of discharge treatment are suitable. The research results point to a fairly simple way to convert salts (calcium carbonates) from an insoluble form to soluble one. Namely, when interacting with NO_3^- anions, insoluble carbonates pass into soluble nitrates.

Keywords: plasma-activated water; barrier discharge; bubble discharge; physicochemical properties; process conditions

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1. Introduction

Interdisciplinary scientific research on the effect of non-equilibrium low-temperature plasma of electric discharges in the air on water and aqueous solutions is being carried out very intensively [1–6]. All research in this direction can be conditionally divided into three groups: (1) Study of the speciation of treated water and the kinetics of reactions in the discharge plasma and aqueous solutions; (2) development and testing of discharge reactors; (3) revealing the useful properties of laboratory-produced solutions, or determination of the degree of purification of such solutions from pollutants of various natures. These lines of research complement each other.

A special place is occupied by the direction associated with the development of equipment for the production of plasma-activated water (PAW). PAW is a result of plasma action on water or aqueous solutions (e.g., phosphate-buffered saline, etc.) in the presence of oxygen O_2 or a mixture of O_2 and nitrogen N_2, at atmospheric pressure [7]. In biomedicine, the benefits of PAW have been demonstrated in the tasks of biofilm removal, wound healing, and bacterial inactivation [8–10]. In agriculture, PAW is proposed to be used to increase the rate of seed germination and subsequently accelerate the growth of seedlings and plants, to inactivate plant-associated pathogens and rescue fungus-infected plants, and to preserve crops [11–15]. In particular, in studies [16,17] it has been shown that treating water with a discharge and then irrigating with this water significantly increases the growth rate of plants such as spinach (*Spinacia oleracea*), radish (*Raphanus sativus* var. *sativus*), strawberry (*Fragaria ananassa*), and Chinese cabbage (*Brassica campestris*). An increase in the concentration of nitrogen in the leaves was also observed.

Currently, it is believed that this PAW activity is due to the action of the following chemicals: reactive oxygen and nitrogen species (RONS), as well as relatively short-lived radicals (•OH, NO•), superoxide (O_2^-), peroxynitrate ($OONO_2^-$), and peroxynitrite ($ONOO^-$). The above effect of improving plant growth with PAW is attributed to the action of aqueous nitrates, nitrites, and ammonium ions, as well as hydrogen peroxide. In addition, the ability of PAW to produce fungicidal and antimicrobial effects is traditionally associated with short-lived reactive oxygen species [7,14,18–20]. PAW is considered a sustainable and promising solution for biotechnological applications due to the transient nature of its biochemical activity and the potential economic and environmental benefits of using ambient air rather than scarcely available or expensive chemicals as the raw material. This approach can potentially reduce the cost of treatment technology.

Methods and devices for water treatment with an electric discharge are of decisive importance for future technology. Authors from different scientific teams classify these methods in different ways, but it is better to give a classification that is most consistent with the processes of obtaining plasma-activated water. These are the following methods of discharge treatment of aqueous solutions: electrohydraulic, heterophase, bubble discharge, as well as some types of remote discharge reactors. Each method has its own advantages, but it also has disadvantages (see, for example [21]), an overview of which, should be briefly discussed.

Electrohydraulic discharge reactors [22] are characterized by the fact that the discharge occurs directly in an aqueous solution; for example, between two pointed or specially shaped electrodes. Or, in a narrow channel between them; for example, a capillary one. Another option may be a barrier configuration, i.e., when the high-voltage electrode is covered with a dielectric (barrier) layer, e.g., polyethylene or ceramic. Most often, pulsed arc and corona discharges are used. The advantage of this approach to the formation of a discharge, is that the plasma is formed directly in contact with water, which accelerates the PAW production. However, when overloaded, this can also lead to the decay of useful chemicals. In addition, the formation of active oxygen and nitrogen species in this case is limited by the low concentration of nitrogen and oxygen dissolved in water. Therefore, in order for hydrodynamic installations for the PAW production to be productive, water must be constantly saturated with the indicated gases or air.

From this point of view, a much more common way to produce PAW is the heterophase method. It consists in the fact that the discharge is formed in a gaseous medium (air and other plasma-forming gases) from an electrode with a small radius of curvature (pin or wire), and closes to the liquid phase (solution). The type of discharge formed in such installations is glow, corona or spark, and supply voltage can be constant, alternative or pulsed. The type of discharge in air and its polarity affect the products of plasma decay and the resulting content of chemicals in an aqueous solution. For example, the positive corona discharge in air saturates an aqueous solution mainly with ozone, while the glow discharge leads to the formation of aqueous nitrates and nitrites, which can be used to stimulate plant development [23].

Another promising method for the PAW production is based on the fact that the discharge occurs directly in the volume and on the surface of air bubbles. There are many options for the execution of a nozzle and its orientation, which ultimately determine the shape and number of bubbles, which in turn affect the distribution of an electric field in the gap. Like heterophase methods, bubble discharges make it easier to initiate a discharge compared to electrohydraulic installations. In addition, the use of bubbles allows better mixing of the resulting PAW and provides better uniformity in the treatment of the liquid. It is also noted that bubbles contribute to a significant increase in the concentration of reactive species, both in plasma and in liquid. In this case, pulse voltage is applied, and the type of particles and their concentration are determined by the type of plasma-forming gas [24]. Equally important is the matching of the discharge supply parameters with the bubble formation process [25].

Devices with discharge treatment of water droplets, or a thin water film to produce PAW, have been studied little. But from the data available in the literature, it can be assumed that their productivity measured in “liters per hour” will apparently be the lowest [2].

The idea of remote discharge reactors is based on the fact that gaseous plasma products are formed by a discharge in a separate zone, and then they are injected into an aqueous solution. Many ozonating plants operate on this principle. If the plasma-forming gas is air (or synthetic air, or humid air), then gaseous plasma products can include not only ozone, but also other chemically-active particles, such as •OH, NO• radicals, H_2O_2 peroxide, and recombination products [26].

Apparently, the simplest way for obtaining PAW is a device that consists of a tube placed in a solution, through which a plasma-forming gas is injected. In this case, a rod electrode is placed inside the tube, to which voltage pulses are applied. Under these conditions, a single-barrier discharge is implemented between the inner wall of the tube and the electrode, and the formed plasma products immediately enter the solution without any losses for transportation. This scheme, as well as options for its implementation, were described in detail in the review article [27].

Despite the abundance of experimental data, when developing a technology, it is necessary to take into account factors that may be out of sight of scientists. One or another method of treatment of aqueous solutions can be effective, in comparison with others, in terms of the yield of chemical products; but at the same time, can be inferior in terms of time and energy costs. Therefore, to create a discharge water treatment technology, comparative studies of various treatment modes are required, one of which will be presented in this article.

The purpose of this study is to compare the ionic composition of water treated with plasma produced with pulsed barrier and bubble discharges. This knowledge will be in demand in the development of specific technologies for the conscious use of certain modes of discharge water treatment. The choice of these types of electric discharges was due, firstly, to their prevalence in applied research [5,12,14]. Secondly, both types of discharge are, quite simply, constructively implemented, which can further facilitate the creation of technological installations. The third reason is their potentially high performance (production of active particles) in the case of using high-voltage pulses, which, however, had to be verified. Based on the results of ongoing research, recommendations will be made regarding the use of these discharges.

2. Experimental Setup and Techniques

During the experiments, distilled water (initial conductivity is 0.2 μS/cm; LLC Oils and Lubricants, Russia), as well as ground water (spring water; initial conductivity is 280 μS/cm; its composition will be discussed below) were treated. The studies were carried out on an experimental setup, a block diagram of which is shown in Figure 1a. Power supply 1 produced voltage pulses with an amplitude of 10 kV, a pulse duration of 1.3 μs, and a rise time of 1000 ns. Voltage pulses following with a pulse repetition rate of 54 kHz were applied to electrode 2 (high-voltage (HV) electrode). It was a metallic rod covered with a polytetrafluoroethylene (PTFE; fluoroplast) shell with a thickness of 0.5 mm. The HV electrode was placed in glass tube 3 connected to membrane pump 5. Ambient air injected through the tube 3 entered water 4, which filled quartz vessel 6. Foil electrode 7 (with an area of 127 cm^2) placed on the outer surface of the quartz vessel and connected with capacitor 8 (capacity is C_0 = 10 pF; charging voltage is U_0 = 20 kV) provided capacitive decoupling between electrode 2 and ground. An inner diameter and a height of the vessel were 2 and 30 cm, respectively. The inner and outer diameters of the glass tube were 5.5 and 7.5 mm, respectively. The glass tube was tapered towards the bottom. The inner diameter of this part was 0.8 mm. In a single experiment, a volume of water treated with plasma was 78 mL.

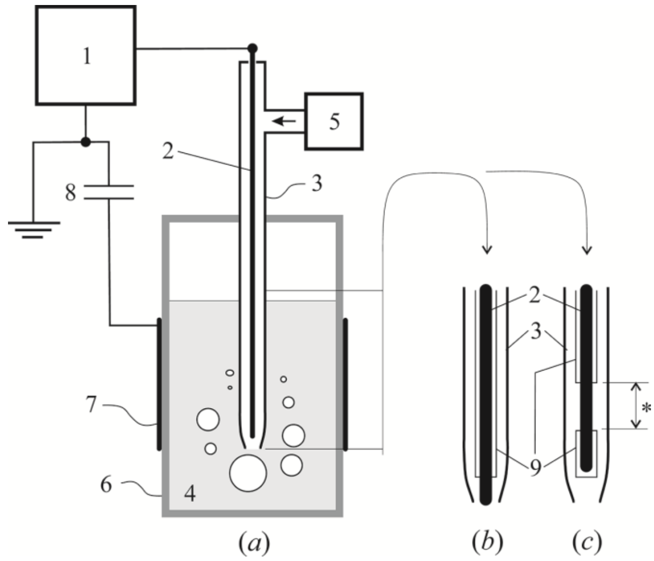

Figure 1. Block diagram of the experimental setup: general view (***a***) and zoomed sketches (indicated by arrows) of two design options of the high-voltage part (***b***,***c***). 1—power supply; 2—high-voltage electrode; 3—air feeding tube; 4—aqueous solution; 5—membrane air pump; 6—quartz vessel; 7—foil electrode; 8—capacitor; 9—dielectric shell; (*)—10-cm-length section freed from the dielectric shell.

Two designs of the electrode assembly were used. In the first version presented in Figure 1b, the metal high-voltage rod was almost completely covered with fluoroplast 9. The part not covered by the dielectric was located in the tapering part of the glass tube. The discharge was ignited at the tube outlet—between the end of the HV rod and the wall of an air bubble formed there. In this case, during the bubble formation, several breakdowns can occur. In other words, the mode of transfer of discharge energy into the bubbles is important here. This mode was chosen in accordance with the data obtained in [26]. With this type of excitation, chemically-active particles (reactive species) are produced both directly in the discharge in air and at the air–water interface. We will call this mode bubble discharge. A characteristic oscillogram of the voltage on the HV electrode for the bubble discharge is shown in Figure 2.

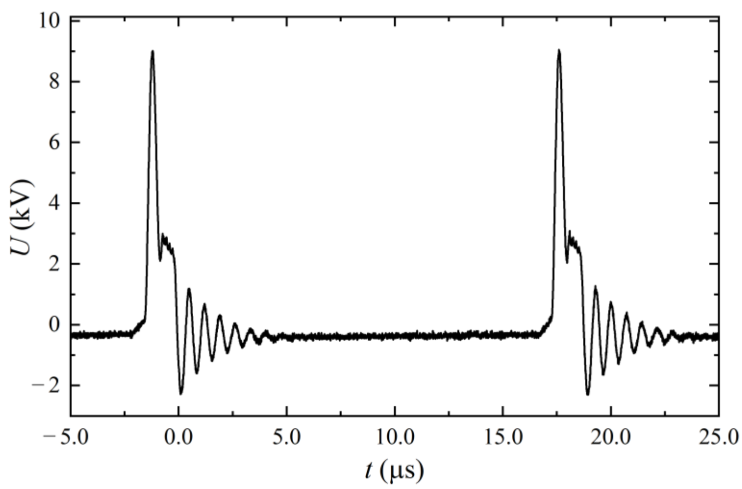

Figure 2. Waveform of the voltage at the HV electrode in the case of the bubble discharge.

In the second version of the electrode assembly design (Figure 1c), plasma enriched with reactive species was formed in a barrier discharge. To do this, the HV electrode had a 10-cm-length section denoted as (*) in Figure 1c freed from the dielectric. The end of the electrode 2 was also covered with PTFE tube. When applying HV pulses, the barrier discharge in the air was ignited in this region, and due to pumping, the chemically-active particles of the discharge plasma were transported into water bulk, saturating it and changing its characteristics. During the entire cycle of studies, the air pumping rate (200 mL/min), water volume, as well as the repetition rate of voltage pulses were fixed. This made it possible to compare the performance and composition of the obtained products for different assemblies.

Since the breakdown in bubbles can occur in different phases of voltage change (during the rise time, at the peak, or during the fall time of the pulse), which at this stage leads to a scatter and ambiguity in the electrical characteristics, the thermodynamic approach was used to estimate the input energy. This approach, in our opinion, makes it possible to quite correctly estimate the energy input for both types of excitation. Therefore, the energy input in this case was estimated by calculating the thermal energy released in the discharge. For this, the heating of the volume of the aqueous solution in the vessel was determined for a fixed time period. The temperature was measured using a temperature sensor built into a tester pH HI98108 (Hanna Instruments Ltd., Nușfalău, Romania). The temperature measurement accuracy was $\pm 0.5^{\circ}$. Measurements for a single sample were carried out three times, and then the obtained values were averaged.

The ionic composition of the aqueous solution after plasma treatment was determined using a "Kapel-105/105M" (Lumeks Co., Ltd., Saint Petersburg, Russia) capillary electrophoresis system for the detection of nitrate NO_3^-, calcium Ca^{++} и magnesium Mg^{++} ions. This device operates on the principle of spectrophotometric detection. The dispersing element is a diffraction monochromator with an operating spectral range from 190 to 380 nm. The accompanying methodological support makes it possible to analyze various anions and cations with a detection limit of 0.5 $\mu g/cm^3$. The hydrogen index (pH) was measured with an "Ionomer I-160MI" (LLC Izmeritelnaya Technika, Moscow, Russia) pH meter using calibration buffer solutions. The electrical conductivity of water was determined using an "ANION-4120" (Infraspark-Analit NPP, Yekaterinburg, Russia) laboratory conductometer. For each sample of water obtained after discharge treatment, measurements of the concentration of NO_3^-, Ca^{++}, and Mg^{++} ions, and electrical conductivity were carried out three times and then averaged. Thus, in what follows, all the data presented are averaged over three measurements.

3. Results and Discussion

Figure 3 demonstrates the change in the concentration of NO_3^- nitrate ions over time for various modes of formation of a chemically-active plasma. It is believed that the formation of NO_3^- anions in an aqueous solution is ensured by the conversion of nitrogen and oxygen molecules present in the air. Their activation and conversion directly depend on the performance of the discharge. It is seen that the discharge in bubbles provides a noticeably higher performance for these anions in ground water; while in distilled water, the best performance is provided by the barrier discharge.

It was found out how the data presented in Figure 3 correlate with the energy release in an aqueous solution. Typically, this characteristic for gas discharge devices is determined by calculating active power based on current and voltage waveforms. However, in our case, this approach was not applicable for two reasons. First, during the formation of a bubble at the end of the glass tube 3 (Figure 1b), several breakdowns of the gap between the rod tip and the inner surface of the bubble occur. Their number can vary from a few to hundreds, depending on the pulse repetition rate and the bubble formation time. Therefore, the energy input for each of these breakdowns will be different, which complicates statistical accounting for individual waveforms. Secondly, in both variants of the treatment, the conductivity and temperature of the solution change. This also entails changes in the energy release. Therefore, a lower estimate of the amount of heat imparted to the liquid during 10-min discharge treatment was made. At the same time, the estimate does not take into account the fact that this heat is also dissipated on the quartz walls of the vessel. In addition, the fact that not all of the energy deposited to water was further turned to heat (a fraction of the energy spent on forming chemical compounds), was not taken into account.

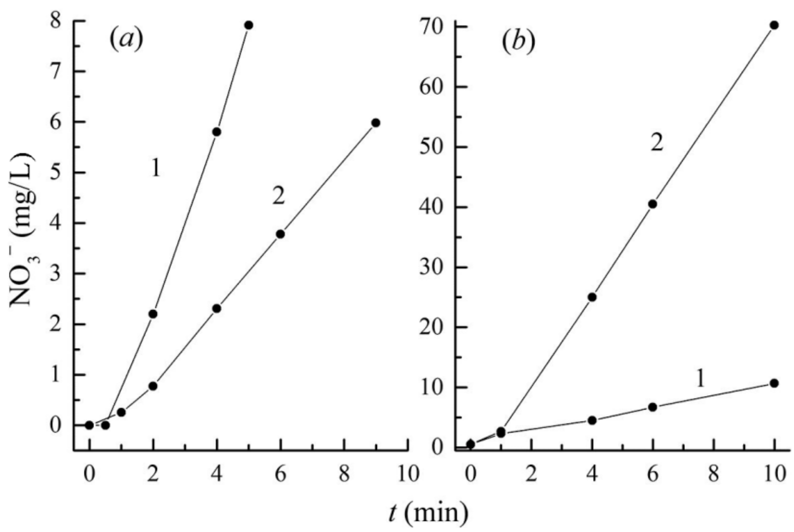

Figure 3. Time behavior of a specific amount of nitrate ions in distilled (***a***) and ground (***b***) water: barrier discharge (1); bubble discharge (2).

Table 1 shows the results of calculating the thermal power for the four cases shown in Figure 3. From the presented data, it can be seen that the higher the thermalized power, the higher the discharge performance in relation to the production of NO_3^- nitrate anions. At the same time, in distilled water, the barrier discharge shows the best performance, and in ground water—the bubble one. From the point of view of electrolytic physics [28], it can be assumed that ground water differs from the distilled one by a significant difference in the concentrations of ions in initial solution (mineralization of water samples). For ground water, a higher concentration of ions in the solution causes its greater conductivity and, consequently, a higher dissipation of the discharge energy in water.

Table 1. The value of the discharge power thermalized in an aqueous solution depending on the discharge type and the type of treated water.

Type of Treated Water	Thermalized Power, W	
	Bubble Discharge	Barrier Discharge
Distilled water	3.3 ± 0.5	4 ± 0.3
Ground water	11.1 ± 0.3	5.7 ± 0.4

When distilled water was treated with both types of discharges, the acidity decreases with time from about 7.5 to 3.5, while the conductivity increases from several to tens of µS/cm. The situation is different for ground water treatment. This is shown in Figure 4. It is seen that when ground water is exposed to both bubble and barrier discharges, the pH index first increases and then reaches a plateau. In addition, during the treatment with a barrier discharge, an anomalous "jump" in conductivity is observed during the first ~6 min. Then the conductivity returns to the starting level. The validity of this anomaly is confirmed by the fact that the conductivity measurements were carried out by two different methods. A similar behavior of the conductivity took place in both cases. When treated with the bubble discharge, no such behavior of the conductivity of the aqueous solution was observed.

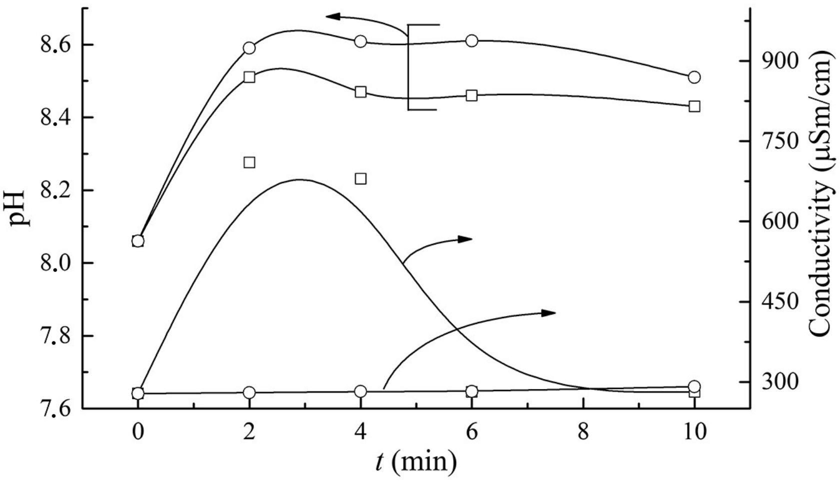

Figure 4. Time behavior of the pH-index and conductivity in ground water: barrier discharge (□); bubble discharge (○).

Table 2 shows the concentrations of magnesium and calcium ions before and after 10-min treatment of ground water with different types of discharge. It should be noted that the calcium concentration reached its maximum value after 2 min of treatment and then remained at the same level. It can be seen that plasma treatment leads to a significant increase in the concentration of Ca^{++} и Mg^{++} in ground water. It is possible to assume that initially these elements are present in the solution in the form of $CaCO_3$ and $MgCO_3$ carbonates. This may be due to the fact that $Mg(OH)^+$ and $Ca(OH)^+$ hydroxide ions are easily converted into carbonates in ground water in the presence of air. Insoluble salts in the form of carbonates in the solution do not precipitate due to the small size of (fine) particles in suspension, and determine the constant hardness of water. Further, under the discharge action, both hydroxide ions and nitric acid (i.e., H^+ and NO_3^- ions) can be formed, as a result of which the formation of ions is possible, including as a result of the reaction $Me(OH)^+ + H^+ \rightarrow Me^{++} + H_2O$. The ion formation process must be accompanied by the consumption of hydrogen ions H^+, since an increase in the hydrogen index from 8.1 to 8.5 for the barrier discharge and to 8.6 for the bubble discharge is observed (Figure 4). It should be noted that the question of the formation of calcium and magnesium ions under these conditions requires a separate, more detailed study. Nevertheless, it can be assumed that the pH index growth is due mainly to the increase in the concentration of calcium and magnesium ions during the first 2 min of treatment.

Table 2. The concentration (mg/L) of calcium and magnesium ions in ground water before and after plasma treatment.

Sample	Ion	Bubble Discharge	Barrier Discharge
Reference	Mg^{++}	0.0526	0.0526
	Ca^{++}	1.1395	1.1395
10 min treatment	Mg^{++}	9.694	9.792
	Ca^{++}	66.43	42.71

In addition to magnesium and calcium ions, potassium and sodium cations were also monitored in water samples. But, as measurements showed, treatment with discharges did not significantly affect their concentration in water; the content of potassium and sodium cations does not change during 10 min of exposure, and amounts to 1.065 and 9.395 mg/L, respectively. Potassium and sodium carbonates are soluble salts and determine the initial conductivity of an aqueous sample at 280 μS/cm. The appearance of additional NO_3^- and

NO_2^- anions, as a result of the action of the discharge, leads to the formation of potassium and sodium nitrates, which are also soluble salts.

The low concentration of Ca^{++} и Mg^{++} in the reference sample (Table 2), and the slightly alkaline environment of ground water, imply the presence of these ions in the solution in the form of insoluble salts. For example, calcium and magnesium carbonates. It is known [5] that when a discharge is ignited in atmospheric air in the presence of water, it leads to the formation of various active particles in the plasma, including NO_3^- and NO_2^- anions, which can interact with water-insoluble carbonates [5,16]. This, in turn, leads to the formation of water-soluble salts—calcium and magnesium nitrates or nitrites, which contributes to an increase in the concentration of their ions in water. However, the alkaline reaction of the solution, which persists throughout the entire time of water treatment with the barrier discharge, indicates that the amount of NO_3^- and NO_2^- anions formed is insufficient to shift the equilibrium to a neutral or acidic reaction.

A further increase in the treatment time led to the fact that the concentration of these anions in the solution increased and the pH began to decrease.

The results described above are of interest from the point of view of access to technological installations for the PAW production, since data on the discharge treatment of distilled and deionized water are usually presented in the scientific literature. Of course, on an industrial scale, the use of such water is impractical. Our data show that, in the case of ground water, we can also obtain high concentrations of NO_3^- anions.

Importantly, ground water treatment also opens the way to the study of relatively easy methods for converting salts (calcium carbonates) from an insoluble form to a soluble one. Namely, when interacting with NO_3^- anions, insoluble carbonates pass into soluble nitrates.

4. Conclusions

As a result of the research, the set goal was achieved—the ionic composition of two types (distilled and ground) of water treated with low-temperature plasma formed by two types of pulsed discharge (barrier and in bubbles) in atmospheric pressure air was revealed. It has shown that the bubble discharge in ground water gives the maximum performance for the NO_3^- anions. In this case, the energy thermalized in the solution is maximum. At the same time, the complex compounds that affect the hardness of water, the most Ca^{++} ions, are released into the solution. These features of the process of water treatment using pulsed discharges should be taken into account when designing installations for large-scale PAW production.

These results are important for an industry such as hydroponic plant growing technologies (see, e.g., [16,17]), where an aqueous solution enriched with NO_3^- anions is required.

The data obtained are of interest due to the fact that data on the discharge treatment of distilled and deionized water are usually presented in the scientific literature. Of course, on an industrial scale, the use of such water is impractical. Therefore, research on discharge treatment of groundwater is needed. Our research starts this process. However, to prove the beneficial properties of plasma-activated water (PAW) produced in a bubble discharge in ground water, laboratory and field studies on the effects of such water on economically-valuable plants are needed.

Author Contributions: V.P., E.S. and V.S. performed the experiments. D.S. helped initiate the research. A.R. and S.K. conducted a physicochemical analysis. E.S., D.S. and S.K. wrote the manuscript. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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