



GrafCompounder

Software Application in Rubber Compounding



Content of this presentation

1. Introduction
2. Program idea
3. Justification of calculation method
4. Comparison with Statistic Experimental Design (DoE)
 - Filler / Oil Design
 - Accelerator Design
 - DoE Simulation
5. Conclusion

Introduction

Program idea

Justification of Method

Comparison with DoE Software calculation

Filler / Oil Design

Accelerator Design


DoE Simulation

Conclusion



Computer Aided Compound Development

- λ **Bridgestone Patent 1994**
Inventor: Akihiko Abe
- λ **Bridgestone Patent 2002**
Inventor: Yukio Nakajima
- λ **Colour Matching Patents from BASF, CyanAmid, DuPONT**
- λ **Empirical DoE Patent: Honeywell**
- λ **Recipe Library Search and Comparison**
CombiChem, GE, Hunt (Private)


US006411945B1

(12) **United States Patent**
Nakajima

(10) Patent No.: **US 6,411,945 B1**
(45) Date of Patent: **Jun. 25, 2002**

(54) METHOD AND APPARATUS FOR DESIGNING MULTI-COMPONENT MATERIAL, OPTIMIZATION ANALYZER AND STORAGE MEDIUM USING LEARNING PROCESS

JP 9-16654 1/1997
WO WO 94/16877 8/1994

OTHER PUBLICATIONS


(75) Inventor: **Yukio Nakajima, Tokyo (JP)**

(73) Assignee: **Bridgestone Corporation, To**

(*) Notice: Subject to any disclaimer, the patent is extended or adjusted U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/051,416**
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EUROPEAN PATENT APPLICATION

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(71) Applicant: **BRIDGESTONE CORPORATION**
10-1, Kyobashi 1-Chome
Chuo-Ku
Tokyo 104 (JP)
(72) Inventor: **Abe, Akihiko**
3-5-5, Ogawa-higashi-cho
Kodaira-shi, Tokyo (JP)
(74) Representative: **Whalley, Kevin**
MARKS & CLERK,
57-60 Lincoln's Inn Fields
London WC2A 3LS (GB)

(34) **Method for designing pneumatic tires.**

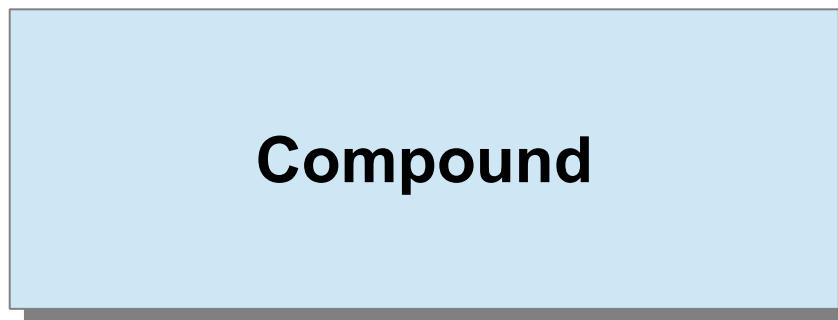
(37) In order to perform tire design and development highly efficiently and provide a tire at low cost, a tire basic model for representing a tire cross-sectional shape including an internal structure and being divided into a plurality of elements, an objective function for representing

FIG. 2A

START

MODELING OF TIRE

100

**Influences:****Factors: Ingredients** F_1 → F_2 → F_3 →**Effects:****Responses: Properties**→ R_1, R_2, \dots, R_n

Objective of an DoE should be the identification of the most important factors (F_1, \dots, F_n) on measurable effects (Responses R_1, \dots, R_n) and to describe there dependency in a mathematical equation:

$$R_{i(1\dots n)} = f(A_0 + A_1 F_1 + \dots + A_n F_n + \dots)$$



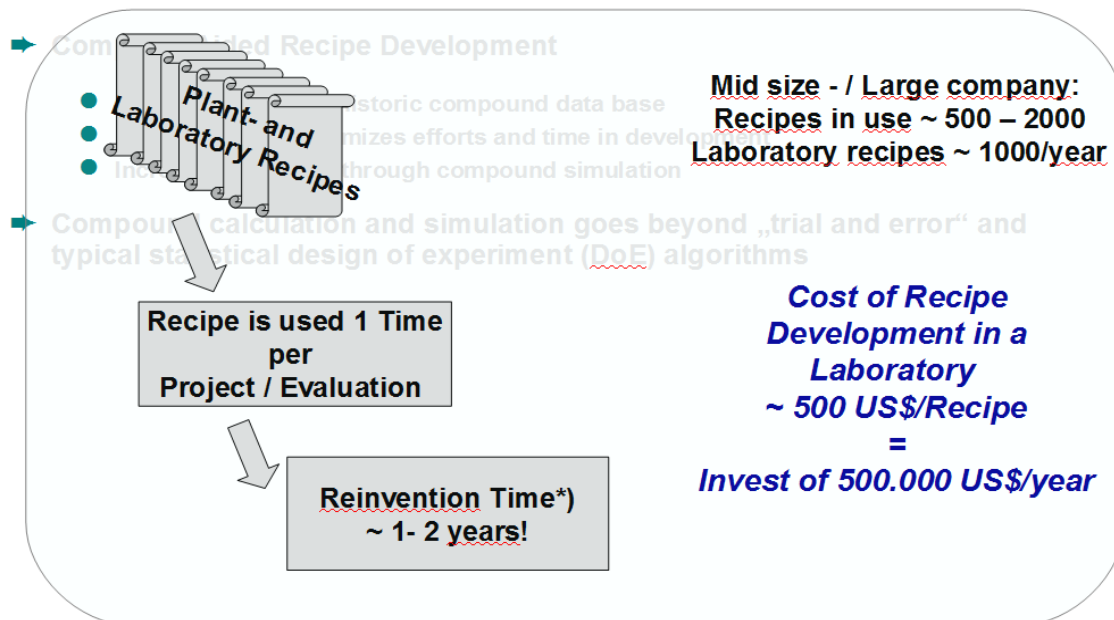
Why Computer Aided Recipe Development ?

- λ Better utilization of historic compound data base
- λ Faster results - minimizes efforts and time in development
- λ Increases creativity through compound simulation

No algorithm describing the relation between ingredients and properties

It gives a better start for a typical statistical “*design of experiment*” (DoE) approach.

Compound calculation and simulation should utilize Compound history, but not in a „trial and error“ fashion.



- **Question:**
- **Why we can hardly take Compound Databases as working capital,
Saving time and effort in our daily work?
Benefits would be:**
 - **Avoiding reinvention**
 - **Increase our compounding knowledge.**
 - **Making room for really new ideas in compound development**



Database created with Statistic Experimental Design (DoE)

- λ Organized / limited size
- λ Variation of few factors according DoE
- λ Optimization, numerical and graphical / prediction Tool available in the software

CARD [Computer Aided Recipe Development] with GrafCompounder - Historically created Database

- Unorganized / Unlimited
- Multiple factor variation
- Prediction according specification

Justification of calculation method with linear dependencies:

- If the majority of factor / response relations are linear the MLI – method gives sufficient accurate results inside 95% confidence interval !



Line call out:

- **SEA J200: AA/BA/CA – NR, SBR, EPDM...and other Material**
- **SAE J200 M4 AA621 A13 B13 F17**

AA 610 Suffix 2	Rubber Hardness Tensile Elongation	NR 60° ShA 21 Mpa 350%
A13	Heat Aging Hardness Change Change Tensile Change Elongation	70h / 70°C + 15° ShA ± 30 % - 50%
B13	C-Set (22h/70)	< 25%
F17	Low Temperature Res. Non Brittle (3Min)	- 40 °C pass



Properties of MB is determined by Polymer, CB and Oil content and the ratio or CB and Oil.

- Unit 2 as a reference (based on Cabot TG RG-135)
- ...

– **CB 550: 55 phr**

– **Oil: 10 phr**

– **Mooney Viscosity: 71 M-Units**

– **Hardness: 60 °ShA**

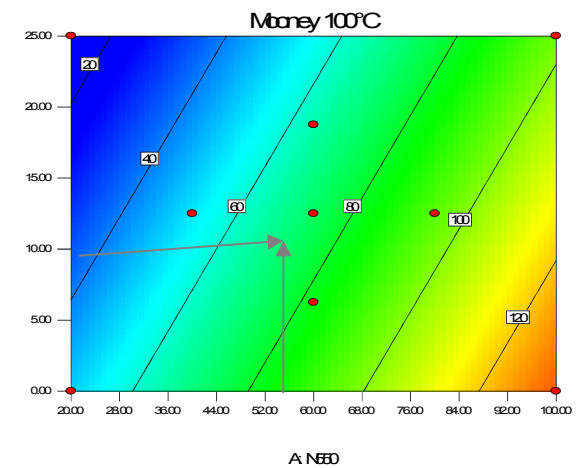
– **Tensile: 21 MPa**

– **Elongation: 460 %**

– **C-Set: 28%**

NR Compound

- **SMR 5CV – 100 phr**
- **CB – Var**
- **Oil – Var**
- **ZnO – 5 phr**
- **StAc – 1 phr**
- **AO – 1 phr**
- **NR 100 phr**
- **MBTS – 0.6**
- **S – 2.5 phr**





Properties of MB is determined by Polymer, CB and Oil content and the ratio or CB and Oil.

– Unit 2 as a reference (based on Cabot TG RG-135)

– ...

– CB 550: 55 phr

– Oil: 10 phr

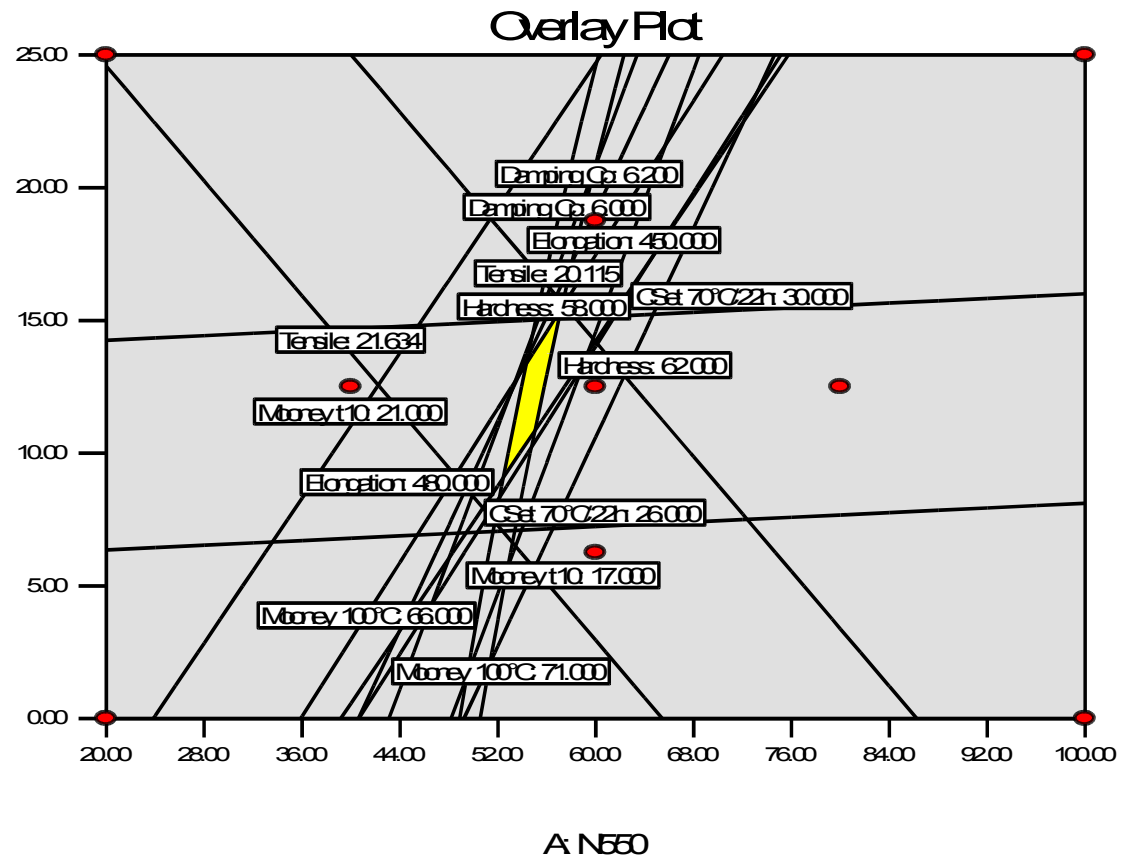
– Mooney Viscosity: 66 – 71 M-Units

– Hardness: 58 – 62 °ShA

– Tensile: 20 – 22 MPa

– Elongation: 450 – 480 %

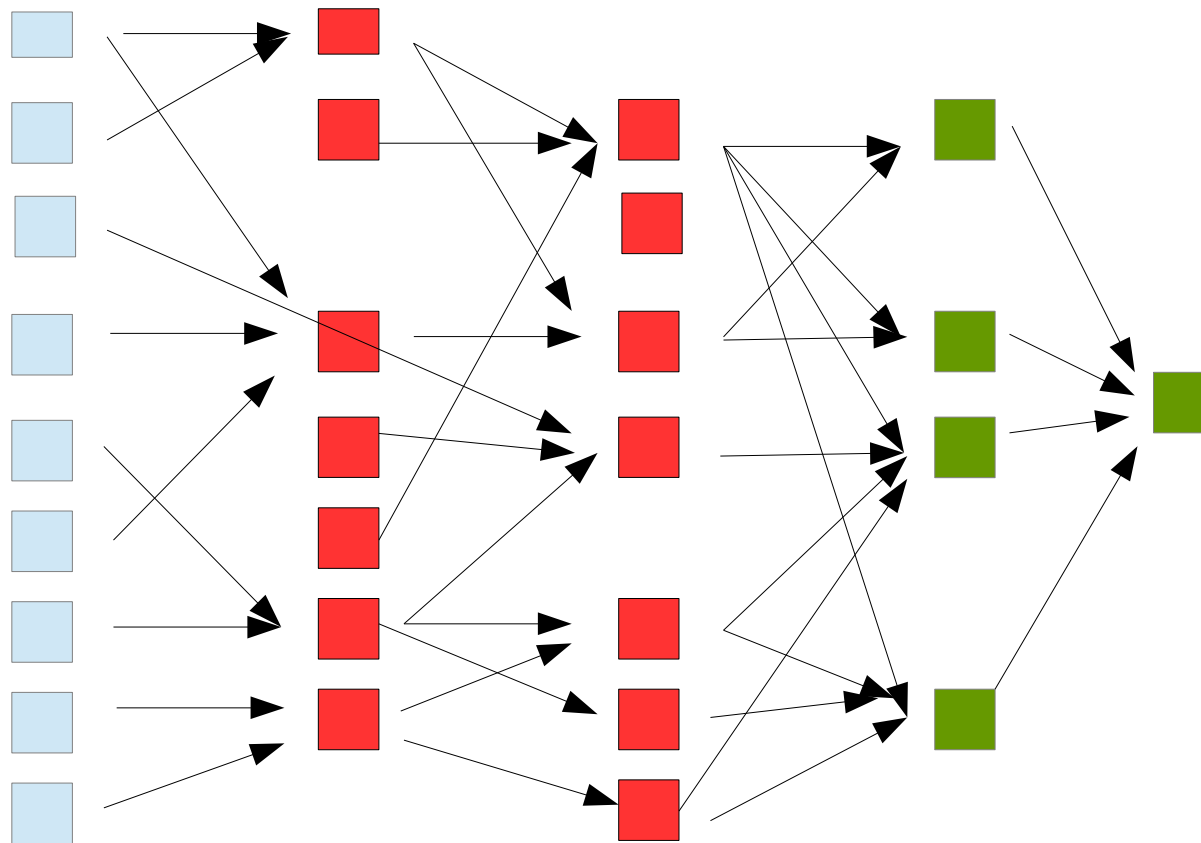
– C-Set: 26 – 30%





Original Data 1. Iteration Level, nth - Iteration Level, Result

Input Data

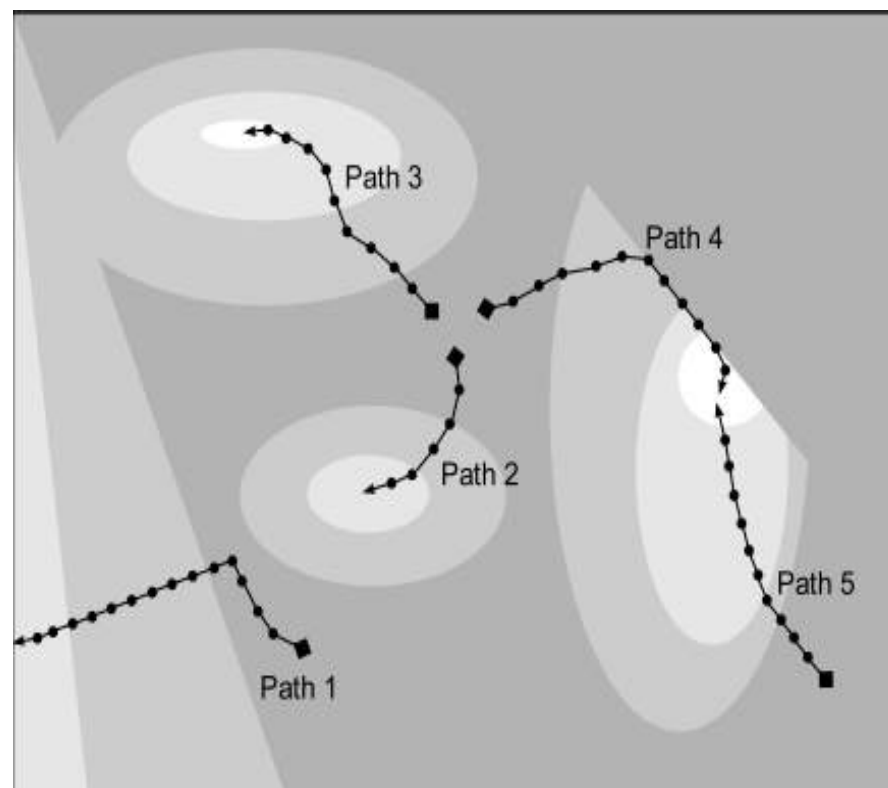


Output Data

→ **Linear Relation between: Ingredient – Property.**
Calculation of compounds with linear algorithm.
Approximisation to target(s) via multiple iteration



- l **The GrafCompounder uses the Multiple Linear Iteration method [MLI] to calculate a new recipe according to properties targeted**
- l **The GrafCompounder enables the user to analyze and improve their compound database via identification of faulty data sets**
- l **Each compound taken into account for the calculation and its influence of each on the final result is visualized.**
 - **Its contribution is given as a ratio**
- l **The GrafCompounder is a fast and easy to use tool without utilizing a complex “hidden” mathematical and analytical method**
- l **The GrafCompounder works with smaller and larger Databases**



Calculation method confirmation

- **Prove with**
 - 1. NR Filler / Oil DoE – most of basic physicals are linear**
 - 2. Filler / Oil DoE**
 - 3. Accelerator DoE**

DoE with 4 Factors

Polymer used: EPDM (Vistalon 8600)

Factor Name	Units	Min	Max
– A C6630	phr	60.00	95.00
B CaCO3	phr	10.00	70.00
C Clay	phr	10.00	50.00
D Oil	phr	70.00	95.00

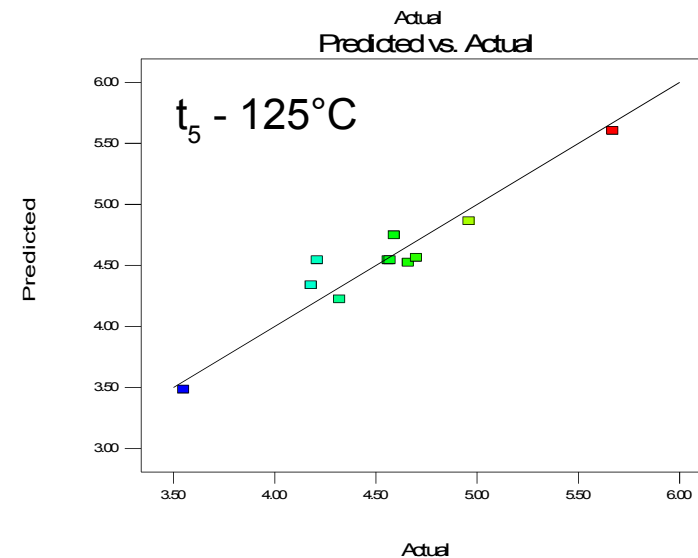
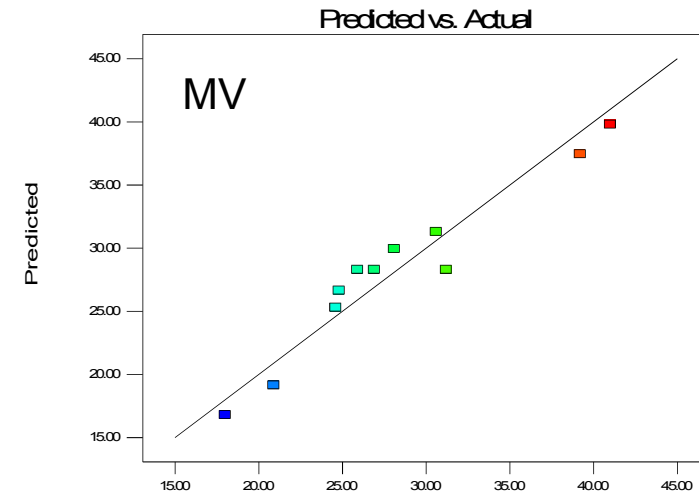


Example 1.

- **A fractional factorial DoE with 11 compounds only!**

Rheology Data are examined

- **MV and $t_5 - 125^\circ\text{C}$ can be measured quite accurate.**
- **Both are significant with a linear model equation**

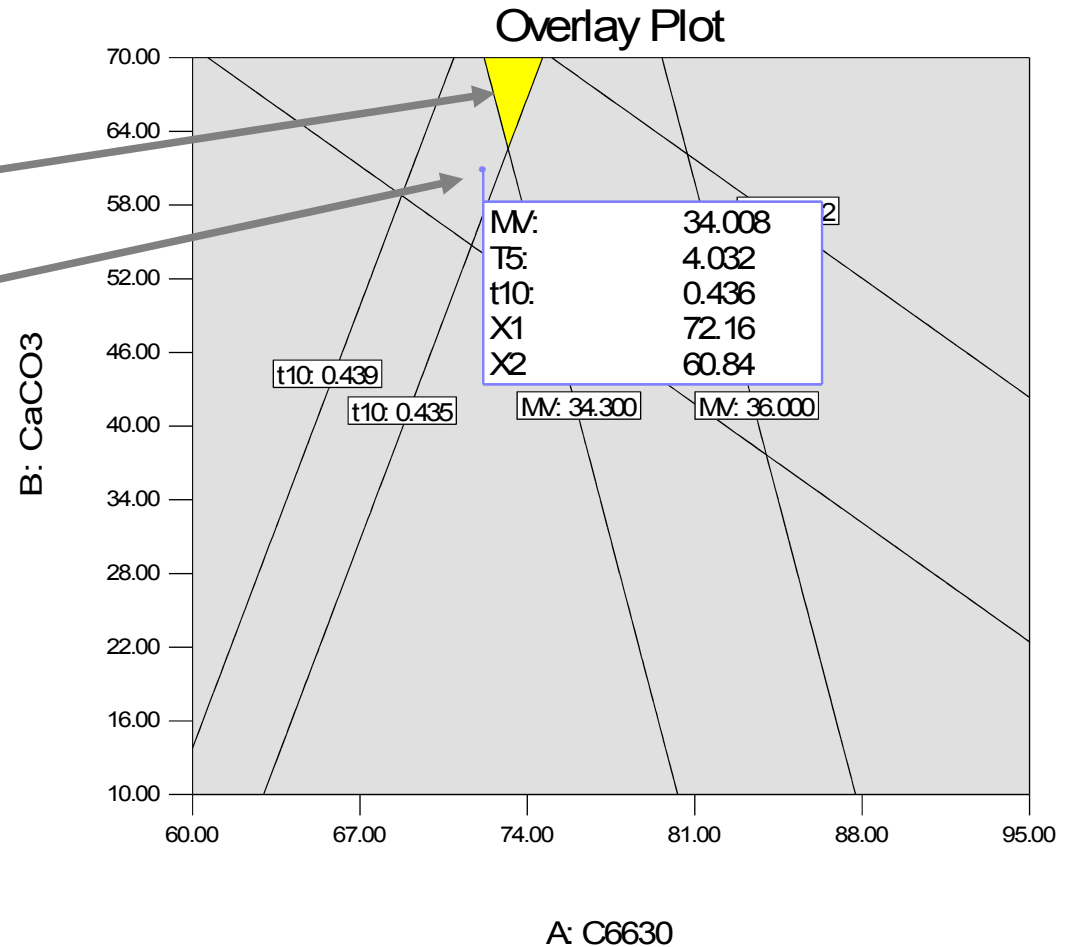


Optimization area

calculated with DoE Software:
 Design Expert®

calculated with
 GrafCompounder

- *boundary condition
 (CC 6630 – 73 phr)*





Example 2

DoE published by DuPont Dow in 1998

- Factors: ENB, DTDC, S, MBT, TiTBD, ZdiBC, DTP
- DoE with 41 Experiments

Tensile at break is significant with linear model

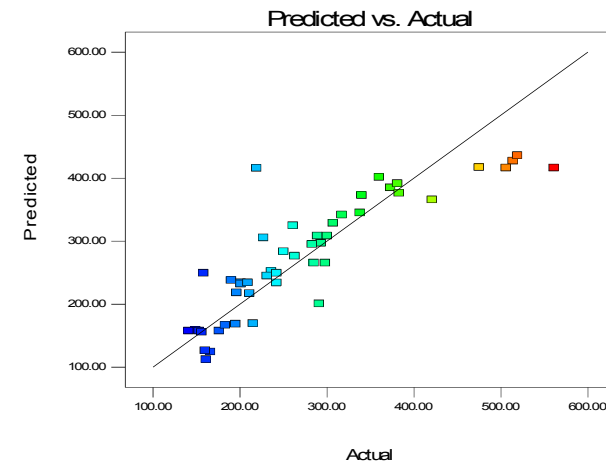
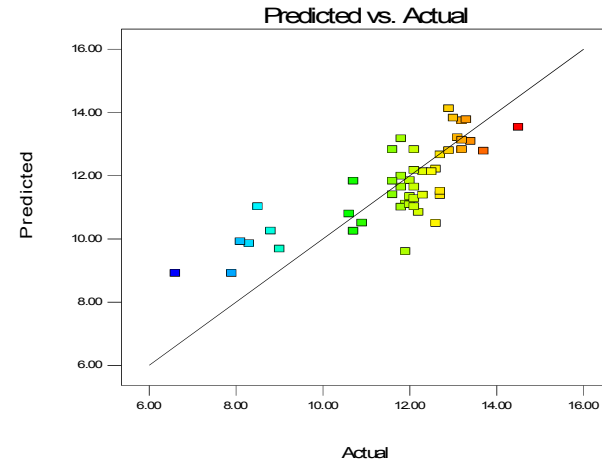
- Sulfur has larger influence followed by DTDC and TiTBD, but negative

Elongation is significant with quadratic model, but linear model is a more than sufficient fit

- Sulfur has the largest influence followed by DTDC

Hardness is sufficient significant with linear model as well

- Main influence Sulfur, DTDC



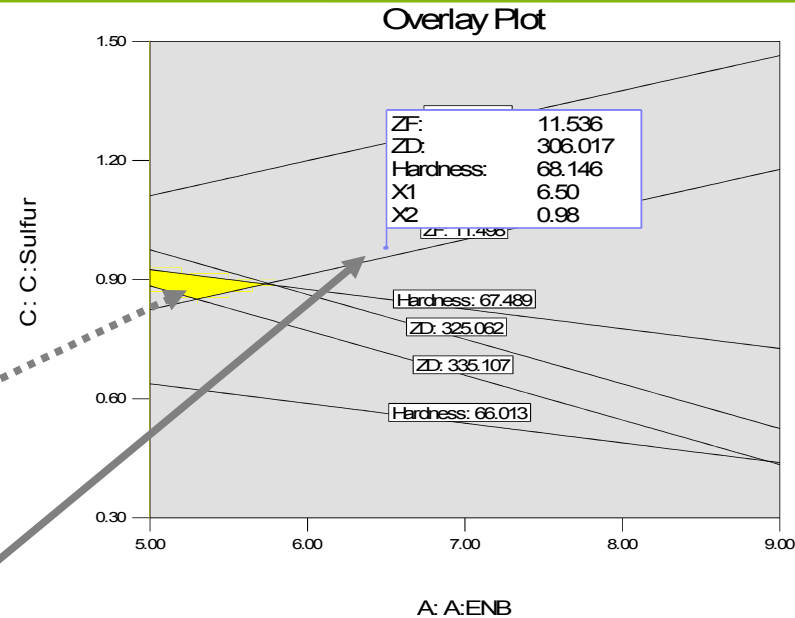


Boundary Conditions

- **Select boundaries**
- **TB-MPa: 11.5 - 12.0**
- **EB-% : 325 - 335**
- **H-°ShA: 65 - 67**

The Design Expert® optimization graph shows the location of the result as a yellow area.

GrafCompounder result is tagged with a flag.



Ingredients	Calculation Method	
	<u>GrafCompounder</u>	Design Expert®
A: ENB	6.5	5.45
C: <u>Sulfur</u>	0.93	0.88
B: DTDC	0.98	0.98
D: MBT	1	1
E: <u>TiBTD</u>	1.51	1.51
F: <u>ZDiBC</u>	1.33	1.33
E: DTP	1.45	1.44
Tensile @Break	11.5	11.5
Elongation @Break	306	330
Hardness	68	67

- **Simulation of a DoE**
- **Experiments made in the Laboratory**



Example 3

■ NR based Compound	LL	UL
■ Filler 1: CB 336	30 phr	70 phr
■ Filler 2: CB 550	0 phr	20 phr
■ Oil: Naphtenic Oil	5 phr	45 phr

- **Type of DoE: fractoinal factorial**
- **Software: Design Expert®**
- **Calculation made with GrafCompounder**
 - **NR Formula index from MRPRA**
 - **Formula data adjusted, but responses taken as is.**

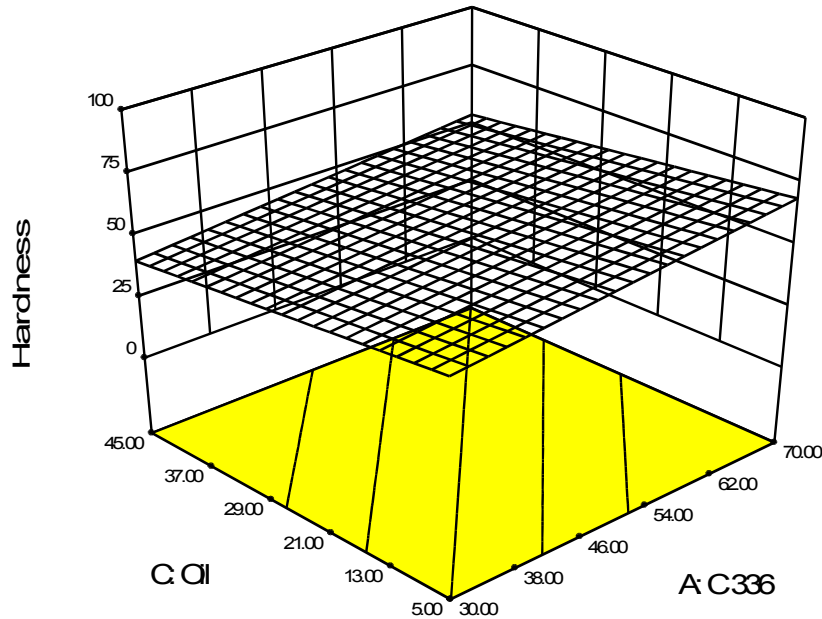
★ **For comparison: Hardness, Tensile - / Elongation at break**

Hardness:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

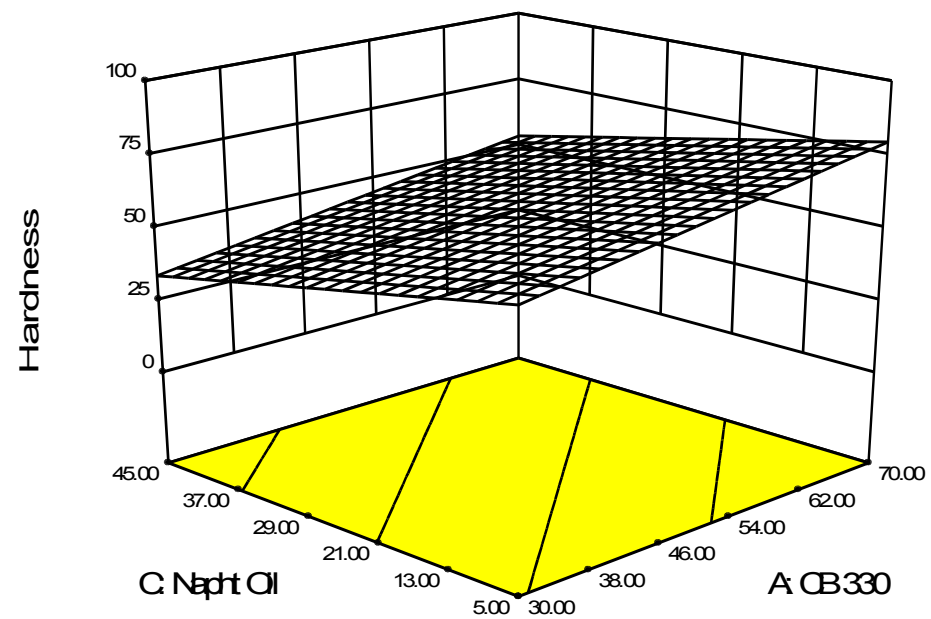


Hardness Simulation

X1 – A: CB 330

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

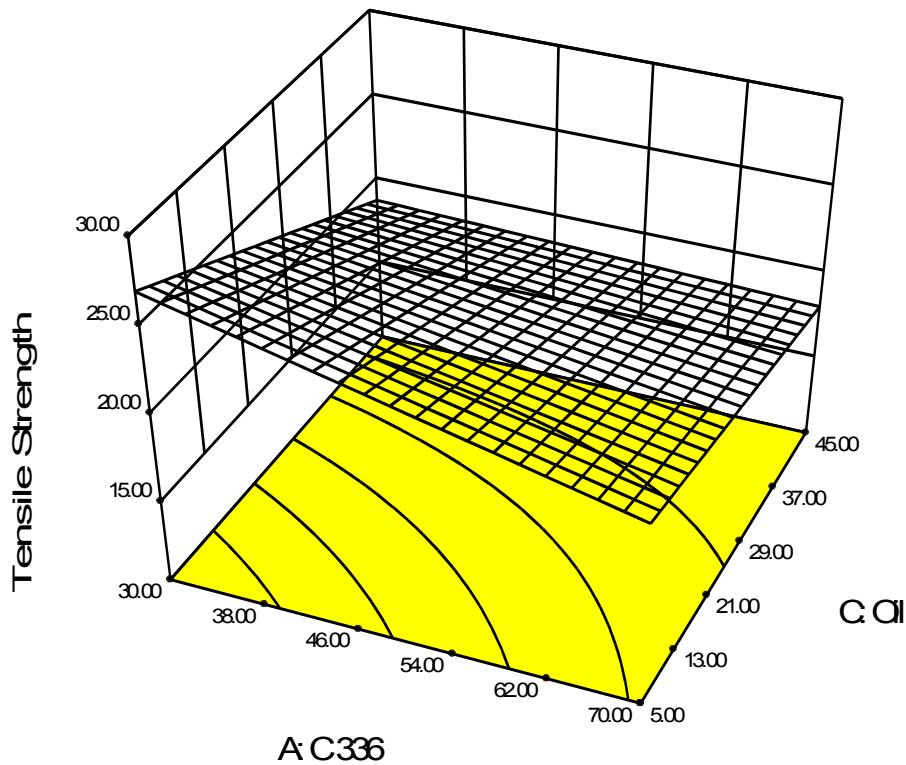


Tensile at break:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

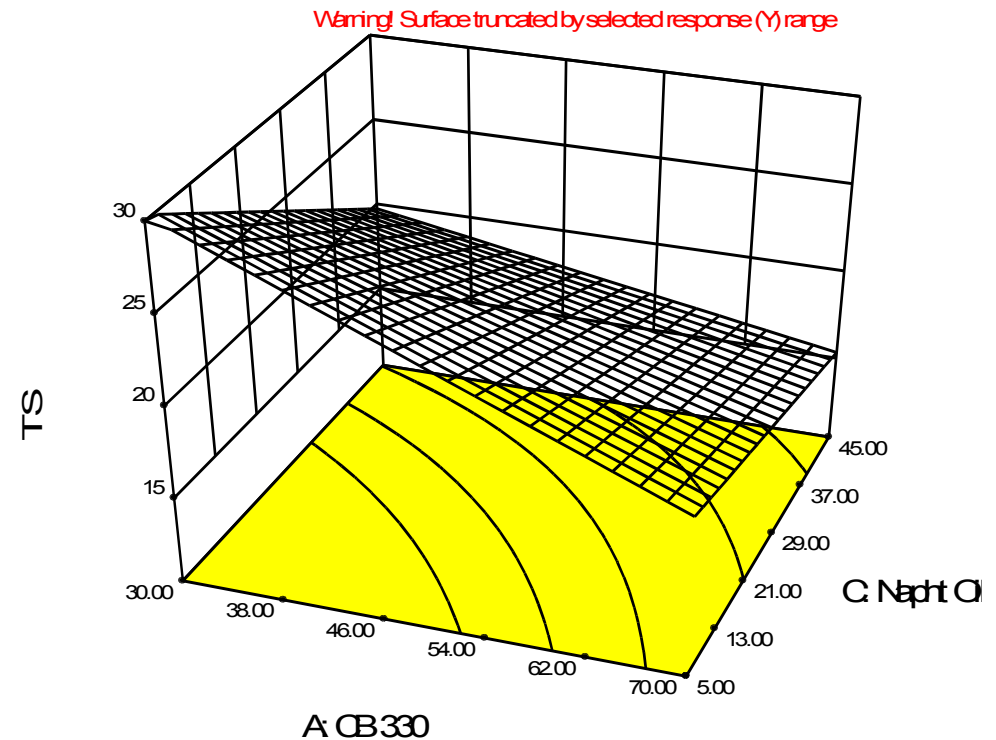


Tensile at break Simulation

X1 – A: CB 330

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

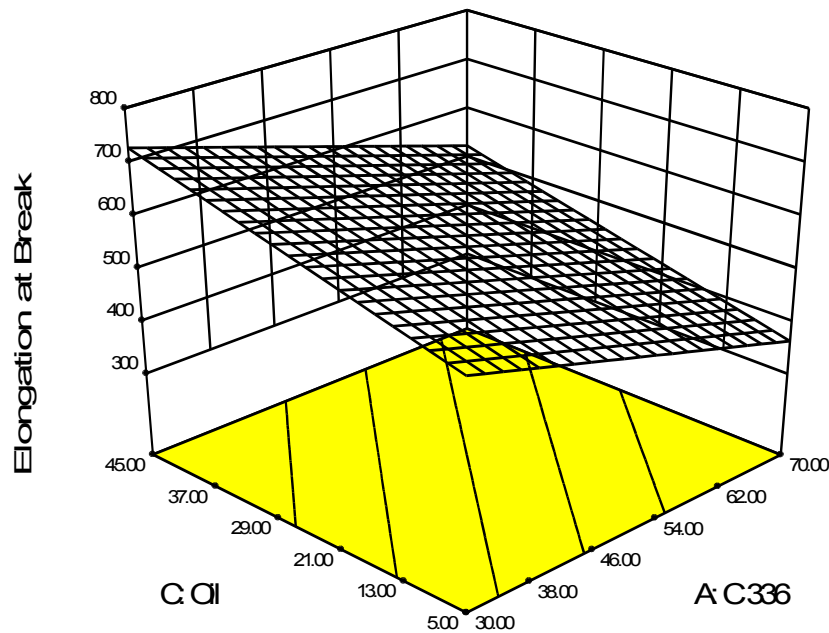


Elongation at break:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

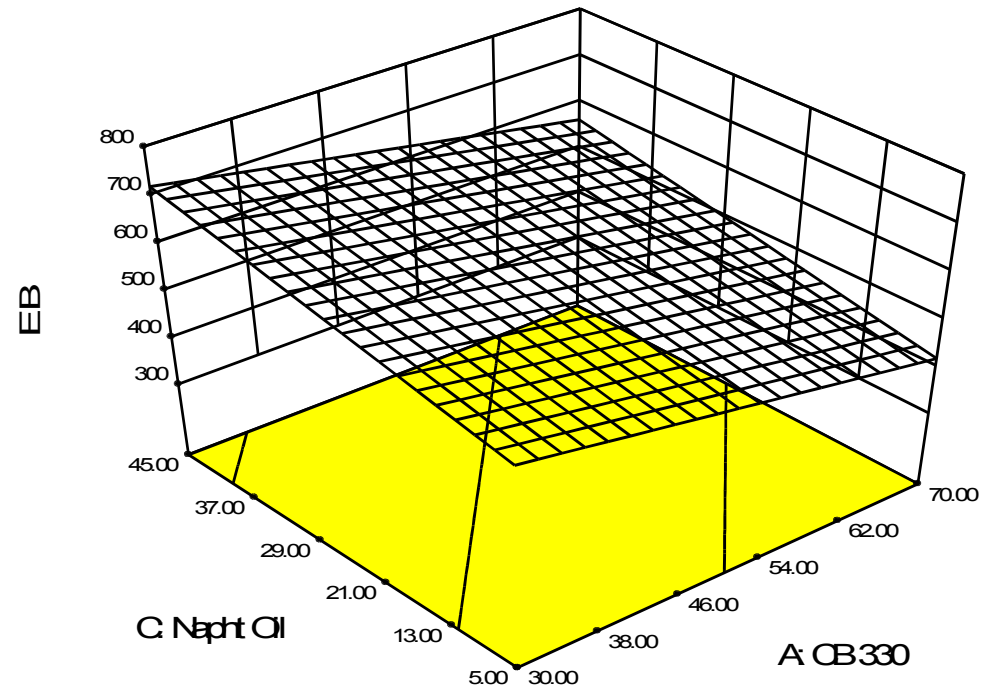


Elongation at break Simulation

X1 – A: CB 330

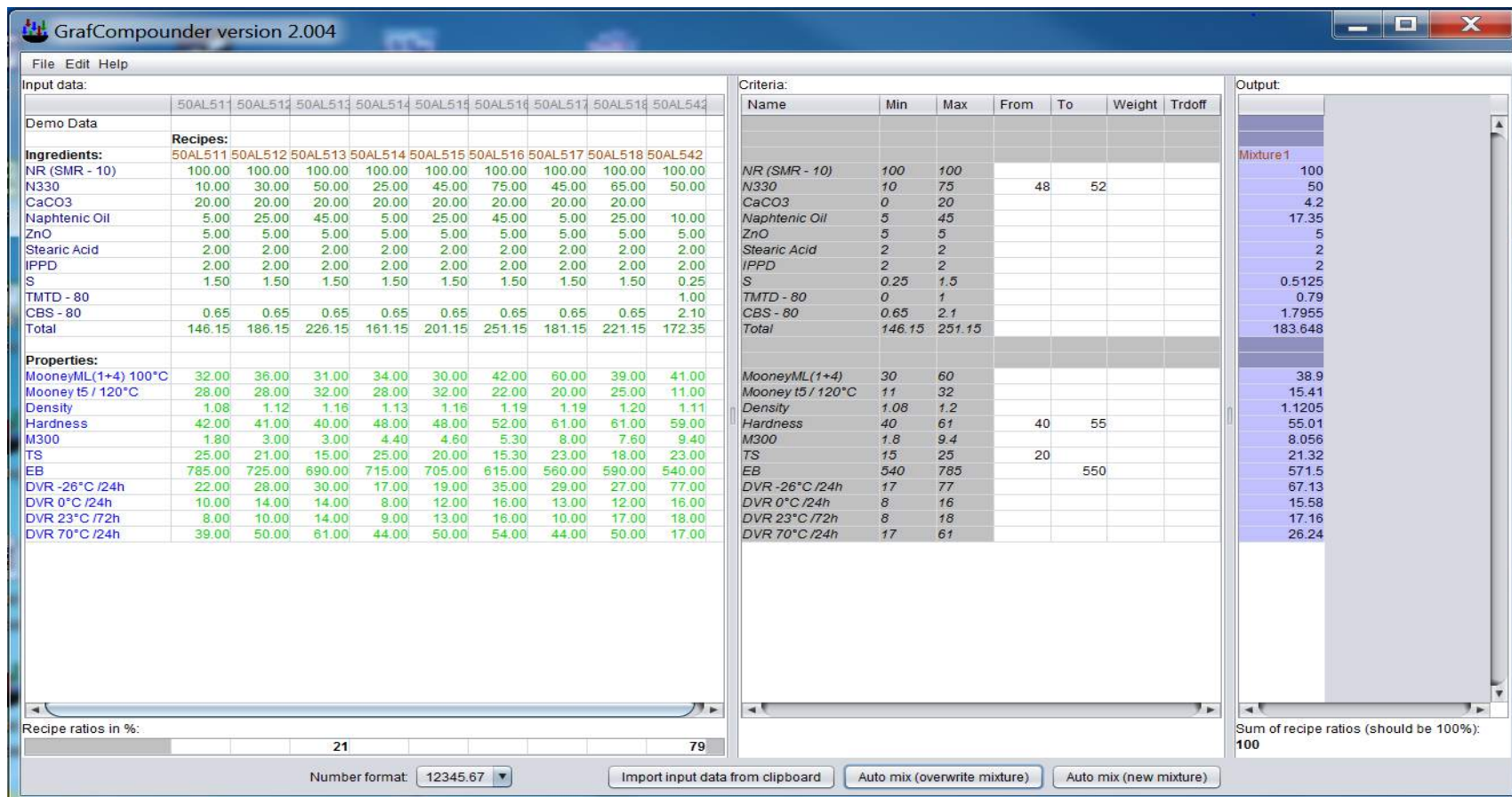
X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr



Outlook

Screenshot of GrafCompounder V 2.004 with demo data, targets and a calculated compound



The screenshot displays the GrafCompounder V 2.004 software interface. It is divided into several sections:

- Input data:** A table showing demo data for various ingredients across ten recipes (50AL511 to 50AL542).
- Ingredients:** A list of ingredients including NR (SMR - 10), N330, CaCO3, Naphtenic Oil, ZnO, Stearic Acid, IPPD, S, TMTD - 80, CBS - 80, and Total, with their respective weights for each recipe.
- Properties:** A table showing target properties for various materials like MooneyML(1+4) 100°C, Mooney t5 / 120°C, Density, Hardness, M300, TS, EB, and DVR at different temperatures and times.
- Criteria:** A table defining the target ranges (Min, Max) and the calculated values (From, To, Weight, Trdoff) for each ingredient.
- Output:** A list of the calculated mixture components and their weights, including Mixture1, NR (SMR - 10), N330, CaCO3, Naphtenic Oil, ZnO, Stearic Acid, IPPD, S, TMTD - 80, CBS - 80, Total, MooneyML(1+4), Mooney t5 / 120°C, Density, Hardness, M300, TS, EB, DVR - 26°C / 24h, DVR 0°C / 24h, DVR 23°C / 72h, and DVR 70°C / 24h.

At the bottom, there are controls for 'Recipe ratios in %' (21 and 79), 'Number format' (12345.67), and buttons for 'Import input data from clipboard', 'Auto mix (overwrite mixture)', and 'Auto mix (new mixture)'.

Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound

The screenshot displays the GrafCompounder V 3.210 interface. It is divided into several sections:

- Input data:** A table with columns for recipes (50AL511 to 50AL542) and rows for ingredients and properties.
- Criteria:** A table with columns for Name, Min, Max, From, To, Weight, and Trdoff.
- Output:** A list of calculated values for the mixture, including Total Ingredients, Density, Cost, and Sum of recipe ratios.

	50AL511	50AL512	50AL513	50AL514	50AL515	50AL516	50AL517	50AL518	50AL542
NR (SMR - 10)	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
N330	10.00	30.00	50.00	25.00	45.00	75.00	45.00	65.00	50.00
CaCO3	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	
Naphtenic Oil	5.00	25.00	45.00	5.00	25.00	45.00	5.00	25.00	10.00
ZnO	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Stearic Acid	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
JPPD	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
S	1.50	1.50	1.50	1.50	1.50	1.50	1.50	1.50	0.25
TMTD - 80									1.00
CBS - 80	0.65	0.65	0.65	0.65	0.65	0.65	0.65	0.65	2.00

Name	Min	Max	From	To	Weight	Trdoff
NR (SMR - 10)	100	100				
N330	10	75	48	52		
CaCO3	0	20				
Naphtenic Oil	5	45				
ZnO	5	5				
Stearic Acid	2	2				
JPPD	2	2				
S	0.25	1.5				
TMTD - 80	0	1				
CBS - 80	0.65	2.1				

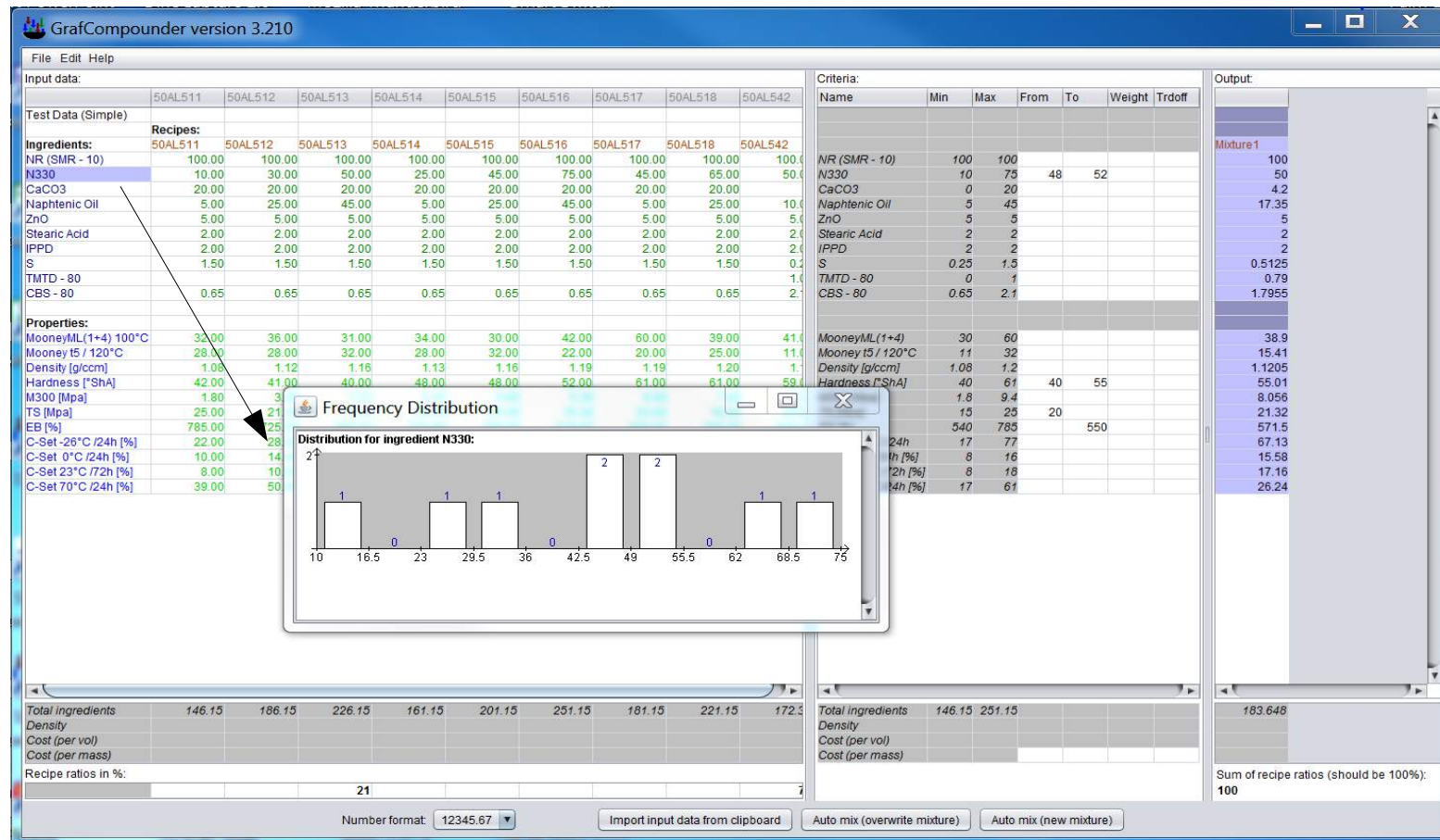
Mixture	Value
Mixture1	100
	50
	4.2
	17.35
	5
	2
	2
	0.5125
	0.79
	1.7955
	38.9
	15.41
	1.1205
	55.01
	8.056
	21.32
	571.5
	67.13
	15.58
	17.16
	26.24
Total Ingredients	183.648
Density	
Cost (per vol)	
Cost (per mass)	
Sum of recipe ratios (should be 100%)	100

At the bottom of the interface, there are summary statistics and controls:

- Number format: 12345.67
- Buttons: Import input data from clipboard, Auto mix (overwrite mixture), Auto mix (new mixture)

Outlook

Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound





Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound

The screenshot displays the GrafCompounder V 3.210 software interface. It is divided into several main sections:

- Input data:** A table with columns for Test Data (Advanced) and Recipes (50AL511 to 50AL515). It lists various materials like NR, CaCO3, Naphtenic Oil, ZnO, Stearic Acid, IPPD, S, and TMTD - 80, along with their costs and densities.
- Properties:** A table listing target properties such as MooneyML(1+4) 100°C, Mooney t5 / 120°C, Density [g/ccm], Hardness [°ShA], M300 [Mpa], TS [Mpa], EB [%], and C-Set at various temperatures and times.
- Criteria:** A table defining the search criteria for the calculated compound, including Name, Min, Max, From, To, Weight, and Trdoff.
- Output:** A table showing the results of the calculation, including the calculated compound's name, its properties, and the recipe ratios.

At the bottom of the interface, there are summary statistics for the total ingredients, density, cost (per vol and per mass), and recipe ratios. The recipe ratios are shown as 0.25 for all five recipes.



Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound

The screenshot displays the GrafCompounder V 3.210 interface. The main window is divided into several sections:

- File Menu:** Includes options like 'Clear All Data', 'Load Demo Data (Simple)', 'Open File...', 'Save As...', 'Merge in Recipes from Clipboard', 'Merge in Recipes from File' (highlighted by an arrow), and 'Exit'.
- Recipe Table:** A table with columns for ingredients (Density, Ingredients) and recipes (50AL511 to 50AL515). It lists various materials like NR (SMR - 10), N330, CaCO3, Naphtenic Oil, ZnO, Stearic Acid, IPPD, S, TMTD - 80, and CBS - 80.
- Criteria Table:** A table with columns for Name, Min, Max, From, To, Weight, and Trdoff. It lists properties like MooneyML(1+4) 100°C, Mooney 15 / 120°C, Density [g/ccm], Hardness [°ShA], M300 [Mpa], TS [Mpa], EB [%], and C-Set at various temperatures.
- Output Table:** A table showing the calculated mixture components and their weights, including 'Mixture 1' and various material weights.
- Summary Tables:** Located at the bottom, showing 'Total ingredients', 'Density', 'Cost (per vol)', and 'Cost (per mass)' for both the recipe and the calculated mixture.
- Bottom Bar:** Contains a 'Number format' dropdown set to 12345.67 and buttons for 'Import input data from clipboard', 'Auto mix (overwrite mixture)', and 'Auto mix (new mixture)'.

Recipe manager

- λ **Creation of a formula according predefined criteria**
 - ∨ **Ingredients**
 - ∨ **Properties**
 - ∨ **Cost**
- λ **Trace ability back to the starting formulas**
 - ∨ **Analysis of outliers and their correction or elimination in the database is possible.**
 - ∨ **Integration of results from statistical experimental designs with merge function.**
 - ∨ **Integration of databases of different origin, provided that an export of the data is possible with table calculation programs.**

Result of the calculations MUST be confirmed by an experiment.

- λ **Probability of a match between calculation and confirmation experiment result is about 90-5% according first experience**

Examples show:

The resulting formulas calculated correspond to the general rules of compounding

- **Differences with calculations based on regression obtained with DoE is marginal**

The formulas will show property scores larger than the 90 % – 95 % confidence interval in confirmation experiment

Only one confirmation experiment would be needed as opposed to multiple trials in case of development targets.

- **Starting formula calculated with GrafCompounder**
- **Optimized formula with Optimization Tool in DoE Software**

More information under: www.grafcompounder.com



Release of the „GrafCompounder“ Version 3.210

Thank you for joining this presentation.

Any question, comment?

More information under: www.grafcompounder.com