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► **To cite this version:**

Bouchra Wahdame, Denis Candusso, Xavier Francois, Fabien Harel, Jean-Marie Kauffmann, et al.. Design of experiment techniques for fuel cell characterisation and development. International Journal of Hydrogen Energy, 2009, 34 (2), pp. 967-980. 10.1016/j.ijhydene.2008.10.066 . hal-00952008

HAL Id: hal-00952008

<https://hal.science/hal-00952008v1>

Submitted on 23 May 2024

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- B. Wahdame, D. Candusso, X. François, F. Harel, J-M. Kauffmann, G. Coquery (janvier 2009). *Design of experiment techniques for fuel cell characterisation and development*. Int. J. Hydrogen Energy. Vol. 34, n°2, pp. 967-980. Ed. Elsevier.

Design of experiment techniques for fuel cell characterisation and development

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Abstract

Designs of Experiments (DoE) can be of immediate relevance for various research works conducted in the Fuel Cell (FC) area. DoE techniques allow efficient test definitions for rapid conceptions and well-organised characterisations of FC materials and components, individual cells, stacks or even complete generators. In the DoE method, some statistic-based models can be proposed in pre-stages of physical models. The statistical / numerical relations are used to predict the behaviour of the investigated systems as a function of various operating parameters. Some control strategies can also

be developed to optimise relevant criteria like FC voltage, fuel consumption, maximal electrical power or stack lifetime.

A review of various DoE applications in the field of FC research is first proposed in this paper. Then, some examples of works are detailed. A conceptual typology of different possibilities offered by the DoE methodology in the FC domain is given in the conclusion.

Keywords: fuel cell; experimental design; design of experiment; response surface methodology; ANOVA

Nomenclature

β_0	Arithmetic mean value of the responses in the RSM model
β_i	Factor effects in the RSM model
β_{ii}	Quadratic effects in the RSM model
β_{ij}	Interactions between the factors in the RSM model
$\beta_{1:k}$	Interaction between all the factors in the RSM model
d. f.	Degrees of freedom in ANOVA tables
DwpT	Dew point temperature of air at stack inlet ($^{\circ}\text{C}$)
ε	Fitting error in the RSM model
$E_{A(i)}$	Average effect of the factor A at the level i on the response
$E_z[y(x, z)]$	Mean response in the DRS method
Fair	Air flow rate ($\text{Nl}\cdot\text{min}^{-1}$)
FH ₂	Hydrogen flow rate ($\text{Nl}\cdot\text{min}^{-1}$)
fs	Fisher statistics
FSA	Anode stoichiometry factor
FSC	Cathode stoichiometry factor
I	Fuel cell current (A)
$I_{A(i)B(j)}$	Interaction between the factors A at level i and B at level j
Mean Sq.	Mean squares in ANOVA tables (= Sum. Sq. / d. f.)
P	Pressure of reactants measured at stack inlets (bar), or mode (pressure mode or open mode)
Pair	Air pressure measured at stack inlet (bar)
PH ₂	Hydrogen pressure measured at stack inlet (bar)
R ²	Multiple regression correlation coefficient
Sum. Sq.	Sum of squares in ANOVA tables
t	Time (h)
T	Fuel cell stack temperature ($^{\circ}\text{C}$)
$U_{\text{stack RSM}}$	Stack voltage computed using the RSM model (V)
x^t	Transpose of x
\bar{Y}	Global average of the responses
$\bar{Y}_{A(i)}$	Average of the responses when A is at the level i
$\bar{Y}_{A(i)B(j)}$	Average of the responses when A is at the level i and B at level j
$\bar{Y}_{\text{FCresistance}}$	Arithmetic mean of the response vector elements $Y_{\text{FCresistance}}$ (FC resistance measured at 1kHz)

Subscript

c	Coded value
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1. Introduction

Fuel Cells (FCs) appear as inherently multi-physic and multi-scale objects [1]. To understand their physical behaviours and to improve their performances, various skills and knowledge are needed: chemistry, electrochemistry, fluid mechanics, thermal, electrical and mechanical engineering... Advances in FC research are obtained by conducting a variety of investigations ranging from fundamental domain and material field (with for instance, the development of new catalysts and new electrolytes) to more application oriented works as the optimisation of FC balance-of-plants to fulfil final operating conditions and requirements (e.g. load current cycles linked with dynamical mission profiles for vehicles) [2]. FC performances estimated at different scale levels (i.e. materials, components, single cells, FC stacks, complete FC systems) are generally highly dependant on different physical phenomena from mixed domains. FCs are difficult to model due to their complex non linear multivariate natures. A high number of input factors that contribute to the FC final output voltage could be mentioned. This can motivate Design of Experiment (DoE) approaches rather than or complementary to first-principles / mechanistic models. To give here a concise overview of factors influencing FC performances, the following simple classification can be adopted: properties and sizes of FC materials and components (platinum loading of electrodes, membrane material and thickness, channel design of gas distributor plates...), operating variables (load current, stack and reactant temperatures, pressures, flow and humidity rates...), technological solutions available for the ancillaries (scroll or screw type compressors, reactant humidification sections based on gas bubbling or enthalpy wheels...). To design or to characterise FCs, the approach that consists in “one-factor-at-a-time” experimentations is really time-consuming and thus quite limited. This quite basic method used to find the best combination of factors leading to the desired response consists in holding all factors constant except one, in varying this factor to find its best level, in holding this factor constant and choosing another factor to vary, in repeating these steps until all factors have been varied. The “one-factor-at-a-time” approach is neither appropriate for in-lab testing of large power stacks or for FC durability tests, which imply large reactant consumptions and therefore higher financial costs. Furthermore, modelling the FC electrical or thermal power outputs by considering the input factors without proper statistical methods for the test analyses is not effective since strong interactions between parameters can exist (the “one-factor-at-a-time” approach does not really take into consideration interactions among the factors.). In this way, statistic-based models can be considered before physical models are developed. So, as FC experiments are generally long and expensive, as complex interrelations between physical parameters exist in FCs, the tests have to be carefully implemented and analysed.

The DoE methodology has the potential to allow efficient test definitions for fast and well-organised characterisations of FCs (components, individual cells, stacks or whole generators). One important aim of the DoE methodology is to predict the behaviour of investigated systems as a function of various operating parameters, and then to propose some control strategies for the optimisation of relevant criteria such as FC output voltage, hydrogen consumption, electrical power, or even stack lifetime. Some design or operating parameters can be selected so that the FC or the FC device will work well under a variety of conditions, that is, so that robust performances will be achieved.

This paper is organised as follows: in section two, a brief description of the DoE methodology is provided. The third section is devoted to an overview of FC researches making use of the DoE methodology. In the fourth part, some examples of FC studies are discussed in more detail. They are related with different FC research purposes and they involve diverse DoE tools and techniques. To conclude, a more conceptual typology is presented with the aim of establishing some relations between the problems encountered in FC research area and the potentialities offered by the DoE methodology.

2. General overview of the DoE methodology

The DoE methodology dates from the beginning of the last century with the work of R.A. Fisher (1925). The first users of this methodology were agronomists and biologists who quickly included the interests of the experimental designs into their works, in particular the possible reduction of the number of tests when many parameters are studied. If Fisher can be considered as the pioneer in the use of statistical methods for experimental design, many other scientists have contributed to the development of the DoE methodology, among them: F. Yates, R.C. Bose, G.E.P Box. In the sixties and seventies, many innovations were brought by G. Taguchi [3-10]. His work enabled the dissemination of the experimental designs in the business world. The purpose of the DoE method is to increase the productivity of the experimental process, especially by minimising the number of test runs and by maximising the accuracy of the results. The method allows the significant factors affecting the studied process to be determined and it can also highlight some possible interactions between the various factors. It is a structured, efficient procedure to plan some experiments and to obtain some data, which can be analysed in order to yield valid and objective conclusions about the studied product or process [11-15].

A large number of experimental tests is generally needed to correctly determine the performances of FC systems or to identify the parameters of FC physical models. The choice of an experimental design depends on the objectives of the experiment and on the number of factors to be investigated [16,17]. Several aspects of the overall DoE method are presented and classified below according to different aims.

- Comparative objective: the primary goal of the experimental design is to conclude on one a-priori important factor (in the presence of and/or in spite of other factors). Here, the question of interest is whether or not that particular factor is significant.
- Screening objective: in this case, the purpose of the experiment series is to select or screen out the few important main effects from the many less important ones. Once the key factors are identified by the screening, the Response Surface Method can be used.
- Response Surface Method (RSM) objective: the experiment is designed to allow the estimation of factor interactions and even quadratic effects. Therefore it gives an idea about the (local) shape of the investigated response surface. The RSM designs are used to find improved or optimal process settings, troubleshoot process problems and weak points, and to make a product or a process more robust against external and non-controllable influences [18-21].
- Optimal fitting of a regression model objective: if the experimental response is modelled as a mathematical function (either known or empirical) of a few continuous factors, then the model parameters have to be properly estimated (i.e. in an unbiased way and with a minimal variance) using a multi-linear regression design. Once a suitable approximation for the true functional relationship between the independent

variables and the surface response is found, the optimisation of the response variable can be made.

3. DoE in the field of FC research

As previously mentioned, the DoE methodology has been widely used in the industry for the development of products and to improve the quality of processes. DoE techniques are also broadly applied in many scientific areas with various research aims. In particular, power sources can benefit from DoE procedures, for the performance estimation and/or the design of electrochemical devices such as capacitors or batteries. In that way, statistical approaches as Principal Component Analysis (PCA) and DoE are adopted by L. Ion-Boussier et al. to characterise Li-ion batteries [22,23]. These approaches are found as well-suited for the development of knowledge concerning complex interrelations between physical parameters like load current, battery temperature and depth of discharge, Open Circuit Voltage (OCV), and internal resistance. After the application of ANOVA (ANalysis Of VAriance) procedures, some reduced models of batteries are proposed. In another work done by J. R. Miller and D. A. Evans [24], an ageing study is conducted on double layer capacitors (also called supercapacitors) using experimental designs and in this case, the effects of temperature, voltage and time on the capacitor properties are determined.

In the specific research area of FCs, the DoE methodology can be used for various technological purposes: development of FC materials and components, analysis and improvement of FC single cell and stack performances, evaluation and development of complete FC systems. Different aspects and tools of the DoE methodology can be of great benefit for the works conducted by engineers and researchers involved in the domain of FCs.

3.1. Development of FC materials and components

The experimental designs are extensively applied by chemists, electrochemists and scientists involved in the field of materials. Obviously, the domain of FCs is also of concern. DoE can be used to develop catalysts for PEMFC (Proton Exchange Membrane Fuel Cell) electrodes. In [25,26], some special catalysts containing cobalt are prepared and their properties are analysed using full factorial designs. MEA structures can be optimised through DoE and Artificial Neural Networks (ANNs). As reported in [27], optimisation of layer structure through DoE using Pt-alloy at cathode can lead to significant MEA performance improvements. In [28], the preparation of gas-diffusion electrodes for alkaline FCs is described. A partial factorial design analysis reveals that PTFE content, milling time and their interactions are the important parameters allowing the best performances to be obtained during polarisation curve records. In [29], graphite composite bipolar plates for PEMFCs are evaluated using DoE. Electrical, mechanical as well as molding properties are investigated. Flow channels are fabricated by compression molding with DoE to evaluate moldability. In [30-32], M. Grujicic et al. have developed some multi-physics models of interdigitated air distributors. The cathode geometries are optimised using orthogonal matrix and statistical tools as ANOVA. The parameters of the geometries are selected to reach higher current densities at constant cell voltages over the complete distributor plate surfaces. Moreover, the robustness of the proposed cathode design is examined with regard to uncertainties in a number of model parameters. In [33], the Taguchi method and variance analyses are used to choose the optimal structural combination of a Membrane

Electrode Assembly (MEA). The parameters studied are the following ones: type of Nafion membrane (with different thicknesses), amount of platinum, amount of ionomer and type of support material of Gas Diffusion Layer (GDL), namely carbon cloth or carbon paper. M. G. Medeiros et al have investigated a magnesium semi-FC [34]. Its performances are optimised by the Taguchi method and considering five critical parameters, namely: hydrogen peroxide (H₂O₂) catholyte concentration, electrolyte temperature, catholyte flow rate, electrolyte flow rate and current density. Using the optimised conditions, high voltages and high efficiencies are obtained.

3.2. Analysis and improvement of FC single cell and stack performances

The DoE methodology can be used to evaluate the respective impacts of physical control parameters (such as load current, stack temperature, gas flows and pressures...) on the FC operation. DoE methods and tools, as statistical sensitivity analyses, can be helpful to design and optimise PEMFCs. Today, a growing number of studies in which DoE are used to reach this aim can be found in the literature. In [35], Jill Williams and Adam Krinke have applied DoE techniques to study the behaviour of a PEMFC. The factors investigated are the mass flows and the relative humidity of hydrogen and air, the temperature of air inflow. The maximum power output is considered in this study as the best indicator for the FC performance estimation. In [36], M.G. Santarelli and M.F. Torchio make use of test matrixes to analyse the performances of a single PEMFC. The effects of cell temperature, anode and cathode humidification temperatures, and reactant pressures are investigated. A combined effect of humidification and operating pressure is notably highlighted by the series of tests. Dante et al. [37] have carried out a fractional factorial design of experiments in order to improve the power output of a commercial PEMFC. The roles of four physical factors (both hydrogen and oxygen pressures and flow rates) on the power supply are studied. In [38], the single and combined effects of independent physical variables (i.e. dew point temperatures of anode and cathode flows; cathode flow stoichiometry) on the cogenerative performances of a PEMFC stack have been analysed by M.F. Torchio et al. In [39], the Taguchi method is applied to determine optimum working conditions (maximum power density) for a PEMFC. The parameters explored are the stack pressure and temperature, the oxygen and hydrogen flow rate ratios, the temperatures of the gas humidifiers. Recently, Sheng-Ju Wu et al [40] have presented an integrated approach combining the Taguchi method with Artificial Neural Networks (ANNs) to optimise the operating conditions of a PEMFC. Temperature, pressure, reactant humidity and stoichiometry rates are the control factors and the maximal FC power is the response studied. Orthogonal array with repetitions and Signal-to-Noise Ratio (SNR) are used to collect some experimental data and to determinate the quality characteristics of the test trials. A parametric analysis of PEMFC performances using DoE is also realised by Wei-Lung Yu et al in [41]. Six primary parameters are investigated (FC operating temperature, operating pressure, anode and cathode humidification temperature, anode and cathode stoichiometric flow ratios). In a first stage, a fractional factorial design is used to determine whether these factors have significant effects on the response (maximum electrical power). Then, an orthogonal array of the Taguchi method is adopted to determine the optimal combination of factors. Factorial designs are also used in [42] with the aim to quantitatively understand the positive and negative tradeoffs encountered in miniature FCs. The parameters linked with the flow structures (channel depth and width, conductive film thickness, reactant gas pressure and cell temperature)

are optimised in particular with regard to the volumetric power density. In [43], M. Ordóñez et al. take advantage of the DoE methodology, first to build up a model for a Direct Methanol FC and then to track the maximum power point. Response Surface methodology (RSM) with Central Composite Design (CDD) and Steepest Ascent Method (SAM), parts of DoE methodology, are used in this work. The specific problem of the PEMFC cold start ability can also be apprehended and treated using DoE techniques [44].

Not only experimental works are concerned by the DoE methodology. Experimental design techniques can also be of important benefit in the fields of modelling and simulation. Indeed, DoE are well-suited for the development of FC models and for the analysis of computing results. In this way, a two-dimensional steady-state model developed using Computational Fluid Dynamics (CFD) is presented in [45]. The FC design depends on five factors: channel width, shoulder width, gas distribution electrode thickness, conductivity and porosity. A full factorial design is used to evaluate the main and interaction effects of the factors on the FC performances (current density obtained for constant cell voltages). In [46], a study is performed to find the optimal operating conditions of a PEMFC. The optimisation approach is based on validated multi-resolution FC simulation tool developed in-house. Through DoE methods, a set of designed simulation runs are carried out using the FC simulation tool. From the simulation results, an analytic metamodel is built using the radial basis function approach [46].

3.3. Evaluation and development of FC systems

DoE techniques are of interest for the design and the evaluation of an entire FC generator, i.e. for a system composed of stack(s) and peripheral ancillaries. In [47], the development and evaluation of a FC test stand incorporating various methods for controlling the temperature and humidity of FC reactants is described. In this work, two test stands are built and evaluated. One of them uses steam injection process for the humidification of reactants and is dedicated to FC stack testing. The other gas humidification solution is based on flash evaporation and is adapted to the operation of individual FCs. The capabilities of both humidification systems and the effects of the humidity rates on the FC performances are studied with DoE tools. The tests of various FC system technologies can be scheduled and analysed using DoE. In [48,49], the operation of a 105kWe tubular SOFC generator dedicated to stationary applications is characterised. The effects of operating factors (fuel consumption, air stoichiometry rate) are analysed in terms of voltage and temperature distribution over the investigated stack. In [50], FC generators are first and rightly presented as complex mechanical systems. Then, the design of a FC generator with a gross power output of 64kW is proposed as an illustrating engineering design problem. The multi-stack configuration is investigated using DoE tools. The air stoichiometry rate is taken into account as an operation variable and six geometric dimensions linked with the stack arrangement are considered in the study. The optimisation is realised from a global cost function balancing functional FC generator performances with FC system cost. Two FC system layouts involving different stack numbers and numbers of cells per stack are compared [51-53]. Furthermore, DoE can be used for the sizing of complete FC vehicle powertrains [54]. Through the use of DoE techniques, University of Waterloo's alternative fuels team has developed some vehicle simulations to obtain clear pictures of various powertrain configurations. Two main fuels options are investigated: hydrogen

and bio-diesel. The simulations are run with primary power sources based on these fuels while varying the power capabilities of the individual vehicle components as well as the vehicle architecture. In [55], a framework for surrogate model of Pt catalyst dissolution in PEMFC is developed. The effectiveness and importance of Response Surface Methodology (RSM) in degradation models is discussed. The relative influences of different variables on platinum catalyst dissolution are highlighted and this information can be employed to design control architecture and power management strategies allowing the mitigation of platinum catalyst degradation in FC hybrid systems composed of FC, batteries and supercapacitors.

4. Examples

Three different examples are selected in order to illustrate various aspects and benefits of the DoE methodology in the framework of FC developments [56]. The first example concerns the in-situ characterisation of particular FC components, namely the membranes that serve as electrolytes in PEMFCs. The evolution of the membrane impedance as a function of three parameters is studied using a full factorial design and modelled. The second example deals with the study of a twenty cell stack whose electrical power is subjected to different reactant flow and pressure values. This example is used to highlight the interests of fractional factorial designs. In the third case, a FC durability test is analysed through RSM. The performance variability of the individual cell voltages in the stack is examined by means of a product design (or cross array).

4.1. FC component characterisation and modelling

Low humidified gas conditions lead to worse performances in FC stacks equipped with conventional perfluorinated ionomer (such as NafionTM) membranes. Proper hydration of these membranes is thus critical to maintain their conductivity. The objective of the study proposed in this subsection is to evaluate how the internal resistance (resistances of the membrane) of a PEMFC reacts for various stack temperatures, air dew point temperatures and pressure operation modes [57]. The investigated FC is composed of twenty cells equipped with Gore membranes. Each cell has an active area of 100cm². The nominal current of the FC is 50A. The stack is fed by dry hydrogen and humidified air and tested on a 1kW test bench developed in-lab [58]. The pure resistive impedance, that reflects the membrane water contents, is measured using Electrochemical Impedance Spectroscopy (EIS). This study of the dynamical FC behaviour is carried out in galvanostatic mode, by considering a constant static operation point (polarisation current of 20A) with a small sinusoidal alternating part (amplitude of 4A). The response studied by the DoE methodology is the stack impedance estimated at 1kHz frequency. Each response is measured when the FC reaches stable performances. The physical factors investigated are: the stack temperature (measured on the cooling loop at stack outlet), the air dew point temperature (temperature of air in the humidification section located upstream of the stack) and the gas pressure mode. Three and five levels are respectively adopted for the stack temperature (T variable) and for the air dew point temperature (DwptT) factors. In the experiments, the stack temperature is always higher than the air dew point temperature, so that the occurrence of cell flooding can be avoided. Two pressures modes (P variable) are also considered (Table 1). Indeed, the gas outlets of the stack can be either kept at ambient pressure (Open mode) or the anode / cathode compartment pressures can be both controlled at a fixed value, 1300 mbar abs.

in this study (Pressure mode). Taking into account the number of factors and levels, the full factorial design includes $3 \times 5 \times 2 = 30$ experiments.

Table 1 - Summary of the factors and levels used in the FC internal resistance study. The levels can be expressed either in actual values (i.e. in the true physical ranges) or in coded / normalised centred values (i.e. in the [-1 ; +1] interval)

Factors (X)	Levels				
	Mini. (i = -1)	Intermediate			Maxi. (i = +1)
DwptT [°C]	-1 : 25	-0.5 : 30	0 : 35	+0.5 : 40	+1 : 45
T [°C]	-1 : 50		0 : 55		+1 : 60
P	-1 : Open mode				+1 : Pressure mode

The typical graphs of average effects and interactions (Fig. 1) can be used to compute the effects and the respective contributions of the three physical factors to the FC internal resistance. The average effect of the factor A (here: P, Tstack or DwptT) at the level i (e.g. -1 or +1 for factor P) on the response Y (here: FC internal resistance) is named $E_{A(i)}$ and is computed as follows:

$$E_{A(i)} = \bar{Y}_{A(i)} - \bar{Y} \quad (1)$$

where $\bar{Y}_{A(i)}$ is the average of the responses when A is at the level i, and \bar{Y} is the global average of the responses collected for all the experiments.

The interaction $I_{A(i)B(j)}$ between the factors A at level i and B at level j can be calculated by Eq. (2):

$$I_{A(i)B(j)} = \bar{Y}_{A(i)B(j)} - \bar{Y} - E_{A(i)} - E_{B(j)} \quad (2)$$

where $\bar{Y}_{A(i)B(j)}$ is the average of the responses when A is at the level i and B at level j.

All the tools that enable the calculation and the graphic representations in Fig. 1 are developed in the MatlabTM environment. On these graphs, with regard to the effect of the A factor, the greater the slope of the segment $[\bar{Y}_{A(-1)} \bar{Y}_{A(+1)}]$, the larger is the influence of the A factor [3,9]. The interaction is physically influential when the lines are not parallel. On the one hand, the low slope of the (red) segment plotted for the average effects of the pressure mode means that this parameter has a poor impact on the resistance evolution. On the other hand, the high slopes of the segments related to the stack and air dew point temperatures reveal the important impacts of these factors.

Moreover, the graph displayed for the dew point factor shows that its effect is almost linear in the selected temperature range. Note that this property could lead to a possible reduction of the test number needed and therefore allow saving time for the FC experimenter. Indeed, if the effect of the air dew point temperature factor is considered as linear, the lowest and highest level values only have to be taken into account for this parameter (i.e. 25°C and 45°C). With this hypothesis, the number of experiments could be reduced to $3 \times 2 \times 2 = 12$ runs. Actually, the duration of the resistance measurement is short (it takes a few seconds only) and thus the gain in terms of time saving would

probably be insignificant. But the time needed to reach the DwptT target temperature from one test run to the other can be significant (about 10min for a change of 5°C) due to the time constants linked with the thermal phenomena involved in the FC test bench (limited dynamics of the dew point control in the air humidification section of the test stand).

In Fig. 1, the stack temperature * air dew point temperature interaction (T * DwptT) is also detected by the difference between the slopes of the related segments. A statistical analysis (ANOVA) [3,9,59] could also highlight a strong and significant interaction between the stack temperature and the air dew point temperature factors [57]. The importance of this interaction can quite easily be understood or even guessed from a physical point of view. As the stack temperature is here always higher than the air dew point temperature, a larger gap between the two temperatures should lead to a drying of the stack and therefore to a more elevated internal resistance value.

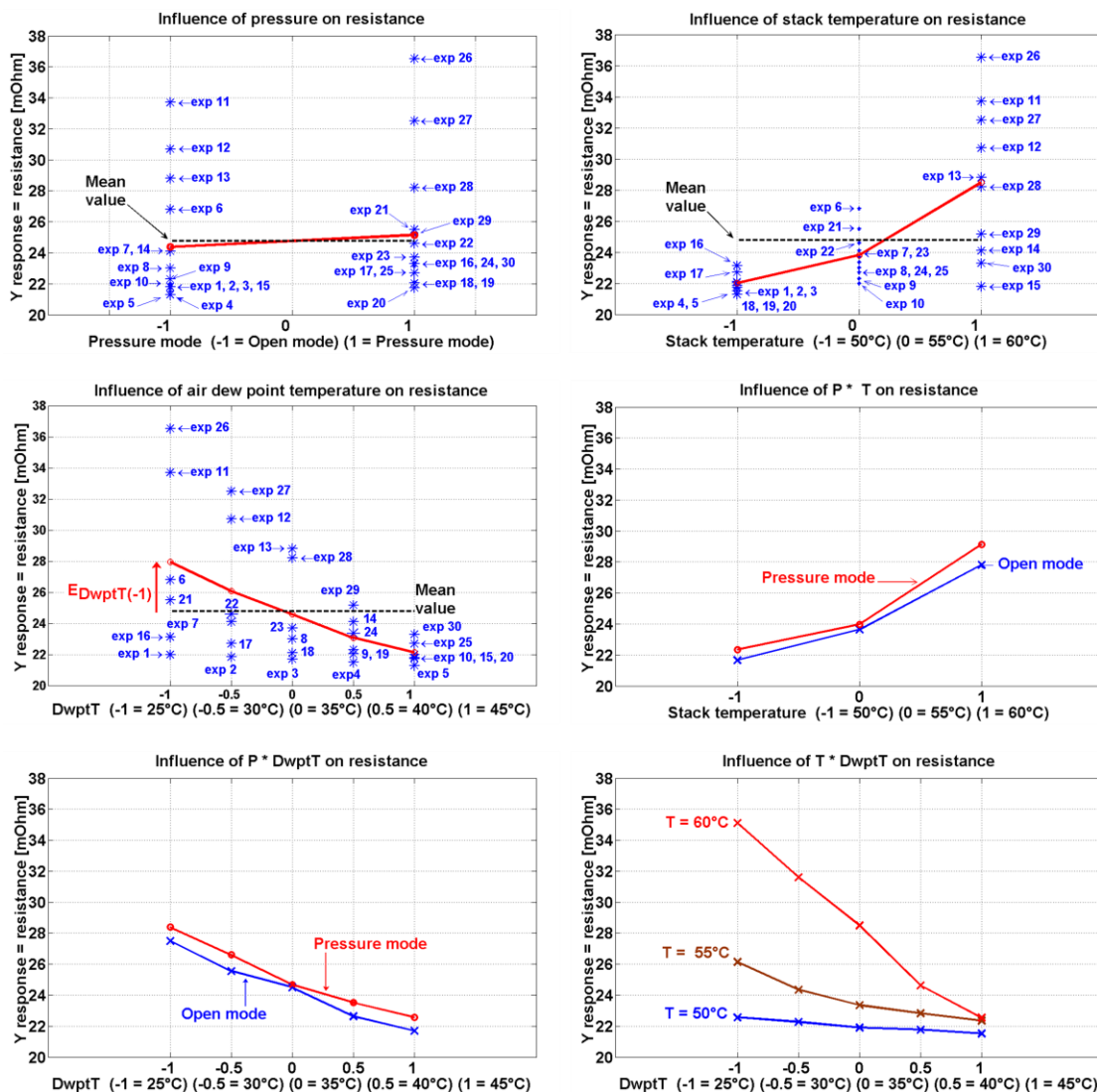


Fig. 1 - Graphs of the average effects for the stack temperature, air dew point temperature and pressure mode factors. Interactions between the factors.

A matrix model (Eq. 1) can be proposed for the expression of the internal resistance ($Y_{FC \text{ resistance}}$) as a function of the three considered parameters. The model is expressed in “coded form” as follows:

$$\begin{aligned}
Y_{FC \text{ resistance}} = & \bar{Y}_{FC \text{ resistance}} + [E_{P(-)} \ E_{P(+)}][P] + [E_{T(-)} \ E_{T(0)} \ E_{T(+)}][T] \\
& + [E_{DwptT(-)} \ E_{DwptT(-0.5)} \ E_{DwptT(0)} \ E_{DwptT(+0.5)} \ E_{DwptT(+)}][DwptT] \\
& + [P]^t \begin{bmatrix} I_{P(-)T(-)} & I_{P(-)T(0)} & I_{P(-)T(+)} \\ I_{P(+)T(-)} & I_{P(+)T(0)} & I_{P(+)T(+)} \end{bmatrix} [T] \\
& + [P]^t \begin{bmatrix} I_{P(-)DwptT(-)} & I_{P(-)DwptT(-0.5)} & I_{P(-)DwptT(0)} & I_{P(-)DwptT(+0.5)} & I_{P(-)DwptT(+)} \\ I_{P(+)DwptT(-)} & I_{P(+)DwptT(-0.5)} & I_{P(+)DwptT(0)} & I_{P(+)DwptT(+0.5)} & I_{P(+)DwptT(+)} \end{bmatrix} [DwptT] \\
& + [T]^t \begin{bmatrix} I_{T(-)DwptT(-)} & I_{T(-)DwptT(-0.5)} & I_{T(-)DwptT(0)} & I_{T(-)DwptT(+0.5)} & I_{T(-)DwptT(+)} \\ I_{T(0)DwptT(-)} & I_{T(0)DwptT(-0.5)} & I_{T(0)DwptT(0)} & I_{T(0)DwptT(+0.5)} & I_{T(0)DwptT(+)} \\ I_{T(+)DwptT(-)} & I_{T(+)DwptT(-0.5)} & I_{T(+)DwptT(0)} & I_{T(+)DwptT(+0.5)} & I_{T(+)DwptT(+)} \end{bmatrix} [DwptT]
\end{aligned} \tag{3}$$

where:

$\bar{Y}_{FC \text{ resistance}}$ is the arithmetic mean of the response vector elements $Y_{FC \text{ resistance}}$,

$E_{A(i)}$ is the effect of factor A at i level on the response $Y_{FC \text{ resistance}}$,

$I_{A(i)B(j)}$ is the interaction between the factor A at i level and the factor B at j level,

each factor vector [A] can be expressed according to the level considered. Let us take the example of factor vector [P]. Then, [P] can be equal either to $[1 \ 0]^t$ or $[0 \ 1]^t$.

With the numerical values, the model of Eq. (3) can be written as follows:

$$\begin{aligned}
Y_{FC \text{ resistance}} = & 24.7635 + [-0.38688 \ 0.3868][P] + [-2.7575 \ -0.9585 \ 3.716][T] \\
& + [3.1789 \ 1.3115 \ -0.1785 \ -1.6885 \ -2.6235][DwptT] \\
& + [P]^t \begin{bmatrix} 0.0508 & 0.2218 & -0.2727 \\ -0.0508 & -0.2218 & 0.2727 \end{bmatrix} [T] \\
& + [P]^t \begin{bmatrix} -0.0557 & -0.1382 & 0.3014 & -0.0548 & -0.0532 \\ 0.0557 & 0.1382 & -0.3018 & 0.0548 & 0.0532 \end{bmatrix} [DwptT] \\
& + [T]^t \begin{bmatrix} -2.62 & -1.0425 & 0.0725 & 1.4525 & 2.1375 \\ -0.8340 & -0.7665 & -0.2765 & 0.7085 & 1.1685 \\ 3.4540 & 1.8090 & 0.2040 & -2.1610 & -3.3060 \end{bmatrix} [DwptT]
\end{aligned} \tag{4}$$

Such a model based on the DoE method contains valuable and quantitative information about the effects of the investigated parameters on the response, and about possible interrelations between the factors studied. Besides, this model can also be incorporated as a sub-model into a global and physical FC system model. The sub-model would then represent local and particular phenomena linked with the membrane impedance which may not be easily described using a mechanistic approach.

From Eq. (4), a more straightforward expression of the FC internal resistance can be deduced. Eq. (5) is computed by considering the factors linear inside the interval [-1 1] and by adopting true actual values for the factors (for instance, $T = 60^{\circ}\text{C}$):

$$Y_{\text{FCresistance}} = 24.763 + 0.387 \cdot P + 3.237 \cdot T - 2.921 \cdot \text{Dwpt}T + 0.162 \cdot P \cdot T - 0.018 \cdot P \cdot \text{Dwpt}T - 2.95 \cdot T \cdot \text{Dwpt}T \quad (5)$$

4.2. Optimisation of FC operation and fractional factorial design

The performances of a 20 cell PEMFC stack are investigated. The FC characteristics are similar to those described in the previous subsection. We only point out the fact that this stack has to be operated at atmospheric pressure (a maximal pressure of 500mbar rel. is authorised). The objective of the DoE proposed is to better appreciate the impacts of the reactive gas flows and pressures on the maximal electrical power delivered by the stack. So, the factors considered are: hydrogen pressure (PH2), air pressure (Pair), hydrogen flow rate (FH2) and air flow rate (Fair). The response is the maximum FC output power. The levels adopted for the four factors are selected taking into account the technological limits of FC stack and test bench (Table 2). The factors can be considered as linear because of the quite restricted ranges of pressures and gas flows investigated which let the FC operate in a homogeneous physical domain (note that this hypothesis can be verified afterwards by performing one or several additional tests inside the experimental domain explored, at intermediate levels). The full factorial design includes therefore $2^4 = 16$ experiments (four factors with two levels each).

Table 2 - Full factorial format for four factors with two levels (actual and coded values)

Test Nr.	PH2 -1 : 1.3 bar abs. +1 : 1.4 bar abs.	Pair -1 : 1.3 bar abs. +1 : 1.4 bar abs.	FH2 -1 : 5.5 NI min ⁻¹ +1 : 8.5 NI min ⁻¹	Fair -1 : 26.3 NI min ⁻¹ +1 : 39.6 NI min ⁻¹	PH2 * Pair * FH2 * Fair
1	-1	-1	-1	-1	+1
2	+1	-1	-1	-1	-1
3	-1	+1	-1	-1	-1
4	+1	+1	-1	-1	+1
5	-1	-1	+1	-1	-1
6	+1	-1	+1	-1	+1
7	-1	+1	+1	-1	+1
8	+1	+1	+1	-1	-1
9	-1	-1	-1	+1	-1
10	+1	-1	-1	+1	+1
11	-1	+1	-1	+1	+1
12	+1	+1	-1	+1	-1
13	-1	-1	+1	+1	+1
14	+1	-1	+1	+1	-1
15	-1	+1	+1	+1	-1
16	+1	+1	+1	+1	+1

Each one of the 16 experiments is performed as follows. The stack is operated at atmospheric pressure with humidified air (dew point temperature of 25°C) and dry hydrogen. Air and hydrogen flows are controlled by flow regulators located upstream of the stack. Pressures at stack inlets are controlled using back pressure valves placed

downstream of the stack. The stack temperature is regulated at 55°C. During each test, the hydrogen and air flow rates are kept constant. For example, during the experiment presented in Fig. 2, their values are respectively equal to 8.5NI min⁻¹ and 39.6NI min⁻¹. The hydrogen and air pressures are controlled gradually with the back-pressure actuators as the load current is incremented by steps of a few Ampere. The FC output power increases progressively and so does the voltage drop-down of the stack. When any one of the 20 cell voltages reaches the threshold of 380mV, the experiment is stopped. The maximal electrical power reached by the stack during the test is recorded.

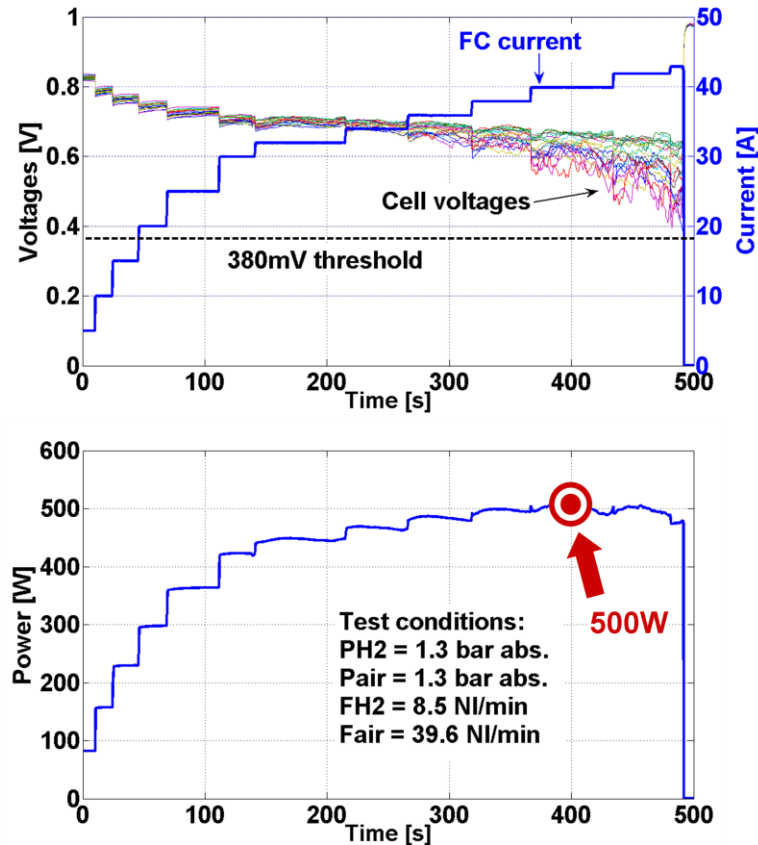


Fig. 2 - Evolutions of parameters (FC current and cell voltage. FC power) during experiment Nr. 13.

In order to compare the effects of the four factors, some graphs of average effects can be used [60]. Statistical sensitivity analyses (ANOVA) can also be used to compute the effects and the respective contributions of the physical factors to the FC maximal power. The ANOVA is a commonly used tool to study and to estimate the factor influences over a process [3,9,59]. It is also used in order to determine if these effects are significant or if these effects are only the expression of the system variability due to not controlled factors (not studied in the design). The ANOVA enables to determine starting from which threshold the effects of factors can be regarded as significant. An ANOVA is done from the 16 tests and maximal powers measured (Table 3). Only first degree interactions between two factors (PH2 * Pair for instance) are taken into account. The ANOVA table has seven columns [59]. The first one shows the source of the variability. The second one shows the sum of squares (Sum. Sq.) due to each source. The third one includes the percentage of contribution for each factor. The fourth one

represents the degrees of freedom (d. f.) associated with each source. The fifth one contains the mean squares, which is the ratio: Sum. Sq. / d. f.. The sixth one shows the Fisher statistics (fs), which is the ratio of the mean squares. The seventh one shows the p-values for the Fisher statistics. The choice of a limit for the p-value, in order to determine whether or not a result is "statistically significant", depends on the application. It is common to declare a result significant if the p-value is less than 0.05 or 0.01 [3,9,59]. The ANOVA of Table 3 shows that the air flow rate (Fair) is the most important factor with a contribution equal to 88.5% of the total variance. The effect of the factor FH2 is the second most significant. The influences of reactant pressures on the maximal power obtained from the FC are low and not statically significant (p-values higher than 0.05) in the experimental domain considered. This study demonstrates that the investigated FC technology needs high stoichiometry rates to operate properly. Especially air flow participates actively to the evacuation of the water produced in the tested FC. This example shows that the DoE method, with its tools (as ANOVA) and models, allow highlighting the relative impacts of the various physical factors considered. DoE techniques can thus be considered as a suitable medium to make well-founded assumptions regarding the different influences of the investigated parameters and to evaluate the level of complexity related with a physical FC model intended for a given application.

A fractional factorial design is now extracted from the full design. A fractional DoE includes selected combinations of factors and levels. It is a carefully prescribed and representative subset of a full factorial design. A fractional factorial DoE is particularly useful when the number of potential factors is large because it reduces the total number of experiments required. A fractional design has to check the following properties: the first one is orthogonality and the second one is linked to the degrees of freedom [16]. In the study, the full factorial design requires 2^4 experiments where 4 is the number of two level factors. From the full factorial design, it is now possible to reduce the runs to 8 ($= 2^{4-1}$) and to generate a fractional factorial design by keeping the tests of Table 2 for which $PH_2 * Pair * FH2 * Fair = +1$ (grey rows of Table 2) [9]. ANOVA is carried out for the 8 maximal powers selected. The interactions are not taken into account (Table 4). It can be noted that the results obtained with the fractional design are close to those computed using the full factorial design. A very significant effect of the parameter air flow rate (Fair) is notably found. It represents 91% of the total of contributions. Since each experimental run takes about 10min (Fig. 2), the total duration required to complete the fractional factorial design is approximately 80min, which is half of the time needed to achieve the full design. Obviously, the benefit of the method to save experiment time would be still greater for longer test runs. The approach shall especially be adopted in the framework of durability studies involving long-term tests to be conducted for a variety of investigated physical parameters and operating conditions. Additional information concerning the work proposed in this subsection can be found in [56,60]. Besides, a similar study has been proposed by Dante et al. in [37].

Table 3 - ANOVA for the full DoE (16 maximal power measurements)

Source	Sum Sq.	%	d. f.	Mean Sq.	fs	p-value
PH2	749.4	0.8	1	749.4	2.99	0.144
Pair	199.5	0.21	1	199.5	0.8	0.413
FH2	5531.6	6	1	5531.6	22.1	0.005
Fair	82010	88.5	1	82010	327	≈ 0
PH2 * Pair	21.4	2e-3	1	21.4	0.09	0.781
PH2 * FH2	78.8	5e-3	1	78.8	0.31	0.599
PH2 * Fair	8.3	≈ 0	1	8.3	0.03	0.863
Pair * FH2	28.9	0.03	1	28.9	0.12	0.747
Pair * Fair	213.9	0.23	1	213.9	0.85	0.397
FH2 * Fair	2512.5	2.71	1	2512.5	10	0.024
Error	1252.1	1.35	5	250.4		
Total	92607		15			

Table 4 - ANOVA table for the fractional factorial design

Source	Sum Sq.	%	d. f.	Mean Sq.	fs	p-value
PH2	247.5	0.52	1	247.5	0.48	0.538
Pair	52.5	0.11	1	52.5	0.1	0.770
FH2	2397.8	5	1	2397.8	4.64	0.120
Fair	42997.8	91	1	42997.8	83.26	0.003

4.3. FC durability and robust FC operation

As stated in Section 3, a variety of works involving the DoE methodology has already been devoted to the study of FC behaviours. In these works, the FC performances are generally analysed during limited time periods and so far, only little information is available concerning specific FC lifetime problems addressed using DoE techniques. As an example of work, the study made by Rajeswari Chandrasekaran et al. can be mentioned [55]. To illustrate the problem of FC durability, the results of an ageing test performed during 1000 hours on a PEMFC and analysed using DoE techniques are presented in this subsection. The RSM is adopted to model the performance degradation over ageing time from various load current – FC voltage curves recorded at regular time-spaced intervals and for various air utilisation rates. In addition, the Dual Response Surface (DRS) approach can be employed to determine the most convenient operating conditions for the cells (load current, air stoichiometry rate levels), leading to a trade-off between high electrical efficiency and low voltage variability versus ageing time and cell positions in the stack.

The investigated PEMFC is analogous to those depicted in the previous subsections (e.g. 50A nominal current) but in this case, the stack has three cells only ($3 \times 100\text{cm}^2$ active cell areas). Some detailed information concerning the test protocol, the characterisation procedures, the cell voltage evolutions over the 1000h can be found in [61,62]. Here, the test analysis is made from polarisation curve data recorded throughout ageing time and for anode/cathode stoichiometry factors (FSA/FSC) equal to 2/5, 2/4 and 2/3. Each polarisation record was realised by incrementing the FC current from 0 to 70A. When either the current value was 70A or when any one of the three cell voltages reached a minimum threshold of 380mV, the polarisation curve test

was stopped. The set of polarisation curves recorded for FSA/FSC=2/5 during the various characterisation sequences is shown in Fig. 3 using a three-dimensional (ageing time – FC current – FC voltage) shaded surface. The projection of the surface, a contour plot beneath this one, is drawn in the time – current plane. This plot is like topological maps, which show elevation versus both longitude and latitude. However, instead of elevation, the map here shows the levels of the measured FC voltage versus two variables: time and load current. An improvement of the stack voltage can be noticed for the first 100 hours. The next 200 hours correspond to a period of relative performance stability. Some degradation appears between the 300th and 400th hour. The small peaks on the voltage contours can be attributed mainly to the effects of the characterisation procedures. Similar response surfaces could obviously be displayed for other gas stoichiometry rates. A graph such as Fig. 3 can provide a first good and sufficient visual representation of the FC ageing. However, it is not really suitable either if the optimal FC operating conditions leading to the highest stack voltage versus time have to be found, or if the impacts of time and cell position in the stack have to be properly estimated. It is also obvious that a large amount of data has to be analysed if the number of factors (with levels) is considered: ageing time with 16 characterisation sequences (at $t = 0\text{h}$, $t = 101\text{h}$, $t = 190\text{h}$, ... $t = 923\text{h}$, $t = 995\text{h}$) [61,62], number of cells equal to three, load current and cathode stoichiometry rate (FSC) ranges. To overcome these issues, some numerical / statistical models of the FC degradation are needed. The RSM can be used to this end.

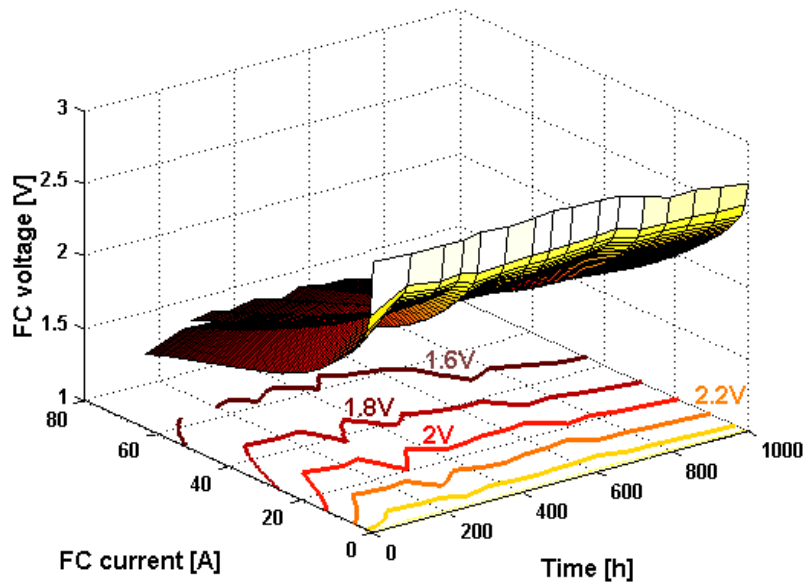


Fig. 3 - Display of polarisation curve set using three-dimensional shaded surface (FSA/FSC = 2/5).

In the study, the experimental design factors considered are the ageing time (t), the FSC and the load current (I). The output voltage of the FC stack ($U_{\text{stack RSM}}$) is the response. The levels adopted for the factors are summarised in Table 5.

Table 5 - Levels of the investigated factors

Factors	Levels			
	Mini.	Intermediate	Maxi.	Number
t	0h	Characterisation instants	1000h	16
FSC	3	4	5	3
I	0A	Steps of 10A	70A	8

One simple and possible response surface equation used in the RSM methodology and relating the studied response y and the i factors x is given by Eq. (6).

$$y = \beta_0 + \sum_{i=1}^k \beta_i \cdot x_i + \sum_{i=1}^k \beta_{ii} \cdot x_i^2 + \sum_{i=1}^k \sum_{j>i}^k \beta_{ij} \cdot x_i \cdot x_j + \beta_{1:k} \prod_{i=1}^k x_i + \varepsilon \quad (6)$$

where β_0 is the arithmetic mean value of the responses, β_i correspond to the factor effects, β_{ii} are the quadratic effects, β_{ij} are the interactions between the various factors, and $\beta_{1:k}$ is the interaction between all the factors. The ε variable stands for the fitting error. The quadratic model for the FC stack voltage generated by the design adopted in the study (depicted by Table 5) has the form of Eq. (7). (multiple regression correlation coefficient R^2 equal to 0.9736). Some normalised centred representations or coded values (hence the “c” index used for the variables in Eq. (7)) are adopted for the factor levels. The -1 and +1 values are respectively related to the minimal and maximal levels. The regression used for the estimation of the polynomial parameters can be made using the *rstool* function of MatlabTM [59]. This function performs the interactive fit and plot of a multidimensional response surface. Even though the model proposed cannot be considered as a physical / mechanistic model (indeed, it is rather a statistical model), it can be helpful to clearly dissociate the respective impacts of the various physical parameters over the investigated response. For instance, the approach allows the dissociation of the cell voltage drop due to the ageing time from the voltage evolutions caused by the setting of different operating conditions (e.g. load current and FSC values). Again, the DoE methodology appears to be particularly well-suited to model complex phenomena (like FC ageing process) which are really difficult to represent through pure mechanistic approaches.

Then, the relationship between the investigated variables can be elucidated using the predicted model (Eq. (7)) together with contour plots (Fig. 4).

$$\begin{aligned} U_{\text{stack RSM}} = & 1.811074 - 0.118371 \cdot t_c + 0.029318 \cdot \text{FSC}_c \\ & - 0.621122 \cdot I_c - 0.061091 \cdot t_c^2 - 0.013025 \cdot \text{FSC}_c^2 + 0.323302 \cdot I_c^2 \\ & + 0.063182 \cdot t_c \cdot \text{FSC}_c - 0.066317 \cdot t_c \cdot I_c + 0.043855 \cdot \text{FSC}_c \cdot I_c \\ & + 0.045029 \cdot t_c \cdot \text{FSC}_c \cdot I_c + \varepsilon \end{aligned} \quad (7)$$

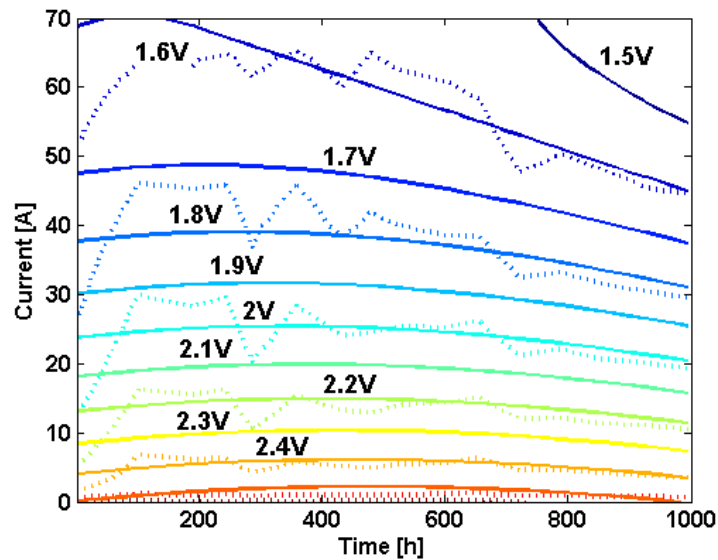


Fig. 4 - Example of contour plots displayed for the experimental measurements (dotted lines) and for the full quadratic model with $FSC = 5$ (full lines).

The model of Eq. (7) can be used to find the optimal air stoichiometry rate (FSC) leading to the highest FC voltage in the time and current ranges explored. The programming problem satisfying the constraint $3 < FSC < 5$ can be solved using the *quadprog* Matlab™ function [59]. Figure 5 shows the results of the optimisation through the contour plot of the response surface: optimal FSC versus time and load. $FSC = 3$ allows the best performances to be obtained at the beginning of the ageing test and for relatively low current values. Then, with time, $FSC = 5$ becomes necessary to reach the highest stack voltage. This result could be taken into account for instance to design a compressor feeding the FC in air. Indeed, the need of air flow increases with time so that the power consumption of this ancillary will be higher at FC end of life.

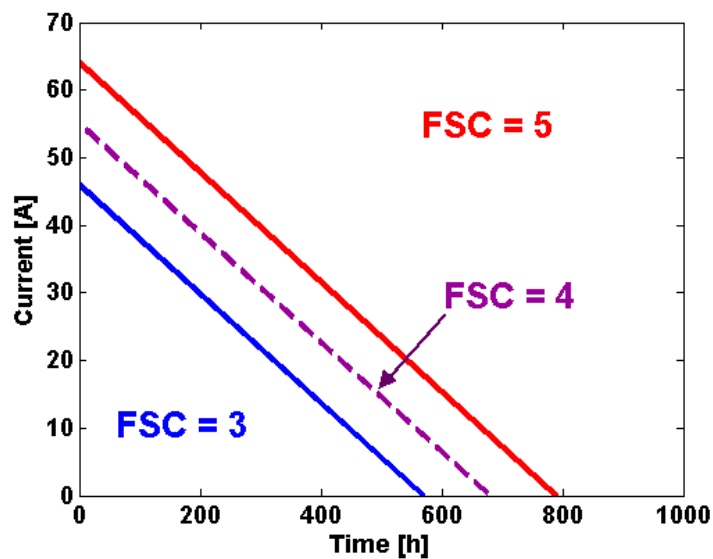


Fig. 5 - FSC leading to the highest FC voltage, vs. ageing time and load current. Contour plot of the computed response surface.

One other possible aim consists in finding the optimal operating conditions that allow both high stack voltage and lower FC voltage variability throughout ageing time. Thus, appropriate load current and FSC levels will be selected to reach high FC voltages and to reduce the influence of operating time on the FC performances. In this case, ageing time can be considered as a noise source and the RSM / Dual Response Surface method (DRS) can be used to solve our robust parameter design problem. Indeed, the DRS approach is very effective in optimising a process by choosing the right levels of controllable factors, so that the needed solution is close to the target and also as robust as possible, i.e. with minimum variation [20,63]. Actually, the DRS method is strongly linked to the crossed array concept. The crossed array, or product array, allows the data collection. It begins with two experimental designs, one for the noise variables and one for the control variables. These designs are then crossed with each other. In our study, the response y refers to the FC stack voltage U_{stack} . The control factors are the load current (I) and the air stoichiometry rate (FSC). These two factors form an (8×3) inner array, while the noise or outer array involves the ageing time (t). The levels considered for the factors are summarised in Table 5. The total experimental design is here a $(8 \times 3) \times 16$ crossed array (Table 6).

Table 6 - Crossed array used for the data collection

Inner array, two control factors: x			Outer array, one noise factor: z			
			n°	(1)	(2)	(16)
n°	FSC	I	t	H0	H101	H995
(1)	3	0		y ₁₋₁	y ₁₋₂	y ₁₋₁₆
(2)	3	10		y ₂₋₁	y ₂₋₂	y ₂₋₁₆
(3)	3	20		y ₃₋₁	y ₃₋₂	y ₃₋₁₆
(22)	5	50		y ₂₂₋₁	y ₂₂₋₂	y ₂₂₋₁₆
(23)	5	60		y ₂₃₋₁	y ₂₃₋₂	y ₂₃₋₁₆
(24)	5	70		y ₂₄₋₁	y ₂₄₋₂	y ₂₄₋₁₆

In the DRS approach, two empirical models are then established to describe the mean and variations in the response. The theoretical development of the general model equations is discussed in [20]. The two empirical relations are used simultaneously to achieve the desired mean response with minimal variation. To reach this aim, the mathematical relations are used in order to display the mean and variance response surfaces. Then, the contour plots of constant mean and constant variance are displayed together on the same figure. To finish, a graphical analysis can be made. The different contour plots are used in harmony to find suggested operating conditions.

Figure 6 shows the DRS brought back to the power of one mean single cell. The contour plots of the mean and variance responses are based on mathematical relations given in [62].

The optimisation of the FC operation can be made by considering some selected electrical power levels to be delivered by the FC. In this case, the optimal FSC and load current values have to be selected on the one hand to reach the desired power level and on the other hand to reduce the impact of ageing time on the FC performance (low variance of the power delivery, i.e. high robustness of the FC performance regarding the operating conditions). So, the optimisation is made considering a double criterion. The

first one is related with the voltage efficiency. For a given power value: the higher the stack voltage is, the lower the load current value. The second one is linked with the performance variability (power variance). Still for a given power level: the lower the performance variance is, the higher the robustness of the fuel cell facing the investigated test conditions (i.e. lower probability to get an unsteady stack voltage obviously linked with a bad FC state-of-health). The optimisation is made inside the experimental domain explored. That means: $3 < FSC < 5$ and load current ranging from 0 to 70A. No other particular constraints have to be taken into account for the optimisation. Since two different criteria have to be taken into account, a multi-objective mathematical function could be defined in order to solve the problem. However, a pure graphical method, as simple as it is practical, will be preferred here to find the optimal load current and FSC. For example, if the objective value of 30W is chosen for the cell power, then Fig. 6 shows that it is better to operate the FC at 57A to get higher stack electrical efficiency (i.e. higher cell voltage). For this current value, $FSC = 5$ will also reduce the impact of ageing time over the stack voltage variability.

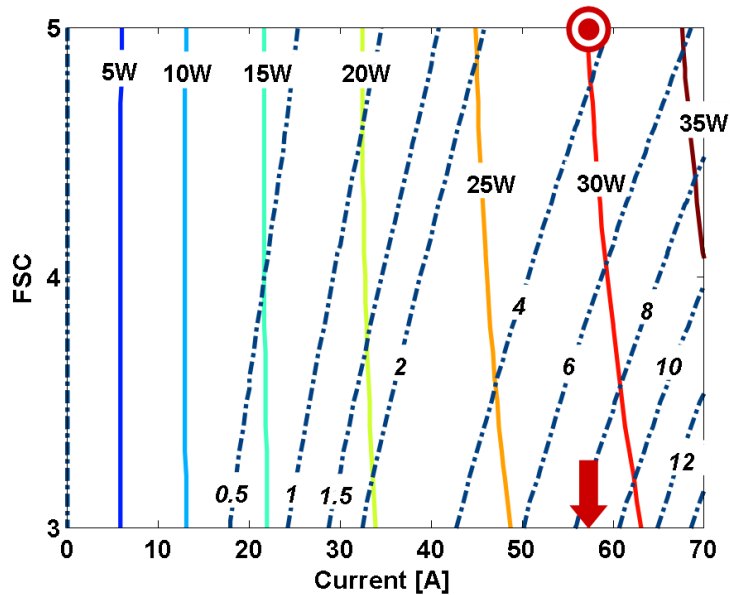


Fig. 6 - DRS computed for the power of one mean single cell (ageing time is the noise parameter).

Not only ageing time can be considered as a noise factor. If the goal is to have a robust FC operation, by avoiding too large cell voltage deviations linked for instance with electrode membrane assembly flooding or drying, the cell position in the stack (i.e. cell number) can be selected as an additional noise factor. As a matter of fact, a lower dispersion in the cell voltage distribution can be considered as a proof of better FC state-of-health. A similar approach to the one used previously can be adapted with the aim of reducing the variability between the cell voltages (three cells for the investigated FC). Again, the control factors will be the FC current and FSC. They will form an (8×3) inner array while the noise or outer array will involve both ageing time and cell position in the stack. Then, the total design will be a $(8 \times 3) \times (16 \times 3)$ crossed array [62].

5. Conclusion

The DoE methodology can be of great benefit for the research in the FC area. As shown in this article, DoE has already proved to be noteworthy for a variety of purposes: development of FC materials and components, optimisation of FC stacks and systems (enhanced design, improved operating conditions, more efficient diagnosis of FC state-of health). DoE techniques can be first applied to organise the tests in a rational way to save time during the experimental characterisation stages but also during the modelling phases in particular when time-consuming simulations due to model complexity are encountered. The DoE methodology with its statistical techniques is then well-suited to analyse the tests conducted on FCs. Indeed, the DoE methodology offers a wide range of practical tools, graphical representations and techniques that can be suitable mediums for FC experimenters and developers. The DoE approach leads to simple and precise models which highlight the impacts of the factors on the response and detect possible interactions between parameters. The DoE based models can be incorporated as sub-models into global physical models to represent a local or a particular phenomenon which cannot be directly and easily described using a mechanistic approach (like the FC ageing process). One other advantage of the models based on the DoE methodology is that they can easily be used for optimisation purposes. Some examples of designs, statistical instruments are reported in Table 7 to give a concise overview of various possibilities offered by the DoE in the FC field.

To conclude, the DoE exercise should be considered as an intermediate and necessary stage between the pure experimentation processes and the next physical modelling phases. With the proposed approach, some mechanistic models can be developed in a more efficient way since some valid hypotheses can be made regarding the different influences of the various investigated parameters.

Table 7 - Brief typology of DoE techniques and tools suitable for advances in FCs

Applications	Designs, techniques and tools	Examples
Preliminary characterisation of FC (material, component, cell, stack, system). Determination of parameter effects and possible interactions. Statistical sensitivity analysis	Screening design (preferably: fractional design with a few additional runs to avoid a full fractional design)	[28,31-39,45,60]
	Graphs of effects and interactions	[33-35,44,45,57,60]
	ANOVAs	[28,30,31,33,37,41,57,60]
	Normal plots	[49,57]
Characterisation of FC with a focus on a few parameters only. Statistical / behavioural modelling.	Response Surface Method (RSM) RSM models	[43,44,48-50,53,55,61,62,64,65]
	Modelling combining DoE with ANNs	[25,26,40]
	Optimal design for the estimation of parameters in empirical model	[66]
Check the repeatability, reproducibility and quality of the tests.	Designs with repetitions. Products designs. Crossed arrays.	[28,29,40,41]
	Signal-to-Noise Ratios (SNR)	[29,40]
Quality and robustness problems. Performance variability through ageing time. Investigations related to spatial distribution of phenomena in FCs.	Crossed arrays, Dual Response Surfaces (DRS)	[62]

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