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ITECH

Using Experimental Design Approach to Understand Interactions of Natural Oils and Waxes in a Lipstick Base

Carmen Cheung , Pascale Cottin, Michèle Lepais, Slobodanka Tamburic

Talk at a glance

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2. Why specific experimental design?
3. Aims and objectives of the study
4. Methodology
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   - Experimental design
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1. Why natural lipstick base?

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93%

6% ↑

£1.5 billion

(2013)

15% lip category

MINTEL

1. Why natural lipstick base?

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Recent Mintel report on Colour Cosmetics (2012):

- Increased consumers' awareness of their impact on the environment
- Increased focus on naturally derived and more sustainable ingredients
- Companies are producing eco-friendlier products

1. Why natural lipstick base?

Technical requirements for natural lipsticks are the same as for traditional ones, e.g.:

- Should have sufficient stick strength
- Should have good pay off
- Should not cause pigment exudation
- Should not crystallise

2. Why specific experimental design?

- Vast number of variables and possible experiments
- The use of experimental design approach
  - increases the *effectiveness* of research
  - reduces *experimentation time*
  - improves the *quality* of the information obtained
- **Mixture Design** enables us to study several variables at the same time, with the minimum number of observations, in the shortest time and the lowest costs.
- Mixture Design was implemented using the programme NEMRODW® (LPRAI, France)

3. Aim and objectives of the study

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Aim:

To assess the applicability of the Mixture Design approach to the formulation of the natural lipstick base

Objectives:

- To gain an understanding of the interaction between different raw materials in the lipid base
- To create an optimised lipid base formula
- To assess its performance against a commercial natural lipstick

4. Methodology

Natural materials

Three most commonly used waxes in natural lipsticks:
carnauba wax, candelilla wax and beeswax

Three most commonly used oils in natural lipsticks:
castor oil, jojoba oil and sunflower oil

The benchmark: product X
from the French market

4. Methodology

Waxes

Wax	Melting Point (°C)	Composition
Carnauba	87	Esters and polyesters of mono and di-hydroxy acids and fatty alcohols
Candelilla	67	esters and hydrocarbon waxes.
Beeswax	63	esters (70%) and free wax acids and hydrocarbons (30%)

4. Methodology

Oils

Oil	Chain Length	Composition
Castor	Long	90% ricinoleic acid means more polar than others
Sunflower	Long	Monounsaturated mix of oleic and linoleic acid
Jojoba	Long	Over 97% liquid wax ester of omega-9 fatty acid and omega-9 fatty alcohol

4. Methodology

Base formula

Phase	%w/w	Functions
Wax	20	- Stick structure - Heat stability - Hard texture on application - Helps with the stick removal
Oil	50	- Immediate properties of lipsticks: ease of application, shine, glide
Butter	15	- Helps the structure - Brings smoothness, melting and shine
Pigment (organic) in castor oil	15	- Colour - Coverage

4. Methodology

Evaluation: instrumental and sensory methods

Every lipstick was evaluated 24 hours after it was produced.

- **Measurement of hardness (break point test)**

  -> An indication of stability – lipstick should not break when force is applied on application

  -> Target: 300-450g

  (corresponds to the weight required to break the stick of lipstick using water)

- **Measurement of softening (softening point test)**

  -> Ring and ball method

  -> An indication of stability at high temperatures

  -> Target: 69-76°C

4. Methodology

Evaluation: instrumental and sensory methods

- **Sensory test - measurement of glide (glide test)**

  Rating scale: 0-10

  Test site: the back of hand

  Panel: untreated volunteers

  Protocol: rating the ease of application (glide) on the scale of 0 to 10 after applying 3 continuous strokes on the back of the hand

Stability test:

Unchanged physical characteristics for one month at:

Room temperature (20-22^{0}C), under northern light

Thermostatic oven (45^{0}C)

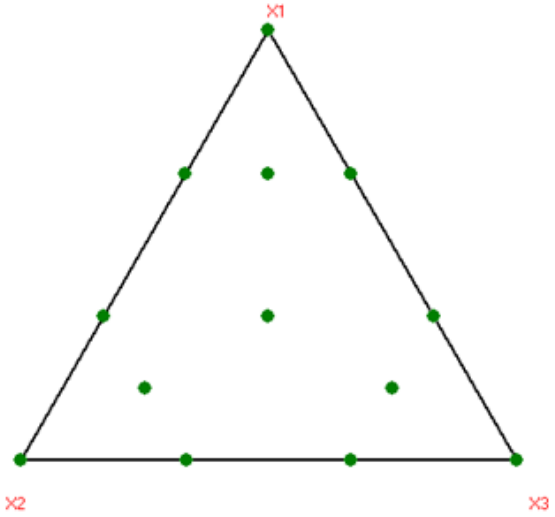
4. Methodology

Optimisation Model: Simplex-Based Mix Design

Method based on the Scheffé's simplex theory

- 16 lipstick formulations were tested (13 from the matrix + 3 at random)

Simplex lattice design for three components



Mathematical Model

$$Y = b1 * X1 + b2 * X2 + b3 * X3 + b12 * (X1*X2) + b13 * (X1*X3) + b23 * (X2*X3) + b1212 * (X1*X2*(X1-X2)) + b1313 * (X1*X3*(X1-X3)) + b2323 * (X2*X3*(X2-X3)) + b123 * (X1*X2*X3)$$

4. Methodology

Experimental design

- Stage 1 - Determination of the optimal wax ratio
- Stage 2 - Determination of the optimal oil ratio
- Stage 3 - Comparative assessment of the optimised lipstick formulation and the market product X

4. Methodology

Stage 1- Determination of the wax ratio

- Total amount of waxes was maintained at 20%, while experimenting with different ratios of waxes
- Oils and pastes were maintained at 50% and 15%, respectively
- 16 samples were generated by the Simplex Lattice Design, using NEMRODW® software
- All experiments were run with three test oils sequentially
- Results were analysed using the NEMRODW® software and the optimum wax ratio was determined.

4. Methodology

Stage 2 - Determination of the oil ratio

- The oil phase was maintained at 50%
- The optimum wax ratio determined in Stage 1 was incorporated in the formula
- 16 samples were generated by the Simplex Lattice Design
- Results were analysed using the NEMRODW® software and the optimum oil ratio was determined
- An optimised formulation was proposed based on the best wax and oil ratios

Stage 3 - Comparative assessment

- The optimised formulation was compared with the market product X in terms of hardness, softening and glide

4. Methodology

Stage 1 - Lipstick formula

Phase	INCI Name	Trade Name	Supplier	%w/w
Wax	Copernicia Cerifera Wax	Cerauba T1 Bio	Baerlocher France	*to find
	Cera Alba	Cerabio W	Baerlocher France	*to find
	Euphorbia Cerifera Wax	Cerilla Raffinée Paillettes	Baerlocher France	*to find
Oil	Ricinus communis Seed Oil/ Simmondsia Chinensis Seed Oil/ Helianthus Annuus Seed Oil	Castor Oil/Jojoba Oil/Sunflower Oil	Aldivia	50.0
Butter	C10-18 Triglycerides	Lipocire A	Gattefossé	12.0
	Butyrospermum parkii (Shea) butter	Shea Butter	Aroma Zone	3.0
Pigment	Red #7, Ricinus communis Seed Oil	COD8001	ITECH Lyon	15.0

4. Methodology

Matrix of experiments: wax phase (20%w/w)

N°Exp	Carnauba wax	Candelilla wax	Beeswax
1	1.00000	0.00000	0.00000
2	0.00000	1.00000	0.00000
3	0.00000	0.00000	1.00000
4	0.66667	0.33333	0.00000
5	0.33333	0.66667	0.00000
6	0.66667	0.00000	0.33333
7	0.33333	0.33333	0.33333
8	0.00000	0.66667	0.33333
9	0.33333	0.00000	0.66667
10	0.00000	0.33333	0.66667
11	0.66667	0.16667	0.16667
12	0.16667	0.66667	0.16667
13	0.16667	0.16667	0.66667

4. Results of Stage 1

Wax interaction with different oils

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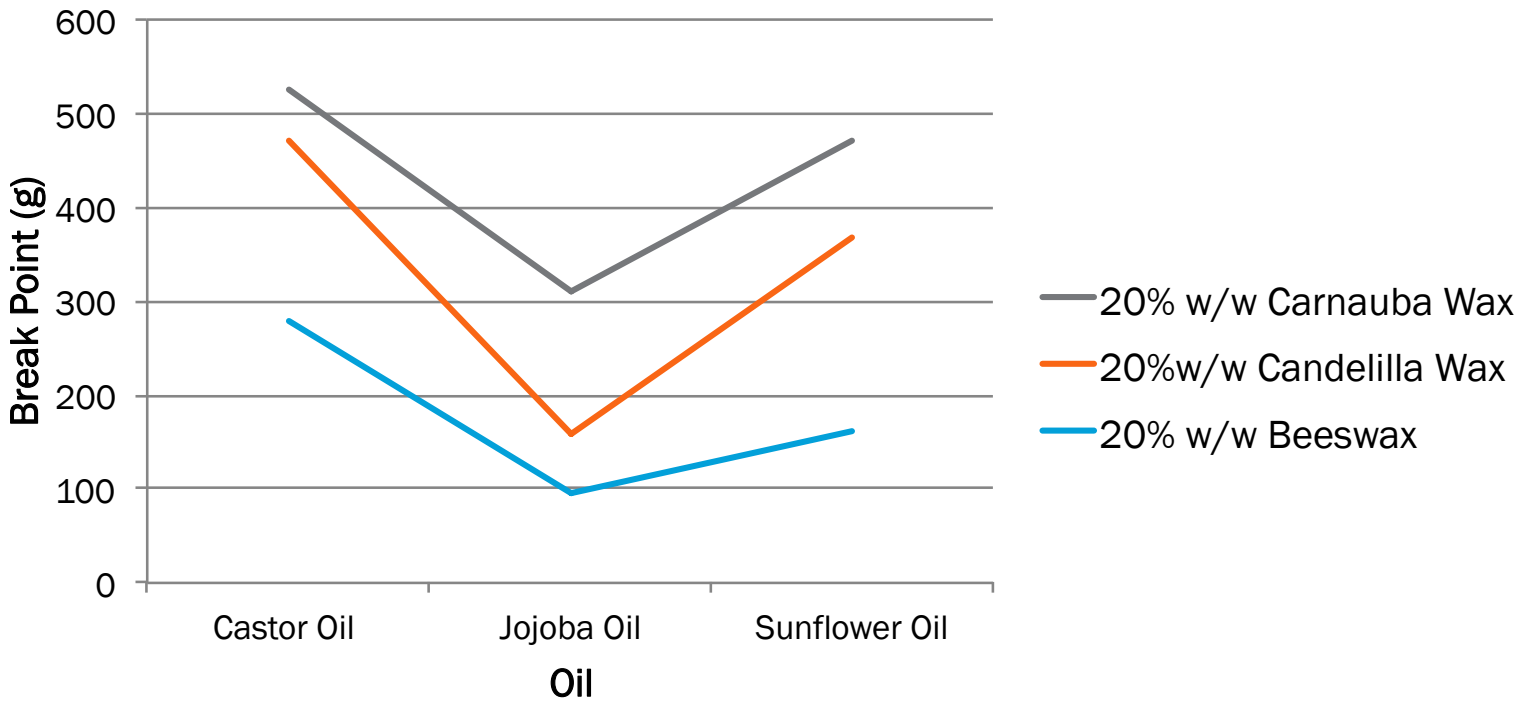
Break Point

Comparing the break point of waxes in different oils



4. Results of Stage 1

Castor oil

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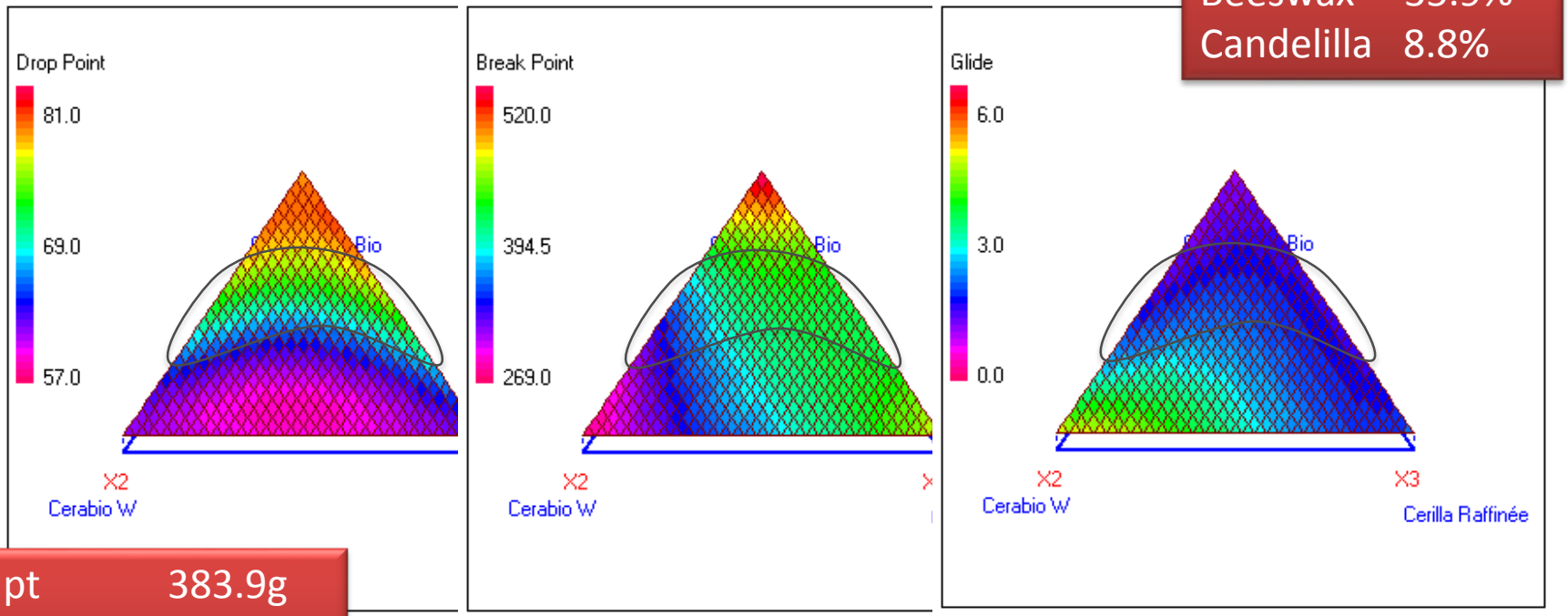
3D Graphs

Carnauba	55.5%
Beeswax	35.9%
Candelilla	8.8%



Break pt	383.9g
Softening pt	70°C
Glide	2

4. Results of Stage 1

Jojoba oil

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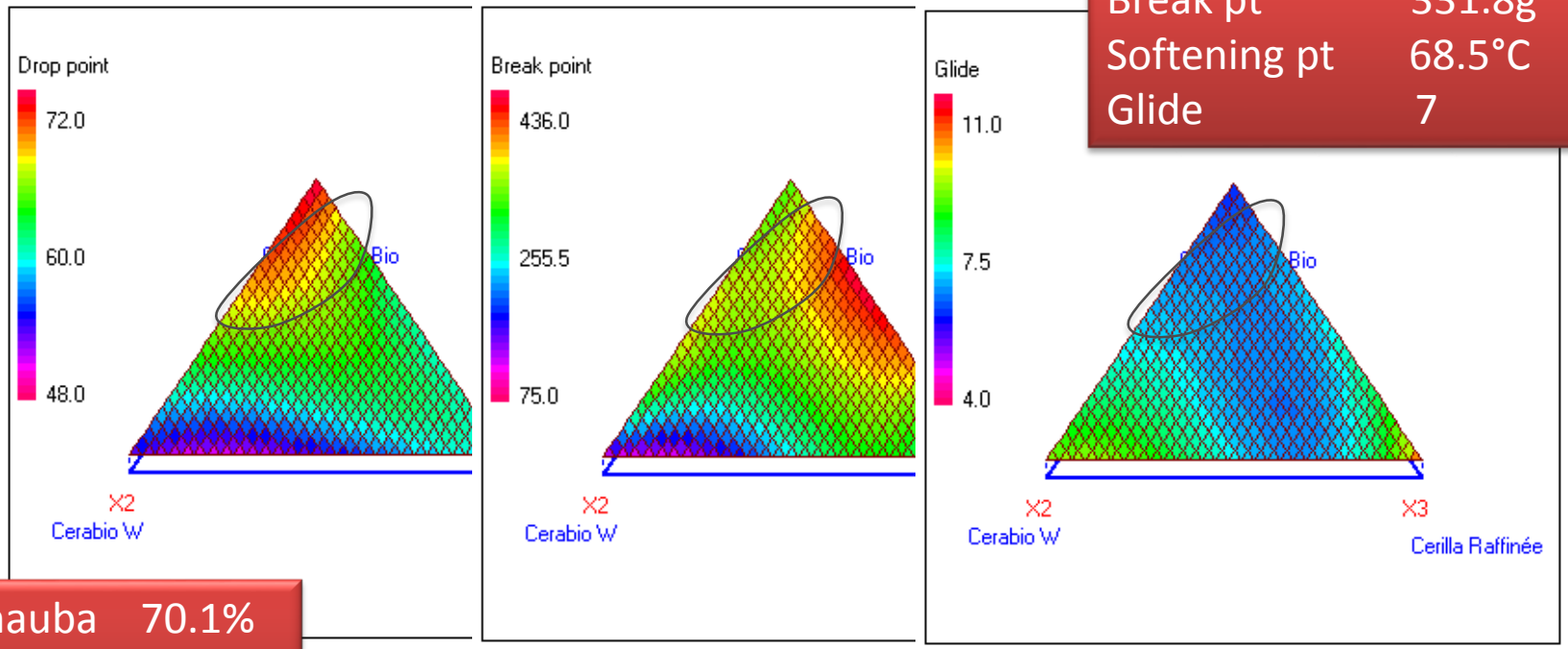
3D Graphs



Break pt	331.8g
Softening pt	68.5°C
Glide	7

Carnauba	70.1%
Beeswax	29.3%
Candelilla	0.6%

4. Results of Stage 1

Sunflower oil

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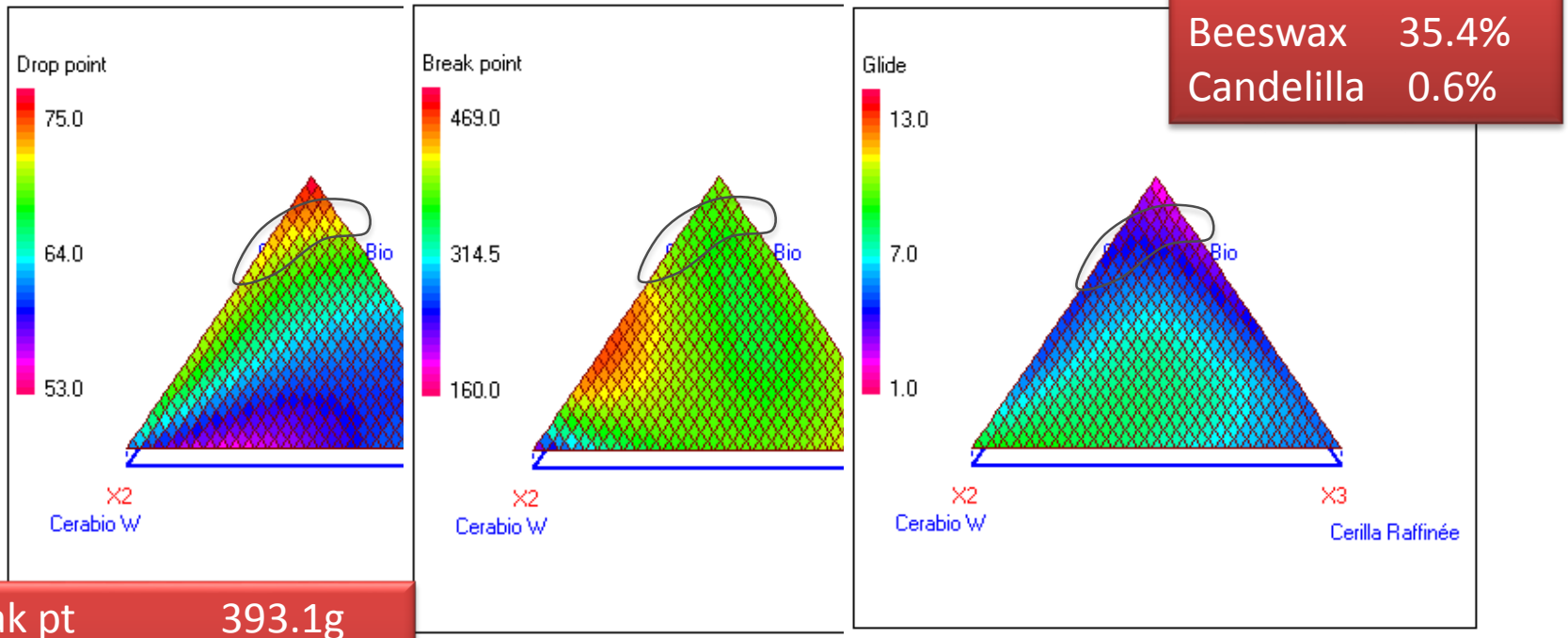
3D Graphs



Carnauba	64%
Beeswax	35.4%
Candelilla	0.6%

Break pt	393.1g
Softening pt	69.2°C
Glide	4.4

4. Results of Stage 2

Lipstick formula

Phase	INCI Name	%w/w
Wax	Copernicia Cerifera Wax	10.7
	Cera Alba	8.3
	Euphorbia Cerifera Wax	1.0
Oil	Ricinus Communis Seed Oil	*to find
	Simmondsia Chinensis Seed Oil	*to find
	Helianthus Annuus Seed Oil	*to find
Butter	C10-18 Triglycerides	12.0
	Butyrospermum Parkii (Shea) Butter	3.0
Pigment	Red #7, Ricinus Communis Seed Oil	15.0

Carnauba 53.5%
Beeswax 41.5%
Candelilla 5%

4. Results of Stage 2

Matrix of experiments: wax phase (20%w/w)

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N°Exp	Castor oil	Jojoba oil	Sunflower oil
1	1.00000	0.00000	0.00000
2	0.00000	1.00000	0.00000
3	0.00000	0.00000	1.00000
4	0.66667	0.33333	0.00000
5	0.33333	0.66667	0.00000
6	0.66667	0.00000	0.33333
7	0.33333	0.33333	0.33333
8	0.00000	0.66667	0.33333
9	0.33333	0.00000	0.66667
10	0.00000	0.33333	0.66667
11	0.66667	0.16667	0.16667
12	0.16667	0.66667	0.16667
13	0.16667	0.16667	0.66667

4. Results of Stage 2

Oil ratio

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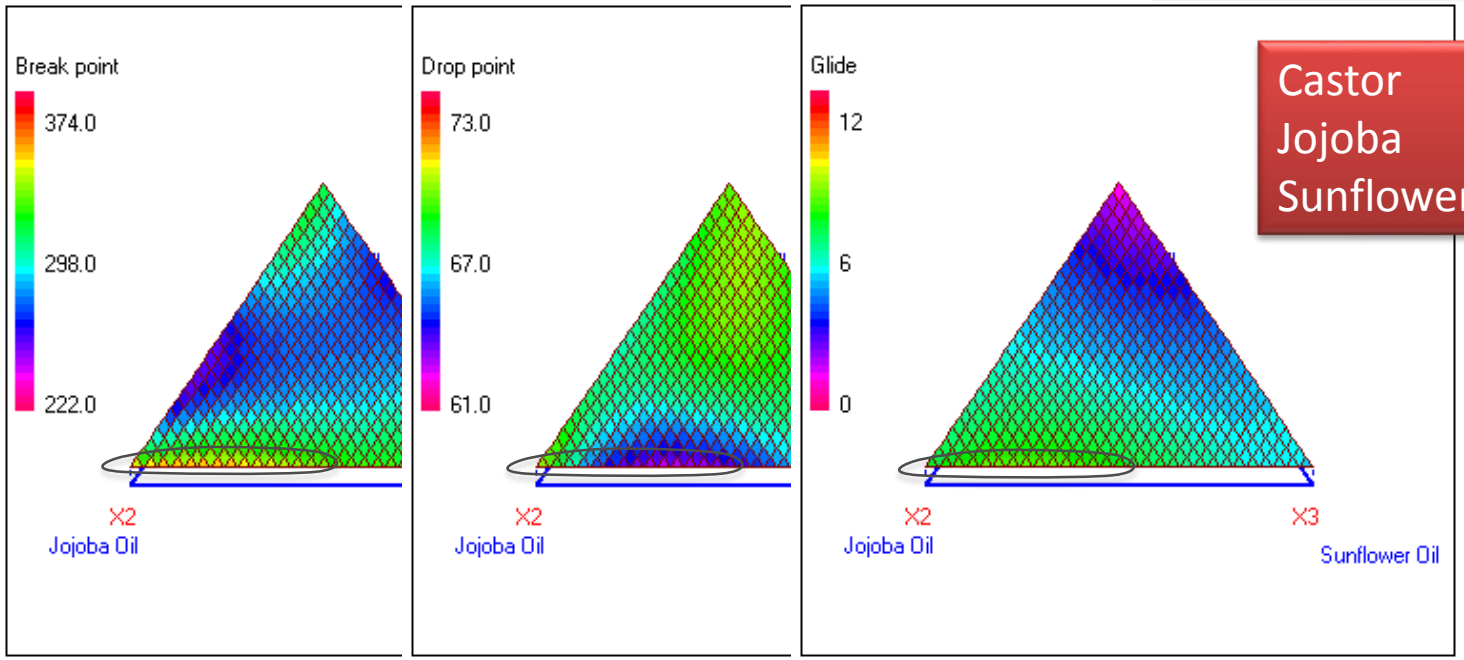
Break pt	352.9g
Softening pt	66.3°C
Glide	8

3D graphs



Castor	1.6%
Jojoba	90.3%
Sunflower	8.1%

4. Results of Stage 2

Optimised formulation

Summary of optimised wax ratios (within a total of 20%w/w) for castor, jojoba and sunflower oil

Wax / Oil	Carnauba wax (%)	Beeswax (%)	Candelilla wax (%)	Break point (g)	Softening point (°C)	Glide
Castor	55.5	35.9	8.8	383.9	70	2
Jojoba	70.1	29.3	0.6	331.8	68.5	7
Sunflower	64	35.4	0.6	393.1	69.2	4.4

4. Results

Optimised formulation

Phase	INCI	Stage 1 (%w/w)	Stage 2 (%w/w)
Wax	Copernicia Cerifera Cera	10.7	10.7
	Cera Alba	8.3	8.3
	Candelilla Cera	0.9	0.9
Oil	Ricinus Communis Seed Oil		0.8
	Simmondsia Chinensis Seed Oil		45.15
	Helianthus Annuus Seed Oil		4.05
Butter	C10-18 Triglycerides	12	12
	Butyrospermum Parkii (Shea) Butter	3	3
Pigment	Red #7, Ricinus Communis Seed Oil	15	15

Castor 1.6%
Jojoba 90.3%
Sunflower 8.1%

4. Results

What did we learn?

- Carnauba wax provided lipstick hardness, but impacted negatively on glide
- Beeswax at higher ratios improved lipstick application (glide) due to its weaker structure and lower melting point
- Candelilla wax showed no correlation with any of the three test parameters
- Castor oil showed negative correlation with glide, but considerably increased stability (hardness and softening)
- Jojoba oil showed positive correlation with glide
- Sunflower oil had little effect on glide, but small positive effect on hardness.

6. Comparative assessment

PARAMETER	FINAL FORMULA	BENCHMARK
Break point (g)	431.32 (±30)	159.83 (±30)
Softening point (°C)	69 (±2)	54 (±2)
Glide	7	6

7. Conclusion

- The use of Mixture Design approach was effective in establishing the optimal ratio of natural waxes and oils in a lipstick base
- The accompanying software NEMRODW® was able to predict the responses of experiments that were not carried out
- This approach offers an efficient way to detect interactions among ingredients and to formulate products to required specifications

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