

Integrated laboratory scale demonstration experiment of the hybrid sulphur cycle and preliminary scale-up

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Abstract

The hybrid sulphur cycle is today one of the most promising processes to produce hydrogen on a massive scale within the scope of high temperature nuclear reactors development. Thus, the Fuel Cycle Technology Department at CEA Marcoule is involved in studying the hybrid sulphur process from a technical and economical performance standpoint. Based on mass and energy balance calculations, a ProsimPlus™ flow sheet and a commercial plant design were prepared. This work includes a study on sizing of the main equipment. The capital cost has been estimated using the major characteristics of main equipment based upon formulae and charts published in literature. A specific approach has been developed for electrolysers. Operational costs are also proposed for a plant producing 1 000 mol/s H₂.

Bench scale and pilot experiments must focus on the electrochemical step due to limited experimental data. Thus, a pilot plant with a hydrogen capacity of 100 NL/h was built with the aim of acquiring technical and technological data for electrolysis. This pilot plant was designed to cover a wide range of operating conditions: sulphuric acid concentrations up to 60 wt.%, temperatures up to 100°C and pressures up to 10 bar. New materials and structures recently developed for fuel cells, which are expected to yield significant performance improvements when applied to classical electrochemical processes, will be tested. All experiments will be coupled with phenomenological simulation tools developed jointly with the experimental programme.

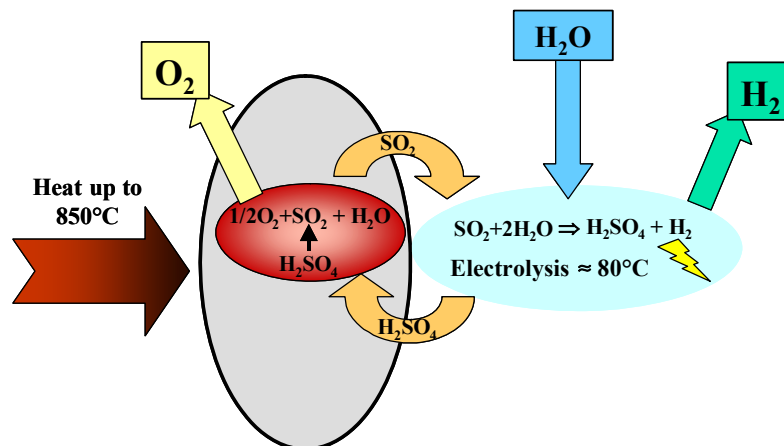
Introduction

Within the framework of the Generation IV forum, one of the primary goals for the very high temperature reactor (VHTR) is hydrogen production. Indeed, VHTR produces heat at about 950°C, which could allow steam electrolysis using solid oxide fuel cell derived technologies, or high temperature thermochemical cycles such as the hybrid sulphur cycle (HyS). These processes are promising candidates for massive hydrogen production, but they require important development.

The French *Commissariat à l'énergie atomique* (CEA) has launched an integrated programme which will assess the most promising way to produce hydrogen using high temperature heat available from a VHTR. This programme includes: development of a methodology for process comparison, acquisition of basic thermodynamic data, flow sheet analysis and development, preliminary design of a hydrogen production plant coupled to a VHTR, including energy distribution and safety issues, efficiency and cost analysis.

CEA has chosen to focus on a limited number of potentially interesting processes, namely the high temperature electrolysis, the iodine/sulphur cycle and the hybrid sulphur cycle (Figure 1). Other options are evaluated on a more limited basis. Rather than building expensive large scale demonstration loops, emphasis is placed on experiments: their goals are to better understand the thermodynamic behaviour of the chemical system to implement more reliable models.

Figure 1: The hybrid sulphur (HyS) cycle

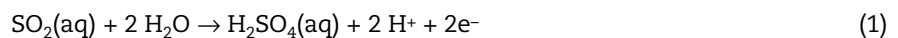


The purpose of this paper is to describe the current progress of the HyS cycle at CEA, based on recent experimental data acquisitions and simulations. We will focus on the field of research and development, taking into account both thermodynamics and economics.

Technical issues

Many variations are possible for each section of the HyS cycle (Brecher, 1977): the temperatures and other operating conditions can be optimised, with a wide variety of solutions in matter of chemical engineering to perform unit operations and to exchange heat. Each section is strongly dependant on the other, meaning that an output may be the input of the next section. Any change in one section may influence directly the whole process.

In the electrolysis step, SO_2 is electrochemically oxidised at the anode to form H_2SO_4 , protons and electrons. The protons are conducted across the electrolyte separator to the cathode where they recombine with the electrons to form H_2 .



One advantage of the HyS cycle is that the standard cell potential for SO₂ depolarised electrolysis is close to 0.158 V at 298 K in water. This value increases to 0.243 V in a 50 wt.% H₂SO₄ aqueous solution which is the most likely anolyte. The main challenge of this step is finding an electrolytic cell technology suitable for the temperature (up to 393 K) and pressure (up to 10 bar). The electrolytic cell development requires an optimisation of the chemical composition of the electrolyte and an optimisation of the cell geometry.

The second step, common to all sulphur processes, is the result of two successive reactions. As sulphuric acid is vaporised (ca 650 K) and superheated (ca 900 K), it spontaneously decomposes into water and sulphur trioxide. The following decomposition occurs by heating the vapour to high temperatures (> 1 000 K) in the presence of a catalyst to produce oxygen and sulphur dioxide.



The main challenge of this step is finding a technology suitable for the temperature (up to 1 150 K) and pressure (up to 50 bar) conditions. In addition, at these operating temperatures most catalysts are severely damaged or destroyed within a short time. Finding the good catalyst is a key point for a sulphur-based thermochemical cycle.

The HyS flow sheet

The ProsimPlus™ flow sheet illustrated in Figure 2 was prepared on the basis of a 1 kmol/s H₂ production rate.

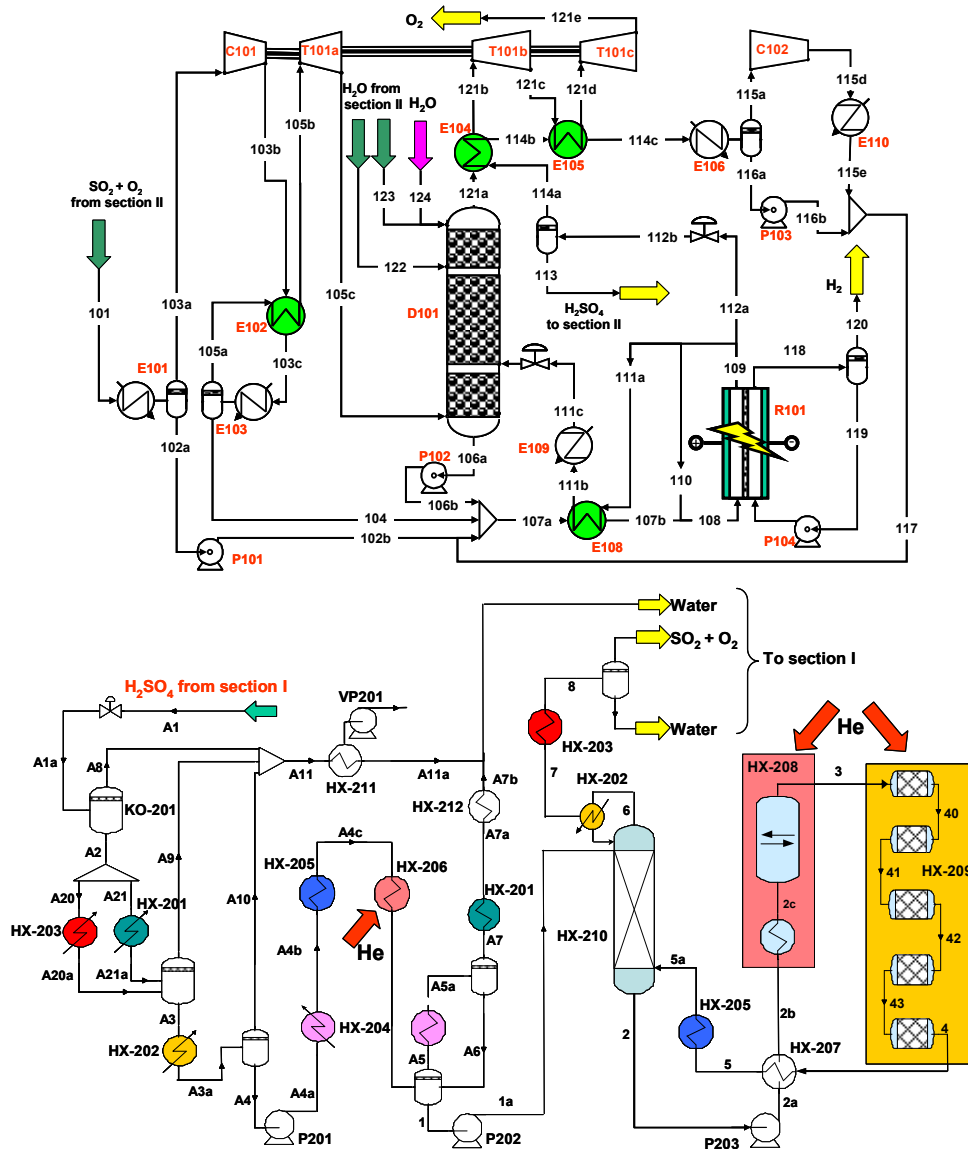
Section I of the HyS process involves two main operations. The first operation concerns electrolysis and the anolyte preparation. The second operation concerns SO₂-O₂ separation.

The following operating conditions corresponding to electrolytic reaction are assumed for the development of the Section I flow sheet: an input sulphuric stream composition (2 mol% SO₂, 49 mol% H₂SO₄ and 49 mol% H₂O), an upstream temperature of 353 K and pressure of 10 bar. The electrolytic cell is treated as a “black box” in which the following changes take place: on the one hand reaction between SO₂ and H₂O via Eq. (1-2) with an assumed conversion of 62% (the anolyte feed contains 62% excess SO₂) and on the other hand calculation of the downstream temperature such that the enthalpy change between the feed and product streams being equal to the electric work with a cell potential of 0.6 V (Lu, 1981).

The anolyte product, containing 0.9 wt.% unreacted SO₂ dissolved in 51 wt.% H₂SO₄, is split sending enough H₂SO₄ to decomposition. The remainder (representing 96% of the anolyte effluent) is split again sending 20% of the flux to the SO₂ absorber at Stage 21 (see below). This stream is cooled by interchange with recycled anolyte involving streams coming from the SO₂-O₂ separation and bottom stream of the SO₂ absorber. The remainder is recycled to the anolyte preparation tank. The pressure of the anolyte product is adiabatically dropped from 10 to 0.2 bar allowing SO₂ be removed in the resulting gaseous stream. This stream is recycled to the anolyte preparation tank via a set of devices including compressor and heat exchangers. The liquid stream is sent to Section II.

The gaseous mixture of SO₂-O₂-H₂O coming from Section II and containing 25.8-12.9-61.3 mol% respectively, is first cooled to 298 K to remove most of the water content. The assumed feed composition resulting from the Section II operating conditions will be justified below. The pressure of the vapour phase is raised from 5 to 10 bar then cooled to 298 K first by interchange with the O₂ product in two heat exchangers. This operation allows about 54% of SO₂ to be liquefied. The residual vapour phase is expanded from 10 to 5 bar with recovery of the expansion work then, fed into the SO₂ absorber at the bottom stage. The SO₂ absorber is a 26-equilibrium stage vapour-liquid fractionation device. It operates at a pressure of 5 bar with an assumed negligible pressure drop. Feed water adding to liquid distillate from the second-stage knock-out of Section II is fed at the top stage. Liquid distillate from the first-stage knock-out of Section II, containing 99.9 mol% H₂O and 0.1 mol% SO₂ is cooled to 298 K in the heat exchanger then routed to Stage 7. The overhead product of composition 99.9 mol% O₂ and 0.1 mol% H₂O is fed to an expander to recover the O₂ expansion work at atmospheric pressure. The SO₂ losses are assumed less than 5.10⁻⁵ mol SO₂/ mol H₂ product.

Figure 2: Hybrid sulphur flow sheet (top Section I, bottom Section II)



Section II of the HyS process involves two main steps. The first one concerns H_2SO_4 concentration to decrease the amount of water which enters the second step with decomposition reactors.

The goal of this step is to concentrate sulphuric acid with minimum external heat requirement. The pressure of the liquid mixture of about 52 wt.% H_2SO_4 , coming from Section I is first dropped adiabatically from 0.2 to 0.1 bar allowing residual SO_2 and O_2 to be removed in knockout drum via gaseous stream, along with some water (KO-201). Next, the liquid stream (52.6 wt.% H_2SO_4) is heated by interchange in a three-heat-exchanger network (HX-201, HX-203, HX-202). The different distilled vapour streams are removed, cooled and condensed at 298 K (HX-211). The residual liquid stream (73.5 wt.% H_2SO_4) is fed into the second vaporisation stage at an operating pressure of 1.5 bar (P201). The stream is first heated by interchange in two heat exchangers (HX-204, HX-205) then in a third exchanger powered by hot stream of helium from the primary heat source (HX-206). The composition of the final stream is controlled by means of a condenser which allows the liquid temperature to be regulated. The chosen parameters are a temperature of 493 K and a pressure of 1.5 bar. With this set of parameters the composition of the liquid phase entering the decomposition step is close to 80 wt.% H_2SO_4 .

At the entry of the decomposition step, volumetric pump raises the pressure to the operating value (5 bar) (P-202). The liquid stream is fed to the top of SO₃ recombination reactor (HX-210). This reactor consists of an adiabatic seven-equilibrium stage vapour-liquid contactor device. It operates at the equilibrium pressure of the process with an assumed negligible pressure drop. The goal of this device is to countercurrently wash the gases coming from the decomposer (HX-209) which contains a hot mixture of O₂, SO₂, H₂O and unreacted SO₃. The gaseous stream is cooled by contact with the liquid sulphuric phase, then SO₃ is converted to H₂SO₄ in the presence of gaseous water. H₂SO₄ in the gaseous stream condenses in contact with the cold aqueous sulphuric stream. Concentrated liquid sulphur acid at about 88 wt.% H₂SO₄ coming from the bottom of reactor at 580 K is sent to the decomposition reactor. The goal is to achieve an equilibrium pressure of 5 bar at the end of the decomposition reactor. The mixture temperature is raised to about 873 K where H₂SO₄ decomposes almost completely to SO₃ and H₂O. Next, the gaseous stream enters a set of catalytic reactors in which the SO₃ to SO₂ decomposition reaction takes place (HX-209). The outlet equilibrium temperature and pressure are controlled at 1 123 K and 5 bar. The heat required for decomposition reactors is supplied by a hot stream of helium from the primary heat source. The gaseous stream is cooled in two heat exchangers which allow for heat recovery. The pinch temperature in the interchange heat exchanger is no less than 50 K and the function of the following heat exchanger is to cool the stream to its dew point to avoid condensation. This procedure prevents water dilution of the feed entering the decomposition step. The cooled gaseous mixture is fed to the bottom of recombination reactor, as described earlier. The gaseous mixture leaving the reactor contains mainly SO₂, O₂ and H₂O. The outlet temperature is controlled to satisfy the material balance of the cycle by means of partial condenser (HX-202).

Flow sheet energy requirements and thermal efficiency

The standard formation enthalpy for water is equal to 286 kJ/mole H₂ relative to the formation of liquid water and corresponding to (HHV) of H₂. The theoretical voltage for pure water decomposition is 1.23 V. However, the majority of conventional electrolysis devices need at least 2.0 V when economically reasonable current densities are maintained. This value translates into a water electrolysis Faraday's efficiency of about 74%. If a thermal-to-electric conversion efficiency of 45% is assumed, the total equivalent heat requirement corresponds to a heat input of 859 kJ/mole H₂.

In the proposed flow sheet, a high temperature heat input is required in only three locations: the vaporiser and decomposer of H₂SO₄ (HX-208), the SO₃ catalytic decomposer (HX-209) and the last heat exchanger of concentration step (HX-206). A total of 413.3 kJ/mole H₂ is supplied by the primary heat source. A grand total of 243 kJ/mole H₂ waste heat is rejected to cooling water. Unfortunately the temperature is too low to allow any use of this heat. A total of 120 kJ/mole H₂ electric energy is used in the flow sheet. Nearly 97% of the electric energy is required in Section I with the greatest part for the electrolysis cell. If a thermal-to-electric conversion efficiency of 45% is assumed, the total electric power requirement corresponds to a heat input of 266.7 kJ/mole H₂.

The proposed flow sheet produces hydrogen at a pressure of 10 bar according to the chosen pressure of the electrolysis cell. Compared to the electrolysis of water, whose pressure of hydrogen product is about 1 bar, the power requirement to raise the pressure up to 10 bar is not negligible. The electric power requirement to do such work is about 9.8 kJ/mole H₂. With this output condition, the new heat requirement for VHTR-powered water electrolysis becomes 881 kJ/mole H₂. With a total equivalent heat requirement of 680 kJ/mole H₂, the proposed HyS process flow sheet compares favourably to VHTR-powered water electrolysis.

Capital assessment method

When studying several hydrogen production processes, it can be important to assess the production cost by means of capital, operational and energy costs. The method of capital cost assessment can be selected according to different criteria. These criteria are: current stage of development, innovation level, available time and assessment cost, wished accuracy. First, the choice of the method is based upon the current stage of development. An estimate of the capital expenditures may consist of a pre-design estimate with few process data or a detailed estimate based upon complete drawings and

specifications. However between these extremes of capital investment estimates, there can be numerous methods which can be divided into the following categories (Chauvel, 2000):

- For a pre-design stage:
 - “Order of magnitude estimate” also called “ratio estimate”. This method considers economic data for reference equipment and supposes that required pieces of equipment are very similar to the reference ones (in size range, materials, temperature and pressure operating range, etc.). The order of magnitude estimate uses ratios based upon specific size or equipment capacity. For such an estimate, the accuracy is about $\pm 40\text{-}50\%$.
 - “Study estimate” also called “factored estimate”. When studies are more advanced, the factored estimate may take into account the detailed equipment characteristics. Once the individual costs are determined, they may be multiplied by a number of factors for installation, piping and services, instrumentation and control and eventually by date and geographic location factors. Probable accuracy of estimate is about $\pm 25\text{-}30\%$.
- For construction:
 - “Design estimate” (budget authorisation estimate or scope estimate) based on a semi-detailed analysis. This approach incorporates a mixture of different methods with probable accuracy within $\pm 15/20\%$.
 - “Detailed estimate” (contractor’s estimate) based on complete engineering drawings, specifications and site surveys. Probable accuracy of estimate is about $\pm 5\%$.

Flow sheets of the chemical plant were prepared with a pre-design of the main equipment. For the capital cost assessment, the factored estimate has been chosen because it considers characteristics of the process: corrosive products, high temperature (up to 850°C for Section II) and high pressure (up to 50 bar for the helium coolant). The factored method is essentially based upon charts and formulas developed over 30 years in the petroleum and chemical industries in France (Chauvel, 2000). It consists of estimating costs of basic equipment (generally carbon steel) and correcting them for materials factors.

Exclusively for shell and tube heat exchangers, the Purohit Method has been used (Purohit, 1983). This method takes into account a large number of technical characteristics according to TEMA standard and allows the use of correction factors for different materials. Material factors compared with carbon steel have been updated in 2008 (Gilardi, 2008).

The hybrid sulphur process requires electrolyzers which are not described in chemical engineering economics literature. A specific approach has been developed by collecting data from literature and constructors of alkaline electrolyzers (Mansilla, 2008). Electrolyser characteristics are also considered (catalyst coating, membranes).

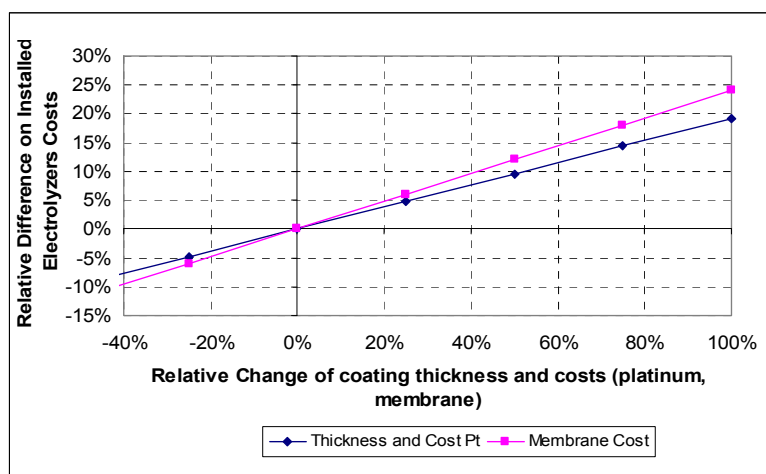
Once the purchased equipment costs are known, the capital investment can be calculated by summing these results and using ratio factors for installation, piping, buildings, instrumentation, electric equipment, thermal isolation, indirect expenses, etc. (Chauvel, 2000).

Economic data of the hybrid sulphur cycle

Considering the equipment design for a plant producing 1 kmol/s H_2 , preliminary calculations suggests unreasonable sizing for the plant. Thus, a plant with 7 to 10 manufacturing facilities is proposed (this number requiring optimisation from an economics standpoint). We consider the hydrogen pressure to be 10 bar. Connection with nuclear reactor is not studied therein.

Due to the lack of SO_2 electrolyzers, alkaline electrolyzers are considered as the reference for economic assessment. Electrodes are assumed to be made of carbon steel with a platinum coating. Electrolysis unit requires an investment of EUR(08) 303.8 M with each electrolyser requiring about EUR(08) 2 920/m².

Coating and membrane costs sensitivities have been studied (Figure 3) in cases of coating thickness or cost doubling (resp. membrane cost doubling). As a result, the installed electrolyzers cost increases up to 20% (resp. 24%).

Figure 3: Cost sensitivities for electrolyzers

Separation SO_2/O_2 requires two absorption columns. Only one column is shown in Figure 2, the second one finishing SO_2 absorption before exhausting (or valorisation of oxygen which is not taken into account therein). This section also requires heat exchangers and compressors. Cost of installed chemical equipment with piping, instrumentation, buildings, etc. is about EUR 223.8 M.

Sulphuric acid concentration, performed in two steps, requires eight heat exchangers, one pump and a knockout drum. A nickel-iron-chromium alloy is used for the majority of equipment and piping due to the presence of hot concentrated sulphuric acid. Capital investment of this section is about EUR(08) 57.3 M (it includes tanks for sulphuric acid).

Concentrated sulphur acid evaporation and dehydration is performed in a group of two heat exchangers with important exchange surface (up to 1 340 m²) (HX-208). The SO_3/SO_2 decomposition reactor (HX-209) is a set of five reactors with two reactive zones. The first one, with a temperature of 875 K requires a platinum catalyst and the second one an iron-oxide catalyst. The operating temperature in the second zone increases up to 1 125 K. Due to operating conditions (temperature, chemical composition), these three devices require a nickel-iron-chromium alloy. Then sulphur trioxide recombination reactor consists of a packed column (HX-210). Required investment for SO_3 conversion is estimated about EUR(08) 508.6 M.

Plant starting requires raw materials, in particular sulphuric acid (1 360 tonnes with a price of EUR(08) 1 200 M per tonne). Battery limits investments for the chemical plant is estimated around EUR(08) 1 095 M, including tanks and sulphuric acid.

Our results can also be submitted in another manner, i.e. showing respective weights of the different equipment. Four major devices represent 88% of the investment: those for SO_3 conversion (HX-208, 28%, HX-209, 16%), electrolysis unit (28%) and compressors/turbines (16%).

Platinum price doubling leads to an increase of 9% of capital investment of the chemical plant due to platinum use in SO_3/SO_2 conversion reactor and for electrolyzers.

Except for shell and tube exchangers, purchased costs were estimated by means of charts from the "Chauvel Method" (Chauvel, 2000), then multiplied by correcting factors (materials for example). Other charts have then been used for purchased costs, the second step requiring correcting factors being the same as previously. These different methods show finally various results with a decrease of about 24% of the total capital investment (Peters, 2003; Ulrich, 2004).

Operational cost

Operational costs include fixed and variable costs per year. The following items have been assessed: maintenance, which is the main contributor, labour and raw materials. Energy costs are not considered in this study. Maintenance is estimated with ratio of capital investment for chemical

application and industrial data for electrolysers (Mansilla, 2008). The maintenance term represents about EUR 73 M/year. Labour costs assessment requires knowledge of wages and operator numbers in a HyS plant. For the first category French data were used and for the second one, analogies with existing chemical and electrolysis units were performed. We obtained a total of about EUR 11.7 M/year. The hybrid sulphur cycle requires water. Off-sites being not included therein, water cost is considered around EUR 1/m³, i.e. EUR 0.5 M/year.

Operational cost, including maintenance, labour and raw materials, represents about EUR 85 M/year. This approach considers HyS process as an ideal one without any losses. The main raw material is sulphuric acid but its price is negligible, in spite of important recent changes. The influence of platinum is certainly more important due to its price and the required quantities: in SO₃/SO₂ conversion reactor and as electrodes coating in electrolysers. Then, time stability of electrode coating is one of the important questions to be solved in future experiments. Considering a loss of 1% platinum during a reconditioning step performed every five years, the supplementary cost to consider is roughly EUR(08) 0.11 M. This value is negligible compared to the other terms. However, it may become more important in case of loss increasing or eventual substitution.

Pilot plant experimentations

With the economic assumptions mentioned above, nearly 30% of capital investment is required for electrolysers. Therefore, our investigations are now focusing on the electrochemical step for which information is still lacking, especially with the SO₂ electrolysers. In order to acquire technical and technological data, CEA has built a pilot electrolysis facility with a hydrogen capacity of 100 NL/h. This pilot plant discussed at ICONE16 (Rivalier, 2008) is designed to handle sulphuric acid solutions with mass concentrations up to 60%, temperatures up to 100°C and pressures up to 10 bar. This pilot plant includes the metered supply of SO₂ in liquid or gaseous form, recirculation flow rates in the range 50 to 1 000 L/h in each electrolysis compartment. Due to the potential risks arising from the operating conditions, the pilot plant is installed in a ventilated cell which allows enough room for a full-scale industrial stack assembly.

The pilot began operating in April 2008. All the tests are currently conducted at room temperature and atmospheric pressure with 20 wt.% sulphuric acid. These tests are performed either:

- with a 64 cm² active area filter-press electrolyser with platinum-coated titanium planar electrodes and cationic exchange membrane (Nafion or Neosepta);
- with a 103 cm² active area proton exchange membrane (PEM) electrolyser using membrane electrode assembly with catalyst sprayed on Nafion membrane, carbon cloth (0.5 mg/cm² Pt loading).

The best results are obtained with the PEM electrolyser: current density of 1 200 A/m² under cell voltage of 1 V corresponding to a hydrogen production of 500 NL.h⁻¹.m⁻². The results from both electrolysers show sulphur deposition at the cathode. This chemical reduction consumes electrons at the expense of hydrogen production causing sulphur to poison the catalyst and modify the membrane conductivity.

So, since the conventional cationic exchange membranes suffer from higher SO₂ transport, CEA has built a test facility to characterise SO₂ transport through membranes. In collaboration with other laboratories, CEA is developing new membranes with reduced SO₂ transport characteristics and high ionic conductivity.

Our test results also show an excessive cell voltage. In order to decrease cell voltage, two approaches have been investigated since early 2009:

- higher activity anodic catalysts;
- porous anodes incorporated in a “flow-through” mode.

Once good performance results with new membranes, new catalysts and porous or PEM electrodes have been achieved, time stability of the components will be evaluated. Future work will focus on improving cell designs.

Accompanying the experimental programme, an increasing effort is today dedicated to the electrochemical process simulation. The modelling task is aimed at: i) understanding the many phenomena involved and their strong coupling; ii) contributing to the design and scale-up of an efficient H₂ production cell (Jomard, 2008; Charton, 2009).

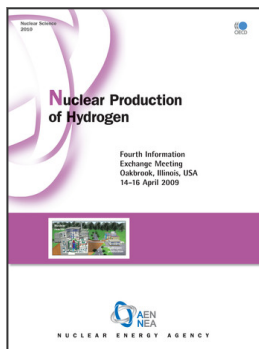
Conclusions

The CEA launched in 2001 an integrated programme to compare the most promising way to produce hydrogen using the high temperature heat available from a VHTR. In order to develop its own expertise on thermochemical cycle assessment, CEA has chosen to develop a scientific approach based on data acquisition (development of devoted devices and specific analytical methods) and modelling (physical models, flow sheet analysis, systemic approach).

Innovative analytical tools and methods have been developed, and dedicated instrumented devices now give access to the necessary reliable data, essential for the optimisation of the process and for the analysis of the potential of the cycle.

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