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Article

Nonthermal nitrogen fixation with air and water by using a low-pressure plasma

Derin Cengizkan[1], Emre Akın[2], Necip Berker Üner[3]
[1]METU DF High School, Ankara, Turkey
[2]Chemistry Department, METU DF High School, Ankara, Turkey
[3]Chemical Engineering Department, Middle East Technical University, Ankara, Turkey

SUMMARY

In the 20th century, the increasing demand for food was addressed successfully by establishing the production of synthetic fertilizers through the Haber-Bosch (HB) process. The HB process utilizes atmospheric nitrogen and hydrogen derived from steam-methane reforming to produce ammonia, which can be transformed into common fertilizers like ammonium nitrate and urea. Despite being energy efficient, the high carbon dioxide footprint of the HB process has led researchers to seek alternative routes for generating small nitrogenous compounds. One of the candidates is using plasma technology, which is generally limited by its energy requirements and small plasma size at atmospheric pressure. We hypothesized that when a nonthermal air plasma is generated over water under vacuum, it could improve nitrogen fixation rates and energy efficiency due to the formation of a larger plasma and the production of more water vapor. At a pressure of approximately 160 Torr, we observed that a high concentration of nitrate, a considerably small amount of nitrite, and ammonium were formed in water in a plasma-liquid cell. We investigated the effect of plasma activation time and the level of total dissolved solids on the amount of fixed nitrogen species and energy efficiency. When compared with literature data, our results suggest that operation under vacuum may increase the feasibility of plasma nitrogen fixation, constituting a step towards the production of synthetic fertilizers without carbon dioxide emissions.

INTRODUCTION

Due to the rapidly increasing population and the limited reserves of natural fertilizers, synthetic fertilizers were introduced into widespread agricultural use in the early 20th century (1,2). According to research conducted in 2011, if the average yield of agricultural land had stayed the same after 1900, four times more land would be needed to feed the world (3). Such an aerial demand corresponds to 15% of the non-frozen continents, hence approximately 60% of the non-frozen land area would have been used to feed eight billion people (3). Fortunately, this is not the case, and the difference was enabled by the Haber-Bosch (HB) process, which is the current industrial process for producing synthetic fertilizers (2).

The HB process increased the productivity of agricultural land by lowering the dependency on natural fertilizers. A fertilizer, either natural or synthetic, must foremost contain nitrogen compounds that can be readily processed by plants (3). Nitrogen is an essential element in life, but despite its abundance in the environment, the forms of nitrogen that are metabolizable by plants are very limited. Atmospheric nitrogen cannot be directly utilized by plants due to the strong triple bond, but the HB process makes atmospheric nitrogen accessible by converting it into ammonia (NH_3), from which nitrogenous fertilizers, such as nitrate (NO_3^-) and urea ($CO(NH_2)_2$) are derived (3). Thus, HB essentially feeds the world population by producing 230 million tons of NH_3 per year as of 2018 to improve the yield of agricultural land. However, it uses 3-5% of world's yearly natural gas at the same time (4,5). This amount is approximately equal to the natural gas consumption of the entire African continent as of 2023, and the energy demand of HB processes makes up 1-2% of the total global energy consumption, which is close to the energy consumption of France in year 2021 (6,7). The dependency on natural gas in HB comes from the need for hydrogen (H_2), the second constituent of ammonia, and H_2 production usually defines the carbon dioxide (CO_2) footprint of the process. This is because H_2 is obtained through an endothermic process called steam-methane reforming (SMR), which yields a mixture of carbon monoxide (CO) and H_2:

$$CH_4 + H_2O \rightarrow CO_2 + 3H_2 \quad \text{(Rxn 1)}$$

Simultaneously with the reaction above, the water-gas shift reaction (WGS) occurs, and it converts CO into CO_2 while producing more hydrogen.

$$CO + H_2O \rightarrow CO_2 + H_2 \quad \text{(Rxn 2)}$$

Stoichiometrically, SMR and WGS results in the release of 5.5 tonnes of CO_2 per one tonne of H_2 produced, but the actual ratio in a real process is more, usually between 7.5 and 12 (8). Since CO_2 is the primary reason behind global warming, with the sheer scale and the prevalence of the HB in mind, production of synthetic fertilizers through HB is a major contributor to climate change. Therefore, the HB process has great environmental and economic impacts on our world due to its large energy consumption, contribution to global warming and heavy reliance on natural resources.

To mitigate the CO_2 emissions of HB, there has been immense research on developing alternative methods of nitrogen fixation that do not release CO_2, and the majority of the efforts focus on processes that are desired to run solely by using electricity. An interesting class of electrified processes rely on using a plasma (9-11). Plasma is the fourth state of matter, formed due to ionization of a gas, and therefore it is electrically conductive. Plasmas are produced when an energy source, such as high-voltage excitation, is applied such that gas molecules are ionized, yielding positive ions and free electrons, along with many excited species. Unless the gas used is not an inert gas (e.g., argon), plasma-based methods offer rapid processing and novel reaction pathways due to presence of ionized and excited molecules. These molecules tend to be highly reactive and this tendency to react may be directed to obtain desired products in a very fast and continuous manner (12,13). For example, in a plasma produced from air, nitric oxide (NO) and nitrogen compounds with higher oxidation states, such as NO_2, N_2O and N_2O_5, are formed from gaseous nitrogen and oxygen (14). In the presence of water, NO_3^-, nitrite (NO_2^-) and ammonium (NH_4^+) can also be produced (15). Both NO_3^- and NH_4^+, commonly combined as NH_4NO_3, are widely used as fertilizers worldwide.

Plasmas are advantageous since they can fix nitrogen with water without the need for hydrogen gas. Water subjected to a nitrogen or air plasma is commonly called plasma-activated water, and it is found to be beneficial to the growth of plants (16). Research on nitrogen fixation with plasmas has been mostly focused on atmospheric pressure plasma for the last 20 years, and companies that provide plasma-based nitrogen fixation technology mostly utilize atmospheric pressure plasmas (17,18). Recent research has shown that, the efficiency of nitrogen fixation processes can be increased with a higher water vapor concentration in the synthesis environment, and basic plasma physics tells us that more energy is required in creating and sustaining a plasma at high pressure (19,20). Based on these observations, we hypothesized that nitrogen fixation with a plasma in a low-pressure environment would yield higher energy efficiency compared to atmospheric pressure applications. More water vapor would be generated under vacuum since a reduction in gas pressure within a closed chamber increases the evaporation and diffusion rates of liquid molecules, and therefore rapidly establishes the vapor-liquid equilibrium at the interface. In addition, the size of the plasma under vacuum would be larger since electrons travel across greater distances without hitting other molecules. In other words, they have a larger mean free path. These physical trends indicate that increasing the efficiency of air-water reactions should be possible at sub-atmospheric conditions.

In this study, we developed a simple plasma-liquid reaction cell for nitrogen fixation under vacuum. Plasma was generated with air over water, and the reaction cell was operated in semi-batch mode, meaning that there was a small but continuous supply of air seeping into the reaction cell under vacuum, and we only used a vacuum pump intermittently to keep the pressure within an operation range. This makes our process neither a continuous process, which involves a steady input and output of materials, nor a batch process, which has no input or output except at the beginning and the end of the process. NO_3^-, NO_2^- and NH_4^+ produced through the plasma-water interaction were measured through colorimetric tests and energy usage was monitored by using a power meter. Dividing the product amounts by the total energy consumption of the process resulted in the energy efficiency. NO_3^- was produced the most and the efficiency for NO_3^- production reached a maximum after 120 minutes of plasma operation. When compared with literature, this efficiency was on par with the previously reported values and the energy used for pumping was found to be constituting a lesser portion of the total energy use (less than five percent of the total), indicating that operation at low pressure might be beneficial for increasing the yield of fixed nitrogen in air-water plasmas (9,10). With these results, we show that a new operation regime for plasma-based nitrogen fixation may exist under vacuum, which is feasible and sustainable, and furthermore allows NO_3^- production with high rates.

RESULTS

Most experiments were performed with tap water, while a few used deionized (DI) and salt water for comparison. Experiments focused on the effects of plasma activation time and the initial amount of total dissolved solids (TDS level) on the amount of nitrogenous compounds formed.

The effect of plasma activation time on product yield

We tested the duration of plasma activation of tap water to investigate the production rates and energy efficiency as a function of time. We did experiments up to four hours and measured the final concentrations of NO_3^-, NO_2^- and NH_4^+. As expected, NO_3^-, NO_2^- and NH_4^+ were generated constantly, leading to a steady increase in the total amount produced as activation time was increased (**Figures 1 and 2**). The overall trends in the production of all these ions share similarities. However, nitrate and nitrite were produced initially with a slightly lower rate, leading to an induction period within the first 30 minutes, as indicated by an upwards curvature in the concentration vs time plots (**Figures 1 and 2**). We observed a significant increase in the NO_3^- and NO_2^- synthesis rate for activation times less than one hour. Later, apart from a slight saturation trend in NO_2^-, ion concentrations increased approximately linearly, indicating constant rates of production and no significant effect of the existing nitrogenous ions on production. We made all experiments with a duration below two hours in triplicates and some of our measurements, specifically those of NO_3^-, resulted in very small error margins. For NO_2^- and NH_4^+, the highest percent deviation between the replicates was 27% and 43% with respect to the average, respectively (**Figure 2**).

We measured the energy efficiency, denoted by $\hat{E}$, of producing each nitrogenous ion individually in units of mol/kWh as a function of time (**Figures 3 and 4**). We calculated $\hat{E}$ values by using the total energy used until the end of an experiment, therefore, $\hat{E}$ represents a time-averaged quantity over the duration of an experiment (**Figures 3 and 4**). We measured the energy consumption rates of the vacuum pump and the power supply to be approximately 33 W and 5.8 W, respectively. We multiplied these values with the experimental durations to calculate the energy usage for the individual processes. The energy efficiency values for NO_2^- and NO_3^- increased with time and eventually reached a maximum (**Figures 3 and 4**). However, the $\hat{E}$ for NH_4^+ decreased with time.

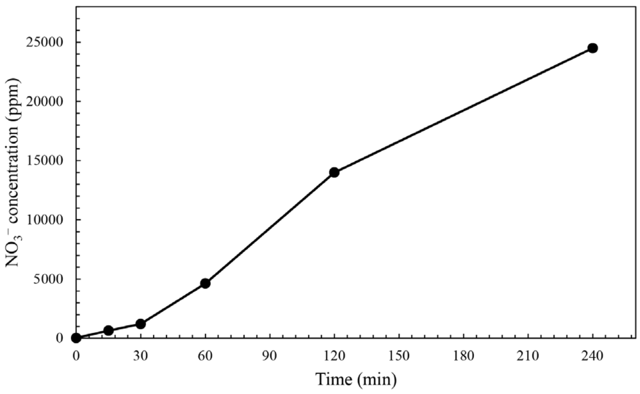

Figure 1. NO_3^- concentration in plasma-treated water measured as a function of time. Plasma nitrogen fixation experiments were done up to four hours of runtime and the final concentration of NO_3^- was measured at the end of each experiment using colorimetric tests. A relatively steady production of NO_3^- was achieved. All experiments with a duration below two hours were made in triplicates and averages were plotted. Measurements resulted in an error margin that was not measurable, primarily due to limited sensitivity and discrete measurement scales involved in the colorimetric tests, hence the absence of error bars.

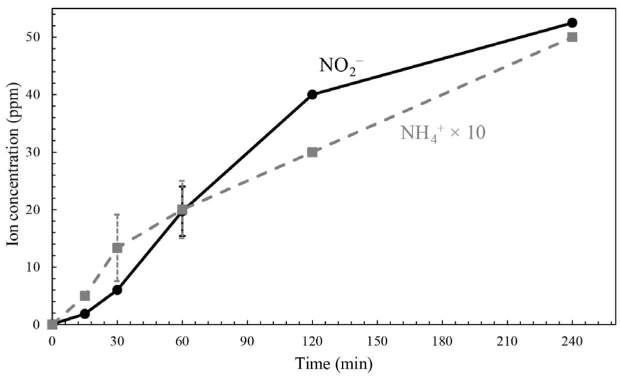

Figure 2. NO_2^- and NH_4^+ concentration in plasma-treated water measured as a function of time. Plasma nitrogen fixation experiments were done up to four hours of runtime and the final concentrations of NO_2^- and NH_4^+ were measured at the end of each experiment using colorimetric tests. The concentration of NH_4^+ was multiplied by 10 in the plot above, indicating a production rate that is an order of magnitude slower than that of NO_2^-. All experiments with a duration below two hours were made in triplicates and averages and associated error bars, representing the ± 1 standard deviation, were plotted. Some measurements resulted in an error margin that was not measurable, primarily due to limited sensitivity and discrete measurement scales involved in the colorimetric tests. For those measurements, error bars were not added.

For NO_3^- and NO_2^-, we observed that an induction period exists for formation (**Figures 1 and 2**). This period appears to be approximately 30 minutes long and after 60 minutes, the production rate increases, which roughly corresponds to the time at which pH is observed to decrease (**Figure 5**). NH_4^+ has similar production trends, but we observed a linear increase for experiment durations beyond 60 minutes. For NO_3^- and NO_2^-, production rate slows down beyond two hours, leading to a maximum in $\hat{E}$ for both ions (**Figures 3 and 4**). We obtained this maximum earlier for NH_4^+, and long experiments resulted in energy efficiencies that decreased with increasing time.

Another significant parameter apart from the energy efficiency of the process is the pH of the produced solution. Direct agricultural application of plasma-activated water may require neutralization if the final solution is highly acidic (21). In our experiments, we saw that the final pH stayed around seven, unless activation was longer than an hour (**Figure 5**). For treatments longer than an hour, we saw that pH decreased steadily. When we used DI water in place of tap water, pH decreased to 3.5 after two hours of activation, whereas pH was equal to 5.5 at the end of an experiment conducted with tap water. Because of the dependency of pH on TDS, we conducted several experiments using solutions with controlled TDS to see the effect of TDS on nitrogen fixation.

The effect of TDS on product yield

TDS of the solution affects the conductivity, vapor pressure and buffering capacity of water. We initially expected conductivity to be an important factor in terms of the amount of fixed nitrogen. Higher solution conductivity should enable the power supply to provide a higher current at fixed voltage, since the equivalent resistance in the plasma-liquid cell would drop.

In experiments testing the effect of TDS on product yield, we kept all operational parameters the same, and the activation time was constant at 30 minutes. We compared three types of water: deionized, tap and salt water. We prepared salt water using NaCl at concentrations of 2,000 and 6,000 ppm with tap water.

Experiments showed that TDS levels do not change the production of nitrogenous ions appreciably unless TDS is very low (**Figure 6**). Tap and saltwater solutions yielded similar concentrations for all ions, but the production of nitrate was severely limited in DI water. Due to the variation of TDS that comes from tap water (see **Materials and Methods**), we detected no appreciable change in TDS after the experiments. The temperature of plasma-treated water did not show any significant change neither (variation of ± 1°C only). The TDS level of the DI water after plasma treatment was not measurable due to the large cell constant of the TDS meter.

DISCUSSION

Results show that dinitrogen can successfully be fixed into nitrogenous ions in water with an air plasma under vacuum (**Figures 1 and 2**). Among the ions of specific interest in fertilizer technology, NO_3^-, NO_2^- and NH_4^+, the concentration of NO_3^- in plasma-activated solutions was orders of magnitude larger than those of NO_2^- and NH_4^+. The large difference between NO_3^- and NH_4^+ production in air-plasma systems is well known in literature (11,22). Removing oxygen from the reactor headspace and conducting plasma activation with pure nitrogen has been shown to promote the production of NH_4^+ and substantially decrease the production of oxidated forms of nitrogen (23). However, since energy efficiencies are generally lower with nitrogen-only synthesis and since using pure nitrogen requires expensive air-separation methods, it appears challenging to produce both NO_3^- and NH_4^+, i.e. ammonium nitrate, at high energy efficiency with an air plasma at low pressure.

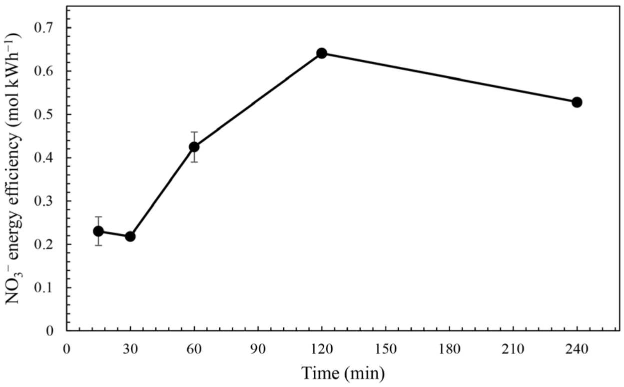

Figure 3. Energy efficiency of NO_3^- production as a function of time. Reported values were obtained by measuring the total energy use and dividing the total amount of NO_3^- formed with this energy. NO_3^- energy efficiency increased to a maximum after two hours. All experiments with a duration below two hours were made in triplicates and averages and associated error bars, representing the ± 1 standard deviation, were plotted. Some measurements resulted in an error margin that was not measurable, primarily due to limited sensitivity and discrete measurement scales involved in the colorimetric tests. For those measurements, an error bar was not added.

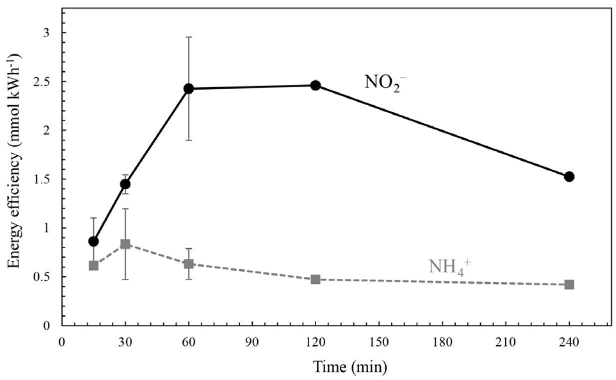

Figure 4. Energy efficiency of NO_2^- and NH_4^+ production as a function of time. Reported values were obtained by measuring the total energy use and dividing the total amounts of NO_2^- and NH_4^+ formed with this energy. All experiments with a duration below two hours were made in triplicates and averages and associated error bars, representing the ± 1 standard deviation, were plotted. These efficiencies were three orders of magnitude lower (note that the unit is mmol/kWh) when compared to those of NO_3^- production (**Figure 3**).

The energy efficiency of nitrate production achieved in this work was comparable to the numbers provided in articles published in literature. While Sun and colleagues measured 0.26 mol/kWh for NOx production, Reniers and his team were limited to values below 0.1 mol/kWh with a dielectric barrier discharge (9,10). We achieved a maximum $\hat{E}$ of 0.64 mol/kWh (**Figures 3 and 4**). Although we expect the measured order of magnitude of the concentrations and efficiency to be reasonably accurate, more advanced methods, such as ion chromatography analysis, are needed for validating the results and for more precise measurements that exclude any potential interference effects which may arise from the use of colorimetric analysis. In addition, the use of electrolytic probes, such as for pH and ion-selective measurements may also help in elucidating the ionic chemistry.

The delayed decrease in pH in tap water was unexpected (**Figure 5**). We initially hypothesized the pH levels would decrease due to high production rates of NO_3^- in comparison to NH_4^+, due to the formation of nitric (HNO_3) and nitrous acid (HNO_2), in combination with water splitting. However, the expected drop in pH due to the formation of acidic compounds was not observed before 60 minutes of operation time. A possible reason for this is the common presence of carbonated compounds in tap water, such as $CaCO_3$, $MgCO_3$, Na_2CO_3 and $NaHCO_3$, and their associated buffering capacity (24). When the cations of the less soluble carbonated compounds bond with NO_3^-, solubility equilibrium suggests that the carbonate is likely to first form carbonic acid, and then carbon dioxide, which would eventually escape from water under vacuum. With this hypothesis, all the carbonate cations would bind to nitrates and then the pH would drop after a sufficiently long operation time.

As seen with the experiments conducted at different TDS levels, TDS does not have a significant effect on the production of nitrogenous ions if it is sufficiently high (**Figure 6**). However, we can say that the presence of TDS can be beneficial in plasma nitrogen fixation since it will delay the decrease in the pH of the solution. A company that uses plasma nitrogen fixation to produce nitrogen-based fertilizers resorts to using potassium hydroxide to neutralize their acidic solution that forms after the plasma activation process (17). Our results suggest that if the tap or irrigation water has sufficient TDS with suitable ions, such a process can be prevented if the plasma activation period is sufficiently short. Therefore, it seems likely that there is a trade-off between obtaining high pH and high NO_3^- concentrations for plasma nitrogen fixation.

The small amount of increase in the NO_3^- production using a solution with high NaCl concentration can be partially

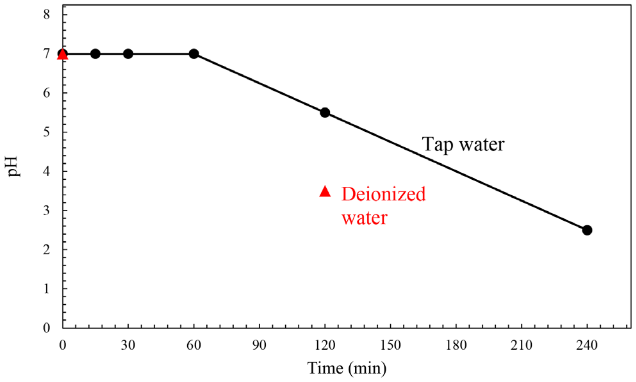

Figure 5. pH of plasma-treated water measured as a function of time. Red data point shows the measurement taken in DI water. With tap water, pH reduced steadily for experiment durations longer than an hour. All experiments with a duration below two hours were made in triplicates and averages were plotted. Considering that the measurable variance in the pH by using strips is small, the pH stayed steadily around seven initially for tap water.

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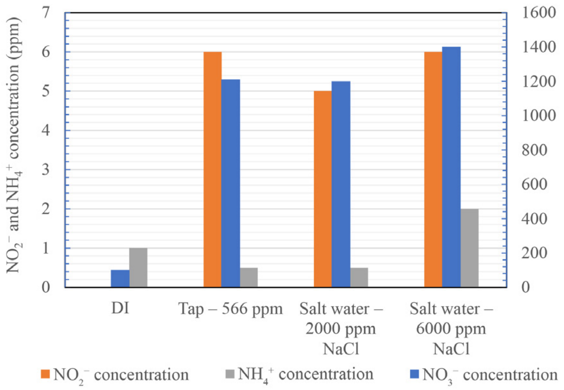

Figure 6. Effect of dissolved substances on the production of nitrogenous ions during plasma nitrogen fixation. All experiments were conducted for 30 minutes. Apart from a slight increase in NH4+ and NO3− concentrations at high TDS levels, TDS appears to be a minor factor as long as the conductivity of the solution is sufficiently high. On the other hand, the use of DI water results in very low concentrations of fixed nitrogen species.

explained by the decrease in the electrical resistance of the water, which is expected to increase the current provided by the compact power source used at fixed total power. Similarly, lower NO_3^- and NO_2^- concentrations measured in the experiment conducted with DI water may be associated with water acting as an additional capacitor in the absence of ions in the water, which would change the power deposition in the plasma. However, more sophisticated tools, such as a high-frequency/high-voltage probe and a large-bandwidth current transducer is necessary to measure the current and the power deposited in the plasma, which may change as a function of TDS level and during plasma treatment.

In summary, we obtained high energy efficiencies for NO_3^- production when the plasma nitrogen fixation process was conducted at low pressure. The semi-batch operation mode, where the vacuum pump is operated in a time-interrupted manner may be one of the reasons for increased energy efficiency. The experiments conducted with varying TDS levels show that TDS does not affect the formation rates of nitrogenous ions unless the conductivity of water is very low. Furthermore, the ionic composition of the water appears to be an important factor that determines the final pH and the content of nitrogenous species in plasma-activated water. Our results suggest that a low pressure non-thermal plasma can be a feasible and energy efficient alternative to other electrified nitrogen fixation processes. The product obtained when tap water is used, a concentrated NO_3^- solution with near neutral pH, may be directly used as a fertilizer, which would further enhance the potential application of low-pressure plasma nitrogen fixation especially when conducted at a small and distributed scale, where CO_2 emissions can significantly be reduced.

MATERIALS AND METHODS

A plasma-liquid reaction cell that can hold vacuum was constructed from simple and available materials. The reaction cell was a modified household glass jar (8.5 cm radius, 10 cm height) with a thick metal lid. In all experiments, the cell contained 35 ml of water, either taken directly from the tap, de-ionized (18.2 MΩ/cm resistivity) or with NaCl added (>%99.5 purity, Sigma Aldrich). To maintain consistency, tap water was used from the same tap throughout the study. The jar was sealed using a 1 mm-thick silicone gasket and vacuum grease (Dow Corning). A hole was made on the lid to create an entrance for copper tubing (50 cm length, 6 cm outer radius), and the tube was fed through an air-tight cable gland, which was further sealed with silicone caulk inside the cell and stabilized with epoxy resin outside. The copper tube extended into the jar and positioned at approximately 1 mm above the liquid surface. The copper tubing acted both as the high voltage electrode and the evacuation port in the reaction cell. The reaction cell contacts a nonthermal plasma with water under reduced pressure and during this interaction, plasma excites and ionizes the air molecules and water vapor in the cell. These excited and ionized molecules lead to the eventual formation of nitrate, nitrite, and ammonium ions in water. The experimental setup is schematically depicted (**Figure 7a**).

A miniature high-voltage (4 kV) and high-frequency (25-35 kHz) power supply (12 W maximum power, MINIMAX40, Information Unlimited) was connected to the copper tubing and the outer end of the tubing itself was attached to a vacuum hose, to which a 16 cm-diameter Bourdon gauge (2.5% accuracy, 0-760 Torr, Pakkens) was connected. The vacuum hose electrically isolated the manometer and the parts downstream of it. The entire assembly was connected to a single-stage rotary vacuum pump (2.55 m^3/hour capacity, VE215N, Value). The jar could be isolated from the vacuum pump with the help of a ball valve positioned in between the manometer and the pump. To create a plasma, a well-defined

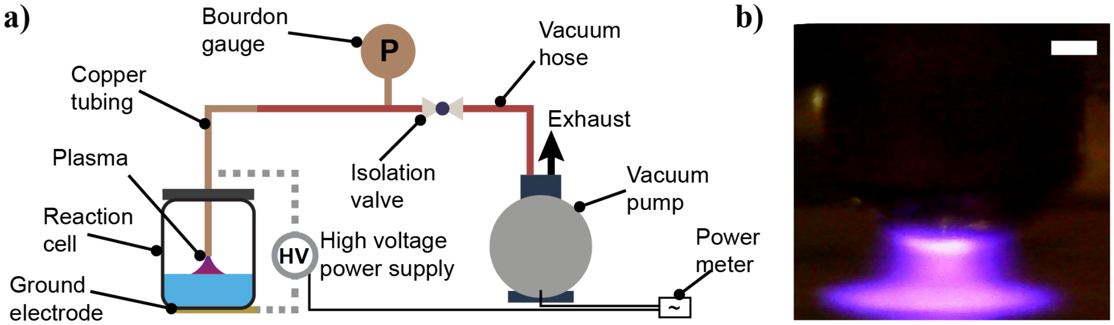

Figure 7. Experimental setup for low-pressure plasma nitrogen fixation and the air plasma. a) Schematic drawing of the setup. **b)** Photograph of an air plasma captured using a digital microscope formed over water at a distance of 1.3 mm from the air-water interface. The white scale bar shows 1 mm.

ground electrode was needed, therefore a thin copper foil attached beneath the jar was used as the ground. It was possible to ignite the plasma when cell pressure was approximately below 260 Torr (**Figure 7b**). All pressure levels reported are given as absolute.

Amount of total dissolved solids (TDS) and the temperature of the water was measured before every experiment using a TDS meter (0-9990 ppm and 0-93.3°C range, TDS-3, Unichrome Zauss). TDS of tap water was found to be 566 ± 51 ppm and experiments were conducted at 16 ± 1°C. After TDS measurement, water was put into the jar and air inside the cell was vacuumed until the pressure was 100 Torr. At this pressure, the ball valve was closed, and the power supply was turned on while the pump was turned off. Over the course of 20 minutes, the pressure inside the cell rose up to approximately 220 Torr due to air leaks. At 220 Torr, the pump was restarted, and the ball valve was opened to obtain 100 Torr pressure. At 100 Torr, the valve was closed, and the pump was turned off again. This process was repeated over the course of the experiment, resulting in an average pressure of 160 Torr. The power used by the power supply and the pump was measured by a power meter (0-16 A and 1-3680 W range, UT230B-EU, Uni-T). To find the energy efficiency of the process, the electric energy used by the vacuum pump and the power supply was measured with the power meter during the experiments. The measured moles of the products (*vide infra*) were divided by the total energy used (kWh) and energy efficiency, $\hat{E}$, was calculated.

After the activation process was completed, the TDS levels, temperature and pH of the plasma-activated water were measured. Test strips (pH 0 - 14 universal indicator, Merck 109535,) were used for pH measurement. In the plasma-treated water, NO_3^-, NO_2^- and NH_4^+ levels were measured using colorimetric test kits (CB 5120 for nitrate, CB 5340 for nitrite and CB 5020 for ammonium, all from Chembio) which can measure up to 100 ppm of nitrate, 1 ppm of nitrite and 10 ppm of ammonium. In order to apply the kits, 5 mL of solution was taken from the plasma-treated water. Then, reagents of the kits were added which resulted in a colored solution. The intensity of the colors was measured using a color legend included in the kits. In the case of the concentrations being higher than the measuring range of the test kits, which appeared as solutions with too dark of a color, the solutions were diluted several times until the color of the solution fell within the range of the color legend. In the tap water, 30 ppm of nitrate and 0.05 ppm of nitrite were measured before plasma treatment, but ammonium was below the measurement level (0.1 ppm).

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