

**G. CAPUTO, A. GIACONIA, S. SAU, C. FELICI**

Energy Technology Department  
Heat and Thermodynamic Solar Division  
Solar Technologies Engineering Laboratory  
Casaccia Research Centre, Rome

**A. POZIO**

Energy Technology Department  
Energy Efficient Production, Conversion and Use Division  
Chemical and Thermo-Fluid Dynamic Energy Process Development Laboratory  
Casaccia Research Centre, Rome

**EXPERIMENTAL STUDY  
FOR  $HI_x$  CONCENTRATION  
BY ELECTRO-ELECTRODIALYSIS (EED)  
CELLS IN THE WATER SPLITTING  
SULFUR-IODINE THERMOCHEMICAL CYCLE**

RT/2016/10/ENEA



ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES,  
ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

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## EXPERIMENTAL STUDY FOR $HI_x$ CONCENTRATION BY ELECTRO-ELECTRODIALYSIS (EED) CELLS IN THE WATER SPLITTING SULFUR-IODINE THERMOCHEMICAL CYCLE

G. Caputo, A. Giaconia, S. Sau, C. Felici, A. Pozio

### Riassunto

L'efficienza del processo di concentrazione/separazione dell'HI da una soluzione  $HI_x$ , (miscela di  $HI/H_2O/I_2$ ) rappresenta un fattore cruciale del processo di scissione dell'acqua per la produzione di idrogeno nel ciclo termochimico zolfo-iodio. In questo lavoro è stata condotta uno studio sperimentale sulla concentrazione catodica dell'HI in una soluzione  $HI_x$  utilizzando una cella di elettrodialisi (RED) alla pressione di 1 atm, a tre temperature diverse (25, 55 e 85 °C) e con una densità di corrente pari  $0.10 \text{ A cm}^{-2}$ . I risultati hanno mostrato che un aumento della concentrazione di HI può essere ottenuto in determinate condizioni. Il numero di trasporto apparente ( $t_+$ ) in tutti gli esperimenti era prossimo ad 1, mentre il coefficiente di elettro-osmosi ( $\beta$ ) era compreso nell'intervallo 1.08-1.16. I test hanno mostrato un aumento dello iodio rilevabile ma lento nel comparto anodico e dell'acido iodidrico in quello catodico.

**Parole chiave:** Reazione di Bunsen, Ciclo Zolfo-Iodio, elettrodialisi, scissione termica dell'acqua

### Abstract

*The efficiency of HI concentration/separation from a  $HI_x$  solution, (mixture of  $HI/H_2O/I_2$ ) represents a crucial factor in the sulfur-iodine thermochemical water splitting process for hydrogen production. In this paper, an experimental study on HI cathodic concentration in  $HI_x$  solution using stacked electro-electrodialysis (EED) cells was carried out under the conditions of 1 atm and at three different temperature (25, 55 and 85 °C) and using a current density of  $0.10 \text{ A/cm}^2$ . Results showed that an increase in HI can be obtained under certain conditions. The apparent transport number ( $t_+$ ) in all the experiments was very close to 1, and the electro-osmosis coefficient ( $\beta$ ) changed in a range of 1.08-1.16. The tests showed a detectable, though slow, increase in both the anodic iodine and cathodic hydriodic acid concentrations.*

**Keywords:** Bunsen Reaction, Sulfur-Iodine cycle, thermochemical water splitting, electro-dialysis



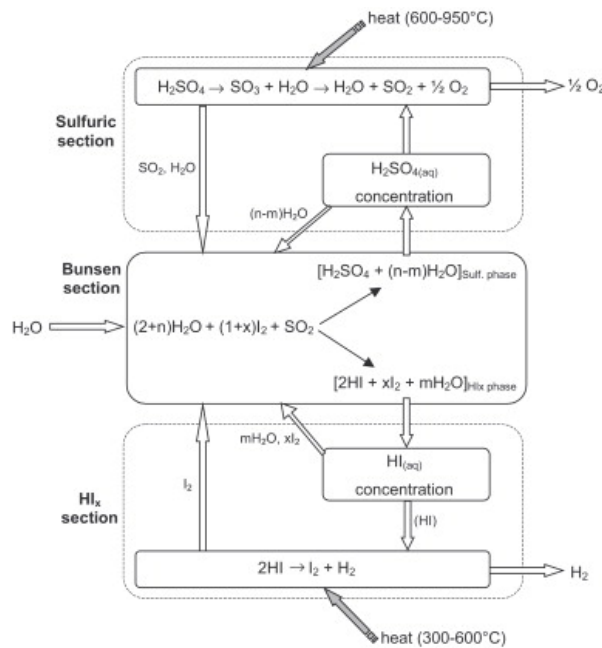
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## 1 - Introduction

Hydrogen is an attractive fuel for the future both for its ability to perform as an efficient energy carrier and its chance to be produced from renewable sources. One of the large scale most promising processes for hydrogen production is represented by the Sulfur-Iodine thermochemical cycle, first proposed by the General Atomics [1], which consists of different steps, leading to water thermal decomposition in its former components, hydrogen and oxygen (Fig. 1):



**Fig. 1- Scheme of the General Atomics sulfur–iodine thermochemical water-splitting cycle [1].**

In particular, the cycle can be schematized with three different chemical reactions, each one representative of a section in Fig. 1:

- $I_2 + SO_2 + 2 H_2O \rightarrow H_2SO_4 + 2 HI$                       Bunsen Section                      (1)
- $2 H_2SO_4 \rightarrow 2 SO_2 + 2 H_2O + O_2$                       Sulfuric Section                      (2)
- $2 HI \rightarrow I_2 + H_2$                                               HI<sub>x</sub> Section                                      (3)

The first reaction is also called “Bunsen reaction” and it is an exothermic process performed in



liquid phase with the adsorption of sulfur dioxide; the reaction proceeds spontaneously in the 20-120°C temperature range [2].

The reaction products are composed by two immiscible liquid-phase systems: a lighter one containing mostly sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and the heavier one presenting a mixture containing  $\text{HI}_x$  species, that is, chemical complexes of HI and  $\text{I}_2$  in aqueous solution.

A correct separation among these two phases is needed in order to obtain a substantial efficiency from the S-I cycle, for this reason, it is necessary to use an excess of both water and iodine in the Bunsen reactor.

The decomposition of sulfuric acid (eq. 2) is an endothermic reaction generally carried out by a two-stage catalytic reactor ( $\text{H}_2\text{SO}_4$  is easily decomposed in  $\text{H}_2\text{O}$  e  $\text{SO}_3$  at 400-450°C and then  $\text{SO}_3$  is further catalytically decomposed in  $\text{SO}_2$  and  $\text{O}_2$  at 850°C).

The section containing a mixture of iodine and iodides ( $\text{HI}_x$  section), represents the most critical point of the cycle, regarding both energy consumption and global efficiency.

In fact, hydrogen iodide (HI), must be separated from the  $\text{HI}_x$  solution (HI/ $\text{H}_2\text{O}$ / $\text{I}_2$  mixture), exiting the Bunsen reaction, before being decomposed for hydrogen generation (3). A simple option would consist of a distillation process followed by HI thermal decomposition, but, due to the presence of an azeotropic composition in the HI/ $\text{H}_2\text{O}$  system (at about 57% w/w), distillation requires high pressures and, consequently, a large thermal burden. Additionally, the low equilibrium conversion of HI decomposition (about 20% at suitable reaction temperatures) imposes a large HI recirculation rate, which results in a further increase of thermal burden and a decrease of the whole thermal efficiency. In order to overcome these problems, General Atomics studied an extractive distillation process using phosphoric acid [1-3]; alternatively, a reactive distillation configuration under pressurized conditions was also investigated [4-6]. More recently, the use of electro-electrodialysis [7-12], pervaporation processes with cation exchange membranes [13] and membrane distillation [14] was proposed to concentrate HI aqueous solutions. In all cases, iodine and water excess are removed from the main stream and recycled to Bunsen reactor.

The HI/I<sub>2</sub>/H<sub>2</sub>O ternary mixture is an exceedingly complex system for the presence of different equilibrium of phases. The H<sub>2</sub>O/I<sub>2</sub> liquid-liquid subsystem has an highly immiscible behavior with a strong tendency of the iodine to crystallize (iodine melting point is 113.6°C). The HI/H<sub>2</sub>O subsystem, composed by strong electrolytes, shows an azeotrope at 57% in weight of HI. The decomposition reaction of HI is limited by the chemical equilibrium, and the conversion rate, at 500°C, reaches only the 22.4%. As reported, in order to obtain a proper separation of phases, an excess of iodine and water is required for the Bunsen section, and, consequently, a large amount of both these species, namely, 8 moles of I<sub>2</sub> and 14 moles of H<sub>2</sub>O, is present in the HI<sub>x</sub> phase for each mole of H<sub>2</sub> produced, leading to a significant value of energy demand. The electro dialysis process here described aims to reduce this thermal burden.

In this work an experimental campaign was carried out to obtain sensible data about this process, in order to show advantages and limitations of this technique.

## **2 - Experimental**

### **2.1 - Chemicals**

The solutions used in the experimental tests were prepared using HI, 57% in weight, distilled and stabilized (Acros Organics) and iodine (Clean Consult International).

### **2.2 - Experimental set-up for EED**

The experimental campaign was carried out using an electro dialysis cell mod. ED-64-002 (PCA Polymerchemie Altmeier GmbH – Germany), whose original electrodes were replaced with home-made rhodium coated titanium electrodes.

Titanium plates (Titania, Italy) with an area of 112 cm<sup>2</sup>, were used to produce two electrodes similar to the ED-64-002 original ones. The electrodes were cleaned by an ultrasonic bath in ethyl alcohol. After 3 minutes of pickling in a HF (Carlo Erba)/HNO<sub>3</sub> (Carlo Erba) solution, in a volumetric ratio of 1:3, and diluted in deionised water until to 100 mL, each titanium electrode was oxidized using a two-electrode cell (the counter-electrode was a platinum gauge), containing a H<sub>2</sub>SO<sub>4</sub> 1 M solution (Carlo Erba) at 25°C, and subjected to a prefixed and optimized density current (40 mA cm<sup>-2</sup>),

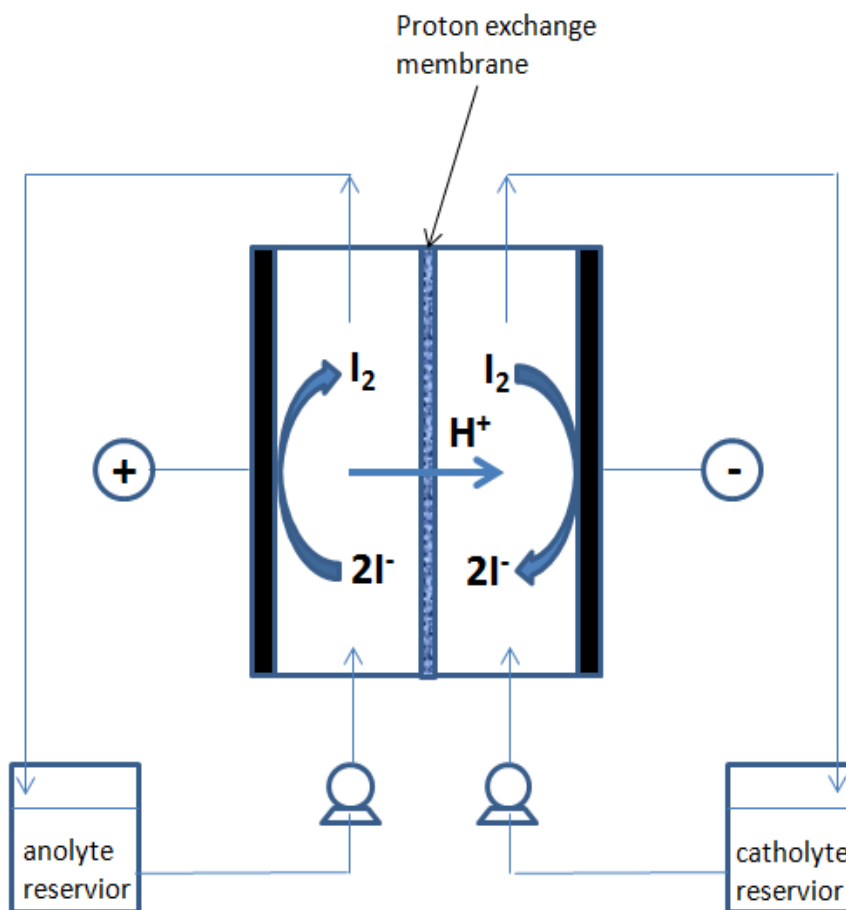
generated by a potentiostat/galvanostat (Ecochemie). The oxidation current was applied to the titanium electrode until a voltage of 100 V was reached and an uniform and dense grey TiO<sub>2</sub> layer was deposited onto the surface. After this anodization treatment, the electrodes were washed in water and carefully dried.

The rhodium coating took place in the same above configuration, by multiple pulse galvanostatic electrodeposition. The electrodeposition bath was Rhodium Sulphate (Rh 5 g L<sup>-1</sup>) in H<sub>2</sub>SO<sub>4</sub> 1 M at 25°C. A pulse current density of -2 mA cm<sup>-2</sup> was applied for 0.25 sec followed by a pause of 0.5 sec. Three thousands pulse were applied in order to obtain a total charge equivalent to a rhodium loading of 0.53 mg cm<sup>-2</sup>. The final Ti/TiO<sub>2</sub>/Rh electrodes' superficial area was 7.5 x 7.5 cm<sup>2</sup>.

Nafion 117 (Du Pont's) cation-exchange membrane with an active surface of 64 cm<sup>2</sup> (8 cm x 8 cm) was used as electrolyte.

Electrodialysis measurements were performed using an Autolab Pgstat30 potentiostat (Ecochemie) interfaced with a current booster BSTR20A, necessary to work with electric currents up to 20 A, and employing a GPES 4.7 control software. Impedance measurements were performed at open circuit voltage (OCV) with an AC signal of 10 mV between 10000-1 Hz using the Autolab Frequency Response Analyser module.

The mixtures subjected to electrodialysis, were contained into two reservoirs, each having a volume of 300 mL, packed in a warming jacket (Fig. 2). Mixture's circulation were assured by two Watson Marlow peristaltic pumps; circulation rate was controlled by changing the pump spinning rate (50 mL min<sup>-1</sup>).



**Fig. 2 - Schematic diagram of the electrodedialysis cell.**

### 2.3 - Concentration by EED

Experimental tests were performed at constant voltage. At the beginning of each test, 300 mL of Hydroiodic mixture were loaded in each reservoir..

Two kinds of iodidric mixture were employed; a former-one called “simil-Bunsen mixture” (HI 3M, I<sub>2</sub> 3.2 M), which composition is very similar to the one obtained from a real the Bunsen reactor, and a latter-one called “mixture II” (HI ≈4.5 M, I<sub>2</sub> 0.8 M) containing only one fraction of the HI and iodine present in the simil-Bunsen mixture.

Mixture II was used to overcome problems of bad circulation due to the presence of crystallized I<sub>2</sub>. Each test lasted 4 hours and, each hour, samples of both anolyte and catholyte were taken for analysis.

Samples composition were determined by dinamic titration (Methrom Titrand), using standardized

solutions of thiosulfate for iodine determination and sodium hydroxide for HI quantification.

Regarding the measurement error, given the high precision provided by the automatic titration technique, the significant detected imprecision can be due to sampling and sample handling problems, especially considering the iodine's volatility. The relative error on the molar values can be roughly estimated around 3% for HI and not less than 12% for iodine determination.

## 2.4 - Transport properties of the membrane and energy consumption

The membrane characteristics were studied by the following two parameters: the apparent transport number of proton ( $t_+$ ) and the apparent coefficient of electro-osmosis ( $\beta$ ).

The apparent transport number shows the ion amount passing through the membrane due to a given driving force.

The electro-osmosis coefficient indicates the water quantity passing through the membrane by means of the solvated ions.

The changes in the measured amounts of respective species, HI and H<sub>2</sub>O, were used to calculate the apparent transport number of proton ( $t_+$ ) and the apparent electro-osmosis coefficient ( $\beta$ ), in the catholyte part. The transport properties were calculated by the following equations, derived from mass balance calculations in the catholyte section, taking into account the changes due to electrode reaction:

$$t_+ = \frac{F \cdot [\Delta mol (HI)]}{i \cdot \Delta t} \quad (4)$$

$$\beta = \frac{F \cdot [\Delta mol (H_2O)]}{i \cdot \Delta t} \quad (5)$$

Here  $\Delta M$ ,  $t$ ,  $i$  and  $F$  indicates, respectively: the molar variation of HI and H<sub>2</sub>O amounts in the catholyte section, the EED process time (s, corresponding to a total of 4 hours in the reported data), the electric current (in Ampere) and the Faraday constant.

Since to obtain the values of  $t_+$  and  $\beta$ , the weight changes, due to the EED process, of anolyte and catholyte solutions have to be measured, and this can be only done at the end of the experiments, by

collecting the anolyte and catholyte solutions, the obtained  $t_+$  and  $\beta$  are average values representative of the all period of the EED experiment considered.

The electric power consumption of the EED cell depends on the potential drop across the cell ( $V$ ), the current applied ( $i$ ) and the current efficiency or the apparent transport number of proton in the membrane ( $t_+$ ) as shown in Eqn. (6):

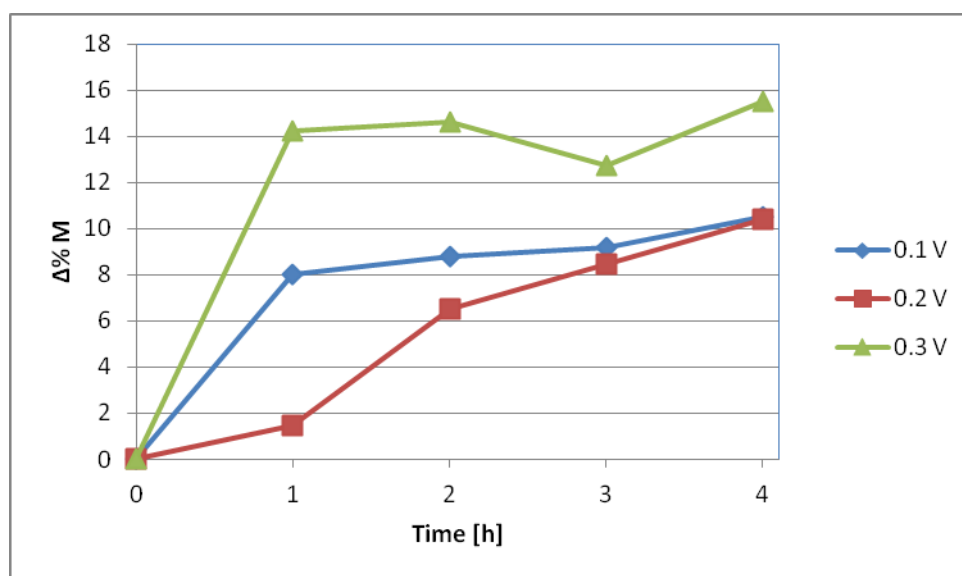
$$E_{eed} = \frac{iV}{t_+} \quad (6)$$

The molarity variations of HI in the catholyte section and  $I_2$  in the anolyte one were reported, in order to obtain trends easy to be compared.

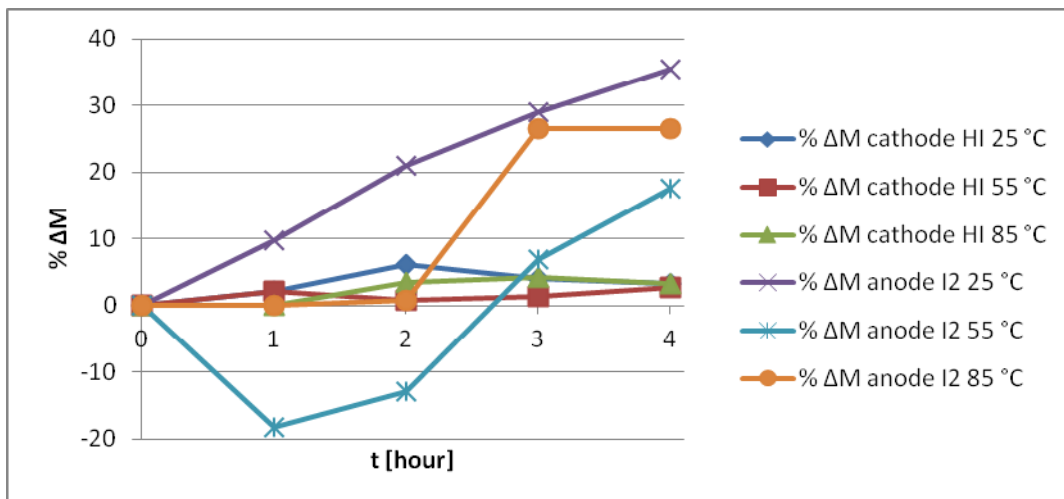
### 3 - Results and discussion

#### 3.1- Concentration by EED

Electrodialysis tests were performed varying the electric voltage applied to cell and mixtures temperature. In the following figures are reported the results obtained with the “mixture II” composition, that is, with relatively low iodine concentration. In Fig. 3 is shown the trend of HI percentage increase respect to the initial molarity versus time, applying three different voltage values at 25 °C, in Fig. 4 temperature was varied, maintain the voltage at 0.1 V.



**Fig. 3 – Cathode HI percentage increase respect to the initial molarity vs time for different cell voltage (T=25 °C).**



**Fig. 4 – Cathode HI and anode I<sub>2</sub> molarity percentage variations vs time for different electro dialysis cell temperature and 0.1 V.**

From fig. 3, the expected proportionality between the applied driving force and the separation rate measured by the change in HI molarity, become significant at 0.3 V. The apparent different behaviour in the first two hours of the tests at 0.1 and 0.2 V, is presumably due to the experimental error. A clearer way to appreciate the voltage effect on the separation process is to consider the apparent proton transport number, as shown below.

Concerning temperature, no detectable effects, considering the experimental error, were present for HI cathodic and iodine anodic concentrations (fig. 4).

The obtained experimental data were used to calculate the apparent transport number of proton ( $t_+$ ) and apparent electro-osmosis coefficient ( $\beta$ ).

**Table 1 – Transport number ( $t_+$ ), electroosmosis coefficient ( $\beta$ ) and specific energy demand ( $E_{eed}$ ) as a function of different electrolysis parameter.**

Voltage V	Temperature °C	Used Mixture	$t_+$	$\beta$	$E_{eed}^*$ J sec <sup>-1</sup>
0.1	25	Mixture II	0.76	1.09	42.4
0.2	25	Mixture II	0.81	1.12	86.7
0.3	25	Mixture II	0.86	1.16	143.6
0.1	55	Mixture II	0.76	1.08	53.2
0.1	85	Mixture II	0.74	1.08	46.6
0.1	85	Bunsen	0.76	1.08	173.5

\*= normalized with  $t_+$

The water contained in each sample was obtained from the difference among the sample's overall weight and the HI and I<sub>2</sub> quantities obtained by means of titration.

For this reason, the calculated water amounts can be affected from a substantial experimental error; anyway, considering that the  $\beta$  parameter can be independently calculated both using cathodic and anodic concentrations, it was possible to cross check the two obtained values, and accept only the electro-osmosis coefficients resulting equal to each other.

The apparent transport number increased with the applied voltage from 0.76 to 0.81 and 0.86 (Table 1), it also virtually didn't change with temperature, remaining constant at a value around 0.75.

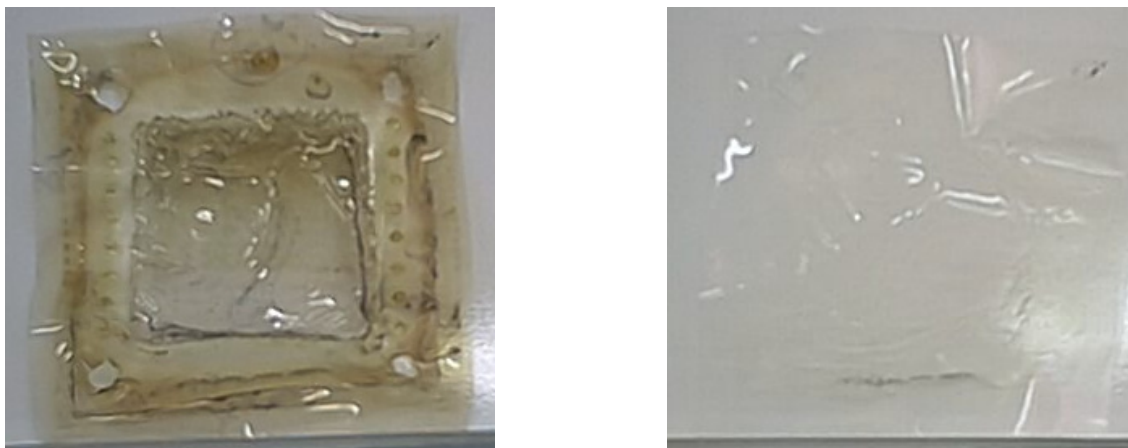
Regarding the electro-osmosis coefficient at 25°C (Table 1),  $\beta$  increases from 1.09 mol F<sup>-1</sup> (at 0.1 V) to 1.16 mol /F<sup>-1</sup> (at 0.3 V). At 0.1 V and in the temperature range 25–85°C, it is possible to observe that the increasing of temperature influenced marginally  $\beta$ , which maintained a value of about 1.08 mol F<sup>-1</sup>; the impact over the system performance was almost null and, as already reported, the trend of concentration was steady at every temperature level for both HI and the iodine.

### 3.2 - Durability of the membrane and of the electrodes in the HI<sub>x</sub> solution

The impedance of the cell was measured before and after each test in order to verify the corrosion of electrodes and/or the membrane degradation through the iodides mixture. The ohmic resistance



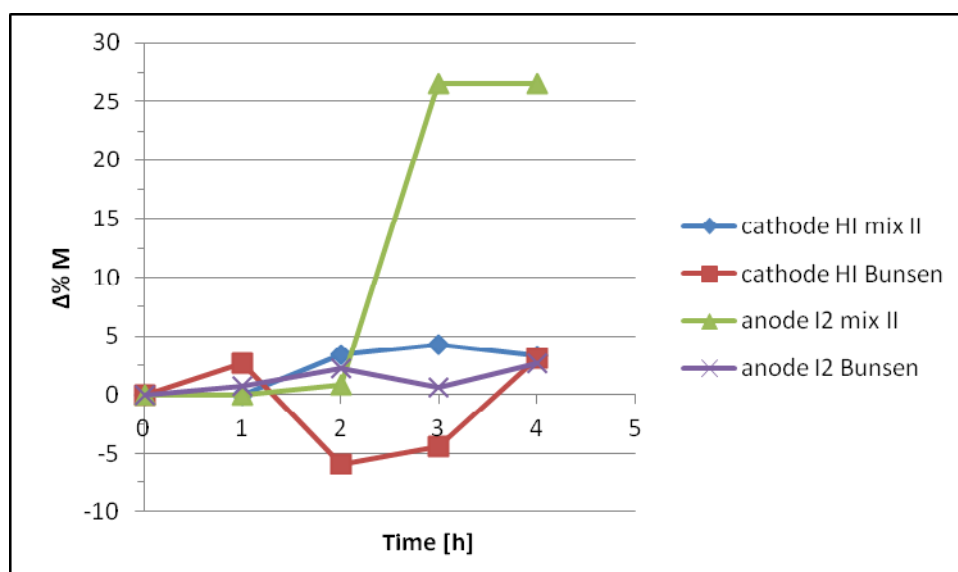
of the cell was constant ( $4.5 \text{ ohm cm}^2$ ), so evidencing the stability of the components. In addition, the examination of the titanium-rhodium electrodes and of the membrane (Fig. 5) after the tests campaign showed the resilient behavior of both elements against the iodide mixture.



**Fig 5 - The Nafion membrane just removed from the cell (left), the membrane after a washing in  $\text{H}_2\text{O}_2$  2 wt% solution (right).**

### 3.3 - Comparison between Bunsen-like mixture and mixture II

As previously said, the Bunsen-like mixture at low temperature, showed some circulation problems during the tests. Consequently, an experiment was carried out with the Bunsen mixture at  $85^\circ\text{C}$  and  $0.1 \text{ V}$  (Fig. 6).

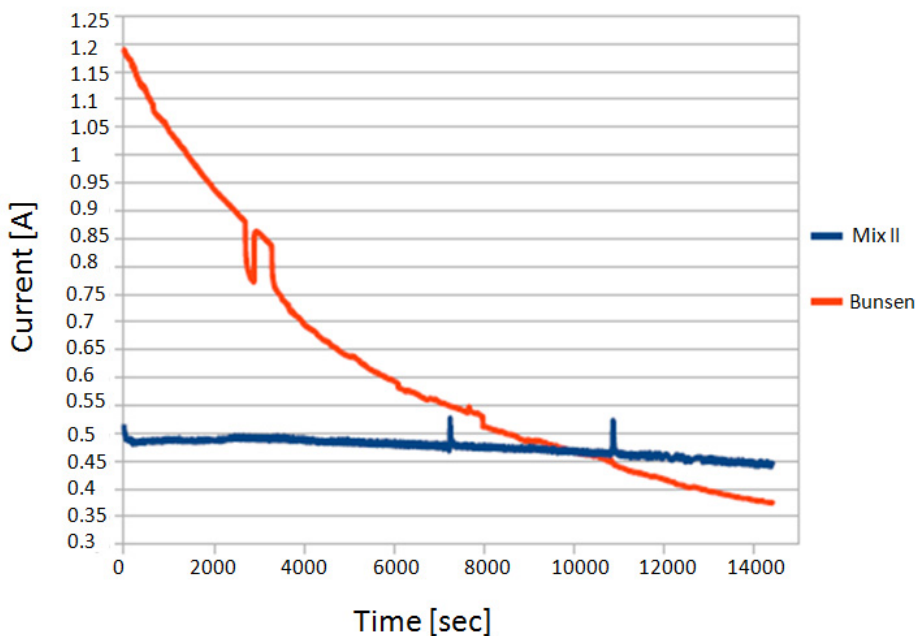


**Fig. 6 – Cathode HI and anode  $\text{I}_2$  percentage increase respect to the initial molarity vs time for two different initial compositions ( $T=25^\circ\text{C}$ ,  $0.1 \text{ V}$ ).**

Because of the sampling difficulty of a solution presenting high  $I_2$  contents at 85 °C, and considering a measurement error of 3%, the increase of cathodic HI can be only qualitatively verified. In any case, it appears that, after 4 hours time, about the same final HI concentration increase is reached both with the Bunsen and the II mixture.

Comparing in table 1 the results with the ones for the “mixture II” composition, it can be noticed that, considering the experimental error, the separation parameters are practically the same respect to the ones obtained with the “mixture II”. The apparent transport number of proton was almost constant varying from the 0.74 (Bunsen-like test) to the 0.76 value (mixture II test).

As expected, a real difference between the two tests is represented by the flow of the electric current in the cell (Fig. 7). The tests with mixture II, furnished a trend of the electric current constant independently from operative conditions. In contrast, the tests with the Bunsen-like mixture, showed a sharp decrease of the electric current in the time.



**Fig. 7 – Electrodialysis cell current vs time for the two different mixture (0.1 V, 85 °C).**

### 3.4 - Energy consumption

An energy consumption estimation ( $E_{eed}$ ) has been calculated starting from applied voltage, electricity and concentration data taken from the various tests (Table 1). The electric current was integrated to calculate the charge developed in the cell. To be able to perform effective comparisons, the energy consumption value has been related with the variation of HI in each test. Results, showed that the energy consumption increased with the growth of applied voltage, although we observed a simultaneous increase of the separation rate.

Working at higher temperatures, it wasn't detected any increasing in the separation rate. So, temperature cannot be considered as a parameters to optimize EED efficiency. Tests performed with the Bunsen-like mixture showed a bigger energy consumption than the correspondent ones with mixture II.

Considering that, within the same time interval, the HI increase at the cathode is basically the same both in the Bunsen and the II mixture, the higher energy consumption could be due to problems in the membrane separation capacity in presence of high iodine concentration that cause partial reactions at the electrodes.

### 4 - Conclusions

1 - The electric current, in tests performed with mixture II, decreases linearly with the time; the electric current variation is enhanced in tests with Bunsen-like mixture.

2 - The apparent transport number of proton shows a linear dependence with the electrodes applied voltage, regardless of the used mixture.

3 - The mixture temperature has little effects on the separation degree. Only the apparent electro-osmosis coefficient varies slightly, this is probably due to the increased thermal agitation of the molecules in the mixture.

4 - The titanium-rhodium electrodes presented a good chemical resistance during tests. The electrodes' surface and the membrane, were shielded very well against the corrosive action of the

solution and, the ohmic resistance of the cell was constant during all of experimental campaign.

5 - Energy consumption increases with increasing voltage applied to the cell, as well as separation rate. Energy consumption at higher temperatures, and at constant voltage doesn't increase significantly, showing once more how temperature is not a sensitive parameter for the separation. The tests carried out with the Bunsen-like mixture show a bigger energy consumption with respect the correspondent tests performed with mixture II.

6 – Experimental results showed that it is possible to concentrate HI at the cathode also starting from a Bunsen like mixture; as a consequence, it is in principle possible to directly concentrate and separate the hydriodic acid from iodine using the EED system downstream of the lower phase of the sulphur-iodine Bunsen section, still having a high energy consumption. Alternatively, it can be possible to use the EED technique to concentrate at low voltages (and with a relatively low energy consumption) a solution where most of the iodine excess has been previously removed by a pre-distillation process.

7 - These tests are been realized on a batch configuration but the results can help to implement the electro-dialysis in a continuous operation.

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