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Optimisation of the wax and oil phases in a conventional lipstick using Mixture Design

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Introduction

Lipstick is essentially composed of oils, waxes and pastes. The type and ratio of these ingredients determine the intensity of their interactions, which directly affects the final quality of the lipstick. Fundamentally, a lipstick must be sufficiently strong to withstand the force during application, but it should also have the appropriate 'pay off' characteristics. Due to the number of variables involved and the two competing requirements, traditional empirical approach may be inefficient in lipstick development. There are several choices of experimental approaches to be employed, including Factorial Design, Cross Design and Mixture Design. In a lipstick mixture, where the proportions of ingredients are more important than the total quantities of ingredients, Mixture Design was deemed most suitable.

Mixture Design is used to model the blending surface with a suitable mathematical equation and to identify the ideal mixture that fulfils the defined responses and their criteria for optimality [1]. One key trait of Mixture Design is that the factors (components) must add up to one. Compared to the traditional one-at-a-time approach, Mixture Design is capable in studying several variables concurrently, with the minimum number of observations, the shortest time and the lowest costs possible.

Aims

1. To evaluate the usefulness of Mixture Design in optimising the wax and oil phases in a conventional lipstick base.
1. To understand the complex interactions between waxes and oils, and the effects of those interactions on the final lipstick characteristics.

Materials and Methods

Materials

A simple formulation containing fixed proportions of waxes (20%), oils (50%), pastes (15%) and pigments (15%) was used. The test waxes were ozokerite wax (OW), beeswax (BW) and candelilla wax (CW); test oils were hydrogenated polyisobutene (HP), octyldodecanol (OD) and sweet almond oil (SAO). As benchmarks, two samples from a contract manufacturer and a French commercial product were used.

Experimental Design

A Design of Experiment software (NemrodW, LPRAI, France) was used to carry out the Mixture Design experiment. In this study, Scheffé's simplex-lattice design was used [2]. There were a total of 13 blend points according to the augmented simplex-lattice design (Figure 1). 3 random points were replicated to estimate the experimental error, giving a total of 16 blend points (Table 1). Scheffé's cubic model (Equation 1) was used for modelling the response data. X1-X3 represent the factor variables, which in this study were the different waxes or oils tested. The response data were analysed by means of ANOVA table, with a significance level of $p<0.05$.

$$\begin{aligned} Y = & \beta 1 * X1 + \beta 2 * X2 + \beta 3 * X3 + \\ & \beta 12 * (X1 * X2) + \beta 13 * (X1 * X3) + \beta 23 * (X2 * X3) + \\ & \beta 123 * (X1 * X2 * X3) + \\ & \beta 1212 * (X1 * X2 * (X1 - X2)) + \\ & \beta 1313 * (X1 * X3 * (X1 - X3)) + \\ & \beta 2323 * (X2 * X3 * (X2 - X3)) \end{aligned}$$

Equation 1. Scheffé's cubic model

This study consisted of two stages. In the first stage, the wax interactions were studied and the best wax ratio was determined. 16 experiments with different wax proportions were conducted following the order of experimental matrix (Table 1). These experiments were run with the 3 test oils sequentially to understand the behaviours of wax mixtures in different oil phases. In the second stage, the oil interactions were studied and the best oil ratio was determined, while using the best wax ratio found in the first stage.

Lipstick Characterisation Control

Breaking point measurement

The breaking point (g) indicates the lipstick hardness. Lipstick sample was secured on the stand and the handle of a bucket was placed on the lipstick. Water was allowed to flow into the bucket until the stick broke and the release of water was stopped immediately. The weight of collected water was measured as breaking point (target: 300-400g).

Softening point measurement

The Ring and Ball method was used to determine the lipstick stability at elevated temperature. The higher the softening point, the better the lipstick stability. The lipstick sample was prepared in a ring and ball assembly set and immersed in a water bath that was heated at a prescribed rate. The temperature at which the ball flows through the melted lipstick sample was recorded as the softening point (target: 68-74° C).

Glide evaluation method

The degree to which the lipstick sample spreads easily and evenly on the skin was evaluated by applying it on the inner forearm in a standard forward-backward motion 3 times. A point scale of 0-10 was used for scoring, with 0 being very difficult glide and 10 for very easy glide (target: 7-10).

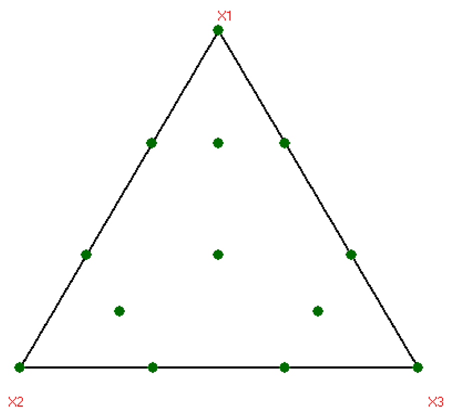


Figure 1. Distribution of 13 experimental points of an augmented simplex-lattice design for three components. The 9 points on the triangle sides calculate the coefficients of the model. The simplex was augmented with 3 interior design points to predict the interior of the simplex and to validate the model used.

N° of experiment	X1	X2	X3
1	1.00000	0.00000	0.00000
2	1.00000	0.00000	0.00000
3	0.00000	1.00000	0.00000
4	0.00000	0.00000	1.00000
5	0.66667	0.33333	0.00000
6	0.33333	0.66667	0.00000
7	0.66667	0.00000	0.33333
8	0.66667	0.00000	0.33333
9	0.33333	0.33333	0.33333
10	0.00000	0.66667	0.33333
11	0.33333	0.00000	0.66667
12	0.00000	0.33333	0.66667
13	0.66667	0.16667	0.16667
14	0.16667	0.66667	0.16667
15	0.16667	0.16667	0.66667
16	0.16667	0.16667	0.66667

Table 1. Experimental matrix showing a total of 16 blend points and their mixture proportions.

Results and Discussion

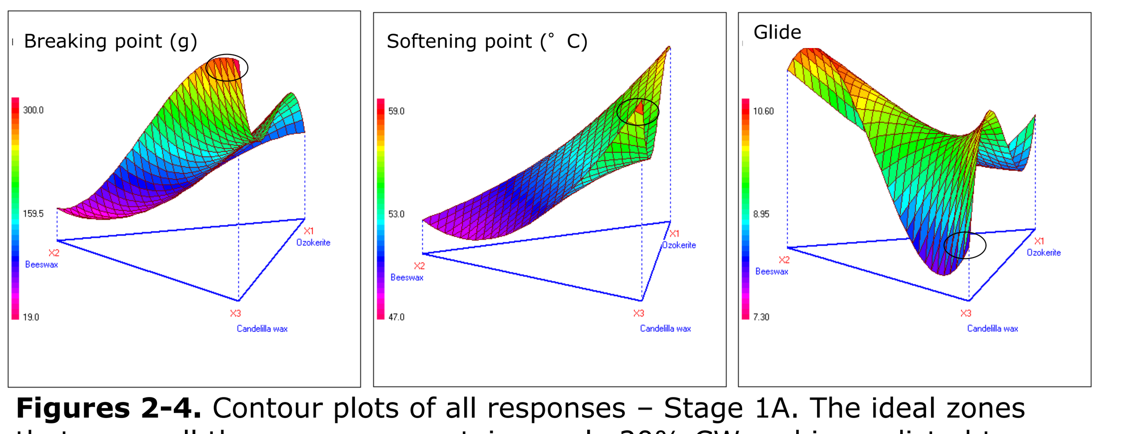
Stage 1A: Optimising the wax ratio with hydrogenated polyisobutene

It was observed that hydrogenated polyisobutene did not provide hardness and structural stability to the lipstick as all the 16 test samples were below the target for breaking point (300g) and softening point (68ºC). It was postulated that non-polar HP forms swollen crystals with non-polar ozokerite, and micelle-like structures with both polar candelilla wax and beeswax, giving less rigid structures than that with a polar base oil [3]. In principle, the lower the oil polarity, the softer the lipstick.

Amongst the test waxes, CW showed the highest breaking and softening points because it contains terpenes, which enables it to retain oil within the structure and improve stability. Contrarily, BW gave the lowest breaking point because of its soft wax structure, which is contributed by its high ester content and very fine crystal structure [4].

Due to its branched structure, HP showed better gliding than other test oils (scores >8). Additionally, BW conferred the best glide amongst the waxes because its complex polyesters make it highly plastic, imparting flexibility and lubricity during application [5]. Overall, HP displayed higher influence on glide than the type of wax used. Hence, HP was not deemed efficient as the main stabilising oil for lipstick, but could be used in low amounts to improve gliding properties.

Referring to the contour plots in Figures 2-4, the best area selected contained almost 20% CW, giving the most optimum results possible for breaking point, softening point and glide.

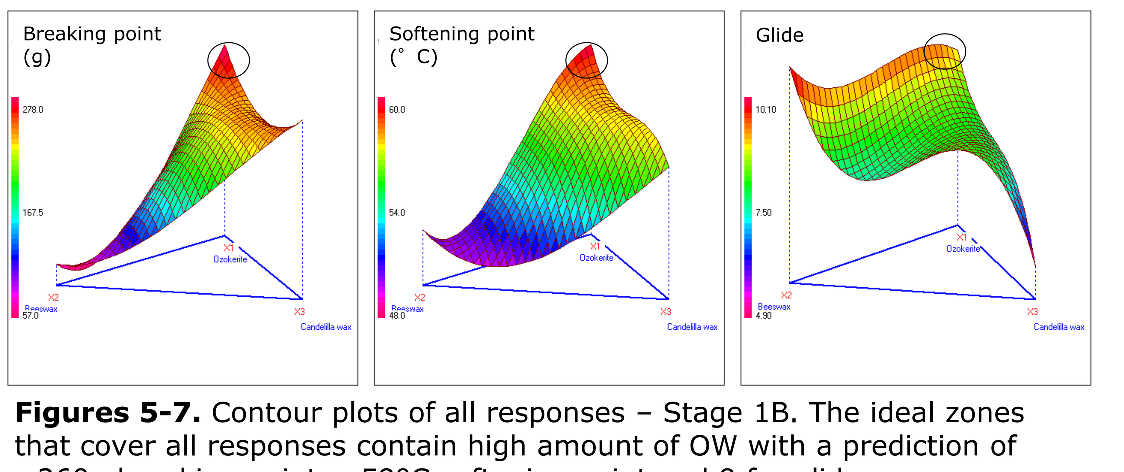


Figures 2-4. Contour plots of all responses – Stage 1A. The ideal zones that cover all the responses contain nearly 20% CW and is predicted to have ~270g breaking point, ~56ºC softening point and 8-9 for glide score.

Stage 1B: Optimising the wax ratio with octyldodecanol

From stage 1A, it was known that oil polarity directly influences the stick hardness. Because of the low polarity of octyldodecanol, all lipsticks were still below the targets for breaking and softening points, although their range of results were generally higher than with HP. Hence, OD was deemed suitable as a secondary oil to aid the formation of stick structure.

In order of stick hardness contributed by the individual waxes, it was OW as the hardest, closely followed by CW and then BW. However, when used in mixture, higher amount of CW in the formula (>13.33%) gave higher breaking point than that of OW. In terms of glide, OD generally interacts well with all the wax combinations to provide good glide (scores 7-10).



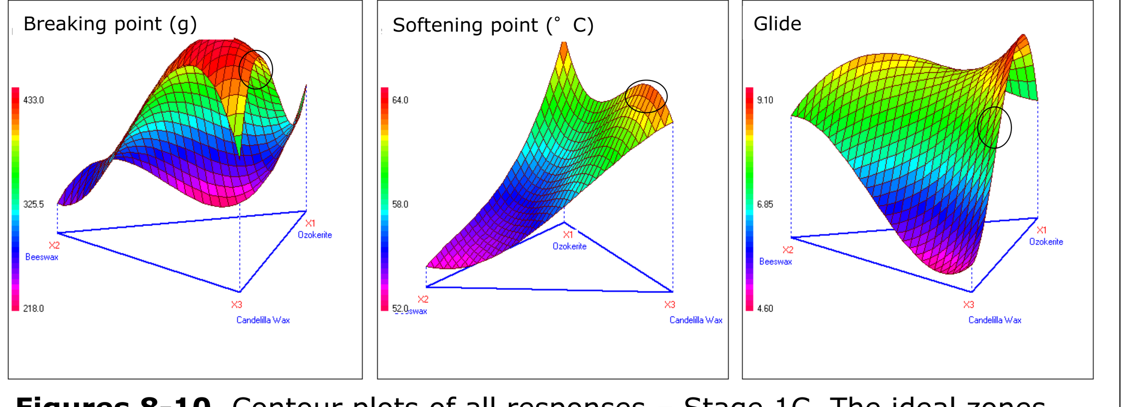
Figures 5-7. Contour plots of all responses – Stage 1B. The ideal zones that cover all responses contain high amount of OW with a prediction of ~260g breaking point, ~59ºC softening point and 9 for glide score.

Stage 1C: Optimising the wax ratio with sweet almond oil

Sweet almond oil generally provided higher breaking point than HP and OD as its high polarity strengthens the stick structure. Collectively, observations from Stage 1A-1C were in line with Takeo's observation [6] that the hardness of oil-wax gel increases with the polarity of oil. This suggests that SAO could act as the primary stabilising oil for structural integrity of the lipstick.

CW showed the best interaction with SAO to provide the preferred stick hardness and stability, both in mixture and as individual. Since CW contains 50% non-polar hydrocarbons and 50% polar esters, it is able to form lamellar bilayers and micelle-like structures respectively with the highly polar SAO. These arrangements reduce any movement within the structure and increase hardness [3]. Overall softening point has slightly increased with SAO but still below the target.

SAO showed an acceptably lower glide scores than with HP and OD, except when candelilla wax in lipsticks exceeded 13.33% (scored 5).



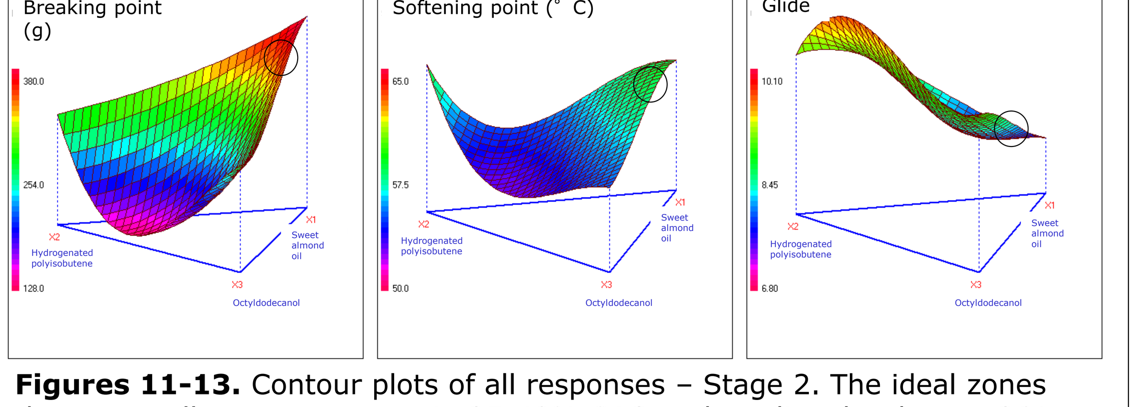
Figures 8-10. Contour plots of all responses – Stage 1C. The ideal zones that cover all responses include higher breaking and softening points, with slightly compromised glide. This area predicts ~419g breaking point, ~61ºC softening point and 7 for glide score.

To obtain the optimality between the competing requirements across all phases, it was deemed important to first achieve the required breaking and softening points, which meant a compromise on glide. Therefore, the optimum wax ratio determined was 16% candelila wax, 2% ozokerite and 2% beeswax.

Stage 2: Optimising the oil ratio

It was found that at least 16.67% of SAO was required in the oil mixture to meet the breaking point target. While higher level of SAO increases the breaking point, the addition of other oils can reduce this parameter. Although some samples did not meet the softening point target, higher reading was observed with more SAO in the mixture.

Generally, all the oil mixtures interacted well with the waxes to meet the glide target of 7-10. While the highest score of 10 was achieved with 33.33% HP and 16.67% OD, the addition of SAO in the formula reduced the glide to the optimal glide score of 8-9.



Figures 11-13. Contour plots of all responses – Stage 2. The ideal zones that cover all responses contain 35-40% SAO and predicted to have ~361g breaking point, ~58ºC softening point and approximately 8 for glide score.

By using the software to identify a precise point on the contour plots, the optimum oil ratio was determined as 38.1% sweet almond oil, 0.75% hydrogenated polyisobutene and 11.15% octyldodecanol. The final formula with the optimised wax and oil phases was predicted to give ~350.8g breaking point, ~58ºC softening point and 7.8 for glide score.

Conclusion

The results of this study have revealed the quantitative relationship between the hardness and stability of a lipstick (expressed as its breaking and softening points respectively) and its 'glide' performance. The use of Mixture Design approach has made it possible to effectively select the samples with the best overall characteristics, on the basis of limited but focused experimental work.

For the given range of ingredients, the optimum wax ratio was found to be: 2% ozokerite, 2% beeswax and 16% candelilla wax, while the optimum oil ratio was: 38.1% sweet almond oil, 0.75% hydrogenated polyisobutene and 11.15% octyldodecanol (making a total of 70% of the lipstick formulation).

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