

Hydrogen production by thermal water splitting using a thermal plasma

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ABSTRACT:

CEA has been working for more than 10 years in plasma technologies devoted to waste treatment: incineration, vitrification, gases and liquid treatment. Based on this experience, CEA experiments since several years an innovative route for hydrogen production by thermal water splitting, using a plasma as heat source. This new approach could be considered as an alternative to electrolysis for massive hydrogen production from water and electricity.

This paper presents a brief state of the art of water thermal plasmas, showing the temperatures and quench velocity ranges technologically achievable today. Thermodynamic properties of a water plasma are presented and discussed.

A kinetic computational model is presented, describing the behavior of splitted products during the quench in a plasma plume for various parameters, such as the quench rate.

The model results are compared to gas analysis in the plasma plume obtained with in-situ sampling probe. The plasma composition measurements are issued from an Optical Emission Spectroscopic method (OES). The prediction of 30 % H₂ recovery with a 10⁶ K.s⁻¹ quench rate has been verified.

A second experimentation has been performed: mass gas analysis, flowrate measurement and OES to study the "behavior" and species in underwater electrical arc stricken between graphite electrodes. With this quench, a synthesis gas was produced with a content 55 % of hydrogen.

KEYWORDS : hydrogen, water, plasma, thermal splitting.

1. Introduction

Hydrogen production from water dissociation is an original process to convert nuclear electricity into chemical vector. This method could be used in response to energy storage problem. Indeed, the total free energy of the compounds issued from dissociation, hydrogen and oxygen, is much higher than the one of the starting water molecule.

Various processes can be used to dissociate H₂O molecules without recombination. The most traditional of them is probably water electrolysis at low temperature; hydrogen and oxygen are released separately in the cathodic and anodic compartments. Application of thermochemical cycles to water decomposition has also been studied; however the development of a practical cycle is a difficult problem due to the great complexity of reaction kinetic, phase separation and corrosion phenomena.

It then appears very interesting to consider another approach of the problem, based on the purely thermal dissociation of water only above 3000 K. Such temperatures can be reached with thermal plasma. Gas separation from dissociation can be considered in two different ways. The first one consists in very high quench (above 10⁶ K.s⁻¹) below 800 K by either water or steam. The second one consists in extracting from reactional medium at high temperature one of the components, for example oxygen, by means of a semipermeable membrane. It is *a priori* possible to carry out water thermolysis at high temperature, using an oxygen semipermeable membrane, characterized by a dual conduction (ionic and electronic).

The aim of this paper is by means of calculations and experiments to point out the interest of this alternative method for massive hydrogen production of hydrogen from water and electricity. In the first part, is presented a brief background linked to water plasma torches. Then, the modelling approach is discussed. Finally, two different experimentations are described: one with water injection in a plasma jet, the other with an underwater electrical arc. The goals were to verify modelling accuracy and to identify ways of work and parameters for designing an industrial process.

2. Background

2.1 Thermal water splitting

Since the XIXth century, studies of hydrogen production by thermal dissociation of steam have been performed. A peak in research was observed reached after the "oil crises" at the end of the seventies but this effort was nearly stopped as soon as the oil barrel price decreased.

A plasma torch is an interesting mean to provide a considerable specific enthalpy to a medium made of either liquid or steam. In this case, the temperature can be set separately from the chemical composition of the plasma forming gas. Water plasma is a very reactive medium and the steam is a particularly economic plasma forming gas /MEILLOT E. 1988/. On factories, large amount of steam are not used.

2.2 Water plasma torch

Plasma generators technology with arc stabilized by water was first developed in Czechoslovakia /GROSS B. and al., 1968/. /KUGLER T. and al., 1972 to 1973/, /HENGARTNER K. 1975/ and /HRABOVSKY M. and al., 2001/. The commercial version of these torches (used for plasma spraying) have a nominal power of 200 kW.

That are interesting in this case : temperature and flow velocity charts measured in the plasma jet (cf. **Figure 1**) /HRABOVSKY M. and al., 2001/.

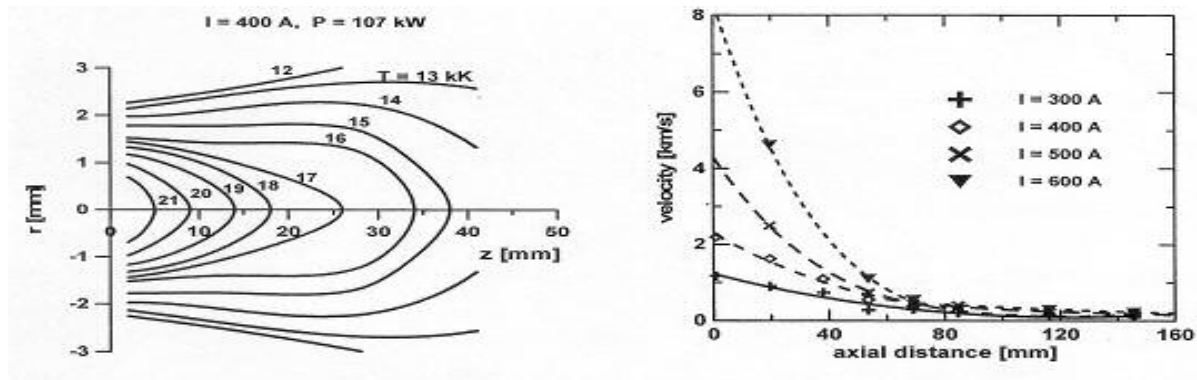


Figure 1: Field of temperature and speeds in the jet of torch from Institute of Plasma Physics of Prague

We can notice that the variation in temperature on the axis of the plasma jet is about $1000 \text{ K}\cdot\text{cm}^{-1}$; while the velocity on the axis is higher than $1000 \text{ m}\cdot\text{s}^{-1}$ leading to a quench rate about $10^8 \text{ K}\cdot\text{s}^{-1}$ that permits an efficient quench of dissociated hydrogen.

2.2 Steam plasma torch

Other plasma torches working with steam for material processing are known. For example, those developed by Plasma Chemistry Laboratory of Limoges University and the Institute of Heat and Mass Transfer of Minsk. Steam is used as plasma forming gas. The tungsten cathode is protected by a low flow of argon (or a not oxidizing gas), the steam being introduced into the arc downstream. The plasma starting gas being air; once the arc is established, it is gradually replaced by steam. With a DC current of 130 A, an argon flow of 30 L/min, the voltage of arc varies from 230 to 320 V when the steam flow varies from 1.3 to 2.5 g/s (2.5 g/s of water corresponds to 200 L/min). At 130 A, the power of the torch is thus around 40 kW with a thermal efficiency of about 70 % /MEILLOT E. and al., 1988/, /KNACK A.N. and MOSSE A.L., 2001/.

3. Modelling of the thermal dissociation of water

3.1 Thermodynamic modelling of the thermal dissociation of water

We assume the steam plasma as an ideal gas mixture at thermodynamic equilibrium. The equilibrium composition is calculated with the software ALEX /SANON A. and al., 1991/, using the free enthalpy minimization method under the constraints of mass conservation and electrical neutrality. Taking into account the experimental device used - a jet of argon plasma produced by a torch with non transferred arc submerged in water -, we present the chemical composition of a plasma argon/water (Ar, 80 % / H_2O , 20 %).

23 species are considered in the calculation : H_2O , H_2 , O_2 , H , O , OH , Ar , Ar^+ , H^+ , H^- , H_2^+ , H_2^- , H_2O_2 , H_3O^+ , HO_2 , O^+ , O^- , O_2^+ , O_2^- , O_3 , OH^+ , OH^- , e^- .

Influence of the temperature

Between 1500 K and 9000 K concentrations of some species never reach more than 10^{-4} moles. Only 11 major species are considered: H_2O , H_2 , O_2 , OH , H , O , Ar , H^+ , O^+ , e^- et Ar^+ .

Water thermal dissociation begins significantly only around 3000 K, to give the radicals and the molecules OH , H_2 , O_2 , H and O . From 4500 K all the chemical bonds are broken and only the radicals H and O are found.

Hydrogen production by water plasma pyrolysis is thus realizable but is it possible to preserve produced hydrogen during the plasma jet cooling? The radical recombination during the quench is studied in the following paragraph, devoted to the chemical kinetic modelling.

3.2 Chemical kinetics modelling of the dissociation products quench

The quench process allows to lead the radical recombination reactions. In the jet of plasma at high temperature take place reactions whose products are stable at ambient temperature.

The kinetic study is carried out thanks to the computer code C3GAZ /BARTHELEMY B. and al., 1999/. To perform the kinetic modelling from elementary processes and a temperature history, we have to identify:

- the starting composition,
- the elementary processes with their kinetics constants,
- the temperature history.

Concerning the temperature history, the maximum reached temperature is calculated from calorimetry balance and thermodynamic calculation. With an electrical power supply from an experimental plasma torch of 6.3 kW and 3 kW lost in the cooling circuit, we can determine an average enthalpic temperature of ~ 4000 K.

Under these experimental conditions, the temperature drop on the axis of the plasma from the torch nozzle is about 5 000 K on a distance of approximately 5 cm. The quench rate is $\sim 5 \cdot 10^7 \text{ K.s}^{-1}$. This order of magnitude is in agreement with /HRABOVSKY M. and al., 2001/.

The outputs, previously calculated by modelling and corresponding quench rate /LAFON C., 2003/, are given in **Table 1**. We can see that for the highest quench speed, the quantity of non recombined hydrogen becomes quantitative.

Speed of quench	Model output
10^6 K.s^{-1}	1,6 %
10^7 K.s^{-1}	7,6 %
10^8 K.s^{-1}	37 %
Table 1: Hydrogen recovery efficiency (Ambient temperature $[\text{H}_2]$ / High temperature $[\text{H}_2]$)	

4. Experimental approach

4.1 Plasma torch

In order to validate the previous theoretical forecasts, we carried out experimental tests.

The experimental device is presented on **Figure 2**. A plasma torch is placed on the vertical axis at the base of a water cooled metallic sealed reactor. The plasma-producing gas is argon or a mixture of argon and helium. Liquid water is injected into this plasma via 8 holes (diameter 0.5 mm) placed around the torch nozzle. The internal diameter and height of the reactor are respectively 40 cm and 60 cm. Three openings are arranged on the side walls. These openings receive quartz discs for the observation of the plasma jet or the diagnosis by OES (Optical Emission Spectroscopy), or allow the passage of an in-situ sampling probe for local measurements of hydrogen and oxygen concentrations.

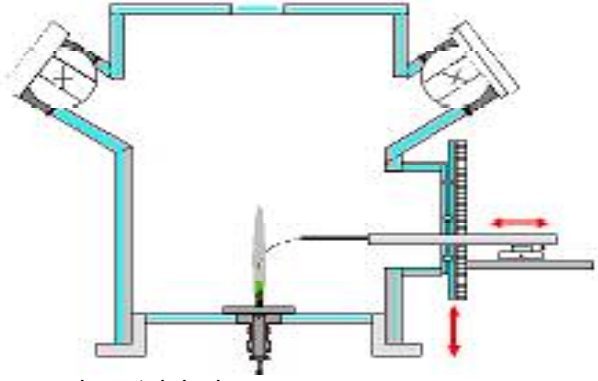
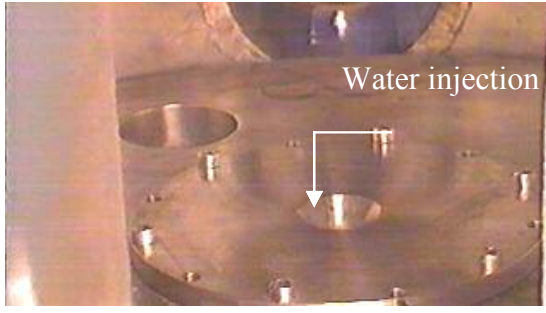


Figure 2: Diagram of the experimental device

Experimental set up

The device used is detailed in /LAFON C. and al., 2003/. We used a multichannel technique of analysis (OMA) including an optical bench, a monochromator, a matrix CCD detector. With this matrix detector, we access two dimensions simultaneously: one corresponds to a diameter of the jet of plasma, the other being the wavelength.

OES measurement technique

Relative emission intensity has been chosen as a tool for OES measurements of the plasma parameters. Plasma temperature has been evaluated using an intensity ratio of two atomic argon spectral lines. Spectral lines of hydrogen and oxygen atoms have been taken to find a concentration of the respective species in the plasma as compared to atomic argon by intensity ratios for corresponding atomic spectral lines. Common plasma diagnostics relying upon relative intensity suppose local measurements of the plasma emission coefficient, which must be actually used instead of intensity, especially at non-homogeneous plasma cases. According to our preliminary study, the jet under consideration is an inhomogeneous and unsteady plasma volume, which generally has no axial symmetry. So, the emission coefficient cannot be measured with a reasonable precision using common Abel inversion. That is why we have used the technique relying upon some approximations of the plasma volume spatial distribution and time behaviour /ERSHOV-PAVLOV E and al., 1999/. According to the approach, plasma temperature distribution in the volume along the observation direction can be presented as follows:

$$T(y) = T_0 [1 + (y/y_0)^\alpha]^{-1} \quad (1)$$

where α is a parameter accounting for a form of the temperature distribution, i.e. the plasma inhomogeneity rate. It makes possible to obtain for a spectral line in the plasma emission spectrum a rather simple relation between the emission coefficient of the plasma $\varepsilon(T_0)$ at the maximum temperature T_0 and the line total intensity I , recorded along y -direction:

$$I = \frac{2y_0}{\alpha} \mu^{1/\alpha} \varepsilon(T_0) \left(1 + \mu/\alpha\right) \Gamma(1/\alpha) \quad (2)$$

Here, $\mu = kT_0/E$, where E is energy of the upper level of the optical transition giving the line under consideration; Γ is Gamma-function.

For unsteady plasmas using the approach it was shown in /ERSHOV-PAVLOV E. and al., 2001/, T_0 fluctuations around its mean value \bar{T}_0 can be chosen to present plasma instability supposing y_0 and α to be constant, as far as namely T_0 influences spectral line intensity more effectively. The following expressions have been found in /ERSHOV-PAVLOV E. and al., 2001/ for the mean intensity values of a spectral line at sinusoidal and sawtooth fluctuation modes, respectively:

$$\langle I_{Sn} \rangle \approx C \sqrt{\frac{2}{\pi}} y_0 \frac{\Gamma(1/\alpha)}{\alpha} \frac{1}{\sqrt{\sigma}} \frac{1}{\bar{T}_0} \mu^{1/\alpha+1/2} \left(1 + \frac{\mu}{\alpha}\right) \exp\left(-\frac{E}{kT_m}\right) \quad (3a)$$

$$\langle I_{Sw} \rangle \approx C y_0 \frac{\Gamma(1/\alpha)}{\alpha} \frac{1}{\sigma} \frac{1}{\bar{T}_0} \mu^{1/\alpha+1} \left(1 + \frac{\mu}{\alpha}\right) \exp\left(-\frac{E}{kT_m}\right) \quad (3b)$$

where $T_m = \bar{T}_0(1 + \sigma)$ is the highest value of the maximum temperature T_0 in the temperature distribution along y during the observation time.

Finally, from the Eqs (2), (3) the following expression can be found for the plasma temperature evaluation by relative intensity of two spectral lines (1, 2) [4]:

$$kT_m \approx (E_1 - E_2) [\ln(C_1/C_2) - \ln(I_1/I_2) - \Omega \ln(E_1/E_2)]^{-1} \quad (4)$$

where $T_m = \bar{T}_0(1 + \sigma)$ is the highest value of the maximum temperature T_0 in the temperature spatial distribution along the line of sight and during the observation time, $\sigma = \Delta T / \bar{T}_0$ is the fluctuation amplitude, is a common parameter combination, $C_{1,2} = (Ag/\lambda)_{1,2}$, for the lines due to transitions from the respective energy levels, $E_{1,2}$. In the temperature region under consideration the parameter $\mu \ll 1$, and the ratio $(1 + \alpha/\mu)_1 / (1 + \alpha/\mu)_2 \approx 1$ is taken when obtaining Eq. (4). The parameter $\Omega = 1/\alpha$ for stationary plasmas ($T_m = T_0$), $\Omega = 1/\alpha + 1/2$ and $\Omega = 1/\alpha + 1$ for plasmas with sinusoidal and sawtooth temperature fluctuations, respectively. Note, when the plasmas are homogeneous ($\alpha = \infty$) and stationary ones, $\Omega = 0$, and Eq. (4) gives a common Boltzmann equation for the plasma temperature measurements by relative intensity of spectral lines /RICHTER and al., 1968/.

Here we have used the Eq. (4) for plasma temperature measurements. We have supposed the plasma components are distributed homogeneously (their partial pressure is constant along the observation line and during the measurements time). It allowed replacing the ratio of the plasma emission coefficients with the relevant ratio of the directly measured intensity of spectral lines at the evaluation the components relative density N_a^1/N_a^2 :

$$\frac{N_a^1}{N_a^2} = \frac{I_{ki}^1}{I_{ki}^2} \left(\frac{E_k^1}{E_k^2} \right)^\Omega \left(\frac{\lambda_{ki} g_f}{A_{ki} g_k} \right)_1 \cdot \left(\frac{\lambda_{ki} g_f}{A_{ki} g_k} \right)_2^{-1} \exp \left(\frac{E_k^1 - E_k^2}{kT} \right) \quad (5)$$

where partition functions of the atoms are replaced with the statistical weights g_i of their ground levels. The atom density relative ratio evaluated using Eq. (5) is strongly influenced by the exponent, and spectral lines with closer upper energy levels $\Delta E_k \rightarrow 0$ have to be taken to minimise the influence. In such a way the ratio dependence on temperature can be practically eliminated.

OH spectrum simulation

Emission of OH radicals has been also used to learn the species distribution in the plasma. The band spectrum has been simulated due to transitions between $A^2\Sigma^+$ and $X^2\Pi_i$ electronic states. The resulting vibrational 0-0 band has a head at 306.1 nm and is degraded to the spectrum red side. The transition principal parameters, as well as the calculation algorithm have been taken from /DIEKE G.H. and al., 1962/.

Results and discussion

Distributions of the plasma parameters across the arc jet have been measured with a step of 0.25 mm at different distances z . The measurements have been made at 250 A arc current, at 30 and 4 NL/min flows of argon and helium, respectively, and at 0.1, 0.5 and 1 L/h of water introduction.

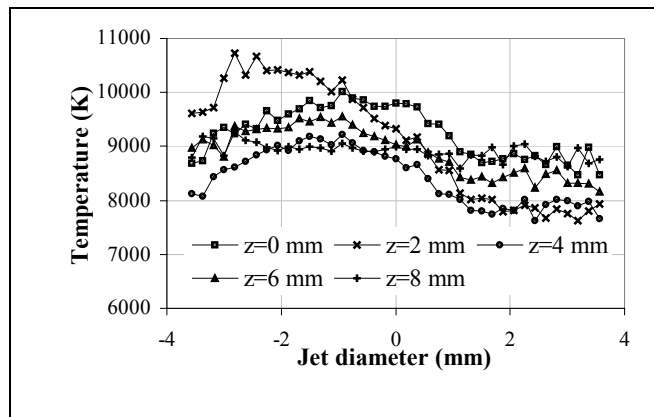


Figure 3: Temperature distribution in the plasma jet at 1 L/h of water introduction

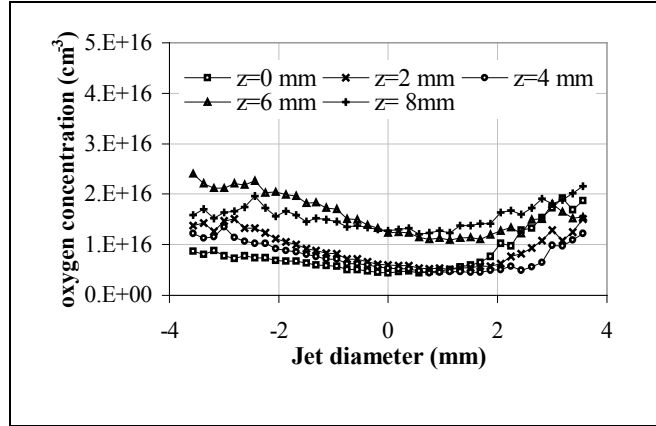


Figure 4: Oxygen density distribution in the plasma jet at 1 L/h of water introduction

The value $z=0$ has been chosen at the cone base, which corresponds to the distance of 9 mm from the torch nozzle.

Parameters of all lines chosen for the measurements have been taken from the NIST tables. Two Ar^* lines at $\lambda=714.7$ and 720.7 nm have been used for the plasma temperature evaluation. According to preliminary observation data, sawtooth fluctuations and $\alpha=2$ ($\Omega=3/2$) have been taken at the plasma temperature and relative composition evaluation. We estimated the uncertainty in the temperature values to be within 15%. For the studied region of 9-17 mm from the torch nozzle, temperature changes in 7000-10000 K interval (cf. **Figure 3**). Some temperature measurement results are presented in Figure 3, where the distributions are shown for the highest water content. Generally, the temperature distributions change not much with water quantity, but a tendency is observed for a temperature decrease at smaller water input.

Relative content of hydrogen and oxygen have been measured using H_α and O^* $\lambda=777.2$ nm lines, respectively, and Ar^* $\lambda=696.5$ nm line as a reference one. Absolute H, O density values have been evaluated from the plasma composition calculated supposing the plasma consists of argon only. The approximation relies upon generally small admixture quantity. An example of the density measurement results is shown in **Figure 4**. According to the observation, hydrogen density is about ten times lower, than argon one. It does not depend much on water quantity introduced and slightly increases with the distance from the nozzle. Atomic oxygen density is about two times lower, than that of hydrogen, which corresponds to the admixture stoichiometry. It also increases with z , which can be due to further water evaporation down along the plasma jet.

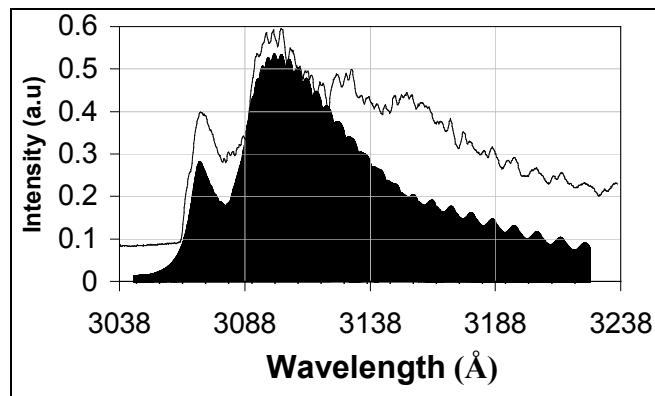


Figure 5: OH band due to transition $A^2\Sigma^+ - X^2\Pi_i$, recorded at the jet axis and $z=25$ mm with 0.5 l/h water admixture (white) and simulated for $T=6000$ K (black).

In the jet emission, molecular OH spectra have been also observed, which are due to water dissociation [2]. **Figure 5** presents OH band spectrum at 310 nm due to transition $A^2\Sigma^+ - X^2\Pi_i$, recorded at the jet axis and $z=25$ mm with 0.5 l/h water admixture. Also in the figure our simulation result for the spectrum is shown supposing the jet plasma to be homogeneous at 6000 K. One can see rather fair general coincidence of the spectra relative intensity. It means a homogeneous OH distribution in the jet, which is also confirmed by OH emission profiles across the jet. Also plasma temperature in these jet regions is evidently around 6000 K.

In situ sampling probe

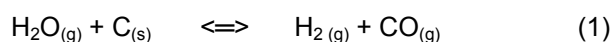
The tests were carried out with an intensity of 250 A (electric output of 5 kW, effective power of 3 kW), flow rate of argon was 30 NL/min and helium flow was 4 NL/min (introduced to increase the measuring accuracy by mass spectrometry of the hydrogen concentration).

In the downstream jet, where the temperature is between 5000 and 1000 K, we could measured (mass spectrometry) a flow of generation of hydrogen ranging between 0.04 and 0.12 L/min; this represents about 7.2 L/h hydrogen.

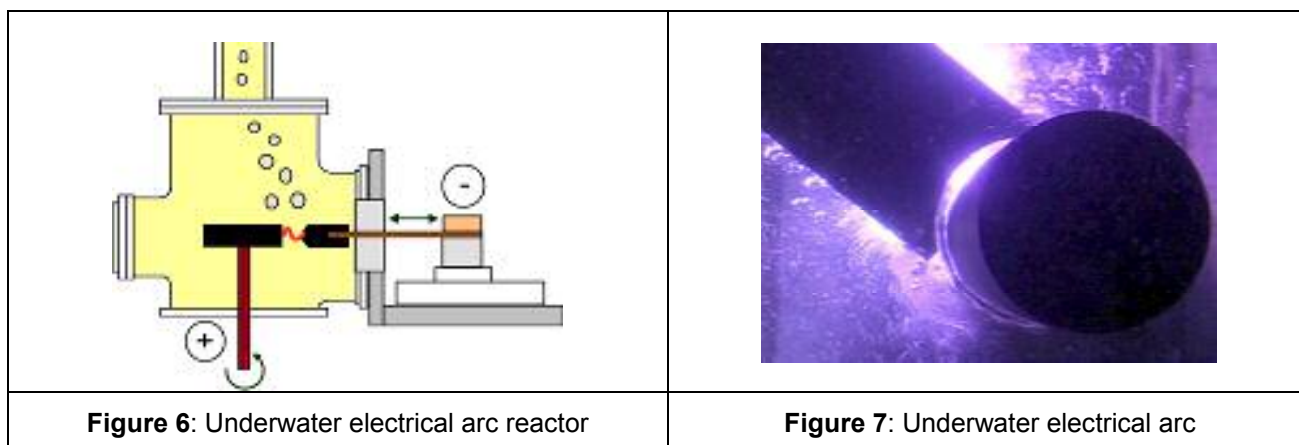
Thanks to the phenomenon of fast quench in the plasma plume, these measurements show that it is approximately 15% of the hydrogen formed in the jet plasma (at high temperature) that is preserved after cooling. This value is in conformity with the previous kinetic forecasts for a quench speed around 10^7 , classical value for the used torch in these condition.

4.2 Underwater electrical arc

Thermal water splitting is dependent of chemical equilibrium so it is theoretically possible to displace the equilibrium and prevent the recombination of hydrogen and oxygen molecules in water by using species like carbon to catch atomic oxygen following reaction (1).



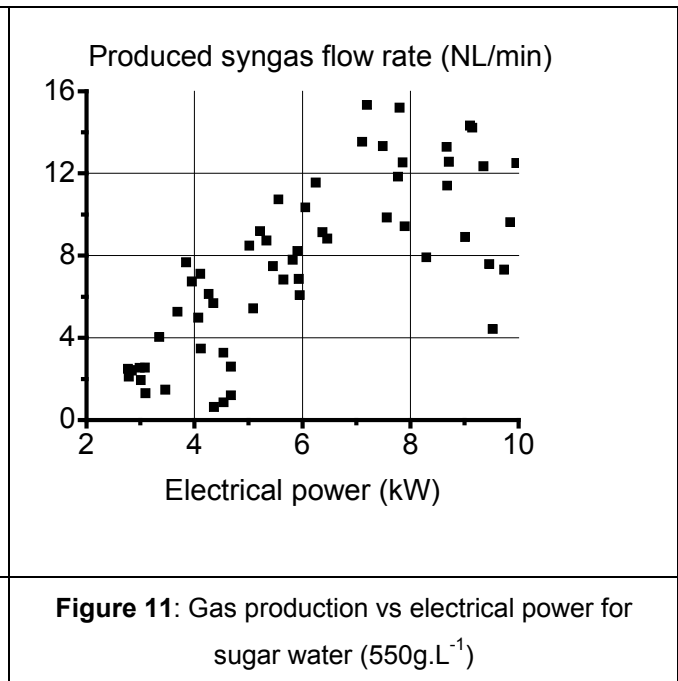
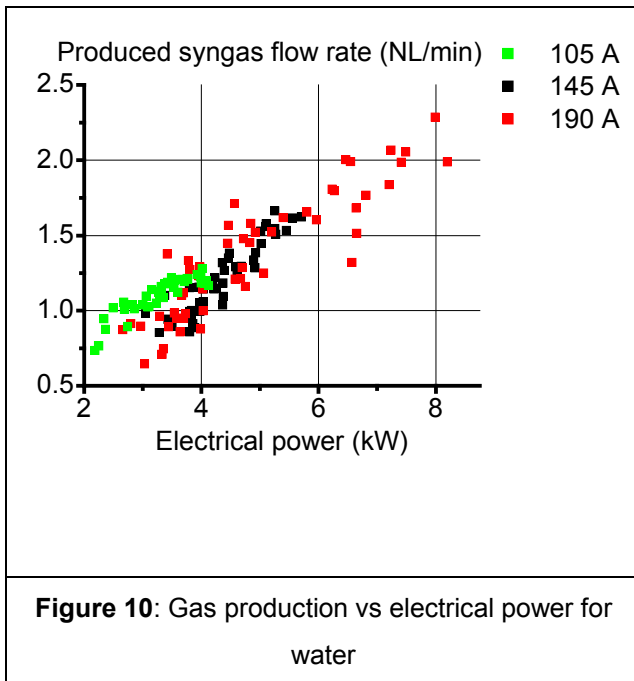
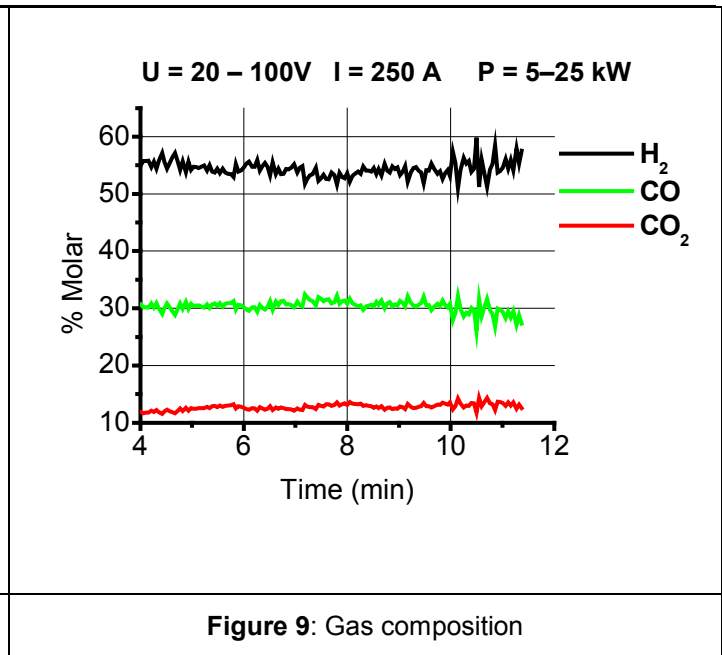
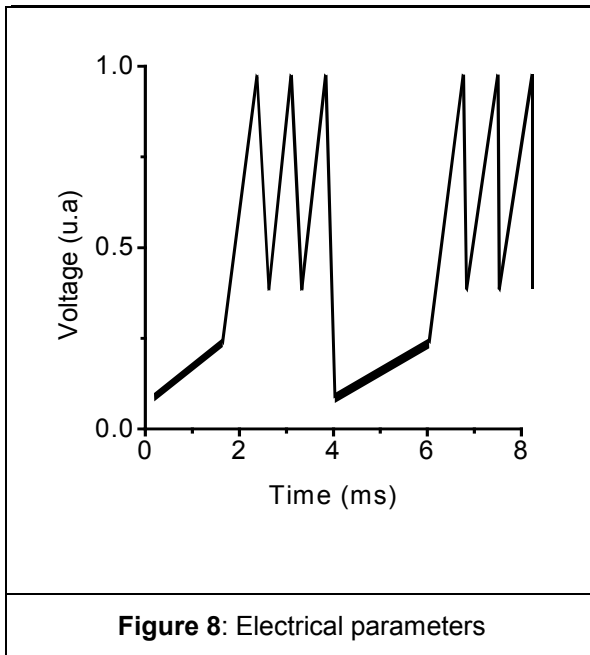
The experimental device uses an electrical arc stricken between two graphite electrodes (cf. **Figure 6**). Their different motions are due to homogenize graphite erosion. DC current about 100 – 400 A is controlled and voltage adjusts itself.



A pulsed gas production is observed. Electrical parameters could explain this phenomenon. Indeed, **Figure 8** represents voltage variation, when the electrical arc becomes longer, voltage and consequently power increase enough to split water in atomic hydrogen and oxygen. The arc lifetime is limited about one millisecond.

Gas analysis is performed by mass spectrometry and the results for trial with "drinking" water is presented in **Figure 9**. With 55 % of H_2 , 30 % of CO and 15 % of CO_2 , the produced gas has a low heating value about 9 MJ.m^{-3} in P,T normal conditions.

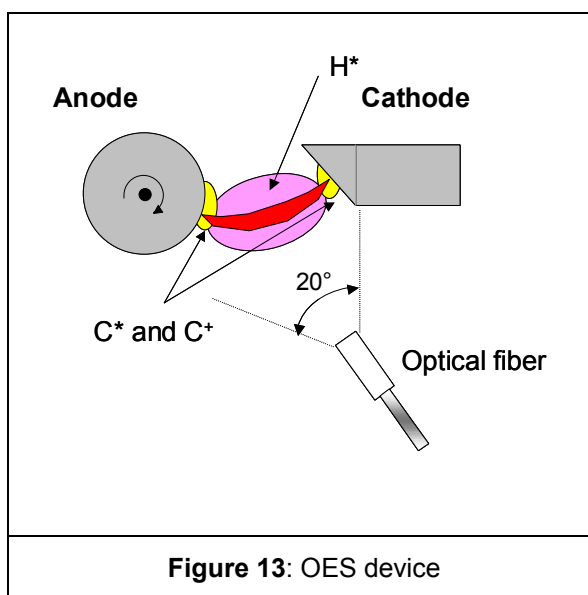
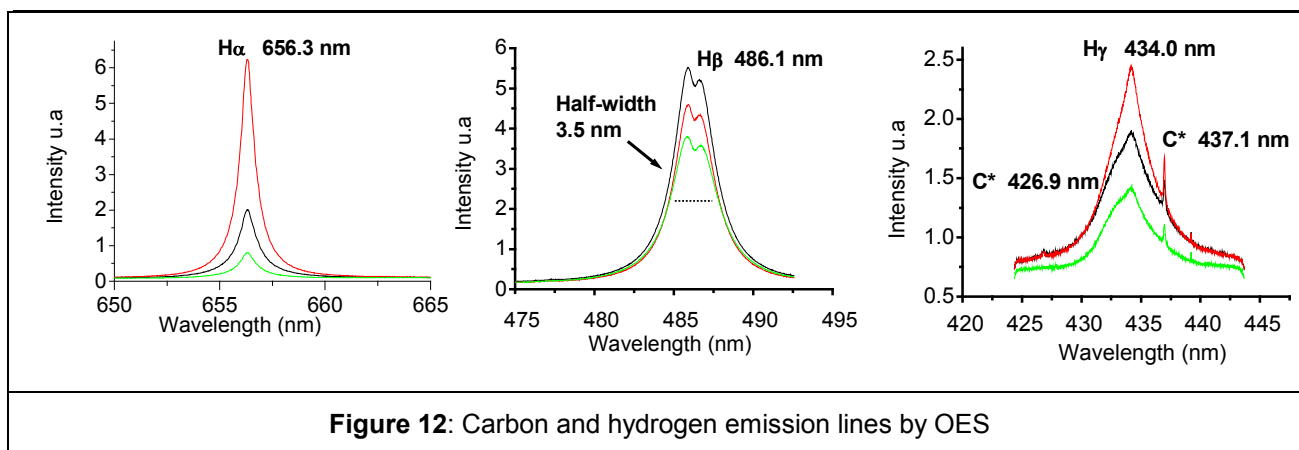
Atomic oxygen reacts with atomic carbon and hydrogen to form steam and carbon monoxide. Steam condensates in liquid water and carbon monoxide bubbles. The hydrogen production is due to the oxygen depletion because of carbon monoxide production.



Synthesis gas flowrate increases with electrical power (cf. **Figure 10**), it is between 0.5 and 2.5 L.min⁻¹. At high intensity, electrical arc is unstable and pulsed behavior of gas production is higher. For electrical power about 5 kW and 8 kW, measurements are very dispersed.

To increase reactions between hydrogen and carbon atomic, trials with sugar water have been done. Flow rate increases significantly with sugar addition (cf. **Figure 11**). Gas composition with 45 % of H₂, 45 % of CO and 10 % of CO₂ varies a little but low heating value is equal. Sugar can be surrogated by organic waste to bring carbon.

OES measurements with an optical fiber instead of optical bench and the same monochromator device has been done. There is no spatial resolution but large fiber angle opening allows observation of whole electrical arc (cf. **Figure 13**). Atomic species as O*, O⁺, C*, C⁺, H* exist in electrical arc. Several emission lines are measured (cf. **Figure 12**) to calculate a temperature estimation.



Electronic density Ne is calculated with half-width of H β (486.1 nm) and Stark parameters. Ne is equal to $5.10^{16} \text{ cm}^{-3}$. Saha-Boltzmann equation with carbon emission line measurements leads to a temperature equals to 17 000 K. Though, Boltzmann distribution equation with hydrogen emission line measurements leads to 7100 K.

The explanation is the existence of two different emission areas. In **Figure 13**, yellow spots are the carbon emission areas at 7100 K and pink column is hydrogen emission areas. So, synthesis gas reaction is limited at the interface of these two areas for the case of water immersion.

5. Conclusion

Thermodynamic and chemical kinetic modelling confirmed the feasibility to produce hydrogen and to preserve it by thermal water splitting if it quench is enough fast. Calculations show that hydrogen preservation is very sensitive to the quench speed : 1,6 % at 10^6 K.s^{-1} , 37 % at 10^8 K.s^{-1} . But, this case is true only if oxygen is free. So, it exists two ways to improve massive hydrogen production by thermal splitting: shift the equilibrium by catching oxygen and increase the quench speed.

With two experimental devices having different technologies, results of modelling are correlated. In the electrical arc reactor, quench by liquid immersion allows to preserve hydrogen in significant quantity. Moreover, carbon introduction with graphite electrodes and sugar solution increases the preservation of hydrogen and produces carbon monoxide. Liquid fuels synthetis as Fischer-Tropsch process requires this kind of gas.

To improve yield of hydrogen production, it will be necessary to design specific devices and associate very fast quench and carbon monoxide production in order to deplete oxygen in plasma. The mix between plasma and carbon source is crucial. A steam plasma torch working in a solution of liquid organic waste could be interesting for coupling these two phenomena.

6. Nomenclature

- A Probability of optical transition [s^{-1}]
- C Constant, dimensionless
- C_{1, 2} Combination of parameters for spectral lines 1, 2 under consideration [$\text{s}^{-1} \text{ nm}^{-1}$]
- E Energy of upper level [J]
- g_f, g_k Statistical weights of atomic ground and upper energy levels, respectively, dimensionless

I	Intensity of a spectral line [$\text{W cm}^{-2} \text{sr}^{-1}$]
k	Boltzmann constant [J K^{-1}]
N_a	Atomic density [cm^{-3}]
T	Temperature [K]
T_0	Maximum value in a temperature distribution along the observation direction [K]
\bar{T}_0	Mean value of the temperature T_0 during the observation time [K]
T_m	Highest value of the maximum temperature T_0 during the observation time [K]
y	Direction of observation, dimensionless
y_0	Effective dimension of the volume [m]
z	Distance from the torch nozzle [m]

Greek symbols

α	Rate of a plasma inhomogeneity, dimensionless
ε	Emission coefficient [$\text{W cm}^{-3} \text{sr}^{-1}$]
λ	Wavelength [nm]
σ	Fluctuation amplitude, dimensionless
Γ	Gamma function
Ω	Parameter, dimensionless

Subscripts

Sn	Sinusoidal fluctuation mode
Sw	Sawtooth fluctuation mode

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