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OPTIMIZATION OF NON-STICK INSECT REPELLENT CREAM FORMULATION

B. ARIFIN
Faculty of Agrotechnology and Food Science Universiti Malaysia Terengganu, Terengganu D.I. Malaysia

A. BONO, D. KRISHNAIAH, M. RAJIN, R. SARBATLY
School of Engineering & Information Technology, University Malaysia Sabah, Sabah, Malaysia.

Abstract: Nowadays, insect repellents are widely used by consumers, especially insect repellents that are produced from natural-based such as citronella, as insects especially mosquitoes are well-known as disease carriers to human. Since the component of insect repellents could not be applied directly to human skin, base cream with insect repellents need to be formulated. The quality of the base cream is directly linked to the basic material used in the formulation. Varying the ratio of the ingredient used determines the final product characteristic. In this work, various compositions of carbopol, triethylamide, glycerine, water, and ethanol were used to prepare the base cream formulations. D-optimal mixture design was performed to obtain the optimum formulation. Twenty-five combination components were selected according to the D-optimal criterion. The consumer acceptance and physical properties of the base cream such as viscosity, drying time stickiness were studied. Three-dimensional surface plots were formed to assess the change in the response surface and to understand the effect of the mixture composition on lipstick characteristics. The result indicates that there are relationships between the processing variables of the lipstick formulation and the consumer acceptance.

KEYWORDS: Base cream formulation, D-Optimal mixture design, insect repellent, lipstick formulation

Introduction

Nowadays, humans face many types of diseases that are caused by insects such as malaria. Everybody knows that insects can transmit virus from the host to the human body through stings. Diseases that are spread by insects can be avoided by knowing the cause of the diseases and how the insects transmit the virus. Insect-transmitted diseases remain a major source of illnesses and deaths worldwide (Borror, 1989).

An insect repellent works by vaporizing the active ingredients such as diethylotoluamide and dimethylphthalate in the insect repellent product (Page et.al., 2002). The effectiveness of the insect repellent depends on the vaporization rate and washed-off properties. An insect repellent can be produced in the form of spray, lotion, or stick. There are two types of the insect repellents; synthetic chemicals and plant derived essential oil (Fradin and Day, 2004). Insect repellents such as the camphor, citronella, cedar, or DEET cannot be applied directly to human skin. Therefore, creams or lotions are used as the base of the insect repellents. The physical properties of the base cream may influence the consumer acceptance.

The challenge faced in formulating the base cream of the insect repellent is to achieve a base cream that has the optimum performance, with the addition of acceptable physical characteristics. In a formulation work, statistical experimental design such as D-optimal mixture design is commonly

Correspondence: Buhri Arifin, Faculty of Agrotechnology and Food Science, Universiti Malaysia Terengganu, 21030 Kuala Terengganu

used to obtain a product with the required characteristics. The primary goal of designing an experiment statistically is to obtain valid results at minimum of effort, time and resources (Myers and Montgomery, 2002).

The objective of this work was to ascertain a base cream of insect repellent formulation, which was assisted by D-optimal mixture design. In this work, carbopol, triethylamide, glycerine, water, and ethanol were selected as the raw materials to produce base cream insect repellent formulations. The formulations were based on the compositions that were suggested by D-optimal mixture design. The consumer acceptance and physical properties of the base cream such as viscosity, drying time and stickiness were studied. Three-dimensional surface plots were formed to assess the changes in the response surface where the relationships between the mixture compositions, physical properties and consumer acceptance of the base cream of the insect repellent were examined.

Materials and Methodology

Formulation Process

The experimental design of the five-component system was conducted by using Design Expert (version 6.10, Stat-Easy Inc., Minneapolis, USA). A set of candidate points in the design space was selected by using the D-optimal criterion (Myers and Montgomery, 2002). In this study, the restrictions on the component proportions X_j can be illustrated as follows:

$$L_j < X_j < U_j \qquad (1)$$

where L_j and U_j represent the lower constraint and upper constraints, respectively. The constraints of the components proportion are shown in Table 1.

Table 1: Constraints of the components proportion

Component, *Xj*	**Lower Limit, *Lj* (%)**	**Upper Limit, *Uj* (%)**
Carbopol, *X1*	0.60	1.50
Triethylamide, *X2*	0.01	0.10
Glycerine, *X3*	0.02	1.00
Water, *X4*	40.00	59.37
Ethanol, *X5*	40.00	59.37

Twenty-five base creams of insect repellent formulations were prepared in a laboratory scale in accordance with the composition percentage as suggested by the mixture design. The ingredients used in producing the base cream formulations were carbopol, triethylamide, glycerine, water, and ethanol. Firstly, water and ethanol were mixed in a 250 ml vessel. Next, carbopol was added gradually to the mixture. The mixture was stirred for two minutes before glycerin was added. Finally, fragrance and triethylamide (TEA) were added into the mixture. TEA acted as an emulsifier to concentrate the mixture. The mixture was then stirred homogeneously at a speed of 50rpm by using a mechanical stirrer. The base cream produced was stored for twenty-four hours before the physical characteristic tests.

Characterisation of Base Cream's Physical Properties

Viscosity

The viscosity of the base cream of the insect repellents is a very important factor in determining its stability [5]. The viscosity measurement was performed by using *Cole-Palmer Rotational Viscometer* with spindle no. R7. A volume of 100ml of base cream was poured into a beaker. The viscometer speed was set to 20rpm. After approximately twenty minutes, the final reading was taken. The process was repeated for three times before the average value was calculated.

Drying Time

In this study, the drying time was defined as the time taken for the cream to vaporise. One gram of the sample was spread on the skin and the vaporisation time was taken in second (Butler, 2000). The test was repeated for three times to obtain the average value.

Stickiness Test

There is no specific equipment to measure stickiness property. Small amount of the sample was taken and spread on the skin. When the sample had dried, the sample was tested for its stickiness. The results of the stickiness test were interpreted as 1 for sticky and 0 for not sticky (Butler, 2000).

Consumer Evaluation

The objective of the test is to investigate the acceptability of the formulated cream. The consumer acceptance of the cream is measured by using nine-point hedonic scale (Meilgaard, Civille, and Carr, 1999). The attributes to be observed are moistness, stickiness, volatility, odour, and texture.

Results and Discussion

Table 2 shows the design layout in terms of actual factor values. A set of candidate points in the design space is selected using the D-optimal criterion. In this work, 25 candidate points have been selected.

Table 2: Design Layout of Non-stick Insect Repellent Cream Formulation

Formultion Number	Design Factor (%)					Response		
	Carbopol	TEA	Glycerin	Water	Ethanol	Viscosity (cP)	Drying time (sec)	Stickiness
1	0.60	0.50	0.02	58.88	40.00	56300	92	0
2	1.50	0.50	0.02	40.00	57.98	89400	92	1
3	0.60	1.00	0.51	40.00	57.89	66200	70	1
4	1.50	0.01	1.00	40.00	57.49	9600	99	1
5	1.26	0.49	0.74	53.01	44.51	122300	87	1
6	1.50	1.00	1.00	40.00	56.50	105100	76	1
7	0.83	0.26	0.26	54.17	44.48	82000	108	0
8	0.60	0.01	0.02	40.00	59.37	8900	103	0
9	0.60	0.50	1.00	40.00	57.90	78100	89	0
10	0.60	1.00	1.00	57.40	40.00	77300	75	1
11	1.28	0.75	0.76	44.48	52.73	100200	90	1
12	0.60	1.00	0.51	40.00	57.89	58000	75	0
13	1.50	1.00	1.00	48.25	48.25	120300	104	1
14	1.26	0.49	0.25	44.51	53.50	80800	106	0
15	1.05	0.01	0.51	49.22	49.21	8100	93	1
16	1.50	0.01	0.02	58.47	40.00	17500	82	1
17	1.05	1.00	0.02	40.00	57.93	102000	82	0
18	0.83	0.26	0.26	44.48	54.17	63200	90	1
19	1.05	0.75	0.75	52.96	44.48	103000	104	0
20	1.50	1.00	0.02	57.48	40.00	120200	103	1
21	0.81	0.49	0.74	44.51	53.46	72000	96	1
22	1.50	1.00	0.02	57.48	40.00	112000	98	1
23	0.60	1.00	0.02	49.19	49.19	73700	62	0
24	0.60	0.01	1.00	58.39	40.00	5700	76	0
25	1.50	0.01	0.02	58.47	40.00	10500	86	1

Effect of the Composition Variation on the Viscosity of the Base cream of Insect Repellent

Since there are significant interaction effects between mixture composition and the viscosity of the cream, the response surface graph is shown in Figure 1. The diagram describes the variation on viscosity 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. In this case, water and ethanol composition were kept constant since these components have less effect on viscosity. The Figure 1 shows that the viscosity of the base cream decreases with a decrease in the TEA composition. This is because TEA acts as an emulsifier to thicken the base cream. However, the viscosity of the base cream increases with the decrease in the glycerine and carbopol composition.

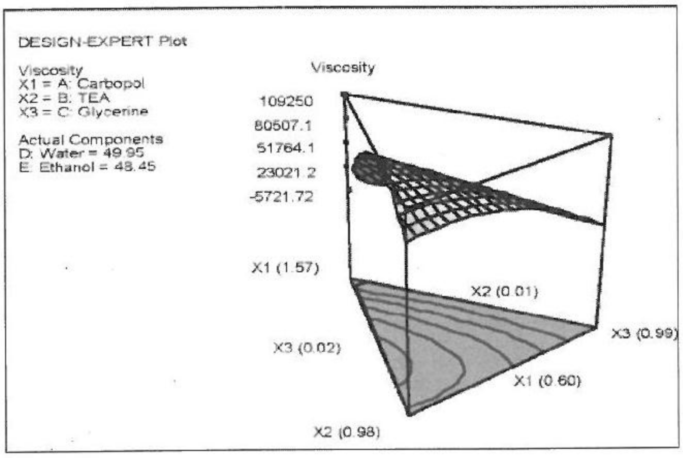

Figure 1: Three-Dimensional Surface of Viscosity (cP) Response

Effect of the Composition Variation on the Drying Time of the Base cream of Insect Repellent

Figure 2 describes the variation on drying time response as a function of the mixture composition. In this case, water and ethanol composition was kept constant since these components have the less effect on drying time. The Figure 2 shows that the moisture content of the base cream decreases with the increase in the glycerine composition. A high quantity of glycerine has resulted in an increase of the vaporisation time of the base cream. On the other hand, the moisture content of the base cream of insect repellent increases with the decrease in the TEA and carbopol composition.

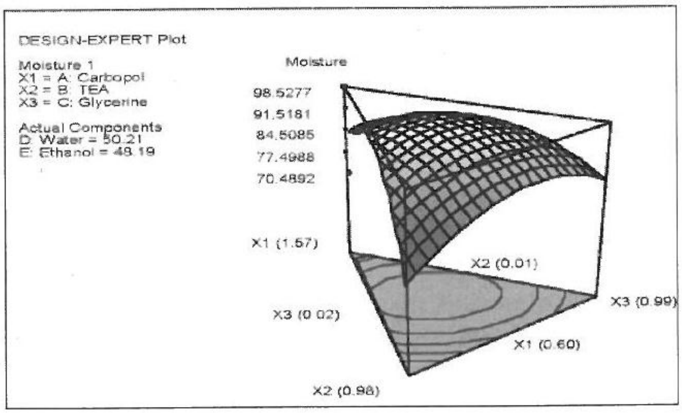

Figure 2: Three-Dimensional Surface of Drying Time(s) Response

Effect of the Composition Variation on the Stickiness of the Base cream of Insect Repellent

Figure 3 describes the variation on stickiness response as a function of the mixture composition. In this case, water and ethanol composition were kept constant since these components have the less effect on stickiness. Figure 3 shows that the stickiness of the base cream of insect repellent increases with the decrease in the glycerine composition. In contrast, the stickiness decreases with the decrease in the carbopol and TEA composition.

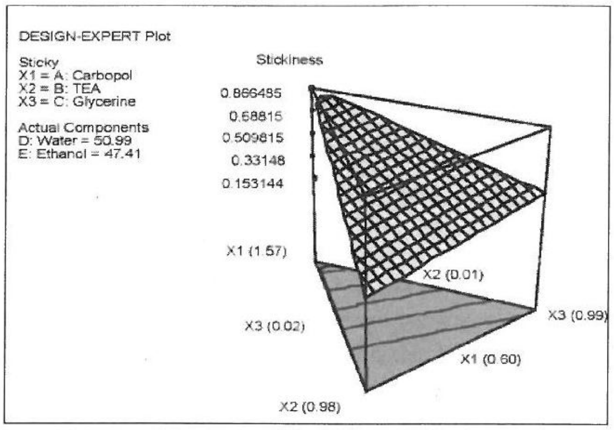

Figure 3: Three-Dimensional Surface of Stickiness Response

Optimization of Base Cream Insect Repellent Formulation

Numerical optimisation is performed in order to obtain the formulation with desired characteristics. The optimisation goal is based on the instrumental measurement and consumer evaluation data. The goal for each response is shown in Table 2.

Table 2: Optimization Target for the Response

Response	Goal
Viscosity	Minimum
Drying time	Minimum
Stickiness	In the target = 0

The result of the optimisation method suggests that the optimum formulation consists of 0.6% carbopol, 0.98%TEA, 0.07% glycerine, 44.07% water and 52.28% ethanol, with the highest desirability of 0.95.

Conclusion

Twenty-five non-stick base creams of insect repellent formulations have been prepared. The effects of the carbopol, TEA, glycerine, water and ethanol compositions on the physical properties and consumer acceptance of the base cream formulation have been studied. The results indicate that the physical properties and consumer acceptance can be manipulated by changing the mixture compositions. The statistical study shows that the fitted model is adequate to describe the viscosity, drying time and stickiness response. Numerical optimisation has been conducted based on the physical properties and consumer acceptability.

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