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Investigation of microwave-induced nitrogen-oxygen plasma for nitrogen fixation

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Abstract

Atmospheric nitrogen fixation is a cornerstone of modern agriculture and industrial chemistry. Typically, the conversion is performed by the fossil-based Haber-Bosch process, and therefore, sustainable and carbon emission-free alternatives are of growing interest. This study investigates the production of nitrogen oxides using a microwave plasma reactor under different oxygen-to-nitrogen ratios, pressures, and energy inputs. The results demonstrate that nitrogen dioxide production peaks at 40 Vol% nitrogen in the input gas, reaching a maximum concentration of 4.86 Vol% at 2.23 MJ mol^{-1}energy input and 0.09 bar pressure. The highest nitrogen conversion of 18.3% was observed under nitrogen-deficient conditions with an energy input of 3.44 MJ mol^{-1}, whereas the highest energy efficiency of 0.36% was achieved at 1.21 MJ mol^{-1}. Additionally, enhancing nitrogen dioxide production is possible by lowering operational pressure, while nitrogen monoxide formation remains relatively pressure-insensitive.
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Keywords: Microwave, plasma, nitrogen fixation, plasma power to X.

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1. Introduction

The conversion of inert atmospheric nitrogen into biologically or chemically usable forms is essential for sustaining life and modern agriculture. Historically, nature's lightning and early industrial processes like the 1903 Birkeland–Eyde electric arc process provided fixed nitrogen (as nitric oxide/nitrates), but with relatively low yield (~1% NO_x) and high energy consumption (~3.5–4.1 MJ per mole HNO_3) [1, 2]. The "Great Acceleration" that began in the 1950s is a part of the global population explosion [3], during which global energy consumption, pollutant emissions, real GDP, and other indicators experienced exponential growth. This population explosion would not have been possible without the development of agriculture, in which one of the most significant changes was the widespread adoption of synthetic fertilizers. The Haber-Bosch (HB) process, developed in the 1910s, soon became the dominant method of artificial nitrogen fixation, producing ammonia (NH_3) from N_2 and H_2 and enabling the mass production of fertilizers that underpin roughly half of global food production [4, 5]. Today, HB provides ~90% of the world's reactive nitrogen supply [4, 6] and is often coupled with the Ostwald process (NH_3 oxidation to NO/NO_2) to produce nitric acid and nitrates. However, HB is extremely energy- and carbon-intensive, accounting for ~2–3% of global energy use and ~300 Mt of CO_2 emissions annually [7]. Its H_2 feedstock (~96%) is still derived from fossil fuels via steam methane reforming, leading to a CO_2 footprint of ~2.5 kg CO_2 per kg NH_3 produced [6]. Decades of engineering optimizations have brought HB close to its theoretical minimum energy input, leaving little room for efficiency improvement. Moreover, if "green" H_2 (from electrolysis) is used, the overall energy efficiency would worsen due to inefficiencies in water electrolysis. The Ostwald process for nitrates has issues, such as the N_2O by-product (a greenhouse gas ~300 times more potent than CO_2). These challenges have motivated the search for alternative, sustainable nitrogen fixation technologies.

In our era, renewable electricity generation is growing rapidly. However, the widespread deployment and intermittent nature of solar and wind power pose significant challenges, particularly regarding energy storage and grid stability [8]. To address these challenges, technologies that can dynamically follow the variability of renewable production and switch on and off easily are required, which is often referred to as Power to X in the literature [9]. Plasma technologies, which can convert renewable electricity into a useful chemical compound (PP2X), offer a compelling solution [10, 11]. This is also why low-temperature plasma technologies have garnered considerable attention from researchers compared to other alternative nitrogen fixation methods (e.g., electrocatalytic, photocatalytic, or biological approaches). Microwave plasma technology is considered as one of the most promising alternatives [12] because it is electrodeless, cost-effective [13], and scalable, making it an optimal plasma source. The ability to rapidly turn on/off the electrical power in plasma reactors also makes them well-suited to intermittent renewable energy, unlike HB process, which demands continuous high-temperature operation [1].

Plasma-based nitrogen fixation can follow two general pathways: reduction of N_2 (typically with H_2 or H_2O) to produce ammonia or oxidation of N_2 (with O_2 or air) to produce nitric oxide (NO) and related oxides (NO_2, etc.). The H_2-based reduction pathway essentially plasma-assists the ammonia synthesis reaction and usually benefits from catalysts (analogous to HB catalysts) to form NH_3 at lower temperatures. The oxidation pathway, the focus of this work, uses air or N_2/O_2 mixtures to generate NO/NO_2 (often collectively termed as NO_x) in plasma. A significant advantage of the N_2/O_2 route is that it eliminates the need for high-purity H_2 feedstock [4]. Instead, air can be used as the input, while the output NO/NO_2 can be absorbed in water to produce nitric acid or nitrates, bypassing the ammonia production step entirely. This simplifies the chemical supply chain and could reduce the costs of feedstock and reactors. Additionally, NO itself is a valuable product for certain medical therapies (e.g., wound healing, vasodilation), so plasma-generated NO has niche biomedical applications as well [14]. For all these reasons, plasma-based N_2 oxidation is an attractive alternative, drawing renewed attention.

Non-thermal plasmas have a theoretical limit as low as ~0.2 MJ mol^{-1} for NO_x production [1, 15] since they could bypass the thermodynamic losses of heat-based processes. Achieving such performance in reality, however, remains a grand challenge, one that modern researchers are actively addressing with improved plasma reactor designs and diagnostics. Several groups have developed microwave plasma reactors operating at atmospheric or moderate pressures to fix N_2 and O_2 into NO_x directly. One of the most significant recent achievements is the work of Kelly and Bogaerts [2], who investigated microwave plasma (2.45 GHz, ~1 kW) with a 50/50 N_2/O_2 flow at atmospheric pressure. By optimizing the gas flow (~20 NL min^{-1} in a tangential swirl) and power coupling, they reported a NO_x concentration of ~3.8% in the effluent, with an energy cost of ~2 MJ mol^{-1}. This is the lowest specific energy consumption documented for atmospheric-pressure plasma N-fixation, marking a major milestone. In another study, Kim *et al.* [16] explored a low-power microwave discharge (80 W) at 1 atm and different ratios of N_2/O_2. They achieved NO_x concentrations of 0.33–0.48% in the output, with the lowest energy cost of ~3.76 MJ mol^{-1} under optimal conditions. Lee *et al.* [17] used a 2 kW magnetron to create microwave plasma at atmospheric pressure in a quartz tube. By operating with N_2/O_2 ratios very close to the N_2-rich (97–99% N_2) region and adjusting the flow rate from 45 NL min^{-1} down to 25 NL min^{-1}, they observed the NO_x concentration climb from ~1612 ppm to ~9380 ppm. Na *et al.* [18] demonstrated a different configuration, a microwave plasma torch operating at atmospheric pressure, to produce NO for biomedical applications. They obtained ~2000 ppm of NO using a 400 W plasma with 5 NL min^{-1} air flow, which was sufficient for fungal spore treatment. Although the yield was modest, this illustrated the versatility of plasma reactors (in this case, a torch design) and the potential for small-scale, application-specific nitrogen monoxide generation.

Researchers have recently revisited pulsed microwave plasmas and low-pressure operation to enhance vibrational excitation and energy efficiency. Samadi-Bahnamiri *et al.* [19] reported a pulsed microwave discharge (2.45 GHz, 0.9 kW average power at 0.5 kHz with 50% duty cycle) in a surfaguide-type reactor. They operated at reduced pressure (~8 mbar) to strongly favour non-equilibrium conditions. Using a specialized FTIR optical diagnostic, they measured the outlet gas composition and found NO as the primary product, with no detectable NO_2 or N_2O byproducts under their conditions. At 50% N_2/50% O_2 feed, the best result was ~7% NO yield at ~8 MJ mol^{-1}, corresponding to an energy efficiency of ~1.1% (defined as the fraction of input energy channeled into the NO formation enthalpy). With a lower flow of 0.5 NL min^{-1}, the yield went up to ~12% NO, though at a higher cost of ~13.7 MJ mol^{-1}. These data highlight the yield/efficiency trade-off: the highest NO fraction came with reduced energy efficiency. In a follow-up study, Samadi-Bahnamiri *et al.* [20] examined the rotational and vibrational temperatures in the same microwave plasma via optical emission spectroscopy. They confirmed that at low pressures, the vibrational temperature of N_2 (T_{vib}) greatly exceeds the rotational (gas) temperature T_{rot}, indicating a strong non-equilibrium, whereas at higher pressures, the two temperatures converge. Notably, they found that NO yield correlated positively with N_2 vibrational excitation: conditions that increased the population of N_2 vibrational levels led to higher NO production, whereas simply increasing the gas temperature had the opposite (negative) effect on NO yield. These observations support the theoretical understanding that vibrationally excited N_2 is the key intermediate for efficient NO synthesis. This kind of plasma kinetic insight is crucial for designing next-generation plasma reactors.

The present work builds upon this background by investigating microwave-induced nitrogen-oxygen plasma under reduced and atmospheric pressure conditions while thoroughly investigating conversion and energy efficiencies. The measurements focus on NO and NO_2 concentrations, with the oxygen concentration of the input gas varying by up to 90 Vol%.

2. Materials and methods

The experimental system (Fig. 1) consists of a magnetron, launcher, circulator, water load, 3-stub tuner, directional coupler, and plasma cavity. The microwave power was generated with a Toshiba 2M248 type magnetron at a 2.45 GHz rated frequency. The generated microwave power can be continuously adjusted in the interval of 0.2–1 kW. The plasma cavity is a mono-mode (TE_{01}) waveguide. At the bottom and the top of the plasma cavity a hole was formed at a distance of $\lambda_g/4$ from its closing plate, where λ_g is the guided wavelength (in our design $\lambda_g = 174$ mm [21, 22]). A quartz tube is placed into the plasma cavity with the possibility of adjusting its location in axial directions. A nozzle for a gas inlet was placed into the quartz tube, and the gas sealing was secured with two rubber O rings. The quartz tube has a 19 mm external diameter, a 16 mm internal diameter, and a 300 mm length. The location of the quartz tube and the nozzle were selected to ensure the highest electric field strength, which was verified by simulation software (Quick Wave v2021:2021.09.10, HN).

Ambient laboratory air supplied by an oil-free compressor blended with 4.5-grade oxygen supplied from a cylinder was utilized for measurements. The flow rates were controlled by mass flow controllers (Sensirion, SFC5500-10SLM [23]). All measurements were performed with a fixed 20 NL/h input gas flow.

The output gas concentration (including NO, NO_x, O_2) was measured with Horiba PG350 gas analyser [24]. The outlet gas was diluted (with 600 NL h^{-1} air) in a controlled manner using a mass flow controller because the Horiba PG350's measurement range for NO and NO_x is limited to 2500 ppm. A vacuum pump was utilized to reduce the pressure during the experiments, while the pressure was adjusted by valves as seen in Fig. 1 and measured with a pressure transmitter connected to a 4–20 mA measurement device type CALOG-TEMP [25].

Before the measurements, the 3-stub tuner was adjusted to form air plasma at the lowest possible value of the introduced microwave power, and all measurements were performed with this fixed setup. Then, the system was ready to adjust the parameters to the desired levels and inject O_2 into the air stream. All the results were recorded after reaching steady-state conditions, typically 5 minutes after the microwave power was applied.
The following reactions were taken into consideration to calculate the nitrogen conversion:

$$\frac{1}{2}\cdot N_2+\frac{1}{2}\cdot O_2 \rightarrow NO\,,\quad \Delta H^0_{NO} = 90.2\,\frac{kJ}{mol-NO} \tag{1}$$

$$\frac{1}{2}\cdot N_2+O_2 \rightarrow NO_2\,,\quad \Delta H^0_{NO2} = 33.2\,\frac{kJ}{mol-NO_2} \tag{2}$$

Following the reactions (1) and (2), the nitrogen conversion (χ_{N2}) can be defined for a nitrogen-oxygen inlet gas mixture as

$$\chi_{N2} \equiv \frac{0.5\cdot n_{NO}+0.5\cdot n_{NO2}}{n_{N2}} = \frac{1}{2}\cdot\frac{n_{NO}+n_{NO2}}{n_{N2}} \tag{3}$$

where n_{NO} (mol) and n_{NO2} (mol) represent the molar quantity of NO and NO_2 in the outlet gas, respectively, while n_{N2} (mol) is the molar quantity of nitrogen gas entering the system. The conversion of nitrogen (χ_{N2}, %) can be expressed as:

$$\chi_{N2} = \chi_{N2}^{NO} + \chi_{N2}^{NO2} \tag{4}$$

where $\chi_{N2(NO)}$ and $\chi_{N2(NO2)}$ are the conversions of NO and NO_2, respectively, and expressed as:

$$\chi_{N2}^{NO} = \frac{y_{NO}}{2k+\frac{k}{2}\cdot\left(\frac{y_{NO}}{100}+\frac{y_{NO2}}{100}\right)} \tag{5}$$

$$\chi_{N2}^{NO2} = \frac{y_{NO2}}{2k+\frac{k}{2}\cdot\left(\frac{y_{NO}}{100}+\frac{y_{NO2}}{100}\right)} \tag{6}$$

where y_{NO} (Vol%) and y_{NO2} (Vol%) represent the volumetric concentration of NO and NO_2 measured by the Horiba instrument after compensation for the dilution gas, respectively. Additionally, k (-) represents the initial concentration of the nitrogen in the supplied nitrogen-oxygen mixture, calculated as:

$$k = \frac{n_{N2}}{n_{N2}+n_{O2}} \tag{7}$$

where n_{O2} (mol) is the molar quantity of oxygen gas entering the system. The molar energy input to the system (E_m, J mol^{-1}), represents the microwave power coupled per unit of gas introduced, and it can be calculated using the following equation:

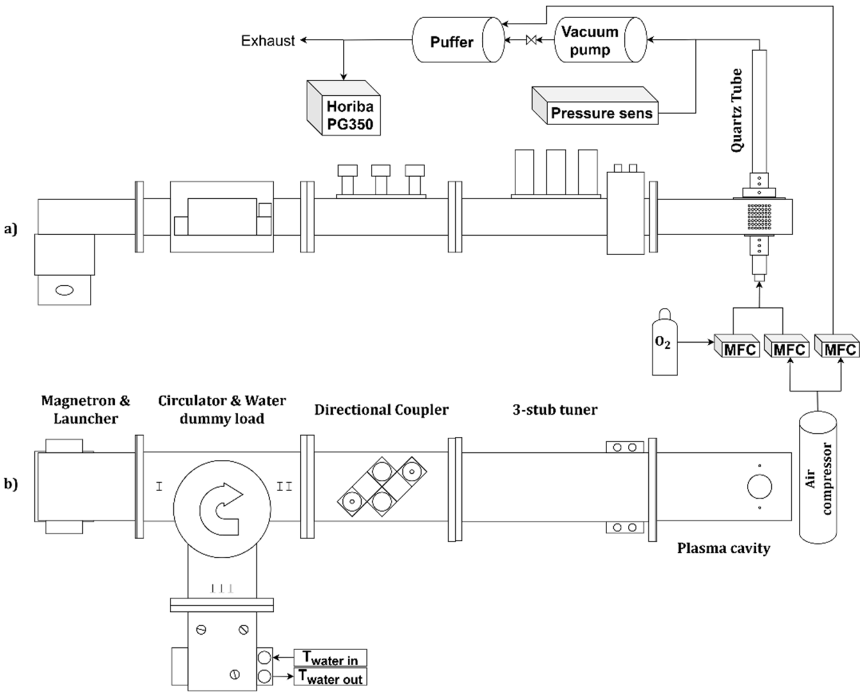

Fig. 1. Schematic diagram of the experimental setup, a) side view, b) top view.

$$E_m = 80640 \cdot \frac{P_{MW}}{Q_P} \tag{8}$$

where P_{MW} (W) denotes the coupled microwave power, and Q_P (NL h^{-1}) represents the standard volumetric flow rate of the gas entering the plasma region. The energy efficiency values corresponding to various cases of nitrogen conversion ($\eta_{N2(NO)}$, $\eta_{N2(NO2)}$, η_{N2}, %) can be calculated as follows:

$$\eta_{N2}^{NO} = \frac{\chi_{N2}^{NO}\cdot\Delta H_{NO}^{0}}{E_m} \tag{9}$$

$$\eta_{N2}^{NO2} = \frac{\chi_{N2}^{NO2} \cdot \Delta H_{NO2}^{0}}{E_m} \tag{10}$$

$$\eta_{N2} = \eta_{N2}^{NO} + \eta_{N2}^{NO2} \tag{11}$$

3. Results and discussion

Fig. 2 summarizes the experimental results by illustrating the concentrations of NO and NO_2 in the product gas measured at different energy inputs and pressures. A maximum point in the NO_2 concentration can be observed at the investigated pressures, peaking at a value of 40 Vol% nitrogen, a result that aligns with stoichiometric expectations. At 0.09 bar, the optimal molar energy input for NO_2 production with a peak value of 4.86 Vol% is determined to be 2.23 MJ mol^{-1}, while further energy increase does not enhance NO_2 concentration and may even lead to its reduction. Notably, the maximum point shifts toward a higher N_2 concentration at lower molar energy input (1.21 MJ mol^{-1} and 1.41 MJ mol^{-1}). This phenomenon is attributed to the plasma approaching equilibrium between the rotational and vibrational temperatures (T_{rot} and T_{vib}), which results in increased energy losses and a subsequent decline in NO_2 concentration.

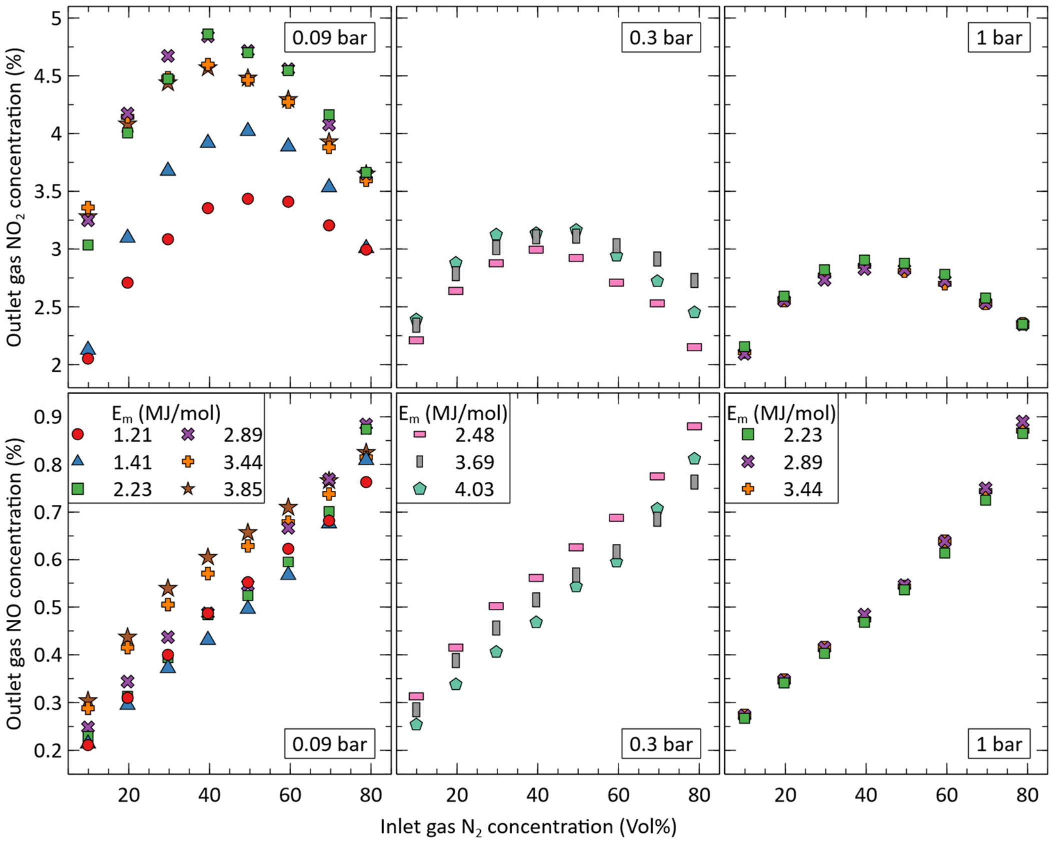

Fig. 2. NO and NO_2 concentration of the product gas at different pressures and molar energy inputs as a function of N_2 concentration.

NO_2 production tends to increase at 0.09 bar compared to a case measured at ambient pressure. The lowest energy inputs of 1.41 MJ mol^{-1} and 2.23 MJ mol^{-1} were achieved at 0.09 bar and 1 bar, respectively, under which maintaining the plasma was not possible. A saturation can be observed in NO_2 as a function of molar energy input; however, it was not possible to measure such a tendency in the case of ambient pressure, presumably attributed to plasma maintaining limits. Based on our measurements, the concentration of NO in the product gas exhibits a slight but discernible increase in sensitivity to the molar energy input under lower pressure conditions. Furthermore, the NO concentration in the product gas was observed to increase with higher inlet nitrogen concentrations.

Fig. 3 presents a comparison of the NO_x concentration in the product gas as a function of energy input, based on literature data. The most immediately striking observation is that the results reported by Azizov *et al*. deviate significantly from all other published experimental findings, and similar results have not yet been reproduced. Another notable exception is the work of Kelly and Bogaerts, whose results, although obtained under atmospheric pressure, align more closely with those typically observed under reduced pressure conditions, likely due to the optimization of flow dynamics. Excluding these exceptions, two distinct trends can be identified in Fig.3: one corresponding to reduced pressure conditions and the other to atmospheric pressure conditions. In both cases, the NO_x concentration in the product gas increases with rising energy input, with a further positive effect observed upon reducing the operating pressure.

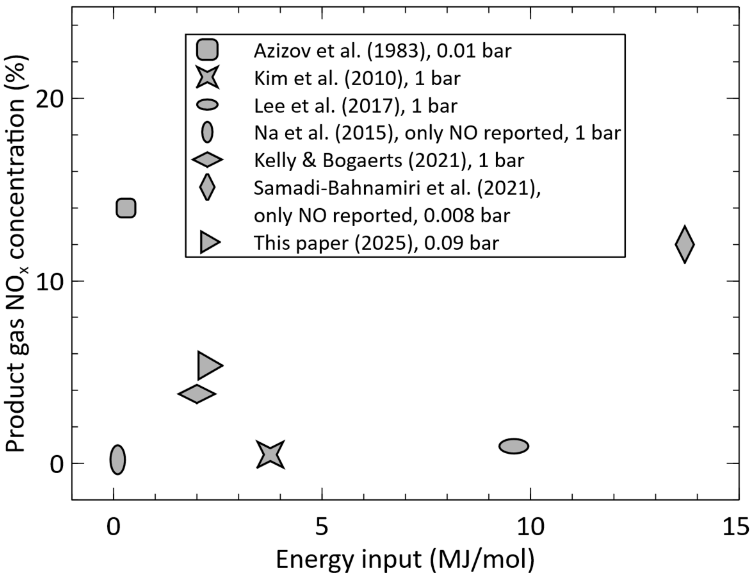

Fig. 3. Comparison of the NO_x concentration values in the product gas, which was found in the literature as the function of energy input.

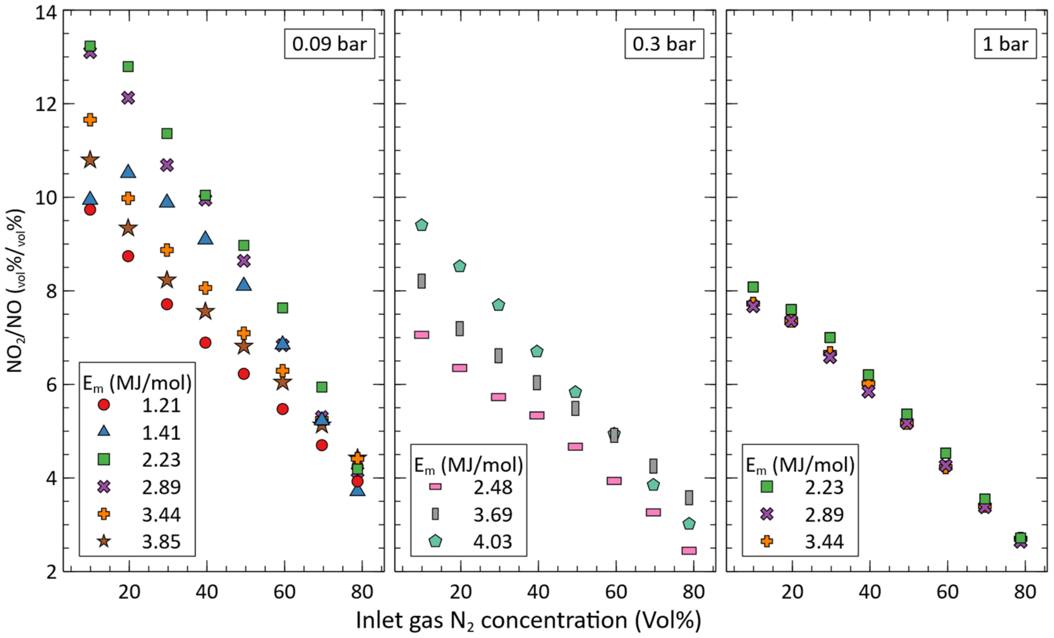

Fig. 4. NO_2/NO ratio in the product gas at different pressures and molar energy inputs as a function of the concentration of N_2 in the gas introduced to the plasma cavity.

Fig. 4 demonstrates that under nitrogen-deficient and reduced pressure conditions, the concentration of NO_2 in the product gas exceeds the concentration of NO by more than a factor of 13 and that the molar energy input significantly influences the NO_2/NO ratio. Conversely, as the system shifts toward nitrogen-rich conditions, the impact of the molar energy input appears to be diminished. Moreover, decreased operating pressure markedly favours NO_2 production over NO production. The results also indicate that the molar energy input does not affect the NO_2/NO ratio at ambient pressure.

Notably, the highest NO_2/NO ratio was recorded at the optimal energy input of 2.23 MJ mol^{-1}. This enhanced NO_2 production is primarily attributed to the pronounced non-equilibrium (non-thermal) characteristics of the plasma under reduced pressure conditions. Specifically, at lower pressures, the plasma gas remains relatively cooler, thus limiting the thermal decomposition of NO_2 back to NO. Furthermore, the observed decreasing trend in the NO_2/NO ratio under nitrogen-rich conditions can be explained by the availability of oxygen becoming limited. As oxygen concentration diminishes, the oxidative conversion of NO to NO_2 is constrained, thereby reducing the difference between NO_2 and NO formation rates, and consequently, a larger fraction of the fixed nitrogen remains as NO.

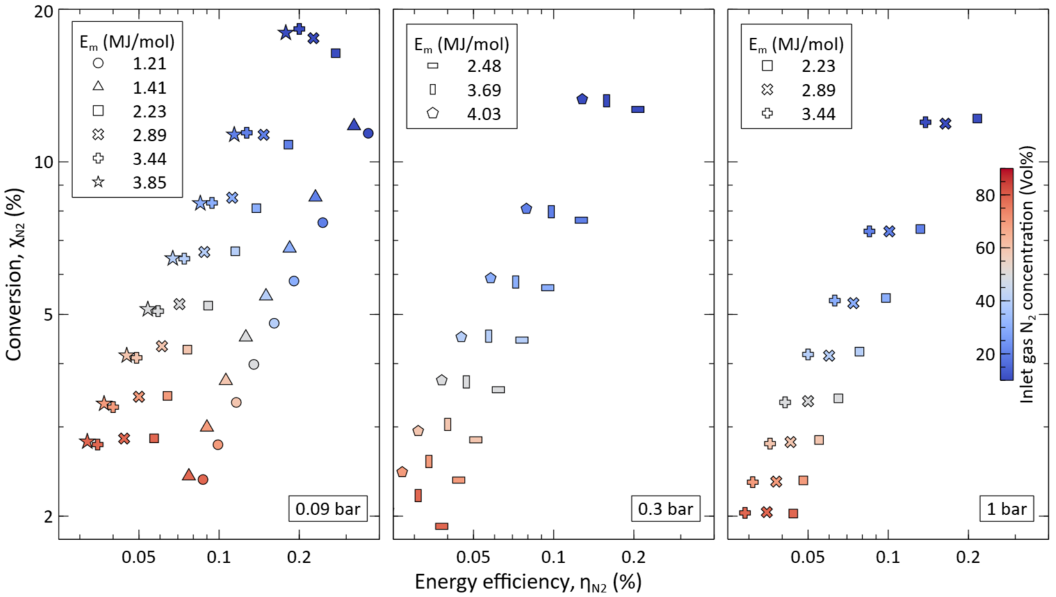

Fig. 5. The total nitrogen conversion vs. energy efficiency according to conversion at different pressures and molar energy inputs. The measurement point colors represent the nitrogen concentration of the input gas.

Fig. 5 presents the nitrogen conversion as a function of energy efficiency, revealing that these two parameters tend to increase simultaneously, in contrast to the commonly observed trade-off between them [17]. However, this observation does not contradict the literature, as the inlet gas nitrogen fraction decreases and is considered during the conversion calculation, i.e. the nitrogen conversion increases by lowering the nitrogen concentration in the input gas if the same NO_x yield can be achieved. Moreover, Fig. 5 shows that decreasing the molar energy input leads to enhanced energy efficiency, while concurrently reducing the conversion. The highest nitrogen conversion of 18.3% was measured at 3.44 MJ mol^{-1} under nitrogen-deficient conditions, while the maximum energy efficiency of 0.36% was attained at 1.21 MJ mol^{-1}, also under a nitrogen-deficient configuration. 2.23 MJ mol^{-1} seems to be an optimal choice as both the conversion and energy efficiency are high, i.e., $\chi \cdot \eta$ resulted in a maximum value. An increase in operating pressure was found to decrease both the conversion and the energy efficiency.

4. Conclusion

This work investigated a microwave-induced plasma as a novel route for nitrogen fixation using N_2/O_2 (air-based) feedstocks. The experiments confirmed that such plasmas can directly generate nitric oxide and nitrogen dioxide from the air. It was found that the NO_2 concentration in the product gas peaks at around 4.86 Vol%, which is attributed to the strong non-equilibrium behaviour of the plasma, with total NO_x around 5.35 Vol%, under optimal conditions of ~40 Vol% N_2 in the inlet, moderate specific energy input (~2.23 MJ mol^{-1}), and reduced pressure (0.09 bar). Under these conditions, a substantial portion of the nitrogen feed (up to 18.3% of input N_2) was converted into NO_x. However, it was also observed that attempting to push the system for higher yields or conversions incurs diminishing returns in efficiency. The highest nitrogen conversion was obtained at a relatively high specific energy input (~3.44 MJ mol^{-1}). In contrast, the best energy efficiency (0.36% of input energy captured as chemical energy in NO_x) occurred at a lower energy input (~1.21 MJ mol^{-1}) and in a nitrogen-deficient feed. This inverse relationship between conversion and energy efficiency reflects a fundamental trade-off in plasma-based nitrogen fixation. Additionally, operation at sub-atmospheric pressure was beneficial for NO_2 synthesis, indicating the importance of non-equilibrium plasma conditions (high vibrational excitation of N_2) for enhancing reaction efficiency, while at 1 bar, the process was less effective in producing NO_x. These results were obtained using a robust and straightforward reactor design, which is expected to facilitate future scaling in both size and capacity. These findings reveal that microwave-induced nitrogen-oxygen plasma can be utilized for nitrogen fixation and can be an alternative solution to recently used fossil-based methods. However, more research focusing on optimizing vibrational excitation should be targeted, with particular emphasis on preventing the plasma from approaching thermodynamic equilibrium, as maintaining strong non-equilibrium conditions can be critical for enhancing energy efficiency. In parallel, efforts should be directed toward exploring flow optimization strategies to simultaneously increase residence time and flow rate, as well as investigating pressure optimization and improvements in reactor design. All these developments should be pursued with a strong emphasis on scalability to ensure the viability of the technology for industrial applications.

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