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Explosive formulation by experimental design

M. Vaullerin[1,*], P. Morand[1], A. Espagnacq[1] and L. Morin-Allory[2]

[1] *Giat Industries, DSAM, 7 route de Guerry, 18023 Bourges Cedex, France*
[2] *ICOA, UPRES-A 6005, Université d'Orléans, 45067 Orléans Cedex 2, France*

Abstract. In order to increase the safety of ammunitions over their full life cycle (from the conception to the disposal), we are developping new explosive compositions called EIDS (Extremely Insensitive Detonating Substances). This work requires substantial investments because some raw materials, the development of the formulations, the preparation of samples and the achievement of the characterization tests are very expensive. To be able to carry out this task at the lowest cost, we focussed on two tools, the **experimental designs** and the **performance predicting_codes**. By using together several experimental matrixes, we could define a very small number of samples which were assessed mainly by the mean of our predicting codes. Then, from the mathematical modelling of observed characteristics we could establish a narrow domain of formulations meeting the expected compromise between the explosive performances and the safety characteristics. The experimental validation test we carried out then, has confirmed the forecast results.

Key words. Experimental design – optimal design – Doehlert matrix – explosive compositions EIDS.

Introduction

Nowadays, in the energetic material field for military use, the major concern in formulation of composition is to develop new explosive compositions whose essential characteristic is a very low sensitivity while preserving a good level of performances. Indeed, to increase goods and persons security in all phases of life of an ammunition, it is necessary in the first place to use very insensitive explosive compositions and notably, those belonging to the family of EIDS (Extremely Insensitive Detonating Substances).

The development of such compositions can be achieved by associating judiciously several energetic and inert components. As part of the present study aimed to develop both low sensitivity and inexpensive explosive substance, we have to define a new composition which could substitute to the H6 composition (TNT/RDX/Al/wax) regarded as too sensitive. As the explosive material formulation requires substantial investments, espacially to buy raw materials but also for the manufacture of compositions or the achievement of characterization tests, we chose to proceed by experimental designs by leaning on test methods of laboratory and predictive computation tools to assess levels of performances. By proceeding like that, we could limit the number of formulations to consider and however estimate performances levels inside a wide experimental field.

This led to the choice of solutions which could cope with both performances and sensitivity aims.

Definition of the studied parameters

The H6 composition made of 2 melted materials (wax and TNT), in which a metallic element (aluminium) and an energetic explosive (RDX) are introduced. If this composition is quite efficient as regards detonation performances, on the other hand it does not cope with sensitivity tests that make it possible to define the membership to the family of EIDS explosives. In order to manage to do this we must intervene at the same time on the nature and on the relative proportions of the different ingredients knowing that in this way the level of detonation performances and the level of sensitivity are simultaneously modified. Then we have to define the good compromise between these two responses families. The parameters that were studied are presented in table I. On the other hand, to ensure a good feasibility of compositions, we decided to keep a stable proportion of melted components (wax + melted explosive) compared to the other components.

Table I. Studied parameters and interest experimental field.

Parameters	*Definition*	*Experimental field of interest*	
		Minimum	*Maximum*
X_1	HMX/NTO	0/100	50/50
X_2 (%)	Wax	4	10
X_3 (%)	Aluminium	8	20
X_4	Nature of melted explosive	TNMA	TNT

Noted: HMX with NTO take the place of RDX.

Experimental interest field

Variations of the proportions of the different components studied are also presented in table I.

Matrix of experiments

So as to learn more about the characteristics inside the experimental field we decided to combine a Doehlert matrix

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processing the 3 continuous parameters (X_1, X_2 and X_3) with a factorial matrix for the only discontinuous parameter (X_4).

The Doehlert matrix [1]

Once the matrix is chosen by means of the NEMROD software version 3.0 [2], it defines a first parameter with 3 levels, a second one with 5 levels and a third one with 7 levels. Given the selected experimental field and our knowledge about the studied parameters, we attributed 3 levels to X_1, 5 levels to X_2 and 7 levels to X_3.

The complete factorial matrix

To process the single discontinuous parameter (X_4), a complete factorial matrix 2^1 was set up.

The result of this is 2 experiments.

Resulting experiment matrix

The next step consists in creating all the experiments resulting from the product of the Doehlert matrix about 13 experiments with the fractional factorial matrix previously described.

This product gives: $2^1 \times 13 = 26$ experiments (Tab. II).

They are presented in figure 1 in the form of a 3-dimensional diagram.

Postulated model

The postulated mathematical model is a polynomial model where interactions are not taken into account. On the other hand, considering that the variation of the response is not inevitably linear, we considered the square terms for the 3 continuous parameters.

The expression of this polynomial mathematical model, where variables X_i are expressed in the reduced form, is defined hereafter:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (1)$$

with:

Y: studied response
X_i: level of the parameter i
b_j, b_{jj}: coefficients of the model.

The comparison of the number of coefficients of this model (8) with the number of experiments to carry out (26) shows that it may be conceivable to reduce the last one so as to curb as much as possible the cost of the works.

Optimization of the matrix of experiments by the FEDOROV algorithm

We tried to optimize the matrix with a FEDOROV [3] exchange algorithm (option of the NEMROD software version 3.0 already mentionned). From the 26 candidates points that constitute the 26 experiments previously defined, this algorithm builds one after the other the potential designs in order to proceed to their comparative and qualitative analysis. To lead to the definition of satisfactory solutions, this algorithm maximizes or minimizes different characteristics of the experiments matrix (Tab. III). The first one is the determinant of the information matrix. Then, it takes the

Table II. Resulting experiment matrix.

Point N°	X_1	X_2	X_3	X_4
1	0	0	0	–1
2	1	0	0	–1
3	0.5	0.866	0	–1
4	0.5	0.289	0.816	–1
5	–1	0	0	–1
6	–0.5	–0.866	0	–1
7	–0.5	–0.289	–0.816	–1
8	0.5	–0.866	0	–1
9	0.5	–0.289	–0.816	–1
10	0	0.577	–0.816	–1
11	–0.5	0.866	0	–1
12	–0.5	0.289	0.816	–1
13	0	–0.577	0.816	–1
14	0	0	0	1
15	1	0	0	1
16	0.5	0.866	0	1
17	0.5	0.289	0.816	1
18	–1	0	0	1
19	–0.5	–0.866	0	1
20	–0.5	–0.289	–0.816	1
21	0.5	–0.866	0	1
22	0.5	–0.289	–0.816	1
23	0	0.577	–0.816	1
24	–0.5	0.866	0	1
25	–0.5	0.289	0.816	1
26	0	–0.577	0.816	1

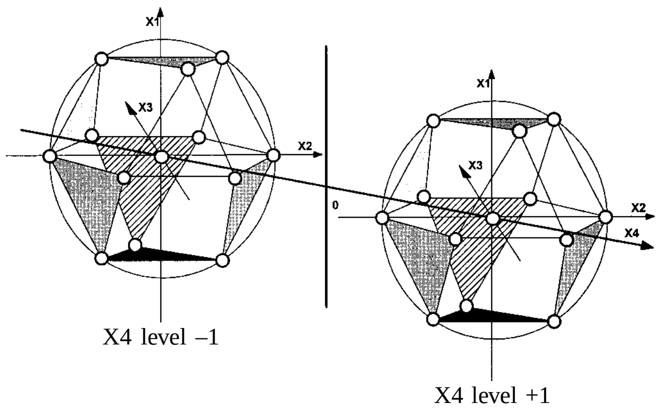

Fig. 1. Product of the Doehlert matrix with 3 parameters (13 experiments) with the complete factorial matrix 2^1 (2 experiments).

Table III. Characteristics of original and optimal matrix.

	Matrix of origin (26 experiments)	*Optimal matrix (12 experiments)*
Log Determinant ($X'X$)	6.453	3.812
Log Determinant (M)	–4.866	–4.822
Log Determinant (M)** 1/p	–0.608	–0.603
Maximal Variance	0.538	0.933
Trace $(X'X)^{-1}$	3.088	4.660
Efficiency G (%)	57.143	71.445

trace of the dispersion matrix and the function of variance into account.

This work led to the construction of a matrix with only 12 experiments (Tab. IV). The figure 2 represents the distribution of these experimental points.

Experimental design

The resulting experimental design is given in table IV. To make its analysis easier, it is divided in two parts, each of which corresponding to one of the levels of the parameter X_4. Moreover, it clearly shows formulations resulting from the experiments matrix defined in table II.

Results and exploitation

The results for the detonation velocity *D* [4] and the sensitivity criterion *CS* [5,6] are given in table IV. The exploitation of these results with the help of the NEMROD software leads to the next equations:

$$D = 7194.5 + 45.3X_1 - 302X_2 + 96.2X_3 - 33.8X_4 + 29.3X_1^2 + 6.7X_2^2 + 27.7X_3^2 \quad (2)$$

$$CS = 102.5 - 11.3X_1 - 13.3X_2 + 8.7X_3 + 1.7X_4 + 0.03X_1^2 + 0.64X_2^2 + 0.28X_3^2 \ . \quad (3)$$

These results are presented in the form of a bar-diagram (Figs. 3 and 4). These equations and diagrams clearly show the very strong influence of X_2 on the response *D* whereas on the response *CS*, the action of X_4 is very low. A detailed analysis of these two equations made it possible to define a solution providing a good compromise between these two responses.

This solution was then subjected to experimental evaluations which confirmed the expected performances given by models.

Conclusion

The research of new compositions presenting both a satisfactory detonation character and a level of the weakest possible vulnerability proves to be a complex compromise. On the other hand, the costs of processing of these new compositions, then their evaluations to energetic as well as to vulnerability levels being extremely expensive, it became imperative to curb the number of tests.

Table IV. Experimental design (12 experiments) and responses.

Point N°	*N° matrix of origin*	X_1	X_2	X_3	X_4	*D*	*CS*
1	1	0	0	0	–1	7248	100.6
2	14	0	0	0	1	7141	104.4
3	15	1	0	0	1	7241	93.3
4	3	0.5	0.866	0	–1	6999	84.3
5	17	0.5	0.289	0.816	1	7202	101.7
6	5	–1	0	0	–1	7206	112.0
7	19	–0.5	–0.866	0	1	7421	122.3
8	20	–0.5	–0.289	–0.816	1	7174	106.5
9	8	0.5	–0.866	0	–1	7516	106.5
10	10	0	0.577	–0.816	–1	6995	86.8
11	24	–0.5	0.866	0	1	6892	98.6
12	13	0	–0.577	0.816	–1	7501	116.3

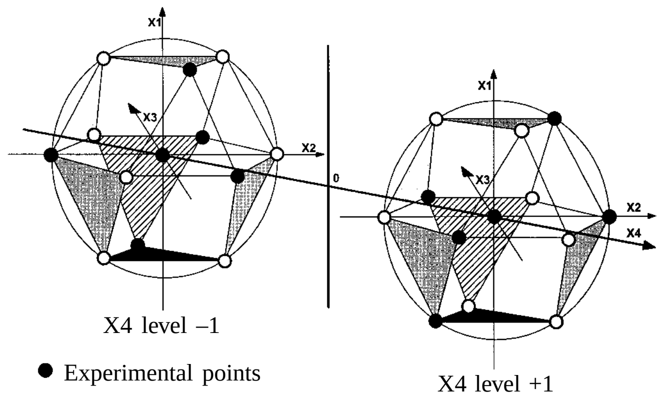

Fig. 2. Representation in 3D of 12 experiments chosen after optimization.

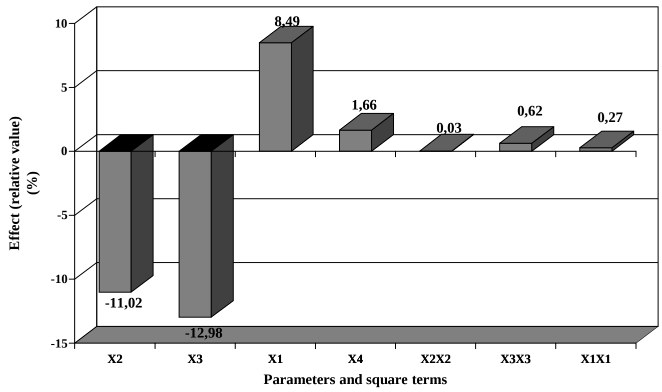

Fig. 3. Effect of parameters on sensitivity criterion *CS*.

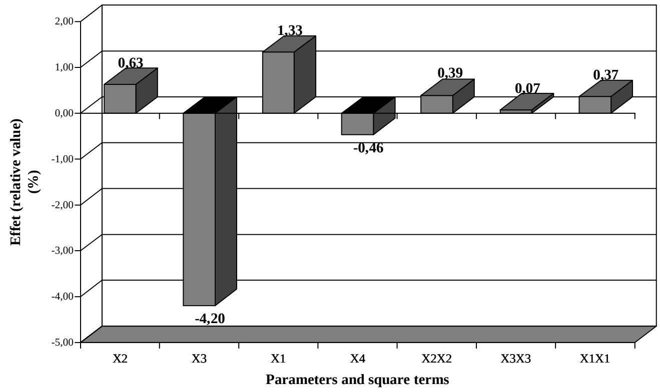

Fig. 4. Effect of parameters on detonation velocity *D*.

That is the reason why the use of an experimental methodology by experimental design is a good answer to these needs.

First of all, the approach by an experimental methodology applied on the code of sensitivity and explosive characteristics prediction makes it possible to know the influence of the different parameters on pressure, temperature and especially on detonation velocity as well as to select the most important ones.

As a result, the application of an experimental design reduced to important parameters allows to define the future formulations which might present the best detonation velocities while taking into account the cost price.

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