UNIVERSITI PUTRA MALAYSIA

OPTIMIZATION AND CHARACTERIZATION OF HALAL LIPSTICK FORMULATION CONTAINING PITAYA (HYLOCEREOUS POLYRHIZHUS (WEBER) BRITTON & ROSE) SEED OIL

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By

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OPTIMIZATION AND CHARACTERIZATION OF HALAL LIPSTICK FORMULATION CONTAINING PITAYA (*HYLOCEREOUS POLYRHIZHUS* (WEBER) BRITTON & ROSE) SEED OIL

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Cosmetic is one of the important industries worldwide. Lipstick is one of the decorative cosmetics that have high demand in the market. In this research, pitaya seed oil (PSO) was extracted using two solvents which are n-hexane and ethanol, solvent extraction. Pitaya seed oil (PSO) from both solvents extraction was used in the lipstick formulation with ratio 3:2 (n-hexane: ethanol). Pitaya seed oil was used in this formulation because it has high content of linoleic acid and linolenic acid which are classified as unsaturated fatty acids (UFAs).

D-Optimal Mixture Experimental Design was utilized to investigate the influence of the main composition in the lipstick formulation; pitaya seed oil (10-35 % w/w), virgin coconut oil (25-45 % w/w), beeswax (5-25 % w/w), candelilla wax (1-5 % w/w) and carnauba wax (1-5 % w/w) on the physicochemical properties of lipstick. The response variable used is melting point which is one of the important properties of lipstick. Optimization of the five independent variables was carried out to determine an optimum lipstick. The D-Optimal Mixture Experimental Design analysis was showed that the variation in the response (melting point) could be depicted as quadratic model of the main compositions of the formulation. The experimental data fitted sufficiently well into a second-order polynomial model. The predicted melting point gave by D-optimal Mixture Experimental Design (MED) was 45.5℃.

Artificial Neural Network (ANN) was designed as another tools to optimize the ingredients in the lipstick formulation. ANN- Batch Backpropagation give the better prediction of melting point compared to D-optimal MED with the value 45.6℃. The optimal ingredients determined by the D-Optimal Mixture Experimental Design and Artificial Neural Network was established to be pitaya seed oil (25% w/w), virgin coconut oil (37% w/w), beeswax (17% w/w), candelilla wax (2% w/w) and carnauba wax (2% w/w). The others ingredients were totaling up to 100% w/w. The actual

melting point of the optimized lipstick is 46.0℃. Hence, ANN-Batch Backpropagation give better prediction compared to D-optimal Mixture Experimental Design.

The other physicochemical properties of the lipstick formulation were also studied. The melting point of the optimized PSO lipstick is 46.0±1.00℃ with the pH of 6.93±0.01. The texture profiles (hardness, stiffness, brittleness) of the optimized PSO lipstick were (29.759±0.00 g; 5.002±0.01 g/sec; 4.675±0.03 mm, respectively) which are almost like conventional lipstick in the market. From the antioxidant analysis, optimized PSO lipstick showed higher 1,1-diphenyl-2-picryl-hydrazine (DPPH) scavenging activity compared to optimized lipstick without PSO. The optimized PSO lipstick was stable after undergoing stability test at 5℃, 27℃, 37℃ and free-thaw cycle for 3 months and the color remained unchanged. The total microbial counts for the optimum PSO lipstick were <1000 $cfug^{-1}$ and complied with Malaysia Cosmetic Directive microbial limit test. The toxic contaminants had also been studied. Three metals were analyzed which are lead, arsenic and cadmium which is the content of the metals in optimum lipstick are 0.09 ppm, 0.034 ppm and 0.027 ppm, respectively. The halal lipstick containing pitaya seed oil was successfully developed and characterized. It is safe and efficient for human lips care.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan Ijazah Sarjana Sains

PENGOPTIMUMAN DAN PENCIRIAN FORMULASI GINCU HALAL YANG MENGANDUNGI MINYAK BIJI BUAH NAGA (*HYLOCEREOUS POLYRHIZHUS* (WEBER) BRITTON & ROSE)

Oleh

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Kosmetik merupakan salah satu industri yang penting di seluruh dunia. Gincu menjadi salah satu kosmetik hiasan yang mempunyai permintaan yang tinggi dalam pasaran. Dalam kajian ini, minyak biji buah naga (PSO) telah diekstrak menggunakan dua pelarut, iaitu n-heksana dan etanol dengan menggunakan kaedah pengekstrakan pelarut. Minyak biji buah naga (PSO) daripada kedua-dua pengekstrakan pelarut digunakan dalam formulasi gincu dengan nisbah 3:2 (n-heksana: etanol). Minyak biji buah naga digunakan dalam formulasi ini kerana terdapat kandungan asid linoleic dan asid linolenic yang tinggi atau dikelaskan sebagai asid lemak tak tepu (UFAs).

Eksperimen Rekabentuk Campuran D-Optimum telah digunakan untuk mengkaji pengaruh komposisi utama dalam formulasi gincu; iaitu, minyak biji buah naga (10-35% w/w), minyak kelapa dara (25-45% w/w), lilin lebah (5-25% w/w), lilin candelilla (1-5% w/w) dan lilin Bogo (1-5% w/w) ke atas sifat fizikokimia gincu.Takat lebur dijadikan sebagai pembolehubah tindakbalas yang mana merupakan salah satu ciri penting dalam formulasi gincu.Pengoptimuman lima pembolehubah telah dijalankan untuk menentukan satu formulasi gincuyang optimum. Analisis eksperimen rekabentuk campuran D-optimum telah menunjukkan bahawa perubahan dalam tindakbalas (takatlebur) boleh digambarkan sebagai model kuadratik daripada komposisi utama formulasi. Data eksperimen dipasang cukup baik kedalam model polynomial tertib kedua.Takat lebur yang diramalkan oleh eksperimen rekabentuk campuran D-optimum (MED) adalah 45.5℃.

Rangkaian Saraf Tiruan (ANN) telah direka sebagai satu lagi alat untuk mengoptimumkan bahan-bahan dalam formulasi gincu.ANN- kumpulan rambatan balik (BBP) memberikan ramalan takat lebur yang lebih baik berbanding eksperimen rekabentuk campuran D-optimum (MED) dengan nilai 45.6℃. Bahan-bahan yang optimum ditentukan oleh eksperimen rekabentuk campuran D-Optimum dan rangkaian saraf tiruan telah dibangunkan, menjadikan minyak biji buah naga (25% w/w), minyak

kelapa dara (37% w/w), lilin lebah (17% w/w) ,lilin candelilla (2% w/w) dan lilin Bogo (2% w/w). Bahan-bahan lain telah ditambah menjadikan jumlah sehingga 100% w/w. Takat lebur sebenar gincu PSO yang optimum adalah 46.0℃. Oleh itu, ANN-kumpulan rambatan balik (BBP) memberikan ramalan yang lebih baik berbanding dengan eksperimen rekabentuk campuran D-optimum.

Ciri-ciri fizikokimia yang lain bagi formulasi gincu PSO yang optimum juga telah dikaji. Takat lebur gincu PSO yang optimum adalah 46.0±1.00℃ dengan nilai pH 6.93±0.01. Profil tekstur (kekerasan, kekejangan, kerapuhan) daripada gincu PSO yang optimum masing-masing adalah (29.759±0.00 g; 5.002±0.01 g/saat; 4.675±0.03 mm,) yang standing dengan gincu konvensional di pasaran. Dari analisis antioksidan, gincu PSO yang optimum menunjukkan aktiviti memerangkap 1,1-diphenyl-2-picyrl-hydrazine (DPPH) lebih tinggi,berbanding dengan gincu tanpa PSO yang optimum. Gincu PSO yang optimum stabil selepas menjalani ujian kestabilan pada 5, 27, 37℃ dan kitaran bebas cair selama 3 bulan manakala warna pada gincu masih tidak berubah. Jumlah bilangan mikrob untuk gincu PSO yang optimum ialah <1000 $cfug^{-1}$ dan memenuh. Peraturan Kosmetik Malaysia terhadap ujian had mikrob. Bahan-bahan toksik logam juga telah dikaji.Tiga logam telah dianalisis iaitu plumbum, arsenic dan cadmium, dimana kandungan logam dalam gincu PSO yang optimum, masing-masing adalah 0.09 ppm, 0.034 ppm dan 0.027 ppm. Gincu halal yang mengandungi minyak biji buah naga (PSO) telah berjaya dibangunkan dan dicirikan. Gincu ini selamat dan berkesan digunakan untuk penjagaan bibir manusia.

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I certify that a Thesis Examination Committee has met on 19 October 2015 to conduct the final examination of Norsuhaili binti Kamairudin on her thesis entitled "Optimization and Characterization of Halal Lipstick Containing Pitaya (*Hylocereous polyrhizhus* (Weber) Britton & Rose) Seed Oil" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree ofMaster of Science. The members of the Supervisory Committees were as follows:

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Signature: _____
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LIST OF ABBREVIATIONS

AAD	Absolute Average Deviation
ANNs	Artificial Neural Networks
ANOVA	Analysis of Variance
APC	Aerobic Plate Count
ANN-BBP	Batch Backpropagation
ANN-GA	Genetic Algorithm
ANN-IBP	Incremental Backpropagation
ANN-LM	Levengberg-Madquaredt
ANN-QP	Quick Propagation
BHA	Butylated-hydroxyanisole
BHT	Butylated-hydroxytoluene
^{o}C/min	Degree/minute
CAGR	Cumulative Average Growth Rate
cfug^{-1}	Bacteria Colony Forming Units
DOE	Design of Expert
DPPH	1,1-diphenyl-2-picryl-hydrazine
ET	Electron Transfer
^{o}F	Fahrenheit
GAE	Garlic acid equivalents
GUI	Graphical User Interface
h	Hours
ha	Hectares
HAT	Hydrogen Atom Transfer
HgS	Mercury (II) Sulphide
HNO_3	Nitric acid
MED	D-optimal Mixture Experimental Design
min	Minute
mL	Milliliter
MLA	Modified Letheen Agar
MLB	Modified LetheenBrooth
MLP	Multilayer Perceptron
mm	Millimeter
MSE	Mean Square Error
p	Probability
PDA	Potato Dextrose Agar
PE	Processing Elements
ppm	Part per million
PSO	Pitaya Seed Oil
R^2	Correlation of determination
RMSE	Root Mean Square Error
rpm	Revolution per minute
RSM	Response Surface Methodology
t	Metric tons
TEWL	Trans-epidermal Water Loss
TGA	Thermo-gravimetric Analysis
UFA	Unsaturated Fatty Acids

v/v	Volume/volume
w/w	Weight/weight
YMC	Yeast Mould Count

LIST OF SYSMBOLS

A	Pitaya seed oil
B	Virgin coconut oil
C	Beeswax
D	Candelilla wax
E	Carnauba wax
Lj	Lower form of proportions
Uj	Upper form of proportions
Xj	Component proportion
Y	Melting point

CHAPTER 1

INTRODUCTION

1.1 Background of study

Cosmetic industry is one of the biggest and successful industries worldwide. Every day, many new cosmetic products are produced and improved as compared to the previous ones (Kamairudin *et al.*, 2014). Cosmetic products comprise color cosmetics and personal care products. Examples of color cosmetics are lipsticks, face powders, eyeliners, eye shadow, and etc. Meanwhile, personal care products are skin care, hair care, oral care and body care products. The increasing approaches to the technologically advances in cosmetics products, which are natural and suitable to use as well as complied to certain requirements of religion, bring this study towards the realization of lipstick from safe and permissible sources (Hashim *et al.*, 2009; Azwanida *et al.*, 2014). Lipstick is one of the color cosmetics which is prepared by molding a dispersion of pigment color in oil-wax base (Schlossman, 1994; Schlossman and Shao, 2014). The lipstick is made up of oily ingredients and addition of various waxes would emphasize the physical ability. The pigment color like inorganic pigment should be finely dispersed in the oily phase. Usually, the lipstick is made in the form of crayon or stick which should be temperature resistance, flawless, smooth, and glossy and should not be crack or break. In the market, there are different characteristics of lipstick is available which is depend on the ingredients used in the formulation (Rajin *et al.*, 2007; Butler, 2013).

Vegetable oils have some beneficial effects on human either used for consumption or external applications (Kamairudin *et al.*, 2014). Pitaya seed oil was introduced as one of the new oil to replace other conventional oils in lipstick formulation. This oil is suitable to replace other oil like jojoba oil because it provides soothing effect and vitamins for cosmetic applications. Pitaya seed oil was used in this lipstick formulation because it contains high content of unsaturated fatty acids (UFAs) which are linolenic and linoleic acids (Ariffin *et al.*, 2009). These UFAs help to balance the skin's metabolism by controlling the flow of oils and nourishing collagen as well as supporting the structure beneath the skin. Incorporating the sought-after essential oil, omega-3, in food or cosmetic products are widely practiced (Darmstadt *et al.*, 2002). Linoleic acids and linolenic acids are examples of antioxidant components.

Hence, these fatty acids would enhance the ability of antioxidant against oxidization process. The onset of unsightly wrinkled chapped lip can be minimized due to presence of antioxidant properties and neutralized the free radical formed on the lips due to the external factor. In other word it prevents aging on the lips. Lipstick with a good properties and safe to users will attract consumer attention as well as efficient for cosmeceutical industry.

The halal market is set to grow rapidly and experience yearly increment (Kassim *et al.*, 2014). Halal cosmetic products in particular the lipstick, from the allowable ingredients are one of the innovations in halal industry which can increase the halal market value. The market of halal industry is not only restricted to halal requirements but also covers

organic, natural, as well as safe product which will be convincing to consumer (Hashim *et al.*, 2009; Azwanida *et al.*, 2014).

The initiative to establish Halal Department/Division, halal cosmetic products manufacturing will further enhance Malaysia's position in global halal cosmetic manufacturing. Lipstick is one of color cosmetic products that is suitable for Muslim and non-Muslim users because halal is not only cover shariah compliance but also include "toyyiban" aspects such as wholesomeness, quality, nutritive value and cleanliness (Alserhan, 2010).

1.2 Problem statements

Pitaya (*Hylocereous polyrhizus*) is one of the fruit that contain a lot of benefits such as increase the cell membrane fluidity, decrease trans-epidermal loss, and improves moisturizing and so on. In this present work, pitaya seed oil (PSO) is used as one of the main ingredients in this lipstick formulation. The common problem in the lipstick formulation is to obtain optimize mixture ingredients aimed to get a product with the required characteristics. This is a very challenging study because to obtain good quality lipstick with required characteristics because it is directly linked to the basic ingredients used in the formulation. To achieve optimization of ingredients, D-optimal Mixture Experimental Design (MED) and Artificial Neural Networks (ANNs) were used as statistical experimental designs as well as to reduce the time and cost consumed. In addition, the focus to produce halal lipstick is a challenge because the ingredients used must be ascertain only from halal sources and shall be safe for the user.

1.3 Significance of study

Formulation and optimization processes are two important issues in the manufacturing of cosmetics. The processes of optimizing ingredients not only increase the utility of the technologist but also the quality of the product as well. Since, the lack of any model or tool to optimize the use of PSO in lipstick formulation which led this study to find and optimal formulation with desirable melting point using MED and ANNs. By using this tool or model, the number of experiments, time and costs can be lowered. From this work, the natural and halal lipstick also had been developed with desirable properties which are same par as the conventional lipsticks in market. This lipstick also meets the criteria of "Halalan Toyyiban" that set by National Pharmaceutical Control Bureau (NPCB) and Department of Standard Malaysia (DSM).

1.4 Scope of study

In this present work, the lipstick containing pitaya seed oil (PSO) was designed and formulated. Various natural ingredients were used in this formulation to produce good and ideal lipstick. The formulation was optimized using D-optimal Mixture Experimental Design (MED) and Artificial Neural Networks (ANNs) statistical experimental designs. The effects of the ingredients were studied through these statistical experimental designs. Then, the optimized lipstick was characterized with respect to melting point, texture profile analysis, pH, antioxidant analysis, thermo gravimetric analysis, stability study and color stability. Safety of the optimized lipstick was evaluated by microbiological and heavy metals analysis.

1.5 Objectives of study

The objectives of this work are as follows:

1. To optimize the ingredients for formulating the lipstick with respect to melting point using D-optimal Mixture Design (MED) and Artificial Neural Networks (ANNs).
2. To characterize the physicochemical properties of the optimized lipstick with respect to melting point, texture profile analysis, pH, antioxidant analysis, thermo gravimetric analysis, stability study and color stability.
3. To evaluate the safety of optimized lipstick by microbiological and heavy metals analysis to comply the "Halalan Toyyiban" requirements.

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