Journal of Applied Sciences

ISSN 1812-5654

science alert

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Journal of Applied Sciences 7 (15): 2099-2103, 2007
ISSN 1812-5654

Optimisation of Natural Ingredient Based Lipstick Formulation by Using Mixture Design

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Abstract: The cosmetic industry in Malaysia is proven to be one of the important economy sources. Lipstick is one of the decorative cosmetic products that command a unique market. The quality of lipstick is directly linked to the basic material used in the formulation. The ratio of the ingredients used determines the final product characteristics. In previous researches, statistical mixture design has been proved to be effective tool to investigate the relationship between variables in formulation work. Contour graphics were formed to assess the change in the response surface in order to understand the relationship between the product cost and consumer acceptance of the lipsticks. In this work, natural waxes, solvents and colorant were used to prepare lipstick formulation. Statistical mixture design has been applied for experimental setting for the components system. The effects of the mixture components on the physical properties and consumer acceptance of the lipstick have been investigated. The results indicate that the physical properties of the lipstick can be manipulated by changing the composition of the base ingredient used in the formulation. It was found that the variation of the mixture component affected the consumer acceptance on lipstick's rub-off characteristic.

Key words: Cosmetic, lipstick formulation, natural ingredient, mixture design, D-optimal

INTRODUCTION

Lipstick contains a variety of emollients, emulsifiers, preservatives, colorants and binders (Sackheim and Lehman, 1998). Previous research proved that the quality of lipstick is directly linked to the basic material used in the formulation. Varying the ratio of the ingredient in formulation, the final product characteristics such as texture, viscosity, hardness and melting point of the lipstick can be varied (Awang *et al*., 2006).

A common problem in pre-formulation of the cosmetic product including lipstick is the optimisation of the mixture composition aimed to obtain a product with the required characteristic. Statistical experimental design commonly used to overcome this problem. The primary goal of designing an experiment statistically is to obtain valid results at minimum of effort, time and resources (Myers and Montgomery, 2002; Allen, 1987). There are several experimental design techniques used for formulation work such as factorial design, cross design and mixture design. However, it should be noted that factorial design could not be used to study such mixtures since the variables are not independent (Hinkelmann and Kempthorne, 1994). All fractions of the components must sum to unity. Thus, mixture design represents an efficient approach for solving such optimisation problem.

Statistical mixture design is more satisfactory and effective than other methods such as classical one-at-a-time or mathematical methods because it can study many variables simultaneously with a low number of observations, saving time and costs. In previous researches, statistical mixture design has been proved to be effective tool to investigate the relationship between variables in formulation work (Kamaun *et al*., 2002; Sabir *et al*., 2001; Cafaggi *et al*., 2003). However, it has not been widely used in cosmetic science.

Therefore, in this work, D-optimal design is applied in order to investigate the relationship between variation composition, physical properties and consumer overall acceptance. By relating consumer data to processing variables and instrumental analyses, the researchers and cosmetic chemists can discover the relationship between product attributes and also the consumer acceptability. In line with Meilgaard *et al*. (1999), these kinds of information will lead to better understanding of the effect of different properties and identify the acceptable limit of the product.

METHODOLOGY

Experimental design: The experimental settings are performed by statistical mixture experimental design. The

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Table 1: Constraint of the component proportion

Ingredient, X_i	Lower limit, L_i (%)	Upper limit, U_i (%)
Castor oil, X_1	37	65
Beeswax, X_2	5	20
Candelilla wax, X_3	1	5
Carnauba wax, X_4	1	5
Solvent, X_5	5	20

experimental design of five-components system is conducted by using Design Expert (version 6.10, Stat-Easy Inc., Minneapolis, USA). A set of candidate points in the design space is selected using the D-optimal criterion. In D-optimal criterion, there are restrictions on the component proportions X_i that take the form of lower L_i and upper U_i constraint.

The constraints of the component proportion is shown in Table 1 and adapted from the experimental results of a previous study (Awang *et al*., 2003).

Lipstick formulation process: Natural ingredient based lipstick formulations are prepared in laboratory scale according to the composition suggested by the D-optimal mixture design. Firstly, the FD and C Red #3 Powder colour is added into the stabilised castor oil for pigment dispersion as suggested (Cunningham, 1992; Alexander, 1973; Bryce, 1993). The solution is heated and homogenised by using high speed homogeniser with speed of 10000 rpm. In another vessel, lipstick mass is prepared. Beeswax, candelilla wax and carnauba wax are dissolved into the solvent, which was used as a heat transfer medium to melt the added waxes. The mixture is then heated to 80-90°C (Knowlton and Pearce, 1993). Finally, the pigment mixture and lipstick mass was mixed together and homogenised at speed 10000 rpm by using high speed homogeniser for 30 min. The hot mixture was then poured into lipstick moulds and left to cool into shape.

Physical properties characterisation: The viscosity measurement for lipstick was carried out by using Brookfield Engineering Rheometer model HA DV-III equipped with small sample adapter with spindle No.28 and speed 10 rpm. Meanwhile, the melting point of the lipstick was directly observed with SMPI Melting Point Apparatus (Stuart Scientific). Lipstick hardness determined by Texture Analyser (Hounsfield) based on European standard method, DIN EN 1426, which specifies the use of needle penetration method for petroleum wax testing at room temperature.

Consumer acceptance investigation: The objective of the test was to investigate the acceptability of the lipstick samples. The consumer acceptance of the lipstick is measured by using nine-point hedonic scale method. In this research, all twenty- five formulations were studied. Balance Incomplete Block Design (BIBD) was applied as suggest by Meilgaard *et al*. (1999). Therefore, the number of respondent required to evaluate 25 samples, which placed randomly in 50 blocks with 3 replications is 150 respondents.

RESULTS AND DISCUSSION

Effect of mixture components on the physical properties of the lipstick: Figure 1-3 show the three dimensional response surface plot of melting point, penetration depth and viscosity, respectively. The diagram describes the variation on physical properties response as a function of the mixture composition. In order to represent the response evaluation in a bidimensional system, two of the variables were to be kept constant.

Figure 1 shows that the higher amounts of candelilla wax and carnauba wax give the higher value of melting point. Candelilla wax and carnauba wax used in lipsticks formulation to confer high melting point upon finished product (Bryce, 1993). On the other hand, the melting point of the lipstick reduced with the increases in beeswax composition. The beeswax does not improve the melting point of the lipstick.

Figure 2 shows that the penetration depth increases with an increase of castor oil and solvent composition, indicating that lipstick formulation with high composition of castor oil and solvent is softer. On the other hand, an increase in beeswax composition reduced the penetration depth, which means that the hardness of the lipstick increases with an increase in beeswax composition.

Knowlton and Pearce (1993) stated that the rub-off characteristic of lipstick is related to its viscosity. The viscosity of twenty-five formulations is in the range of 57,057 cP to 62086 cP. Fig. 3 shows that the higher amounts of candelilla wax and carnauba wax give the higher value of viscosity. On the other hand, the viscosity of the lipstick reduced with the increases in beeswax composition.

Effect of mixture components on the overall consumer acceptance of the lipstick: Figure 4 shows the consumer acceptance on lipstick's overall acceptance. The diagram describes the variation of consumers' response as a function of the mixture composition. The Fig. 4 shows that the higher amount of candelilla wax gives the higher score in consumers' response. On the other hand, consumer overall acceptance reduced with the increase in carnauba wax and solvent composition.

Furthermore, the result indicates that the binary blending of castor oil and solvent and beeswax and solvent have binary synergistic effects. The binary blending of these components enhances the overall consumer acceptance towards the lipstick formulation.

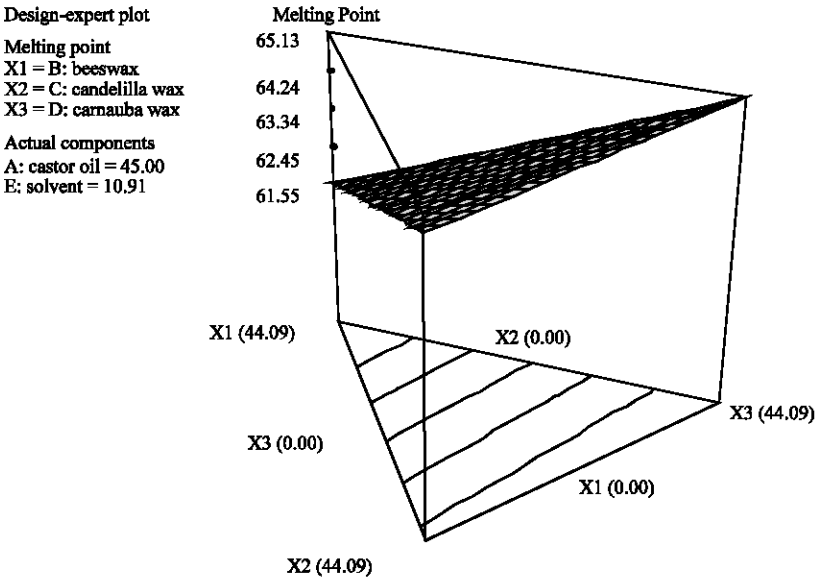

Fig. 1: Three Dimensional Response Surface plot of Melting Point

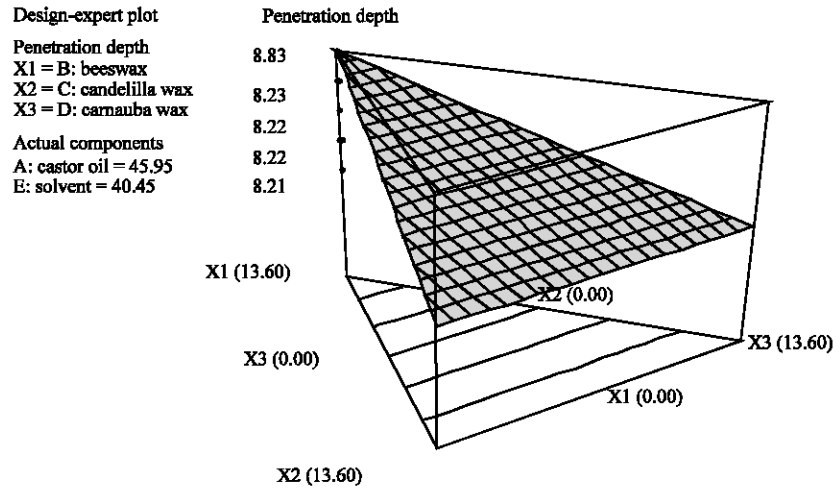

Fig. 2: Three dimensional response surface plot of penetration depth

On the other hand, when castor oil and candelilla wax and beeswax and candelilla wax are combined, the overall consumer acceptance is reduced. The binary blending of these components has antagonistic blending effect on the overall consumer acceptance.

Optimisation of natural ingredient based lipstick formulation: Numerical optimisation is performed in order to obtain the formulation with desired characteristics at the minimum cost. The optimisation goal is based on the instrumental measurement and consumer evaluation data. The goal for each response is shown in Table 2. The melting point of lipsticks is generally within the range 55-75°C (Bryce, 1993). All twenty-five formulations met this requirement with the melting point within 59.8 to 64.0°C. Furthermore, by referring to consumer data, it was found that the acceptable limit for lipstick's melting point was in the range of 60.6 to 64.0°C. In line with Bryce (1993), the melting point of lipstick must be high to avoid it from technical deterioration when expose to anticipated environmental temperature and humidity during preparation and use. Consequently, the maximum melting point is desirable for lipstick formulation.

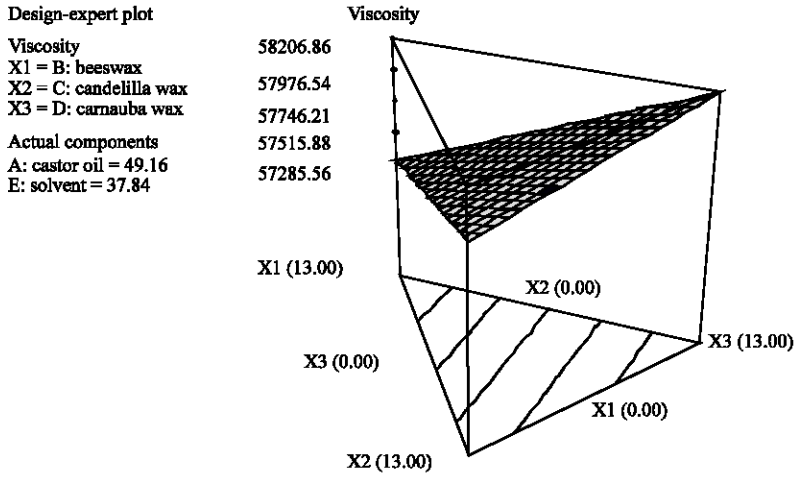

Fig. 3: Three Dimensional Response Surface Plot of Viscosity

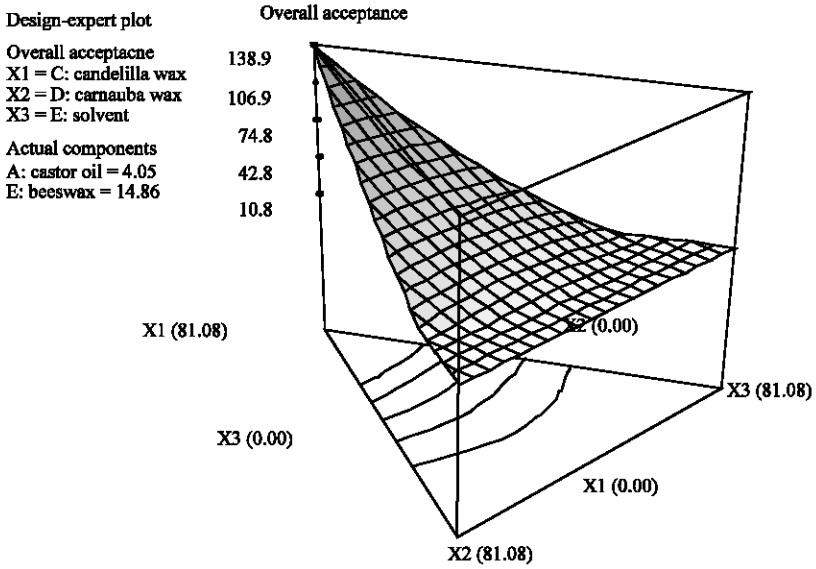

Fig. 4: Three dimensional response surface plot of consumer overall acceptance

Table 2: Optimisation target for the response

	Response	Goal/Target	Acceptable limit
Physical	Melting Point (°C)	Maximum	55-75
Properties	Penetration Depth (mm/5sec)	8.14-8.19	8.14-8.19
	Viscosity (cP)	59486-62086	59486-62086
Consumer	Hardness	Maximum	6-9
Evaluation	Consistency	Maximum	6-9
	Stickiness	Maximum	6-9
	Overall Acceptance	Maximum	6-9
Cost	Cost	Minimum	none

On the other hand, for hardness and viscosity of the lipstick, there is no specific requirement. In this case, consumer acceptance for lipstick's hardness and consistency is used to estimate the acceptable formulation in term of hardness, which is measured by penetration depth. Whereas, the acceptable viscosity range for lipstick formulation is determined by the consumer acceptance for lipstick's stickiness and consistency. Therefore, it is found that the goal and acceptable limit for penetration depth and viscosity for the lipstick is in the range of 8.14-8.19 mm/5sec and 59486-62086 cP, respectively.

Consumer acceptability in term of hardness, consistency, stickiness and overall acceptance for the lipstick has been evaluated by using nine-point hedonic scale. The acceptability limit for the consumer score is within 6 to 9. The highest score indicates the most acceptable formulation. Therefore, the formulation with the highest score is desirable in consumer acceptance.

Based on the criteria stated in Table 2, the optimum formulation suggested consist of 39.40% castor oil, 20.00% beeswax, 5% candelilla wax, 5.00% carnauba wax and 17.60% solvent, with the highest desirability of 0.84.

CONCLUSION

The effects of the mixture components on the physical properties and consumer acceptance of the base cream formulation have been investigated. The optimum formulation has been obtained by relating the instrumental analysis and consumer data. This research has provides a guideline on improving or achieving specific desirable characteristics by using mixture experimental design which is capable to study many variables simultaneously. The research finding also provides a guideline on effects of ingredients towards the physical properties and consumer acceptance of the lipstick formulation.

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