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Article

Absorption Chemistry of Plasma Generated NO_x Gas for Green Nitric Acid Production

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Abstract

Nitric acid (HNO_3) is predominantly produced in large production plants using the Ostwald process. In view of its widespread application as synthetic fertilizer, small scale and local production becomes of interest. The chemical precursor of nitric acid is NO_x gas which can be produced from air at percentage-level concentration using small-scale electrically powered warm plasma reactors. The absorption and disproportionation chemistry of concentrated NO_x streams under pressure and in minimal amounts of water, as in the Ostwald process is well documented. However, the chemistry at lower NO_x concentration levels, without pressurization and in presence of excess water, relevant for nitric acid production with NO_x streams from warm plasma has received much less attention. In this work, the NO_x absorption chemistry is investigated at pH values of the scrubber solution in the range of 1 to 5. The kinetics of NO_x absorption are found to be strongly pH dependent, requiring up to hours of recirculation to reach steady state conditions. Once at steady state conditions, the NO_x removal efficiency turns out to be rather pH independent. The formation of nitrous acid (HNO_2) byproduct reaches a constant value based on a dynamic equilibrium of its formation and decomposition. Approaches to minimize undesired nitrite and nitrous acid byproduct formation are discussed.

Keywords: green nitric acid production; warm plasma; plasma NO_x; NO_x absorption

1. Introduction

Ammonium nitrate fertilizer, an essential element in our global food production system is currently produced in large chemical plants [1,2]. Ammonia (NH_3), being the base chemical, is produced in the Haber-Bosch process. Ammonia synthesis, using hydrogen gas produced from fossil methane is responsible for a significant percentage of the global carbon dioxide emissions [3–5]. In the subsequent Ostwald process, NH_3 is catalytically oxidized with O_2 to form NO_2, which upon reaction with water forms HNO_3. The chemistry of this industrial absorption processes is complex. The process entails about 40 entangled reaction steps with intermediates and by-products such as HNO_2, N_2O, N_2O_3 and N_2O_4 [6]. NO oxidation and NO_2 absorption are performed under pressure of up to 10 bar and at NO_x gas concentrations of around 10%. The commercial HNO_3 product is typically a concentrated aqueous solution containing 65 wt.% HNO_3 [7–9].

Electricity-powered warm plasma technology is an emerging alternative for nitric acid production and even has the potential to fixate N_2 into NO_x with a lower net energy consumption than the combined Haber-Bosch and Ostwald process [10]. In the plasma electrons and gas molecules are excited such that nitrogen and oxygen gas from atmospheric air react to NO_x [10,11]. Like in the Ostwald process, NO_x needs to react with water to form HNO_3. However, in a plasma process NO_x is produced at concentrations in the range of a few percentages, which is substantially lower than in the Ostwald processes (ca. 10%), leading to different absorption conditions [7,8,10].

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Besides serving as a precursor for HNO_3, NO_x gas is also a known air pollutant emitted at parts-per-million levels during combustion processes [12,13]. NO_x scrubbing is an end-of-pipe solution to eliminate it from flue gas [14,15]. Under the near ambient temperature and pressure conditions, the low aqueous solubility of NO and the slow kinetics of its spontaneous oxidation to the better dissolving NO_2 often necessitate the use of oxidizing agents like H_2O_2 and O_3 [15–19]. The partial pressure of NO_x and the NO_2 - to - NO ratio significantly influence the absorption performance and the formation of HNO_2 versus HNO_3 [12,20,21]. Alkaline scrubbing solutions are often deemed more effective for NO_x removal compared to neutral of acidic solutions due to the acidic character of NO_x [12].

Given the current relatively small size of plasma reactors for NO_x production, a scrubber unit for producing HNO_3 from plasma-generated NO_x should be simple and operated without pressurization and added chemicals. The operation conditions of such NO_x scrubbers are in between those of the Ostwald process and NO_x emission abatement technologies. While NO_x absorption is extensively modelled for example by Patwardhan et al. [22], only limited experimental research has been performed in NO_x to HNO_3 conversion, specifically under reaction conditions relevant to plasma-NO_x. Notably, much of the experimental research in this area focuses on short-duration tests, often with recirculation times for the scrubbing solution limited to an hour [20,23].

In this work the absorption reaction network of NO_x-containing gas, simulating the gas mixture produced from ambient air in a warm plasma reactor is experimentally investigate for long term recirculation. Insight is gained on the influence of scrubbing pH on NO_x absorption kinetics, capacities and on the formation of undesired nitrous acid side product without the addition of oxidizing agents.

2. Results

2.1. NO_x Scrubbing Experiments

NO_x scrubbing experiments were performed in a column with an empty bed volume (EBV) of 780 mL with 400mL recirculation solution of which the pH was kept fixed at values of 1, 2, 3, 4 and 5 by dosing KOH (**Figure 1**). A gas mixture consisting of 1% NO, 20% O_2 and 79% N_2 enters the column in counter current with the solution at a flowrate of 3 L/min. In order to ensure sufficient contact area the column is filled with an inert packing material. In- and outgoing NO_x gasses are analyzed in-line using a UV detector. The HNO_3, NO_2^- and HNO_2 content of the scrubber solution was analyzed on regularly taken samples, therefore HNO_2 was deprotonated to NO_2^- by adding buffer solution of pH 7 to enable quantification with ion chromatography. The combination of NO_2^- and HNO_2 will later on be referred to as total nitrate concentration. For more information see section Methods.

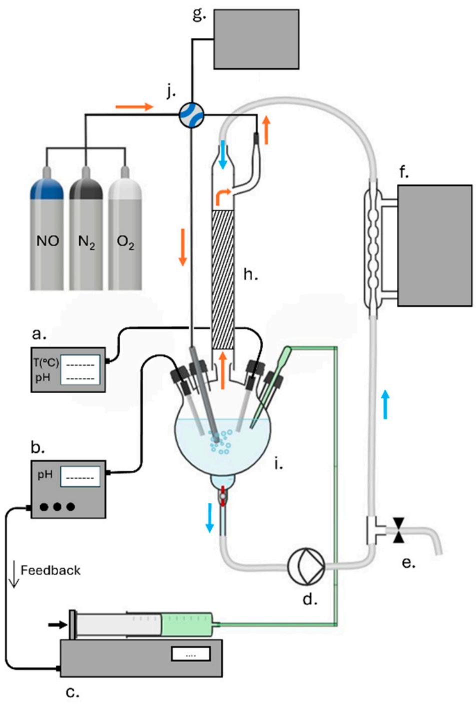

Figure 1. NO_x scrubber experimental setup. a) pH and temperature monitoring; b) pH controller; c) KOH solution pump; d) recirculation pump; e) scrubber solution sample valve; f) cryostat; g) NO_x gas analyzer; h) absorption column filled with packing material; i) round bottom flask with probe inlets; j) bypass valve →) direction of gas flow →) direction of liquid flow.

2.2. *Scrubber Liquid Analysis*

The nitrate, nitrite and total nitrite concentration, standing for the sum of NO_2^- and HNO_2 in the scrubber solution were determined at 1 h time intervals (**Figure 2**). The experiments at pH 4 and 5 were run over two days. No liquid samples were taken during the night. The nitrate concentration steadily increased throughout the five experiments at different pH values. After 6 h scrubbing, the nitrate concentration in the scrubber solution reached values of 12.6 mM at pH 1; 14.0 mM at pH 2; 10.4 mM at pH 2; 4.72 mM at pH 3, and 2.97 mM at pH 5 (**Figure 2**). The nitrate accumulation decreased with increasing pH value.

The total nitrite concentration displayed increasing concentrations at higher pH value. At pH 1, 2 and 3, the total nitrite concentration reached a plateau after 2 to 4 hours (**Figure 2**) at ca. 3.37 mM, 3.86 mM and 5.41 mM for pH 1, 2 and 3, respectively (**Table 1**). At pH 4 and 5 the total nitrite concentration in the circulating solution did not reach a plateau, even when the scrubbing was prolongated to 30 hours. The total nitrite concentrations rose to 21.6 and 38.4 mM, respectively at pH 4 and 5. At pH 5 nitrite is the main NO_x-derived compound in the scrubber liquid.

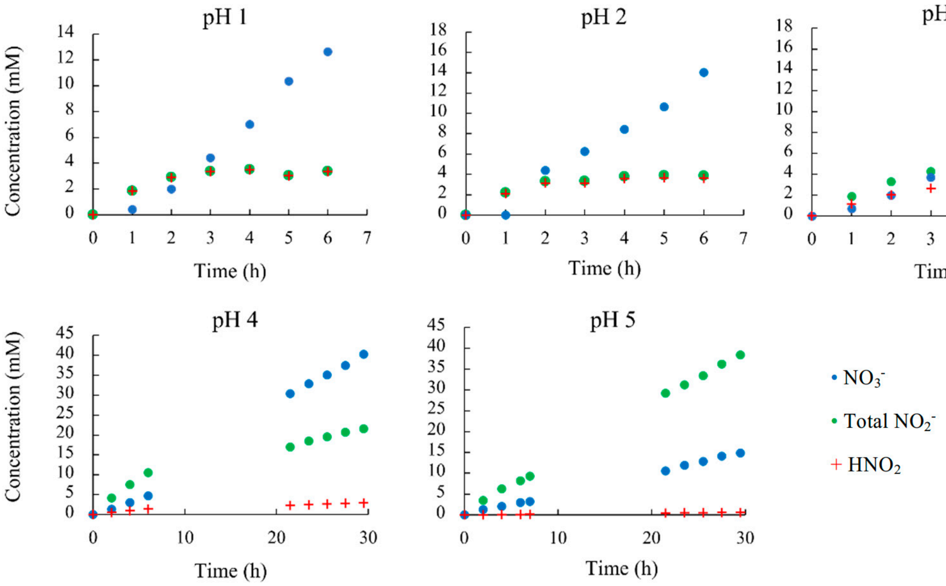

Figure 2. Nitrate, total nitrite and nitrous acid concentration in the scrubber solution during NO_x scrubbing experiment at pH 1, 2, 3, 4 and 5. For experiments at pH 4 and 5 the scrubber solution was not sampled during the night in the period from 9 to 20 h.

After the total nitrite concentration plateau was reached at pH 1, 2 and 3, the calculated HNO_2 concentrations reached very similar concentrations of 3.35, 3.63 and 3.32 mM, respectively (**Table 1**). At pH 4, the HNO_2 concentration reached 2.95 mM at the end of the 30 h testing period and did not fully reach a plateau yet, suggesting it could still increase to reach a value similar to that reached in the experiments at lower pH. At pH 5 the final HNO_2 concentration reached only 0.599 mM, which is substantially below the plateau value reached at lower pH. Under this condition, it may take a very long time to reach the plateau.

Table 1. Total nitrite , actual NO_2^- and HNO_2 concentrations in the scrubber solution at the end of the experiments of **Figure 2** at different pH.

pH	Time (h)	Total NO_2^- (mM)	NO_2^- (mM)	HNO_2 (mM)
1	6	3.37	0.0211	3.35
2	6	3.86	0.229	3.63
3	6	5.41	2.09	3.32
4	30	21.6	18.6	2.95
5	30	38.4	37.8	0.599

2.3. Gas Phase Analysis

The NO_x concentration in the gas exiting the scrubber under the different pH conditions is presented in **Figure 3a** and **b**. In experiments at pH 1, 2 and 3, NO_x outlet concentrations initially were around 800 ppm, and increased during the experiment until a plateau value of ca. 1,800 ppm was reached. The corresponding NO_x removal efficiency decreased from ca. 95% initially, to 82.5%, 83.9% and 83.0 % at pH 1, 2 and 3, respectively (**Figure 3c**).

At pH 4 and 5, the outgoing NO_x gas concentrations were significantly lower after 7 hours, and still increasing (**Figure 3b**). When prolonging the scrubbing to 30 hours at pH 4, a plateau value of ca. 84.2% removal efficiency was reached (**Figure 3c**), which is in the range of values obtained at pH 1, 2 and 3. At pH 5 even after 30 hours the removal efficiency kept on decreasing slowly. Very long times would have been needed to verify the reaching of a plateau. Gaps in data are due to a set-up malfunction leading to an artefact in the data, irrelevant for further interpretation.

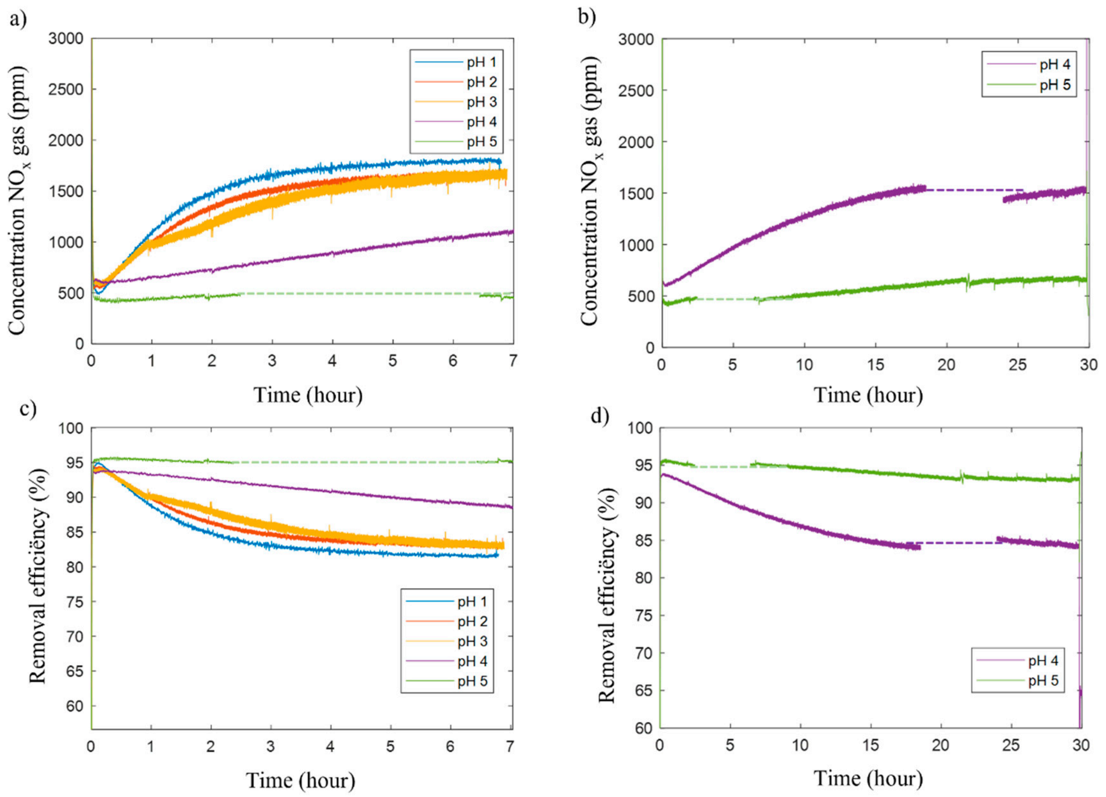

Figure 3. a) NO_x outlet concentrations (ppm) for experiments over a time span of 7 hours at pH 1, 2 and 3; b) over a time span of 30 hours at pH 4 and 5;. c) NO_x removal efficiencies at all pH values over a time span of 7 hours; d) Removal efficiency in scrubbing experiment at pH 4 and 5 over a time span of 30 hours. For representation, the moving average over 20 points of each curve is taken. Standard deviations of the curves of pH 1, 2, 3, 4 and 5 were 16, 68, 100, 38 and 28 ppm, respectively.

3. Discussion

The reaction network of NO_x gas absorption in aqueous media is complex and involves dissolution as well as chemical reactions involving reaction intermediates and reversible and irreversible reactions in gas as well as liquid phase. The reactions considered most important in the gas phase are presented in Eqns. 1-4 [6,23–25].

$$2\ NO + O_2 \rightarrow 2\ NO_2 \tag{1}$$

$$NO + NO_2 \rightleftharpoons N_2O_3 \tag{2}$$

$$NO + NO_2 + H_2O \rightleftharpoons 2\ HNO_2 \tag{3}$$

$$2\ NO_2 \rightleftharpoons N_2O_4 \tag{4}$$

According to Eqns. 1-3, NO gas is converted into NO_2, N_2O_3, and HNO_2, respectively, which are more water-soluble. NO_2 also dimerizes to form N_2O_4 (Eqn.4).

The main reactions in the liquid phase are given in Eqns. 5-8. Dissolved N_2O_3, and N_2O_4 are converted into either HNO_2 or a combination of HNO_2 and HNO_3 [22,25,26]. The reactions of Eqns. 5-8 are often assumed irreversible since the rate of the back reactions, being bimolecular, is considered negligible in the present diluted, aqueous solutions [23,27–29].

$$2\ NO_2 + H_2O \rightarrow HNO_2 + HNO_3 \tag{5}$$

$$N_2O_3 + H_2O \rightarrow 2\ HNO_2 \tag{6}$$

$$N_2O_4 + H_2O \rightarrow HNO_2 + HNO_3 \quad (7)$$

$$3\,HNO_2 \rightarrow H_2O + 2\,NO + HNO_3 \quad (8)$$

In liquid phase nitrous acid is prone to dismutation leading to HNO_3, NO and H_2O formation according to Eqn. 8 [22,26,30,31]. The poorly soluble NO molecules desorb from the solution and return to the gas phase, where they re-enter the reaction network [32]. The HNO_2 decomposition reaction mechanism is often assumed as described by Schwartz et al. to be 4th order in HNO_2, but accurate rate equations remain matter of discussion [26,28,29].

The observation of a total nitrite (NO_2^- and HNO_2) concentration plateau in the scrubber liquid at fixed pH values of 1, 2 and 3 (**Figure 2**) can be explained as follows. Nitrous acid decomposition is a dismutation reaction involving several HNO_2 molecules and a reaction order higher than 1 (Eqn. 8) [28,29]. With increasing HNO_2 concentration, the HNO_2 dismutation reaction rate will increase until it equals the rate of HNO_2 formation (Eqns. 5-7), leading to a steady state HNO_2 concentration. At a constant pH, this also means a steady state NO_2^- concentration.

This is in agreement with what is seen in the analysis of the gas phase. The decrease of the NO_x removal efficiency with time at constant pH (**Figure 3**) could be attributed to this formation of NO by HNO_2 dismutation (Eqn. 8). Here as well it is remarkable that once the system reaches its steady state, NO_x removal efficiencies are very similar for pH 1, 2, 3 and 4. The similar limitation of the NO_x removal efficiency is also likely to be a consequence of the similar HNO_2 concentration (**Table 1**) and the equal rate of the HNO_2 decomposition reaction (Eqn. 9).

While at steady state the pH does not seem to influence the removal efficiency, in the transient period the pH does matter as observed in earlier studies [12,21,23,33]. The time after which the steady state condition is reached, is pH dependent. For tests at pH 1, 2 and 3 the HNO_2 concentrations reaches steady state after approximately four, five and six hours, respectively. At pH 4 and 5, the accumulation of HNO_2 in the scrubber solution progresses much slower (**Figure 2**). This can be attributed to the acid dissociation reaction. The higher the pH, the more HNO_2 directly dissociates into NO_2^- and the longer it takes for HNO_2 to accumulate enough for the dissociation reaction (Eqn. 8) to balance the formation rate. Per pH unit, ten times more HNO_2 is converted to NO_2^-, which is reflected in **Table 1**. The test at pH 4 nearly reached its steady state after 30 hours, but the test at pH 5 did not reach this level within the tested timespan. An important conclusion to draw is that running experiments for shorter timespans than necessary to reach steady state, could wrongly lead to the assumption that increasing the pH of the scrubbing solution also increases the removal efficiency on the long term.

Nitrate, being a strong acid and assumed always in its dissociated form, accumulates over time in the scrubber solution (**Figure 2**). In the transient period the concentration increases more than proportional with time. This can also be attributed to the increasing rate of the HNO_2 decomposition reaction (Eqn. 8) which also forms HNO_3. Given the steady state NO_2^- and HNO_2 concentrations and the ongoing NO_3^- accumulation there is no point in expressing a nitrate selectivity in these tests as it is strongly time dependent. What can be expressed is the steady state NO_2^- amount that remains present. In practical applications of nitrate fertilizer production, nitrite and nitrous acid are undesired side products. Even though maximal allowed nitrite concentration in nitrogen fertilizer is in many countries not explicitly regulated, effects of nitrite toxicity are reported at concentrations starting from 0.11 mM [34]. It is however known that nitrite is in soil processes rapidly transformed in nitrate [35]. This study hints at a few options expected to lower the steady state HNO_2 concentration without sacrificing the NO_x removal efficiency.

A first approach to consider, is to increase the liquid volume. For a constant EBRT but larger liquid volume, a slower increase in HNO_2 concentration is expected for the same absolute amount of NO_x absorbed. This means that also the dismutation reaction is slowed down, resulting in a lower steady state HNO_2 concentration and NO formation. For experiments at pH 1 with a liquid volume of 250 mL and 400 mL, similar removal efficiencies of 83.6% and 82.5% were reached, respectively.

Total nitrite concentrations stagnated after 6 hours at 4.07 and 3.37 mM for 250 mL circulation volume and 400 mL. Respectively, 4.04 mM and 3.35 mM of this were in the form of HNO_2. Increasing the volume of the scrubbing liquid 1.6 times, reduced the nitrite content with around 17%. Increasing the liquid volume as well as slowing down the dismutation reaction, also decreases the rate of NO_3^- formation. After 6 hours a nitrate concentrations of 19.6 mM and 12.6 mM were obtained for 250mL and 400mL scrubbing solution. However, this can be remedied by increasing the time over which the experiment in ran.

As shown in other experimental contexts an important factor influencing the nitrite content is the NO_x oxidation ratio [12,20]. By oxidizing NO to NO_2 and forming N_2O_4 instead of N_2O_3, reactions of Eqns. 4, 5 and 7 are favored over reactions of Eqns. 3 and 6, lowering in this way the HNO_2 formation and dismutation rate. The NO_x oxidation ratio is dependent on the composition of the plasma produced NO_x gasses but also by the oxidation reaction of NO (Eqn. 1), which is second order dependent on NO partial pressure and first order dependent on O_2 partial pressure [22,36]. As often done, pressurizing the air in or at the outlet of the plasma process will improve the NO_2 content of the NO_x and the nitrate selectivity in the scrubber as showcased by Tsonev et al. reaching an OR of 94% [37]. Another solution is to prolong the residence time of the gas in the scrubber.

A final option to further lower the nitrite content would be to store the scrubbing solution in a storage tank, after the desired nitrate concentration is reached. The dismutation reaction of HNO_2 proceeds spontaneously but is slow. Favored by a low pH, undesired nitrite and nitrous acid are expected to be eliminated over time, producing nitric acid and NO gas. The gas phase enriched with NO could be recovered from the storage tank and again injected in the scrubber to ensure no NO_x in released to the environment.

4. Materials and Methods

The experimental set-up for NO_x absorption experiments is depicted in **Figure 1,** see section Results. A mixture of NO, O_2 and N_2 gases delivered from gas cylinders was fed to the absorption column. Absorption liquid was circulated in such a way that gas and liquid are running in counter current. Large contact area was assured between liquid and gas by filling the column with packing material consisting of 6 mm glass Raschig rings. The column was kept at constant temperature of 293K by a cryostat. The liquid outlet of the column is connected to a round flask with openings for the gas inlet, base solution inlet, pH probe (Mettler Toledo, SevenCompact) and pH control system (Hannah instruments, HI510 and HI1006-1850).

The inlet gas mixture was composed of 1% NO, 20% O_2 and 79% N_2, and fed from gas cylinders using mass flow controllers (Bronkhorst, EL-FLOW). The flow rate was 3 L/min. NO and NO_2 concentrations in the gas mixture were measured with an UV-analyzer (ABB AO2020 Limas11HW). Before starting the absorption, the feed gas was run during 30 minutes in bypass mode and sent directly to the analyzer without passing through the column to allow the gas composition to stabilize and to be verified. The NO and NO_2 removal efficiency (RE) was estimated as the difference between the in- and outlet concentration divided by the inlet concentration (Eqn. 9):

$$RE\ (NO_x)\% = 1 - \frac{\int_{t_{end-5min}}^{t_{end}} (C_{NO_x\,out})}{\int_{t_{end-5min}}^{t_{end}} (C_{NO_x\,bypass})} * 100 \tag{9}$$

In the bypass phase of each experiment some NO oxidation already occurred in the gas lines, resulting in a mixture of NO and NO_2 to reach the scrubber column. The oxidation ratio (OR) defined as the ratio of the concentration of NO_2 over total NO_x is determined according to Eqn. 10. Oxidation ratios were very similar for all tests with OR values of 0.56 ± 0.008.

$$OR = \frac{C_{NO_2\,in}}{C_{NO_2\,in} + C_{NO_{in}}} \tag{10}$$

The pH of the scrubbing solution was kept constant as follows. The round flask was filled with acidified aqueous solution of H_2SO_4 (Acros Organics, 96 wt%) or H_3PO_4 (Acros Organics, 85 wt%) at the intended pH, in the range from pH 1 to pH 5. During operation of the absorption column, the pH was monitored and kept constant by dosing 0.2 M KOH (Sigma-Aldrich, ≥85%). Liquid samples were taken every hour. Liquid samples taken from the round flask were diluted 100 times and buffered at pH 7 using an equimolar mixture of 0.005 M NaH_2PO_4 (Merck, > 99%) and Na_2HPO_4 (Merck, 98-100%). Nitrous acid is a weak acid being only partially deprotonated under acidic conditions. Protonation-deprotonation equilibrium of nitrous acid was considered to be established in the scrubber solution under all conditions. Estimates of the actual concentration of nitrous acid based on total nitrite analysis were made assuming a nitrous acid pK_a value of 3.20 (Eqn. 11) [38].

$$[HNO_2] = \frac{[NO_2^-] * 10^{-pH}}{10^{-pK_a}} \tag{11}$$

At this pH value nitrous acid is deprotonated which is needed for quantification using anion chromatography. The nitrate and nitrite content of the liquid samples were analyzed using ion chromatography (Metrohm Eco IC, A Supp 5 column, 850 IC conductivity detector).

5. Conclusions

This study provides experimental data on the NO_x absorption chemistry in aqueous solution under conditions relevant for small scale fossil carbon-free nitric acid production based on thermal plasma technology producing NO_x gas at concentrations of around 1%. An aqueous solution with constant pH in the range 1 to 5 was recirculated over a packed scrubber column ensuring large contact area.

The NO_x removal efficiency is observed to be higher for increased pH at the onset of the experiment, yet decreases over the course of the experiment to a plateau value of 83%, independent of pH. The explanation lies in a corresponding increase in the HNO_2 concentration until a plateau is reached, and a negative correlation between HNO_2 concentration and removal efficiency. The steady state HNO_2 concentration and, therefore, the HNO_2 decomposition rate are observed to be very similar for all pH values. It is thus concluded that pH does not influence the removal efficiency when running experiments sufficiently long for steady state to be reached. However, pH does remain an important parameter concerning the NO_2^- concentration as it is the determining factor in the $NO_2^- \leftrightarrow HNO_2$ equilibrium.

This paper indicates the importance of HNO_2 as a key compound in the systems transient period as well as in the reaction steps which contribute to ultimately reaching a steady state removal efficiency.

Supplementary Materials: Not applicable

Author Contributions: Conceptualization, L.H. and J.M.; methodology, A.V. and R.D.W.; software, L.H., A.V. and R.DW.; validation, A.V. and R.D.W; formal analysis, A.V and R.D.W.; investigation, A.V. and R.D.W.; resources, J.M.; data curation, A.V. and R.D.W.; writing—original draft preparation, A.V., R.D.W. and L.H.; writing—review and editing, J.M..; visualization, A.V.; supervision, J.M.; project administration, L.H and A.V.; funding acquisition, L.H. and J.M. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest

Abbreviations

The following abbreviations are used in this manuscript:

OR	Oxidation Ratio
EBV	Empty Bed Volume
RE	Removal Efficiency

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