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Optimal design of experiments for the modelling of food processes

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ABSTRACT

In this paper, we present an original approach and implementation of the A-, D- and E-optimality on the estimation of unknown coefficients in dynamic models. This approach is validated over 4 applications, 2 of which being taken from literature: (A) Reproduction of the pharmacokinetic results from [1]: diffusion of a drug between 2 compartments; (B) Reproduction of the fermentation results from [2], non sequential optimal design of 6 fermentations trials. (C) Order 1 reaction of ascorbic acid in stewed apples; (D) Rice drying, most complex case with no analytical solution: optimal use of experimental device to identify unknown heat and mass transfer coefficients

The objective is to diminish the experimental effort needed to make this identification within acceptable confidence ranges. After each experiment, the next experiment is A-, D- or E-optimally designed. Our design of experiments can take into account all experimental constraints and experimental results of all previous experiments to calculate best experimental conditions and to obtain smallest uncertainties on estimated parameters. In all contexts (A-D), this methodology is applied to a simulated noised experiment, and its stability and convergence is shown to be effective. In the last context (D), this methodology is also applied to a drying pilot plant; the identification made with only three real experiments with non-constant drying conditions are shown to be as effective as an identification based on two-factor three-level grid of nine experiments at constant conditions.

Keywords: design of experiments (DOE); non-linear system; drying model; parameter identification; uncertainties

INTRODUCTION

The experiment design can be defined as the optimization of an experimental strategy in order to minimize the confidence intervals of the parameters to be identified with minimal experimental effort.

Design of experiments (DOE) techniques are well known and widely adopted for the identification of unknown parameters of very simple black box models of algebraic type as in response surface methodology (RSM) method. Things are completely different when it comes to tackle with — strongly non linear — differential models.

As a matter of fact, most of the DOE related publications are restrained to classical static models (e.g. polynomials). Hence, there is very little work done concerning the design of experiment considering dynamic (i.e. based on ODE) models. The usual method for this identification consists in making many experiments (kinetics) each at different, but constant conditions. This is to be compared with doing only two or three experiments with highly unsteady conditions.

There are very few interesting works concerning applications of DOE techniques. For instance, Luc Pronzato, the reference author in this domain, detailed several pharmacokinetic applications in a recent publication [1]. In addition, [3] wrote a review of available techniques. One can also find an application of D-optimality on a fermentation model [2].

In this work, we present sequential DOE of dynamic system; the originality of this work is the construction of a detailed methodology to apply DOE to a dynamic model. This methodology implements, as a Matlab toolbox, the A-, D- and E-optimality on the estimation of unknowns coefficients. First experiment is thus optimized on the basis on an initial (plausible) guess on unknown parameters while further identified parameters are used to optimize further experiments. In this methodology, the identified parameters are expected to converge rapidly (usually in 2-3 exp.) to true values with smallest possible uncertainties.

MATERIALS & METHODS

Prerequisites and general overview. The methodology of design of experiments[4] has a number of prerequisites:

- (a). A vector (a "protocol"), φ , formalizing[5] the degree of freedom in the experiments.

- (b). A computer modelization ("model") of experiments taking the protocol φ in account.
- (c). An initial guess for each unknown parameter of this model, θ^0 .
- (d). Information on the measurement error of y (standard deviations σ_y),

All above prerequisites are also prerequisites to generic parameter identification with gradient method. A general experiment design method, given the above four elements (a)-(d), is the following[4]:

- (i). Set i to 1.
- (ii). Find the A-optimal protocol φ_i , by optimization (experiment design).
- (iii). Make an experiment as close as possible to the calculated protocol φ_i . Collect real operating conditions and measurement times in vector $\tilde{\varphi}_i$, and collect measurements $\tilde{y}(\tilde{\varphi}_i, n_t)$ for $n_t = i, 2, \dots, N_{\tilde{\varphi}_i}$.
- (iv). Identify, by optimization, i -th parameter vector θ^i with which the model best predicts all available measurements.
- (v). Quantify the parameter confidence region and intervals around θ^i . Depending on the acceptability of parameter confidence interval, the algorithm may loop back to (ii).

For the results presented in this paper, step (iii) is replaced, except in second part of example (D), by simulations with random added noise (using standard deviation σ_y) on measures predicted with another set of parameters, named *true* parameters. These *true* parameters are the expected results of the DOE algorithm in this simulated environment.

To allow the use of standard optimization routines, the protocol vector and the parameter vector should be reconditioned (normalization) such that:

- (1). the domain of variation of the vector elements (the coefficients of the vector) have an order of magnitude close to one,
- (2). a variation of vector element of smaller order of magnitude does not influence qualitatively the model predictions,
- (3). there are no vector regions, wider than one in order of magnitude, in which the model prediction does not depend on the vector.
- (4). the total vector region is parallelepipedic.
- (5). couplings between vector elements are reduced.

This leads to much faster computations. However, for the sake of unbiased comparison with published results, no normalization were performed on applications (A) and (B).

Confidence regions and intervals. It is important to describe how the prerequisite (d), *i.e.* knowing measurement errors, is used in point (v) to quantify the acceptability of parameter confidence. For that purpose, the sensitivity (or jacobian) matrix, $M_{Jacobian}$, and the Fisher information matrix, M_{Fisher} must be defined.

The sensitivity matrix, $M_{Jacobian}$ gives the sensitivities of the model prediction in the parameter vector. It is a rectangular matrix, whose coefficient line i column j is the derivative by i -th parameter of the model prediction of the j -th available and ongoing design experimental data. In the design of the third experiment, this matrix $M_{Jacobian}$ is:

$$\begin{pmatrix} \frac{dy_\theta}{d\theta_1}(\tilde{\varphi}_1, 1), \frac{dy_\theta}{d\theta_1}(\tilde{\varphi}_1, 2), \dots, \frac{dy_\theta}{d\theta_1}(\tilde{\varphi}_1, N_{\tilde{\varphi}_1}), \frac{dy_\theta}{d\theta_1}(\tilde{\varphi}_2, 1), \dots, \frac{dy_\theta}{d\theta_1}(\tilde{\varphi}_2, N_{\tilde{\varphi}_2}), \frac{dy_\theta}{d\theta_1}(\varphi_3, 1), \dots, \frac{dy_\theta}{d\theta_1}(\varphi_3, N_{\varphi_3}) \\ \frac{dy_\theta}{d\theta_2}(\tilde{\varphi}_1, 1), \frac{dy_\theta}{d\theta_2}(\tilde{\varphi}_1, 2), \dots, \frac{dy_\theta}{d\theta_2}(\tilde{\varphi}_1, N_{\tilde{\varphi}_1}), \frac{dy_\theta}{d\theta_2}(\tilde{\varphi}_2, 1), \dots, \frac{dy_\theta}{d\theta_2}(\tilde{\varphi}_2, N_{\tilde{\varphi}_2}), \frac{dy_\theta}{d\theta_2}(\varphi_3, 1), \dots, \frac{dy_\theta}{d\theta_2}(\varphi_3, N_{\varphi_3}) \\ \vdots \\ \frac{dy_\theta}{d\theta_P}(\tilde{\varphi}_1, 1), \frac{dy_\theta}{d\theta_P}(\tilde{\varphi}_1, 2), \dots, \frac{dy_\theta}{d\theta_P}(\tilde{\varphi}_1, N_{\tilde{\varphi}_1}), \frac{dy_\theta}{d\theta_P}(\tilde{\varphi}_2, 1), \dots, \frac{dy_\theta}{d\theta_P}(\tilde{\varphi}_2, N_{\tilde{\varphi}_2}), \frac{dy_\theta}{d\theta_P}(\varphi_3, 1), \dots, \frac{dy_\theta}{d\theta_P}(\varphi_3, N_{\varphi_3}) \end{pmatrix} \quad (1)$$

where $y_\theta(\varphi, n)$ the prediction by simulation of the n -th measurement of experiment ϕ , and where θ_i is the i -th coefficient of the parameter vector. Using the method of adjoint modeling[6], the sensitivity matrix can be computed by numerical integration of a set of $N \times (P + 1)$ ODEs: the original N ODEs in the system, and their derivatives in the P parameters.

The Fisher information matrix, M_{Fisher} , is a $P \times P$ matrix, which can be computed by

$$M_{Fisher} = (M_{Jacobian} \times \Sigma^{-1} \times M_{Jacobian}^T)^{-1} \quad (2)$$

where Σ is the variance/covariance matrix, whose coefficient line i column j is the covariance of the i, j -th available measures (actually recorded in past experiments, or currently planned), which is prerequisite (d). It is a diagonal matrix when the noise of measures are independent. Confidence intervals and confidence regions can be computed using this Fisher information matrix. First of all, the confidence region of the parameter is an ellipsoid, centred on the parameter vector, whose axis are the eigenvectors of the Fisher information matrix, and whose axis lengths are proportional to the square of the respective eigenvalues[7]. The proportion coefficient is twice the square root of $\chi^2(P)$ value at 95%. Then, the specific confidence interval length of each estimated parameter is proportional to the square root of the diagonal coefficients of the Fisher information matrix[4]. This proportion coefficient 3.92 is twice the square root of the product of the $\chi^2(1)$ value at 95%.

Step (ii): optimizing the experimental protocol φ . The A-, D- and E-optimal criteria are respectively the trace, the determinant, and the spectral radius[4] of the Fisher information matrix. A protocol is A-, D- and E-optimal when the respective criterion is minimal. The optimal protocol φ is computed using a non-linear optimization routine according to the selected criterion.

During the real protocol design, before experiment number i , the protocol φ_i was A-optimally designed by minimizing the trace of the inverse of the 5×5 matrix whose element line n_l and column n_c is

$$\frac{1}{\sigma_y^2} \sum_{n_e=1}^{i-1} \sum_{n_t=1}^{N_{\tilde{\varphi}_{n_e}}} \frac{\partial y_{\theta}(\tilde{\varphi}_{n_e}, n_t)}{\partial \theta_{n_c}} \times \frac{\partial y_{\theta}(\tilde{\varphi}_{n_e}, n_t)}{\partial \theta_{n_l}} + \sum_{n_t}^{N_{\varphi_i}} \frac{1}{\sigma_y^2} \frac{\partial y_{\theta}(\varphi_i, n_t)}{\partial \theta_{n_c}} \times \frac{\partial y_{\theta}(\varphi_i, n_t)}{\partial \theta_{n_l}}$$

Step (iv): optimizing model parameters θ . A parameter θ is considered to be identified when it is found to be the only one minimizing the residuals. The residual is $(M_r \times \Sigma^{-2} \times M_r^T)^{-1/2}$, where M_r is a line vector whose j -th coefficient contains the difference between j -th available experimental measure, and its model prediction.

The computation were done with `Matlab` version 2009b. The estimated parameters are found by Nelder-Mead optimization[8]. They are further enhanced with Levenberg-Marquardt optimization[9]. To avoid local minima, a stochastic optimization strategy is chosen, leading to lengthy computation times: numerous various initial guesses for parameter vector are randomly picked. The minimized residual other the dataset is:

$$\frac{1}{\sigma_y^2} \sum_{n_e=1}^i \sum_{n_t=1}^{N_{\tilde{\varphi}_{n_e}}} |y_{\theta^i}(\tilde{\varphi}_{n_e}, n_t) - \tilde{y}(\tilde{\varphi}_{n_e}, n_t)|^2$$

(A) *Diffusion between two compartments.* Pronzato [1] gave an example of D-optimal design; the model is solved algebraically through spectral decomposition. This greatly speeds up the protocol design and the parameter identification.

(B) *Six fermentations for bacterial growth.* In [2], van Derlinden and van Impe present a D-optimal experiment design corresponding to the following model:

$$\begin{aligned} \frac{dn}{dt} &= Q(t)\mu(T(t))(1 - e^{n-n_{max}}) & \frac{dQ}{dt} &= Q(t)\mu(T(t))(1 - Q(t)) \\ \mu(T) &= \frac{\mu_{opt}(T - T_{min})^2(T - T_{max})}{((T_{opt} - T_{min})((T_{opt} - T_{min})(T - T_{opt}) - (T_{opt} - T_{max})(T_{opt} + T_{min} - 2T)))} \end{aligned}$$

These equations have been enhanced with a reparameterization of the physiological state of the cells Q , improving the stability of the dynamic system.

(C) *Ascorbic acid in stewed apples.* This is the classical first order reaction of degradation of vitamin C. The reaction rate is given by

$$\frac{dy}{dt} = -yk_0 e^{-E_a/(RT)}$$

with E_a the activation energy in J/mol, k_0 a pre-exponential coefficient in s^{-1} , temperature T in kelvins, and $R = 8.31$ J/mol/K the perfect gas constant. The identification algorithms work on vector

$$p = [\log_{10}(k_0 e^{-E_a/(R(273.15+25))}), (\frac{1}{273.15+25} - \frac{1}{273.15+90}) \frac{E_a}{R \log 10}] \quad (3)$$

which is the logarithm of the reaction rate at min temperature, and the difference between logarithms of reaction rates at min and max temperatures. The following boundaries have been added: $-9 < p_1 < 10$ on reaction rate at 25°C , and E_a kept in 30 - 300 kJ/mol range.

For each experiment, the heat treatment temperature must be constant, this constant can be chosen between 25°C and 90°C . One assumes that there are five samples per experiment, taken between one minute and four hours after the start of the experiment. The first guess is $k_0 = 1.77 \cdot 10^9 \text{s}^{-1}$, $E_a = 99.2 \text{kJ/mol}$ which has been identified by [10] for apricot.

There is no real experiment. They have been replaced by numerical simulations using parameters $k_0 = 3.927 \cdot 10^7 \text{s}^{-1}$, $E_a = 72.813 \text{kJ/mol}$.

One should note that one isothermal experiment is not enough to calculate an activation energy. Hence, using variable conditions looks like a promising strategy, see application (D).

(D) Rice samples. In this work, the validated rice drying model from [11] is considered. This 2-compartment dynamic model is composed of 3 ODE with strong non-linearities brought mostly by a_w and P_{sat} equations. The state vector is composed of inner and outer compartment moisture contents, and one global temperature. Moisture is evaporating from outer compartment. Only the mean of the two compartment moisture contents is effectively measured online. All parameters are known, except two transfer coefficients, h (convective heat at the surface) and D (apparent water diffusion inside the kernel; It should be noted that in the original publication, it was presented in a slightly different formalism with water exchange coefficient.), and the volume fraction τ_1 of inner compartment.

Each experiment lasts $t_{\max} = 2\text{h}$, and is divided into five periods. The protocol vector contains:

- the air temperature set points (divided by T_{\max} for normalization) of each of the five periods, greater than the ambient temperature T_{\min} .
- the set points of water additions divided by maximum available value (saturation or maximum capacity reached) for each of the five periods. These set points can be easily converted in relative humidity set points.
- the duration of each first four periods divided by remaining time of current experiment.

An experimental dryer used is installed at the INRA's associated laboratory at the AgroParisTech. The inlet air temperature, relative humidity and velocity are controlled by three independent PID controllers, under the supervision of the master PC. Product (rice here) is weighted every minute. The current mean moisture content can be computed from this weight; it is the only product related variable that is measured online.

Definition of the parameter vector for prerequisite (c) p. 1. Using the same reasoning (see items (1)–(5) above), the five (unknown) parameters are grouped in a parameter vector as follows:

$$\theta = \left[\log_{10}(D(X, T = 0)), \log_{10} \left(\frac{D(X_{\max}, T_{\max})}{D(X, T = 0)} \right), \log_{10}(h(T_{\min})), \log_{10}(h(T_{\max})), \tau_1 \right] \quad (4)$$

RESULTS & DISCUSSION

Thanks to the re parameterization of the parameter and protocol vectors and to the stochastic enhancement of the A-, D- and E-optimization, the initial guess for the unknown parameters was found to have no influence on the final results.

(A) Diffusion between two compartments. We found protocol [1.0003, 1.0004, 10.4131, 10.4273, 67.3876, 70.8813, 718.9642, 719.7539] (values in minutes) which is very close to D-optimal protocol [1, 1, 10, 10, 74, 74, 720, 720] proposed by [1]. Even better, it has a D-optimal criterion which is 0.31% smaller. This validates our toolbox of D-optimality for constant conditions and varying sampling times.

(B) Six fermentations for bacterial growth. Table 1 shows that our methodology is able to compute independently the six D-optimal protocols proposed by [2], hence validating our implementation of DOE for experiments with non-constant temperature profiles.

Table 1. Results of our methodology in case (B), compared to [2, Table 2].

D-optimality		initial temperature (°C)		final temperature (°C)		duration of initial temperature (h)		temperature decrease (°C/h)	
published	this work	published	this work	published	this work	published	this work	published	this work
$6.8154 \cdot 10^5$	$8.482 \cdot 10^5$	45	44.983	15	15.042	3.835	3.0785	5	4.6126
96949	$1.117 \cdot 10^5$	45	44.999	15.94	15.2	2.854	3.5405	5	4.9986
66129	81980	45	44.935	16.62	15.833	2.938	3.2592	5	4.8999
$1.7983 \cdot 10^6$	$1.8 \cdot 10^6$	40.85	40.987	16.91	16.952	1.78	1.7432	5	4.9981
76796	76740	31.15	31.174	16.87	16.871	4.382	4.2245	2.388	2.2608
$1.5282 \cdot 10^6$	$1.547 \cdot 10^6$	45	44.979	16.67	16.751	2.801	2.6751	5	4.9956

Table 2. Experiment design convergence. Test with experiments done on drying pilot plant. (a) A-optimal design in three iterative steps, (b) standard two-factor three-level design. Last column gives σ_f/σ_y which is the lack of fit σ_f , divided by the mean experimental error σ_y .

#Exp. done	$D(0) (\mu\text{m})^2/\text{s}$	$D(T_{\max}X_0) (\mu\text{m})^2/\text{s}$	$h(T_{\min}) \text{ W/Km}^2$	$h(T_{\max}) \text{ W/Km}^2$	$\tau_1 \text{ m}^3/\text{m}^3$	σ_f/σ_y
(a) A-optimal design, experimental run						
1	20 ± 5	$100 \pm \infty$	[3, 20]	[6, 23]	$.55 \pm 0.02$	1.09
2	10.0 ± 1.2	[30, 13000]	[4, 30]	[7, 30]	$.64 \pm 0.02$	1.12
3	10.0 ± 0.7	[40, 8000]	13.2 ± 0.7	$10.2 \pm .5$	$.549 \pm 0.005$	1.40
(b) two-factor three-level design, experimental run						
9	12.7 ± 0.7	[14, 5000]	15.5 ± 1.3	12.5 ± 1	$.674 \pm .006$	1.84

(C) *Ascorbic acid in stewed apples.* With A-optimality, the total computation times does not exceed 20 minutes, usually less than 5 minutes. The results are not sensitive to the numerical integration error of ODEs.

A-optimality, D-optimality and E-optimality give close results, with usually 5 or 6 iterations. A-optimality results are slightly better. This number of iterations increases by two when the first experiment is replaced by three experiments corresponding to a central composite experiment design.

Obtained precision on estimated parameters is sufficient after only 2 or 3 experiments.

(D) *Rice.* The computation time of an A-optimal protocol is three hours on a cluster of 12 processors (about a day on one processor).

The first objective here is to choose between A-, D- and E-optimality, and to proof check the feasibility (mostly as a quick error-checking procedure) at no experimental cost. In these favorable conditions, the A-optimal algorithm converges after only 3 — simulated — experiments while D-optimality performs significantly worse in terms of quantity of bad confidence intervals than A-optimality and E-optimality. Even if it is not a generic conclusion, on the basis of this pure simulation run, A-optimality is preferred to E-optimality because it produced less false confidence interval. A possible reason for this is that the A-optimal criterion is smoother than the E-optimal criterion.

The second objective is to apply it in a real experimental setup. The A-optimal protocols are actually used on the experimental pilot, leading to estimated parameters shown in Table 2 (a). Observed error is less than 1.4 times the measurement error on average, and uncertainties on identified parameters are low (see Table 2 (a)).

To validate the results shown in Table 2 (a), they are compared to the identification obtained by a two-factor three-level grid of nine experiments, combining three temperature levels of 50°C, 70°C and 90°C, and three levels of relative humidity 0%, 20% and 40%. The cross-validation comparison between this validation and the validation of A-optimal designed experiment is hence only marginally in favor of 9 experiments on static conditions, it has about 15% smaller fit errors. Our methodology let us divide by three the time of experimentation and the amount of used product. The confidence intervals on the identified parameters were smaller. On the other hand, the error of fit of prediction gets 15% bigger.

CONCLUSION

Thanks to the normalization of the parameter vector, and use of stochastic optimizations in DOE algorithm, the convergence is less sensitive to the initial value of the protocol and the initial assumption of parameter ϕ_i .

Our implementation of the DOE for dynamic systems give results comparable to optimal designs already published in literature, in the context of fixed profile by Pronzato [1] for our case (A), and in the context of variable non-constant temperature profile by vanDerlinden et al. [2] for case (B).

Our methodology of sequential DOE was tested in a virtual environment, and its convergence was established, in the case (C); the A-optimality was shown to be somehow better than the E-optimality and far better than the D-optimality in the context (C) and (D). It is observed in the context of respectively (C) and (D) that respectively first two and first experiment reaches directly the right parameter neighborhood in terms of order of magnitude.

In the experimental environment, experimental temperature profile will differ to some extent from the designed one; this methodology was shown to give nonetheless good results: identifying simultaneously four transfer-related constants in three A-optimally designed experiments gave a precision only 15% worse than the classical identification based on nine experiments with static drying conditions. Hence, this methodology let us divide by three the time of experimentation, and the amount of used product, for a similar quality of identification.

It is generally observed that the computed A-optimal protocol tends to maximize the amplitude of the variations of experimental conditions. The proposed methodology has shown, on the example (D), its ability to minimize the experimental work needed to identify unknowns in a given non-linear dynamic model. Furthermore, it optimizes confidence levels on the estimates while classical strategies cannot give any guarantee on that point.

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