THE EFFECTS OF MIXTURE COMPONENTS ON THE PHYSICAL PROPERTIES AND CONSUMER ACCEPTANCE OF THE NATURAL INGREDIENT BASED LIPSTICK FORMULATION

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DECLARATION

The materials in this thesis are original except for quotations, accepts, summaries and references, which have been duly acknowledged.

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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. 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 characteristics. In previous researches, statistical mixture design has been proven to be effective tool to investigate the relationship between variables in formulation work. Therefore, in this work, statistical mixture design has been applied in order to investigate the effects of mixture components on the physical properties and consumer acceptance of the formulated lipstick. Statistical mixture design has been applied for experimental setting for five components system, which are castor oil, beeswax, candelilla wax, carnauba wax and solvent. Twenty-five natural ingredients based lipstick formulations have been prepared based on D-optimal mixture design criterion. The effects of the castor oil, beeswax, candelilla wax, carnauba wax and solvent compositions on the melting point, hardness and viscosity of the lipstick have been studied. The results indicate that the physical properties of the lipstick can be manipulated by changing the composition of the castor oil, beeswax, candelilla wax, carnauba wax and solvent in the formulation. Linear model has been suggested for all physical properties studied for lipstick formulation. The results shows that the melting point and viscosity of the lipstick are affected most by candelilla wax, carnauba wax and beeswax. Whereas, the hardness of the lipstick is affected most by castor oil, beeswax and solvent composition. Furthermore, the consumer acceptance of the lipstick has been measured by using nine-point hedonic scale method, with 150 respondents. Quadratic model has been suggested for consumer response on lipstick's hardness, stickiness, consistency and overall acceptability. The compositions of candelilla wax and carnauba wax have the strong effect to consumer acceptance for all attributes studied including the overall acceptance. On the other hand, solvent composition has the strong effect only on hardness, stickiness and overall acceptability of the lipstick. Meanwhile, beeswax composition has the strong effect only on consistency acceptability. Finally, the numerical optimisation has been 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 formulation, which has the acceptable physical characteristic and gained the maximum score in consumer evaluation at the minimum cost, is desirable. It is found that the optimum formulation suggested consist of 39.40% castor oil, 20.00% beeswax, 5.00% candelilla wax, 5.00% carnauba wax and 17.60% solvent, with the highest desirability of 0.84. Comparing to another two commercial formulation, the optimum formulation found to be harder and more viscous at room temperature. However the melting point of the optimum formulation is lower compared to commercial formulation.



ABSTRAK

KESAN KOMPONEN CAMPURAN TERHADAP CIRI-CIRI FIZIKAL DAN PENERIMAAN PENGGUNA UNTUK FORMULASI GINCU BERASASKAN BAHAN SEMULAJADI

Industri kosmetik di Malaysia telah dikenalpasti sebagai salah satu sumber ekonomi negara yang penting. Gincu merupakan salah satu produk kecantikan yang mendapat permintaan yang menarik di pasaran. Kualiti gincu sangat berhubungkait dengan bahan-bahan asas yang digunakan dalam formulasi. Masalah yang lazimnya dihadapi dalam proses formulasi produk kosmetik termasuk gincu adalah untuk mendapatkan formulasi yang optima, dimana ia memenuhi ciri-ciri yang dikehendaki. Dalam kajian terdahulu, statistical mixture design telah dibuktikan dapat menyelesaikan masalah tersebut selain dari mampu mengenalpasti hubungan antara pembolehubah yang terlibat dalam proses formulasi sesuatu produk. Oleh yang demikian, dalam kajian ini, statistical mixture design telah digunakan untuk mengkaji kesan komponen campuran terhadap ciri-ciri fizikal produk dan penerimaan pengguna terhadap produk gincu yang terhasil. Statistical mixture design telah digunakan untuk rekabentuk eksperimen bagi sistem lima komponen iaitu minyak kastor, lilin lebah, lilin candelilla, lilin carnauba dan pelarut. Dua puluh lima formulasi telah terhasil berdasarkan kepada rekabentuk D-optimal. Kesan minyak kastor, lilin lebah, lilin candelilla, lilin carnauba dan pelarut ke atas takat didih, kekerasan dan kelikatan gincu telah dikaji. Keputusan menunjukkan bahawa ketiga-tiga ciri-ciri fizikal tersebut boleh diubahsuai dengan mengubah komposisi minyak kastor, lilin lebah, lilin candelilla, lilin carnauba dan pelarut dalam formulasi. Model linear dicadangkan untuk menerangkan keputusan untuk ciri-ciri fizikal. Keputusan menunjukkan bahawa takat didih and kelikatan gincu banyak dipengaruhi oleh lilin candelilla, lilin carnauba dan lilin lebah manakala kekerasan pula banyak dipengaruhi oleh komposisi minyak kastor, lilin lebah dan pelarut. Seterusnya, penerimaan pengguna terhadap gincu yang terhasil daripada kajian ini telah dikaji menggunakan skala hedonik 9 titik oleh 150 respondan. Model kuadratik dicadangkan untuk menerangkan kesan komposisi campuran terhadap penerimaan pengguna dari segi kekerasan, kelekitan, keseragaman dan penerimaan keseluruhan gincu yang diformulasikan dalam kajian ini. Akhir sekali, pengoptimaan berangka telah dijalankan untuk mendapatkan formulasi yang memenuhi semua ciri-ciri yang dikehendaki dengan kos yang minimum. Sasaran untuk pengoptimaan adalah berdasarkan data dari pengukuran peralatan dan penilaian pengguna. Formulasi yang mempunyai ciri-ciri fizikal yang diterima serta mendapat skor penilaian pengguna yang tertinggi pada kos yang minimum adalah dikehendaki. Keputusan menunjukkan bahawa formulasi optimum mengandungi 39.40% minyak kastor, 20.00% lilin lebah, 5.00% lilin candelilla, 5.00% lilin carnauba dan 17.60% pelarut, dengan desirability 0.84. Perbandingan dengan dua produk sedia ada dalam pasaran mendapati bahawa pada suhu bilik, formulasi optimum yang dihasilkan daripada kajian ini lebih keras dan lebih likat tetapi mempunyai takat didih yang lebih rendah.



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NOMENCLATURE

%	Percent
g	Gram
L	Litre
mm	Millimetre
S	Second
rpm	Rotation per minute
°C	Degree Celsius
сР	Centipoise
BIBD	Balance Incomplete Block Design
RSM	Response Surface Methodology
ANOVA	Analysis of Variance
DF	Degree of Freedom
SSR	Sum of Square
SSE	Residual Sum of Square
SST	Total Sum of Square
PRESS	Prediction Error Sum of Square



CHAPTER 1

INTRODUCTION

1.1 Overview

According to the Federal Food, Drug and Cosmetic Act of 1938 (sec 17.14), a cosmetic is anything intended to be applied directly to the human body for cleansing, beautifying, promoting attractiveness or altering the appearance (Synder, 1995; Whittam, 1987; Shai, Maibach and Baran, 1999).

The rise of the worldwide living standard has created a path for the increasing use of cosmetic products. This is supported by the research done by Mulhern *et al.* (2003) which found that the belief of cosmetics do improve female attractiveness,has led the cosmetic industry to be one of the most successful worldwide. Multinational companies and our own local companies are among the leading investors for cosmetic market, which provide a wider product range. The cosmetic industry in Malaysia is proven to be one of the most important economy sources. From the year of 1998 to 1999, Malaysian import for personal care products, which includes skin care, makeup, hair care and fragrance, was RM1255 million while export was RM 592 million (Afiza, 2000). Meanwhile, the total annual sale for cosmetic products in 2003 was RM 8.2 million. This market is expected to grow more than fifty percent for the year ahead (Beautycontrol, 2003).



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In the last quarter of twentieth century, cosmetic industries exist with technology of their own. Every year, users were introduced with various new cosmetics products of the latest trend. The ingredients and basic material used in cosmetic formulations become the important criteria for costumers in choosing the cosmetic product, as their interest in health and safety issues grew. Recently, there has been a marked increase in consumer awareness towards the issue and importance of natural products. Natural products are felt to be safer and more environmentally friendly (Middleton, 1996; Joeston and Wood, 1996; Maccioni *et al.*, 2002). Consequently, natural-ingredient based products are getting popular (Josten & Wood, 1996; Grievson *et al.*, 1992; Fridd, 1996). Cosmetic manufacturers have invested in technology and researches to offer a wide range of products that meet this requirement.

Generally, cosmetics can be grouped by product into seven categories namely skin care and maintenance, cleansing, odour improvement, hair removal, hair care, care and maintenance of mucous membranes and decorative cosmetics (Rieger, 1983). Lipstick is one of the decorative cosmetic products that command a unique market. Lipstick contains a variety of emollients, emulsifiers, preservatives, colorants and binders (Sackheim and Lehman, 1998). Williams and Schmitt (1992) stated that the quality of lipstick is directly linked to the basic material used in the formulation. Varying the ratio of the ingredient used in formulation determines the final product characteristics such as texture, viscosity, hardness and melting point of the lipstick (Awang *et al.*, 2003).

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 characteristics. 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 were several experimental design technique 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. 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, Evans and Jain, 2001; Bodea and Leucuta, 1997; Campisi *et al.*,1998; Cafaggi *et al.*, 2003; Brandvik and Daling, 1998; Gaspar, Laureano and Sousa, 1998; Ruohonen *et al.*, 2003; Lee and Gilmore, 2004; Samet and Chaabouni, 2004). However, it has not been widely used in cosmetic science.

Therefore, in this work, statistical mixture design will be applied in order to investigate the relationship between variation composition, physical properties, and consumer overall acceptance. By relating consumer data to processing variable and instrumental analyses, the researchers and cosmetic chemists can discover the relationship between product attributes and also the consumer acceptability. These



kinds of information will lead to better understanding of the effect of different properties and identify the acceptable limit of the product.

1.2 Research Objectives

The main objective of this research is to produce a natural ingredient based lipstick formulation. This research also conducted to investigate the effects of various mixture components on the physical properties and consumer acceptance of the lipsticks by using statistical mixture design. Major work involves the formulation, evaluation and optimisation of lipstick that meets the technical requirement and consumer acceptability.

1.3 Scope of Research

Statistical mixture design will be applied for experimental setting and data analysis. Various formulation of natural ingredient based lipstick will be produced. The effect of mixture components or ingredients towards the physical properties and consumer acceptance of the product will be studied. Finally, the optimum formulation will be obtained by relating the physical properties and consumer acceptability of the lipstick.

1.4 Research Methodology

Systematic steps and iterations of work are carried out to accomplish the research objective. Thus, the research methodology is carefully laid out as follows:



Literature and biography of natural ingredient based lipstick formulation including its raw material, formulation process technology and important characteristics are investigated. The application and analysis of statistical experimental design, and the techniques of consumer acceptance observation for cosmetic product are also studied.

1.4.2 Experimental Design and Formulation of Natural Ingredient Based Lipstick

The experimental setting is performed by mixture experimental design. Natural ingredient based lipstick formulations is prepared in laboratory scale according to the composition suggested by the D-optimal criterion. Various compositions of natural waxes, oils and solvents are used to prepare natural ingredient based lipstick formulation.

1.4.3 Characterisation of Natural Ingredient Based Lipstick Formulation

Once the lipstick has been properly formulated, its physical properties are studied. The most important properties to be studied are melting point, hardness and viscosity. Melting point is directly observed by using Stuart Scientific SMPI Melting Point Apparatus, whereas the viscosity of the lipstick is measured by using Brookfield Rheometer. Needle Penetration Method by using Hounsfield Texture analyser is used to measure the lipstick's hardness.

1.4.4 Consumer Evaluation Test on Natural Ingredient Based Lipstick

The consumer acceptance of the lipstick is measured by using nine-point hedonic scale method. The Central Location Test is conducted by using 150 respondents.



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