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Short Communication

Hydrogen production from ammonia using a microwave plasma torch at atmospheric pressure

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ABSTRACT

An electrodeless, catalyst-free microwave (MW) plasma torch has been investigated to produce hydrogen (H_2) from ammonia (NH_3) dissociation, with NH_3 admixed in nitrogen up to 90 vol.% using a total inlet flow rate of 10 sLm at atmospheric pressure. Conversions beyond 99% are obtained for MW powers between 1.50 – 1.75 kW. The largest H_2 energy yield $\simeq 447\,L_{H_2}\,kWh^{-1}$, or lowest energy cost of $25\,kWh\,kg_{H_2}^{-1}$, is obtained for 90 vol.% [NH_3], which corresponds to outlet concentrations of H_2 and NH_3 around 69 vol.% and below 1 vol.%, respectively. These performances are highly promising for the application of plasma technology for the production of hydrogen from ammonia.

1. Introduction

Hydrogen (H_2) production from ammonia (NH_3), for example to be used to feed fuel cells for power generation [1,2], has received strong interest from the scientific community and industry. NH_3 is a carbon-free molecule, it has high volumetric energy density (around $10\,MJ\,L^{-1}$, comparable to compressed natural gas and methanol) as well as high hydrogen storage capacity (17.8 wt.%), and a worldwide infrastructure for ammonia transportation already exists [3,4]. Ammonia dissociation is an endothermic reaction ($2\,NH_3 \rightarrow N_2 + 3\,H_2$, $\Delta H = +91.88\,kJ\,mol^{-1}$ [5]), thermodynamically favoured at high temperatures and low pressures [6]. The thermal decomposition of ammonia is enhanced in presence of catalysts, the most active of which are ruthenium-based [7,8]. Despite its high activity for ammonia dissociation, ruthenium is an expensive metal, which requires the need to develop alternative, cheaper catalysts that enable complete dissociation at low-to-moderate temperatures ($\simeq 650$ K) [9,10]. Complete conversion of pure amonia flowing through a catalytic reactor would result in 75 vol.% of H_2 in the outlet stream, which makes the use of downstream separation/purification steps necessary. An example where high-purity H_2 streams (above 99.9 vol.%) from NH_3 cracking are achieved is with catalytic membrane reactors, in which palladium-based membranes allow the in-situ extraction of H_2 by permeation [11,12].

Plasma conversion technology, owing to its inherent compatibility with intermittent electricity, highly reactive environment, tunable selectivity, and wide range of gas temperatures can be a promising route to produce H_2 from NH_3. Non-thermal dielectric barrier discharges, due to the acceptable surface temperatures, allow increasing conversions by placing the plasma in contact with a catalyst at atmospheric pressure. For instance, Wang et al. reported an increase in conversion from around 7.8% with plasma alone to 99.9% using an iron-based catalyst in presence of plasma at 683 K [13]. Rotating gliding arcs, which are discharges that favour gas temperatures of several thousand kelvin, have been reported to achieve conversions of 80% without catalyst, albeit with large uncertainties due to a significant erosion of the electrode used in this plasma process [14]. Microwave (MW) plasmas are sources that also yield high gas temperatures with relatively high gas throughputs and in principle do not require electrodes. MW plasma torches, which have been mainly studied for the endothermic reaction of CO_2 dissociation by different groups, yield thermal plasmas at atmospheric pressure with gas temperatures at around 6000 K [15–18]. These sources enable large conversions for CO_2 and their relative flexibility and ease of operation motivate its use towards other endothermic reactions such as NH_3 cracking. Recently, a microwave plasma torch has been studied for the dissociation of ammonia admixed with nitrogen for which gas temperatures between 5000 and 6000 K and conversions of 65 – 85% for relatively low ammonia concentrations in the feed gas (17 to 42 vol.% NH_3/N_2) are reported [19].

This short communication aims at reporting very large conversions beyond 99% obtained with a microwave plasma torch at atmospheric pressure without catalyst and with gas mixtures up to 90 vol.% NH_3 in N_2.

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2. Experimental

Fig. 1 displays the experimental setup used in this work. The microwave plasma torch consists of four tangential gas inlets at the bottom (inner diameter 1.5 mm), a coaxial (pin) and cylindrical resonators. The tangential inlets produce a gas flow that swirls inside a quartz tube (140 mm long, 30 mm wide, 2 mm wall thickness), which is sealed on the bottom to the resonator and connected on the top to a water-cooled stainless steel piece by O-rings. The plasma is ignited by coupling 2.45 GHz, magnetron-generated microwaves through a three-stub tuner (SAIREM, GMP G4 60K, 6 kW of nominal power) and the hot effluent gas is cooled down through a 2 m long heat exchanger. The cold gas is sampled to a quadrupole mass spectrometer (MS, Pfeiffer Vacuum, QMG 220 PrismaPlus) for gas composition analysis, calibrated for NH_3, N_2 and H_2 following the procedure described in [20]. The whole setup is connected to a vacuum pump (Leybold, TRIVAC D16B) to ensure safe operation at quasi-atmospheric pressure ($\simeq$ 940 mbar), which is equivalent to operation at atmopsheric pressure as demonstrated in a previous work [21]. The base pressure of the entire setup is $\leq 10^{-1}$ mbar and the pressure inside the reactor (measured by Pfeiffer Vacuum, PCR 280) is regulated by a bellows valve. Nitrogen (5.0 purity, Air Liquide) and ammonia (5.0 purity, Air Liquide) are injected into the reactor by means of mass-flow controllers (Brooks Instrument, GF40). With the setup used in this work, the discharge is first initiated at around 100 mbar with pure N_2, and NH_3 is injected at elevated pressures until the desired composition is reached. The pure N_2 plasma is characterized by a pink column, which is characteristic of the intense N_2^+ first negative system emission in these types of sources [22]. The visible column shrinks with the addition of NH_3 and at large [NH_3] a yellow glow forms around it and extends beyond the top of the resonator (Fig. 1b). This glow is attributed to the emission of NH_2^* [19]. The experiments are carried out at $\simeq$ 940 mbar and with a total inlet flow rate of $F_{\text{total,in}} = 10$ sLm (sLm: standard Liter per minute at 273.15 K, 1013 mbar). With the increase of [NH_3] in the plasma, a larger power is required to sustain it. For 10 – 90 vol.% [NH_3], the net MW plasma power (given by $P_{\text{MW,net}} = P_{\text{MW,forward}} - P_{\text{MW,reflected}}$) was varied between 1.0 and 1.75 kW. The only molecules detected in the effluent are NH_3, H_2 and N_2 and their concentrations are determined by averaging $\simeq$ 15 min of constant MS current signals and the standard deviation used for their uncertainty.

To quantify the performance of the MW plasma torch, two key parameters, the NH_3 conversion χ_{NH_3} defined by Eq. (1) and the H_2 energy yield Y_{H_2} in L_{H_2} kWh^{-1} given by (2), are used. $[NH_3]_{in}$ and $[NH_3]_{out}$ are the ammonia concentrations at the inlet and outlet, respectively. $F_{H_{2,out}}$ is the total hydrogen outflow rate in sLm, $[H_2]_{out}$ is the outlet concentration of H_2 and $F_{\text{total,out}}$ is the total outflow rate, calculated by: $F_{\text{total,out}} = F_{N_2,in} + (1 + \chi_{NH_3})F_{NH_3,in}$, with $F_{N_2,in}$ and $F_{NH_3,in}$ being the total inlet flow rates of N_2 and NH_3 in sLm, respectively. Note that for $\chi_{NH_3} = 1$, $F_{\text{total,out}} = F_{N_2,in} + 2F_{NH_3,in}$ which accounts for gas expansion upon dissociation of 1 mol of NH_3 into 0.5 mol of N_2 and 1.5 mol of H_2. The uncertainties for χ_{NH_3} and Y_{H_2} are determined by propagating the uncertainties for the concentrations and flow rates.

$$\chi_{NH_3} = \frac{1 - \frac{[NH_3]_{out}}{[NH_3]_{in}}}{1 + \frac{[NH_3]_{out}}{[NH_3]_{in}}} \quad (1)$$

$$Y_{H_2} = \frac{F_{H_{2,out}}}{P_{\text{MW,net}}} \times 60 = [H_2]_{out} \frac{F_{\text{total,out}}}{P_{\text{MW,net}}} \times 60 \quad (2)$$

3. Results and discussion

Conversions above 99% have been achieved for 50–80 vol.% NH_3 in N_2 with an electrodeless and catalyst-free microwave plasma torch, as displayed in Fig. 2a. With the increase of the ammonia concentration in the feed gas, a larger MW power is required to promote high conversions. A clear example of this is the drop in conversion from 98.9% to

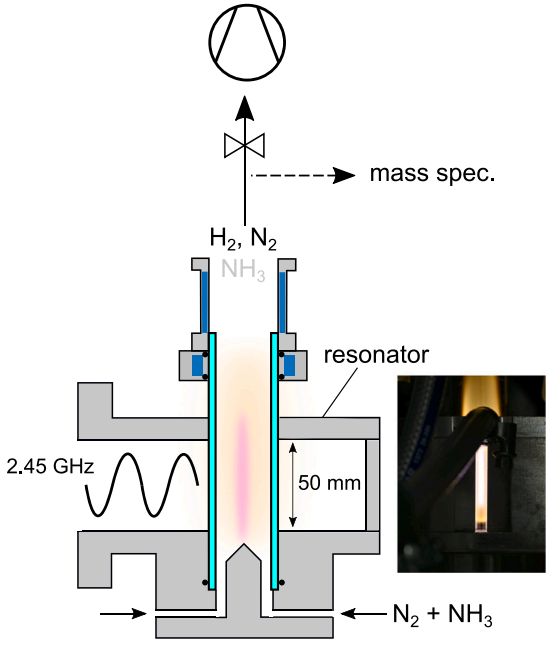

Fig. 1. Experimental setup (a.) and photo of the discharge with 40 vol.% NH_3 in N_2 with $P_{\text{net}} = 1.00$ kW (b.).

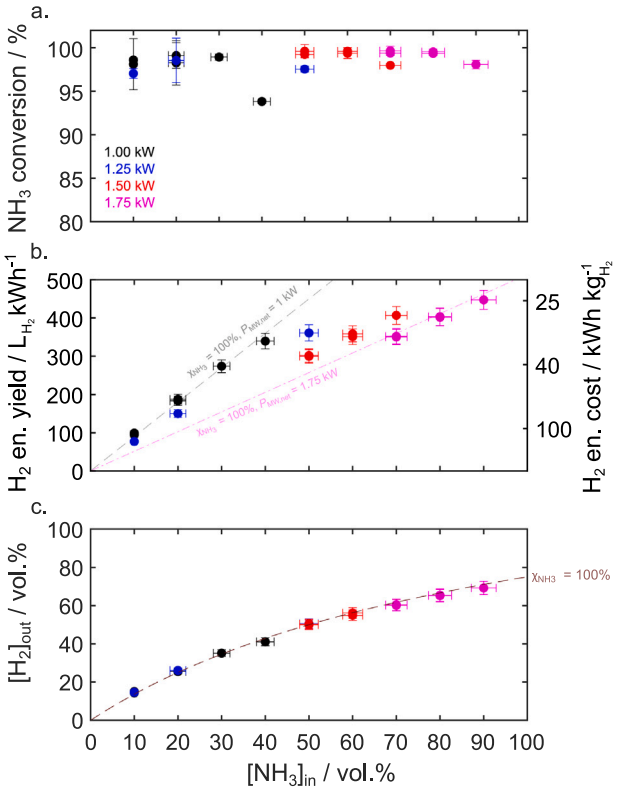

Fig. 2. Ammonia conversion (a.), H_2 energy yield (b.) and $[H_2]_{out}$ (c.) as a function of the ammonia inlet concentration in nitrogen. Black circles: $P_{\text{MW,net}} = 1.00$ kW; blue circles: $P_{\text{MW,net}} = 1.25$ kW; red circles $P_{\text{MW,net}} = 1.50$ kW; magenta circles: $P_{\text{MW,net}} = 1.75$ kW. Total flow rate: $F_{\text{total,out}} = 10$ sLm. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

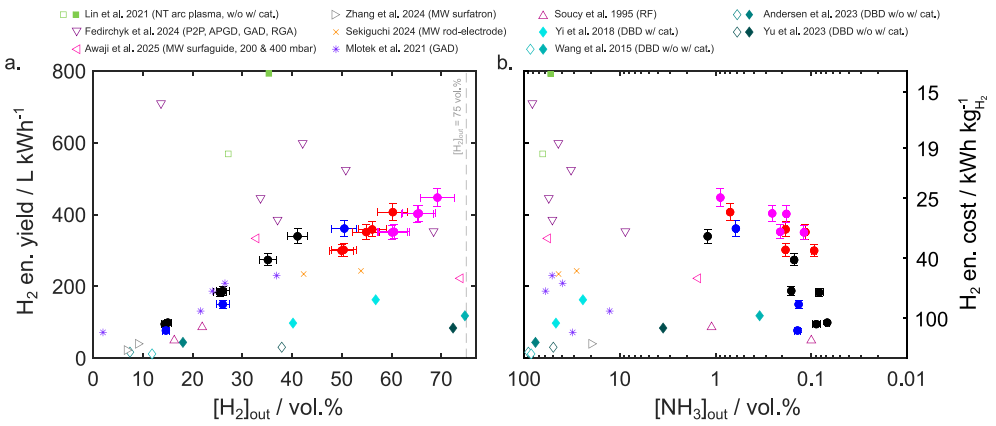

Fig. 3. H_2 energy yield as a function $[H_2]_{out}$ (a.) and $[NH_3]_{out}$ (b.) compared with literature data: Lin et al. [23], Fedirchyk et al. [14], Awaji et al. [24], Zhang et al. [25], Sekiguchi et al. [26], Mlotek et al. [27], Soucy et al. [28], Yi et al. [29], Wang et al. [30], Andersen et al. [31], Yu et al. [32]. P2P: pin-to-pin, APGD: atmospheric pressure glow discharge, GAD: gliding arc, RGA: rotating gliding arc, DBD: dielectric barrier discharge. Unless stated otherwise, all data points were obtained at atmospheric pressure. The circles correspond to the values presented in Fig. 2. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

93.8% when increasing the ammonia content from 30 to 40 vol.% with 1 kW. With larger ammonia concentrations, MW powers of 1.50 kW and 1.75 kW enable conversions above 99%. The decrease in conversion from 99.5% to 98% with the increase of $[NH_3]$ from 80 to 90 vol.% suggests that powers above 1.75 kW are needed to maintain higher conversions. The large conversions translate into H_2 energy yields close to the theoretical maximum at a given power. This can be appreciated in Fig. 2b, in which the H_2 yields obtained with 1.0 kW and 1.75 kW closely follow the values for 100% dissociation and the same applies for the other microwave powers. In general, an increase in $[NH_3]_{in}$ allows an increase in energy yield. However, note that, although an increase in power results in larger conversions, it can also lead to a reduction of the energy yield, as for example for 50 and 70 vol.% $[NH_3]_{in}$. The largest energy yield $\simeq$ 447 L kWh^{-1} is obtained at 90 vol.% $[NH_3]_{in}$, corresponding to an energy cost of 25 kWh $kg_{H_2}^{-1}$. The concentration of H_2 at the outlet of reactor must be considered as well and it can be appreciated in Fig. 2c that the $[H_2]_{out}$ values lie close to the maximum values that can be achieved for each gas composition. The largest $[H_2]_{out}$ is around 69 vol.%, which is remarkable considering that the highest possible concentration corresponding to 100% dissociation with pure NH_3 is 75 vol.% $[H_2]_{out}$.

The results displayed in Fig. 2a suggest that the amount of dissociated ammonia is dependent on the energy delivered per ammonia molecule, or specific energy input. However, the increase of the net MW power can also have a direct impact on the conversion, for instance by increasing the gas temperature in the effluent, which contributes to further dissociation of NH_3. Such a direct impact between the microwave power and gas effluent temperatures has been previously reported for CO_2 plasmas using this plasma torch [33,34]. Moreover, the increase in MW power is expected to yield larger electron densities in the plasma, which may enable further dissociation of NH_3 by electron-impact dissociation. Indeed, Niu et al. reported an increase in emission of excited N_2^+ and NH from the plasma core with the increase of MW plasma power [19]. Further insights about the plasma chemistry can be gained with optical emission spectroscopy, which will be used in the future to investigate gas temperatures and electron densities under relevant conditions. The limitation on the maximum $[NH_3]$ concentration of 90 vol.% and maximum power of 1.75 kW is imposed by the heat management of the current plasma torch resonator. A similar limitation was encountered in plasma conversion of CO_2 molecule, which has been resolved by using a reverse vortex and fast cooling of the effluent gas [17]. Similar approaches focused on the heat management are envisaged as a next step towards removing the limits on the maximum ammonia concentration and microwave power.

Contrary to its impact on the conversion of CO_2, fast cooling is not expected to affect ammonia decomposition, whose stable dissociation products (mainly N_2, with its triple bond and dissociation energy of 945 kJ mol^{-1} [35]) are very unlikely to react back to form NH_3 at atmospheric pressure. As discussed by Fedirchyk et al. gas quenching in ammonia plasmas will likely not improve conversions [14].

For the application of plasmas for hydrogen production it is most relevant to consider the energy used to produce a given amount of H_2 (i.e. the H_2 energy yield) and the outlet concentrations of H_2 and NH_3. While high H_2 purities ($\geq$ 99.97 vol.%) are required for fuel cells in road vehicles [36], the presence of NH_3 in the feed gas at concentrations as low as 0.1 ppm has a negative effect on fuel cell operation [1]. Consequently, a downstream separation/purification system is required and its size is directly impacted by the gas composition at the outlet of the reactor. It should be pointed out that N_2 is also an undesired impurity, but, apart from diluting the incoming hydrogen, it does not have a degradating effect [37]. The energy yields obtained in this work are plotted in Fig. 3 as a function of both $[H_2]_{out}$ and $[NH_3]_{out}$ and compared to results reported in the literature (note the inverted x-scale in Fig. 3b). Large energy yields as high as 800 L_{H_2} kWh^{-1} have been reported by Lin et al. using a non-thermal arc plasma with a NiO/Al_2O_3 catalyst [23], however with only 35 vol.% H_2 at the outlet. Another interesting example is the H_2 energy yield of 524 L_{H_2} kWh^{-1} reported by Fedirchyk et al. using a gliding arc plasma [14], albeit with an outlet stream containing $[H_2]_{out}$ = 51 vol.% and $[NH_3]_{out}$ = 32 vol.%. Overall, the results reported so far seem to suggest a trade-off between the H_2 energy yield and $[NH_3]_{out}$ (Fig. 3b), while the MW plasma torch enables relatively large yields for $[NH_3]_{out} \lesssim$ 1 vol.%.

Energy costs for H_2 production from NH_3 cracking using thermocatalytic processes lie in the range of 6 – 16 kWh $kg_{H_2}^{-1}$ [38] and around 9 kWh $kg_{H_2}^{-1}$ for electrochemical conversion [39], placing these technologies closer to the thermodynamic limit of 4.2 kWh $kg_{H_2}^{-1}$. However, the results presented here demonstrate that reduction in energy costs by further increase of NH_3 flow rates at increased MW powers should be expected. Further improvements in the process efficiency could be achieved by implementation of heat recovery strategies. By eliminating the need of catalysts and electrodes, the MW plasma torch presents a promising advantage for sustained, long-term use.

4. Conclusions

A microwave-drive plasma torch operated without electrodes or catalyst materials has been studied for the decomposition of NH_3 mixed in N_2 up to 90 vol.%. Very high conversions exceeding 99% have been

obtained for 50 – 80 vol.% of ammonia in the feed gas. At 90 vol.% NH_3 the conversion slightly decreases to 98% for a MW power of 1.75 kW, corresponding to a hydrogen yield of $\simeq 447\,L_{H_2}\,kWh^{-1}$ or an energy cost of $25\,kWh\,kg_{H_2}^{-1}$, and outlet concentrations of 69 vol.% H_2 and $\lesssim 1$ vol.% NH_3. Future work will include gaining insights into the plasma chemistry by spectroscopic methods and improvements to the reactor design to cope with the high gas effluent temperatures.

CRediT authorship contribution statement

R. Antunes: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **A. Meindl:** Writing – review & editing, Investigation. **C. Kranig:** Writing – review & editing, Investigation. **A. Hecimovic:** Writing – review & editing, Resources, Project administration, Funding acquisition. **U. Fantz:** Writing – review & editing, Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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