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In Situ Investigation of Water Splitting Mechanisms in Non-Thermal Plasma

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Abstract

Understanding complex reaction pathways is essential for efficient generation of energy carriers, including in hydrogen production and waste-to-energy processes. This work presents an *in situ* diagnostic framework for plasma-assisted water splitting, quantitatively monitoring species concentrations and temperatures, including for radicals and intermediates. Optical emission spectroscopy, online mass spectrometry, and modeling together characterized capacitively coupled Ar/N₂/H₂O non-equilibrium plasma, with optimal hydrogen production conditions of 100 SCCM Ar, 0.01 mL/min water injection, and 100–150 W plasma power. Reaction pathway analysis revealed power-dependent H₂ production routes, shifting from electron-impact dissociation of H₂O toward dissociative ionization of H₂O followed by charge-transfer with increasing power. At 150 W, water conversion reached 56.8% with an energy yield of 0.254 gH₂/kWh, while 100 W achieved comparable conversion (54.4%) and higher energy yield (0.365 gH₂/kWh). *In situ* spectra revealed strongly non-equilibrium behavior: OH exhibited two rotational populations (~650 K for intermediate *J* and ~7500 K for high *J* levels), N₂ a vibrational temperature of ~5500 K, and H₂ remained near room temperature. These results shed light on species-dependent energy transfer, linking operating conditions, radical chemistry, and hydrogen production. The methodology and insights for fundamental plasma reactive intermediates should extend to more complex feedstocks, including plasma-assisted waste valorization.

Introduction

Waste-to-energy strategies have the potential for enormous impact in environmental chemistry by simultaneously addressing both waste accumulation and the growing demand for sustainable energy. However, current treatment approaches are often limited by secondary pollution, low efficiency or high energy demands, restricting large-scale applicability [1-6]. These challenges highlight the need for more efficient conversion strategies, particularly when managing complex waste streams. While converting waste into hydrogen represents a promising approach towards clean energy carrier production [7-8] current technologies struggle with the inherent moisture present in real-world waste streams. Rather than energy-intensive pre-drying, utilizing water as a co-reactant offers a more practical alternative. Consequently, understanding the foundational plasma chemistry of water to hydrogen as a model system en route to co-converting water and waste is vital for optimizing energy yields.

Cold plasma integrated with pyrolysis offers a potential pathway to energy-efficient waste treatment, leveraging low power inputs to generate high densities of reactive radicals and ions [9]. Feedstock humidity produces hydroxyl (OH) radicals, which drive secondary reaction pathways [10] leading to molecular hydrogen (H₂) formation [11-13], a carbon-free green energy carrier [14-15]. However, the non-equilibrium nature of cold plasma introduces complex reaction mechanisms that complicate optimization.

Water splitting provides an ideal model system because of its relatively simple reaction network and relevance to moisture-containing waste streams. While plasma-driven hydrogen production has been widely reported [11-12, 16-19], intermediate species, reaction pathways, and *in situ* temperatures remain poorly understood. The goal of this study is therefore to investigate the water-splitting reaction in a non-thermal plasma to elucidate its reaction mechanism, primarily by characterizing reactive intermediates *in situ*. This work builds on our previous methodologies for species identification and temperature determination in Ar, N₂, and Ar/N₂ plasmas [20-21].

To detect intermediates and products, we combine *in situ* optical emission spectroscopy (OES) with online mass spectrometry. Absolute species concentrations obtained from calibrated mass spectrometry are compared with species-density plasma modeling to assess reaction pathways. Water-to-hydrogen conversion and energy efficiency are evaluated under different operating conditions. Although the efficiencies remain below those of dedicated water electrolysis, they demonstrate high conversion and competitive energy efficiency compared with other non-thermal plasma systems, highlighting the potential of plasma-assisted waste treatment [22]. The resulting mechanistic insights provide a basis for extending this approach to alternative reactor designs and more complex waste-to-energy systems.

Methodology

Experimental

The experimental setup is based on a capacitively coupled plasma (CCP) reactor and workstation described previously [20-21]. The reactor was operated at pressures of 0.1-1.0 Torr and at applied powers ranging from 50 to 300 W. OES spectra were acquired at both low (FLAME-S, Ocean Optics, 200-1000 nm, 0.5 nm) and high resolution (Jobin-Yvon HR800, 2400 l/mm, 330 nm blaze and Synapse detector). Online gas analysis was performed using a quadrupole mass spectrometer residual gas analyzer (RGA) (Extorr XT300M, 70 eV electron impact energy) with 75 and 10 μ m sampling orifices. Liquid was introduced via a syringe pump (Chemyx Fusion 100x, 0.5 μ L–60 mL) with a 3 mL syringe and 12 cm Hamilton needle (7748-1) sealed by a 1/8" Vespel/graphite ferrule (Restek 20254) to an evaporation vessel maintained at atmospheric pressure by a downstream needle valve. To prevent condensation and ensure complete vaporization, the gas lines and evaporation vessel were maintained at 75 °C using a temperature controller and heating coil (Box-3216, HT-1-1.5, MRC) with thermal insulation. A schematic is shown in Fig. S1.

Simulations and modelling

Rotational and vibrational temperatures were determined by contour fitting of experimental spectra in PGOPHER [23], as in our recent studies [20-21]. For N₂, temperatures were extracted from the (0,0) and (1,0) bands of the second positive system (SPS) $C^3\Pi_u - B^3\Pi_g$. OH rotational temperatures were obtained from the (0,0) band of the ($A^2\Sigma^+ - X^2\Pi$) system using reported molecular constants [24-25]. Due to spectral congestion and overlap of spin-orbit components [26], selected sparse regions (308–310 nm and 304–305 nm) were fitted separately. H₂ rotational temperatures were estimated from the Fulcher- α system ($d^3\Pi_u - a^3\Sigma_g^+$) using the Q₃, Q₄, and Q₅ transitions with values from [27-28].

The plasma conditions and chemistry were simulated with a drift-diffusion model similar to that used previously [20-21] and described in detail in [29]. Details regarding data analysis of water splitting intermediates and products are given in the Supporting Information (SI) [Secs. S2-S3].

Results and discussion

Water splitting species identification

In situ optical diagnostics

The emission spectrum of the Ar plasma, with and without water vapor, was recorded to identify water-splitting products and elucidate reaction pathways (Fig S3). The spectrum (Figure 1) is dominated by OH emission, exhibiting $A-X(0,0)$ and $(1,0)$ vibronic bands [2], together with H_α and H_β Balmer lines confirming atomic hydrogen [30]. Weak H_2 Fulcher- α band emission [31], provides unambiguous identification of molecular hydrogen, supporting the quantitative mass spectrometry measurements. These key molecular water-splitting species (OH and H_2) were subsequently analyzed at higher resolution to determine their rotational and vibrational temperatures. Trace air is indicated by the N_2 SPS, while atomic oxygen emission appears at 844 nm in both cases [32] and weakly at 777 nm only with water. No O_2 emission from either the Schumann–Runge bands [33] or the atmospheric band [34] was detected, since its strongest transitions lie outside the spectrometer range or are masked by intense Ar emission.

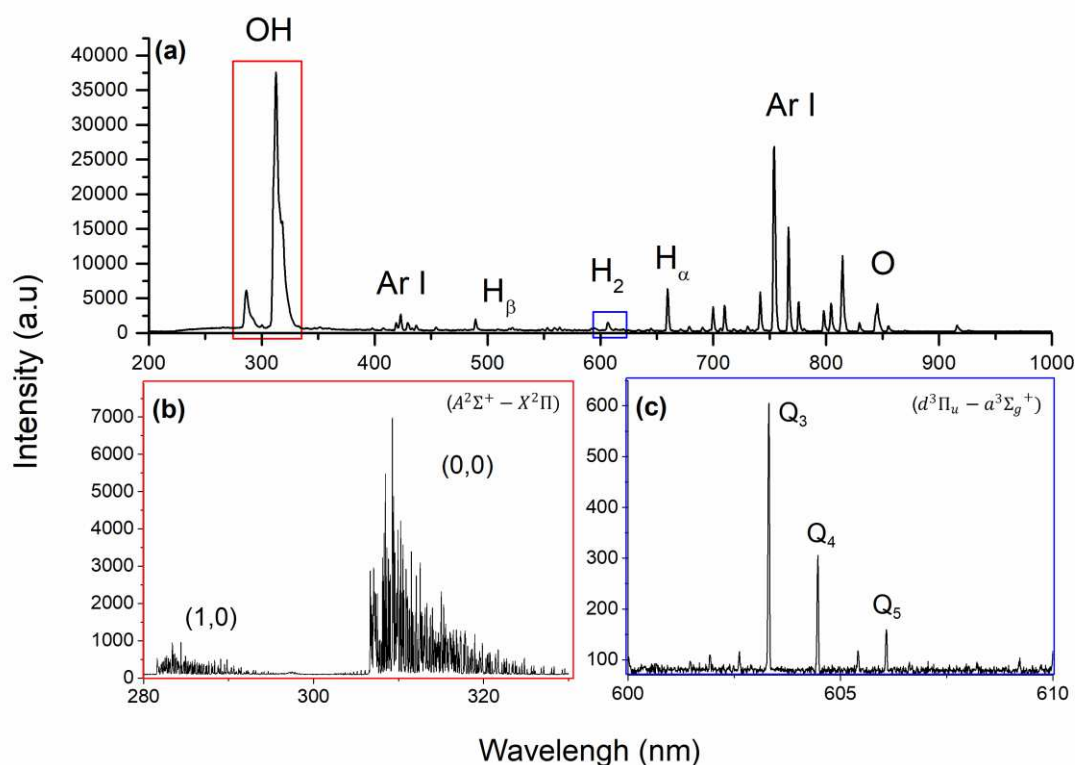


Figure 1. Emission spectra recorded at 100 SCCM Ar, 0.01 ml/min, 100 W (a) Low resolution overview (b,c) High resolution spectra of OH and H_2 , respectively.

Mass spectrometry measurements

OES and RGA provide complementary diagnostics: OES detects emitting species, whereas RGA measures all gas-phase species, albeit potentially with fragmentation via dissociative ionization. Together, these techniques enabled identification and quantification of all major water-splitting species: H_2O , O_2 , OH , H , H_2 .

The RGA results (Fig. S4) support the OES species identification. Although some signals arise from dissociative ionization at the RGA filament, we found that it was possible to extract parent species concentrations by a combination of calibration/control experiments with the use of known partial ionization cross-sections. The RGA thus enables convenient monitoring of species concentrations under changing reactor conditions.

Qualitative and quantitative trends

Effect of carrier gas

The reactor operating parameters were varied to maximize hydrogen production and investigate the underlying reaction mechanisms. Fig. S5 compares the effects of carrier gas and water injection rate on H_2 production, with the residual H_2O signal indicating unreacted water. Argon plasma produced significantly more hydrogen than nitrogen plasma, consistent with the emission spectra (Fig. S6). The lower OH emission in $\text{N}_2/\text{H}_2\text{O}$ plasmas is attributed to OH quenching and electron energy loss through N_2 vibrational excitation [21], and competing reaction pathways that suppress hydrogen formation. The highest hydrogen yield was obtained at a water injection rate of 0.01 mL min^{-1} , which was used for the subsequent experiments.

Effects of water injection rate and gas flow

The water injection rate was varied from 0.005 to $0.025 \text{ mL min}^{-1}$ under fixed operating conditions (Ar 100 SCCM, 100 W) to evaluate its effect on hydrogen production. As shown in Fig. 2(a), hydrogen production peaked at water injection rates of 0.01 – $0.015 \text{ mL min}^{-1}$. The close correspondence between hydrogen production and water consumption rates indicates that H_2 is produced predominantly by plasma-induced water dissociation. The model reproduces the measured H_2 density well but slightly underpredicts the H-atom density (Fig. 2b). The parabolic trend reflects a balance between the increasing reactant water concentration and its countervailing effect on plasma reactivity; at higher water content, additional electron energy-loss pathways through collisions with H_2O lower the electron temperature, thereby decreasing the efficiency of water dissociation and H_2 production. Compared with the water injection rate, the effect of Ar flow rate (i.e., pressure) was less pronounced under the intermediate water-content conditions investigated here, with maximum H_2 production observed at 100 SCCM Ar [29].

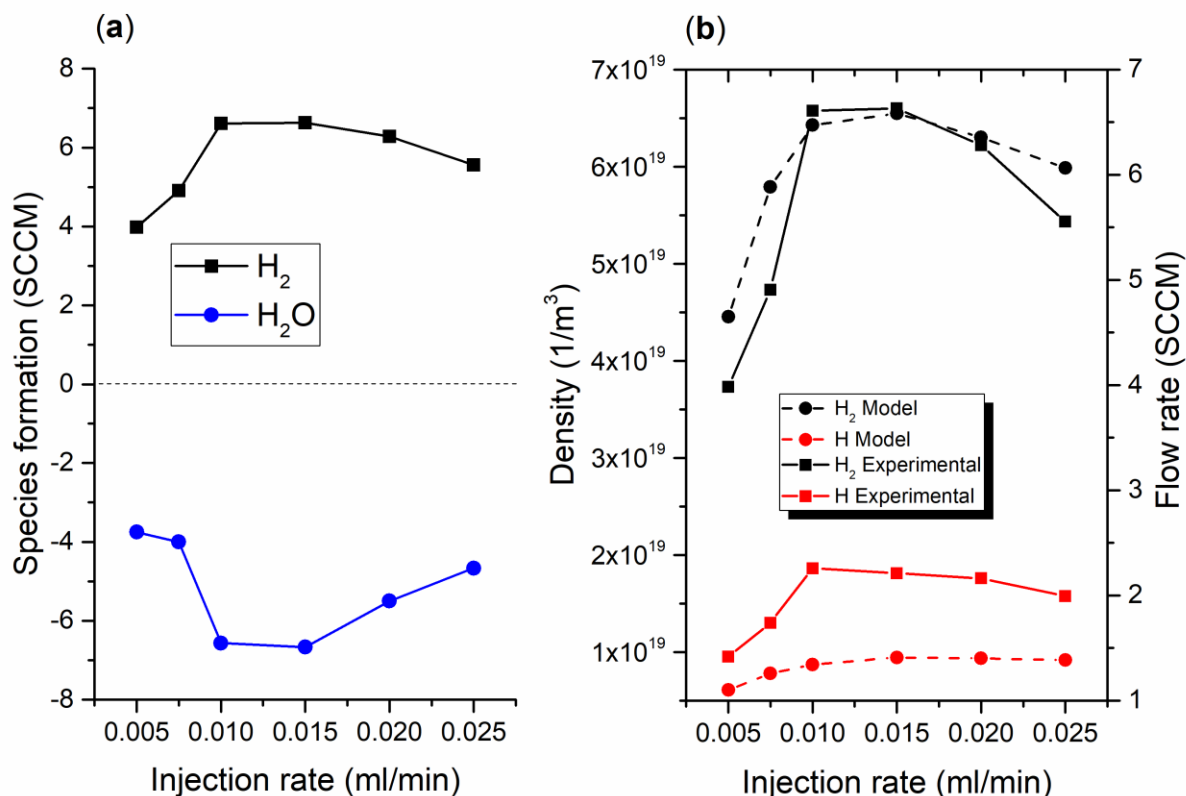


Fig. 2- (a) Hydrogen production and water consumption as a function of water injection rate. Water consumption was calculated from the difference between the injected and residual H_2O . Positive values represent H_2 production, whereas negative values represent H_2O consumption. (b) Measured H_2 and H densities as a function of water injection rate compared with the calculated density model.

Effect of plasma power

The plasma power was varied from 50 to 300 W under the optimized conditions (Ar 100 SCCM, water injection rate 0.01 mL min^{-1}). The measured species densities (solid lines) and model predictions (dashed lines) are shown in Fig. 3. The H_2 and H densities increase with power up to ~ 150 W and then remain constant or decrease slightly. O_2 exhibits a similar trend, peaking near 200 W, whereas OH and O show only a weak power dependence, with OH decreasing slightly at higher powers. Overall, the model reproduces the experimental trends for H_2 , OH , and O reasonably well, while the amount of H is underestimated and the measured O_2 density exceeds predictions, likely due to residual air.

The maximum H_2 concentration observed at 150 W appears to result from the power-dependent interplay between H_2 production and loss pathways, as revealed by reaction network analysis of the plasma simulations [29]. At low plasma power, H_2 production is dominated by electron-impact dissociation of H_2O . As the power increases, the contribution of this pathway diminishes, while an H_2^+ mediated pathway becomes increasingly significant, involving the formation of H_2^+ via electron-impact dissociative ionization of H_2O (or through ionization/charge transfer recycling of existing H_2), followed by charge transfer with H_2O to form H_2 . Regarding H_2 loss, electron-impact dissociation remains the dominant pathway throughout the investigated power range, although its relative contribution decreases with increasing power. In parallel, the ion-molecule reaction of H_2 with H_2^+ leading to H_3^+ formation becomes increasingly important; subsequent surface neutralization of H_3^+ regenerates a single H_2 and produces an additional H atom, such that the overall effect of this pathway is to dissociate H_2 . Further information regarding these reaction trends is shown in Table S1.

The radical chemistry provides additional insight into the water-splitting mechanism. Hydroxyl is produced primarily by electron-impact dissociation of H_2O , with an additional contribution from the reaction of H with HO_2 . Its consumption is dominated by OH recombination to form H_2O_2 , together with secondary reactions with H_2O_2 and HO_2 (Table S2). H atoms (ground state and electronically excited) are formed predominantly through electron-impact dissociative excitation of H_2O [29]. Their main loss pathways involve associative detachment with OH^- to form H_2O and release an electron, and reaction with HO_2 to produce OH radicals (Table S3). We note that the agreement between the model and experiment for H is less quantitative than for H_2 and OH , suggesting that one or more H formation or loss pathways may have their rates inaccurately represented or even be missing in the current plasma chemistry reaction mechanism.

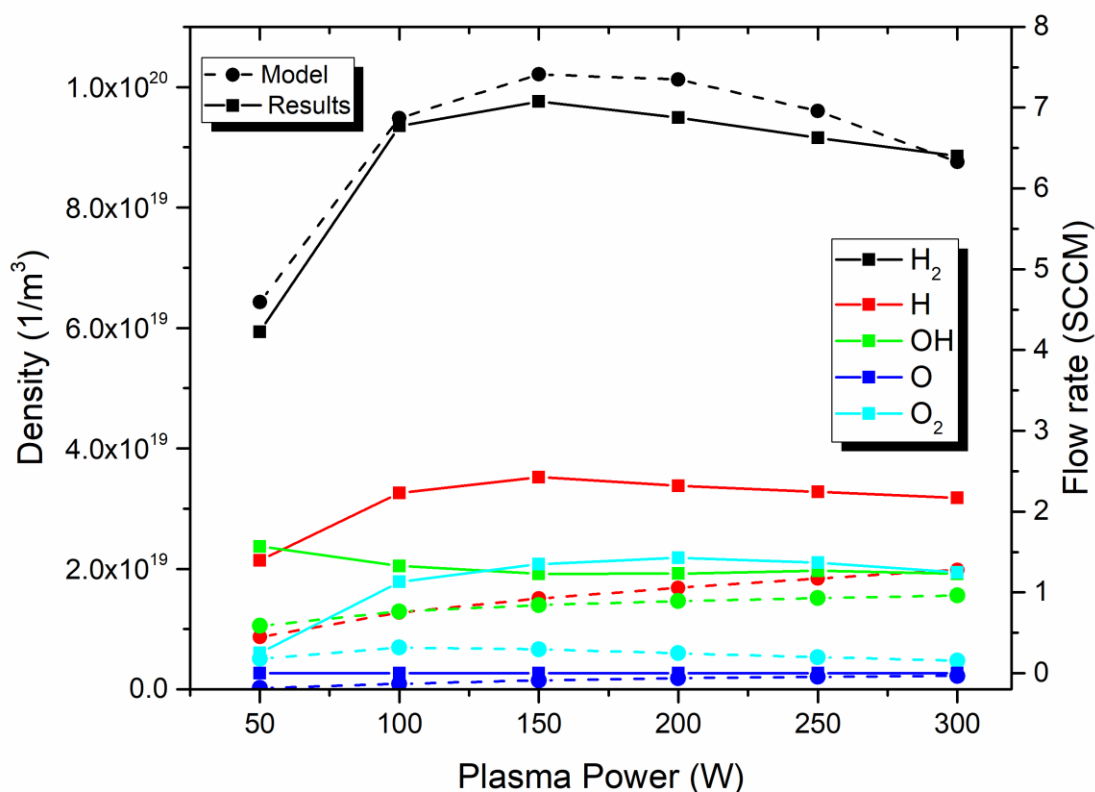


Fig. 3. Measured species flow rates (SCCM) and corresponding model-predicted species densities as a function of plasma power under optimized conditions (Ar 100 SCCM, water injection rate 0.01 mL min^{-1}). Solid lines represent experimental data; dashed lines represent model predictions.

Water conversion and energy yield

Water conversion and energy yield were evaluated under optimized conditions (Ar 100 SCCM, water injection rate 0.01 mL min^{-1}). At 150 W, water conversion reached 56.8%, corresponding to an energy yield of $0.254 \text{ g H}_2 \text{ kWh}^{-1}$. Since H_2 production changed only marginally between 100 and 150 W, operation at 100 W achieved a comparable conversion (54.4%) with a higher energy yield of $0.365 \text{ g H}_2 \text{ kWh}^{-1}$. This value is comparable with previous plasma-based water-splitting studies [12,35]. According to the performance metrics proposed in Cook *et al.*, our system achieved an energy efficiency of 1.45% and an H_2 mass yield of $1.1 \times 10^{-7} \text{ g J}^{-1}$ at 100 W, within the range of reported plasma water-splitting technologies. Although the reactor was designed for plasma-assisted waste treatment rather

than optimized hydrogen production, these results demonstrate the potential of cold plasma H₂ generation for competitive performance towards an added-value product.

In situ characterization of the plasma conditions

Additional mechanistic insight can be obtained by determining the temperatures of different species and molecular degrees of freedom. Since reaction pathways depend on how the available energy is partitioned within the reactants, resolving these temperatures provides a deeper understanding of species formation mechanisms.

Plasma temperatures were characterized using N₂ as an actinometer [36] following our previous approach [21]. A small N₂ admixture (2 SCCM) was added to the optimized Ar/H₂O plasma (100 SCCM Ar, 0.01 mL min⁻¹ H₂O), and rotational and vibrational temperatures determined as a function of plasma power. Figs. S7-S8 present the rotational temperatures (e.g. ~470 K) that slightly increase with power and are higher than in pure N₂ plasma (~355 K from 0-100 W), while the vibrational temperature was significantly lower (~5500 K) than in pure N₂ plasma (~6250 K from 0-100 W) and decreased with power, likely due to N₂/OH quenching [21]. The H₂ rotational temperature from the Fulcher- α (0,0) spectrum was surprisingly cold at approximately 300 K [37-39]. No significant power dependence was observed, and vibrational temperature could not be determined due to the absence of additional transitions.

The OH A-X (0,0) band was analyzed at plasma powers of 50–150 W. Previous studies have extracted OH rotational temperatures under various plasma conditions, including atmospheric pressure, plasma jets and reduced- pressure RF discharges [26, 40-41]. Owing to its pronounced non-equilibrium behavior [26], the intermediate- and high J rotational populations were fitted separately, yielding rotational temperatures of ~650 K and ~7500 K, respectively. Both components remained nearly constant over the investigated power range. The coexistence of these two populations indicates strongly non-Boltzmann behavior, with the hot component associated with dissociative OH formation from H₂O [42-43] and the colder component resulting from rotational relaxation. Together with the N₂ vibrational temperature (~5500 K) and the near-ambient H₂ rotational temperature (~300 K), these results demonstrate the strongly species-dependent, non-equilibrium energy distribution in the plasma. A summary of the extracted temperatures is presented in Fig. 4.

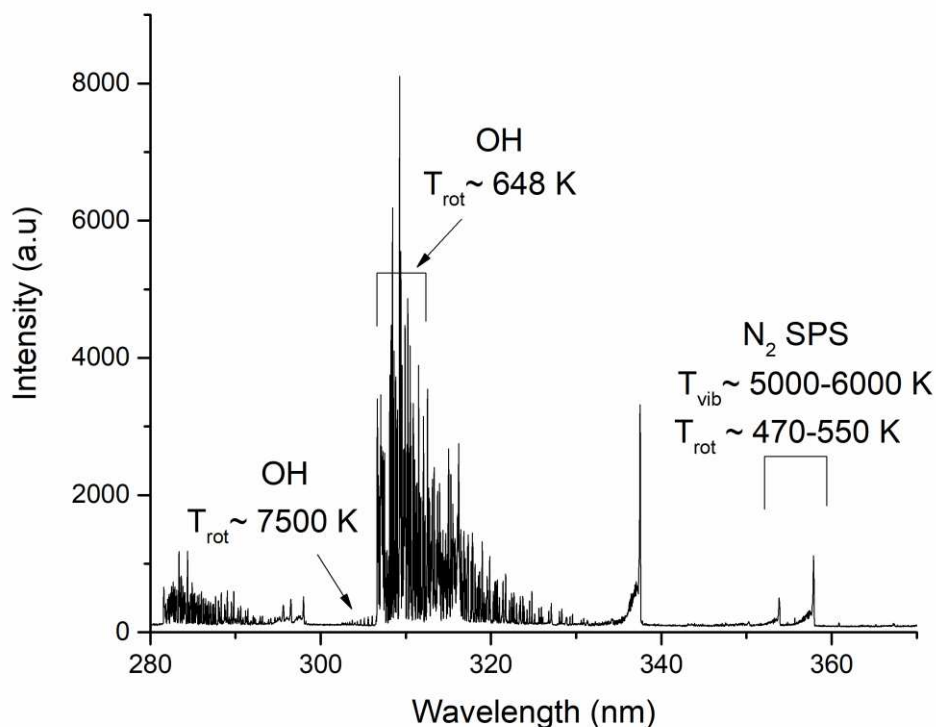


Fig. 4. Summary of extracted plasma temperatures: OH rotational temperatures (high- J and intermediate- J), and N_2 rotational (T_{rot}) and vibrational (T_{vib}) temperatures from the SPS using the (1,0) and (2,1) bands.

Conclusions

This work establishes a comprehensive *in situ* diagnostic framework for plasma-assisted water splitting by combining OES, mass spectrometry, and plasma modeling to correlate operating conditions with reaction pathways and hydrogen production. Quantitative H_2 and reactive intermediate monitoring, together with species-resolved temperature diagnostics, provides direct insight into plasma chemistry under non-equilibrium conditions.

The combined experimental and modeling results reveal a power-dependent redistribution of the dominant H_2 reaction pathways. At low power, H_2 formation is dominated by electron-impact dissociation of H_2O , whereas at higher power, H_2^+ mediated formation and recycling pathways become increasingly important. H_2 consumption remains dominated by electron-impact dissociation, while the contribution of the ion-molecule pathway leading to H_3^+ formation increases with power. These changes in the underlying reaction chemistry coincide with the maximum H_2 production observed near 150 W. The model also reproduces the behavior of OH and O reasonably well, while the discrepancy in the experimentally observed H power dependence highlights H-atom chemistry as an area requiring further refinement of the reaction mechanism.

A key finding is the simultaneous observation of distinct temperature population distributions across different molecular species and degrees of freedom. While H_2 remained near room temperature (~ 300 K), N_2 exhibited vibrational temperatures of ~ 5500 K, and OH displayed two rotational populations (~ 650 and ~ 7500 K), revealing pronounced species-dependent energy partitioning. These juxtapositions

provide mechanistic insight into radical formation and relaxation processes that highlight the strongly non-equilibrium nature of plasma-driven water dissociation.

Under optimized conditions, the reactor achieved a water conversion of 56.8% and an energy yield of 0.254 g H₂ kWh⁻¹, increasing to 0.365 g H₂ kWh⁻¹ at 100 W with only a minor reduction in conversion. Together, these results demonstrate how combining quantitative species diagnostics with reaction modeling can connect reactor performance to the underlying plasma chemistry. This framework can be extended to more complex plasma systems, particularly plasma-assisted waste valorization, where understanding reactive intermediates and reaction pathways is essential for improving process efficiency and environmental outcomes.

Author contributions

S.HL., A.K., and J.H.B. were the main contributors to the design and conception of the study. S.HL. conducted experiments, with technical assistance from A.L., and analyzed the data. S.A. performed the modeling. I.R. contributed to project management. The manuscript was written by S.HL., with assistance from A.K and J.H.B., and all authors read and approved the final manuscript.

Declarations

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Consent to participate: Non-applicable

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Availability of data: Non-applicable

Code availability: Non-applicable

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