Evaluation of Computer Aided Rubber Compound Development

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Introduction:

There is a short history of an evaluation of possibilities to calculate compound properties based on the content of ingredients. Methods included Neuronal Network Mathematics (NNM) and None Linear Regression (NLR) for example. A couple of patents granted on different compounds like recipes for color master batches but on rubber compounds as well (**1,2,3,4**). Testing a program based on NNM to predict the properties of a rubber compound formula failed even a larger database was created and implemented (**5**) On the other hand, the prediction of compound properties with a much smaller database created with Statistic Experimental Design (DoE) is successful but limited to the boundary conditions of this experiment. In a DoE is it called optimization and the result of the overlay plot. **[Figure 1]**

The analysis of the failure to predict even with a larger database is the intolerance of NNM to experimental error or mistype errors in the database and that the data in the database can not be corrected, respective excluded from the calculation. There is no ability to of tracking bag. Any regression model obtained from DoE can not handle any happenstance database.

On the other hand most DoEs evaluated during investigations of ingredient / rubber property relation show clearly linear correlations, at least second order type of regression. In particular a more careful investigation of the statistics indicate a data fit with linear regression equations is sufficient. In most cases a none linear regression is indicated by the DoE-program used for evaluation, but in particular under consideration of measurement error in rubber testing and statistical noise in compound mixing it may be an over interpretation.

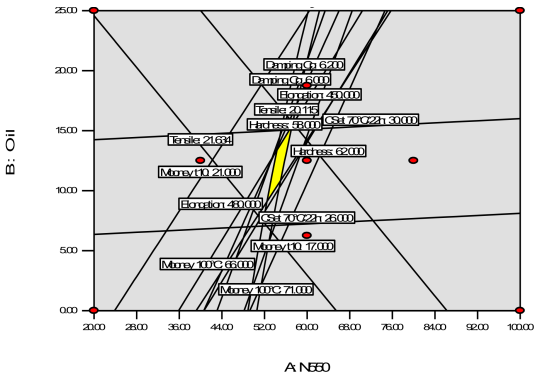

Fig. 1: Overlay Plot of physical properties - upper and lower limits

As a consequence the prediction of a compound with its properties should be possible with sufficient precision if an approach is used based on linear regression in combination with iteration. This method is called Multi-Linear-Iteration (MLR). To demonstrate the precision of predictions based on MLR two experiments were performed: Data from experimental designs taken – an oil/filler and on the other hand an accelerator design from literature (**6**) - and recalculated with MLR.

Experiments

As a first example, a simple oil/filler DoE, performed as a fractional factorial design, was chosen to show in principle, how those tools react given a limited set of data.

Eleven experiments are necessary to perform the DoE with three replicates of the center point. Responses chosen for evaluation were Mooney Viscosity (MV), the scorch time at 125°C (T5) and the vulcameter time to ten points rise at 175°C (t10). All responses are significant using a linear model regression equation.

Ingredients	Unit	DoE Optimization	GrafCompounder
CB 6630	phr	73	73
CaCO3	phr	68	61
Clay	phr	39	32
Paraffinic Oil	phr	72	70
MV 120	MU	34	34.1
T5 (120°C)	min	4.04	4.1
t10 (170°C)	min	0.45	0.45

Fig. 2: Comparison of DoE with GrafCompounder: Table of Results

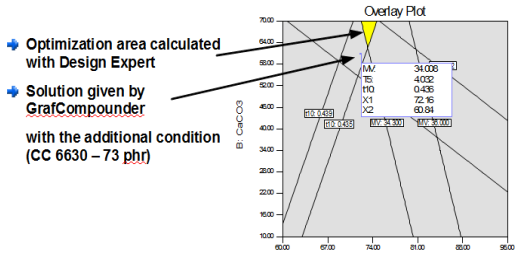

Fig. 3: Comparison of DoE with GrafCompounder: Optimization Plot

The optimized factors resulting from the Design-Expert® software are somewhat different compared to those seen using the "GrafCompounder software **(Figure 2, 3)**. The main differences are the amount of CaCO3 and Clay. Even with MLR there is no data fit via regression for calculation, it is shown, that the result are in good agreement inside the 95% confidence interval.

Now using the point prediction tool, we are able to set the two factors CaCO3 and Clay at the same values as calculated with the GrafCompounder. The analysis of the two different results should show whether they are in the 95% confidence interval. In the following **Figure 4** it is shown that the results from both software systems are not only inside the confidence interval but also even inside the measurement errors of the methods.

Ingredients	Unit	DoE Optimization	GrafCompounder	DoE Point Prediction
CB 6630	phr	73	73	73
CaCO3	phr	68	61	61
Clay	phr	39	32	32
Paraffinic Oil	phr	72	70	70
MV 120	MU	34	34.1	34.2 ± 3
T5 (120°C)	min	4.04	4.1	4.01 ± 0.25
t10 (170°C)	min	0.45	0.45	0.43 ± 0.07

Fig. 4: Comparison DoE with GrafCompounder: Table of results after optimized calculation

To go one step further an experiment was conducted based on NR compounds taken from a historic database (5) and at the same time preparing an Oil / Filler 1 / Filler 2 DoE in the laboratory. Database compounds were imported in the GrafCompounder Software to simulate the DoE. That means, a set of compounds were calculated according to the DoE scheme and analyzed with the DoE software (**Figure 5**).

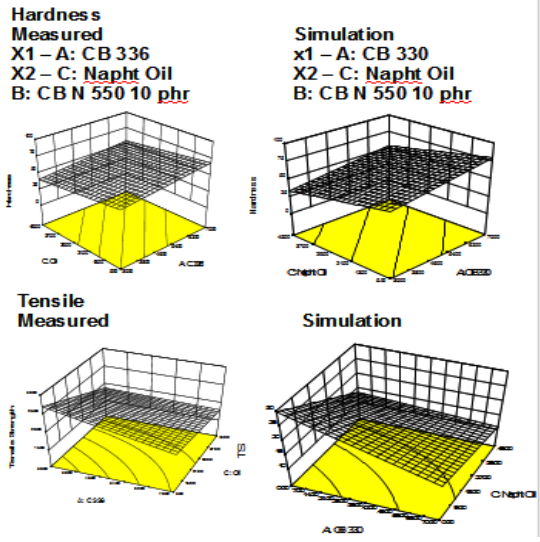

Fig. 5: 3D Contour plots of DoE - Measured vs Simulated

Even the preparation of the compounds and some of the materials used are different the result show clearly that all tendencies are the same but very few physical properties show characteristic onset like the tensile at break for example.

Summary

With this examples it is demonstrated, that such tool is helpful to create a compound and predict it physical properties using databases with either historic or new data. Such simulation is helpful to save development time and effort and allows to concentrate on solutions of compounding challenges.

Literature:

(1) US Patent 6,411,945 B1 (2002) Yukio Nakajima

(2) US Patent 6,671,661 (2003) Christopher Bishop

(3) US Patent 6,714,924 (2004) Craig J. McClahnahan

(4) US Patent 7,451,122 B2 (2008) Paul F. Dietrich

(5) N.N.: The Natural Rubber Formulary and Property Index, MRPRA, Printed by Luton Ltd, GB, 1984

(6) R. Ohm, R. Vara, T.M. Buckley, Sulfur Cure System Development for EPDM Produced via constrained Geometry Catalyst Tehcnology; Paper presented at RD of ACS Meeting, Indianapolis, IN, May 5-8, 1998