Research Article

Formulation of Sunscreen Cream of *Germanicol cinnamate* from the Leaves of *Tabat barito* (Ficus deltoides Jack) and an Assay of its' Sun Protection Factor

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ABSTRACT

The purpose of present task was to evaluate the sun protection factor (SPF) of Germanicol cinnamate in two types of sunscreen formulations FA (W/O) and FB (O/W) by ultraviolet spectroscopic method. The SPF values of the sunscreen cream containing 0.025% germanicol cinnamate were evaluated by UV spectroscopic method. The SPF values of FA were 3.48; 6.90; 7.54 and 7.15 after UV radiation exposure for 0, 1, 3 and 5 hours respectively which showed a maximum protection. FB demonstrated a better protection with the SPF values of 8.98; 14.25; 9.62 and 8.67 after UV exposure for 0, 1, 3 and 5 hours respectively, which was a maximum protection. FB demonstrated a better protection than FA.

Keywords: Sun protection factor (SPF), Sunscreens, Ultraviolet spectroscopy, Germanicol cinnamate, *Ficus deltoidea* Jack

INTRODUCTION

Sunlight composed of various wavelengths ranging from ultraviolet light. Some of which have negative effects on the human skin. Long term exposure of sun radiation could make degenerative changes in the skin cells which leads to premature ageing, sunburns and skin cancers.[1,2]

Sunscreens prevent or minimize the harmful effects of the solar radiations on the skin. The application of sunscreens is an efficient method of protecting skin against UV radiations.

The efficacy of a sunscreen is usually expressed by the sun protection factor (SPF).[3,4] The SPF is a numerical rating system to indicate the degree of protection provided by a sun which is defined as the UV energy required to produce a minimal erythema dose (MED) on protected skin, divided by the UV energy required to produce a MED on unprotected skin.[5]

$$SPF = \frac{minimal\ erythema\ dose\ in\ sunscreen - protected\ skin}{minimal\ erythema\ dose\ in\ nonsunscreen - protected\ skin}$$

The minimal erythemal dose (MED) is defined as the lowest time interval or dosage of UV light irradiation sufficient to produce a minimal, perceptible erythema on unprotected skin.[6,7] The higher the SPF, the more effective is the product in preventing sunburn.

Germanicol cinnamate is the main chemical constituents of the leaves of plants Tabat barito (*Ficus deltoid* Jack).[8] SPF (Sun Protection Factor) experiment showed that this compound provided protection against UV light, ranging from a minimum protection to ultra.[9]

This paper reported the preparation of sunscreen formulations using active substance germanicol cinnamate and evaluation of the effectiveness of test preparations cream and sunscreen preparations by determining the value of SPF (Sun Protection Factor) *in vitro*.

MATERIALS AND METHODS

Table 1: The formula of sunscreen containing germanicol cinnamate[10,11]

Ingredients	Composition (%)	
	FA (O/W type)	FB (W/O type)
Germanicol cinnamate	0.025	0.025
Paraffin liquidum	-	43.6
Cetyl alcohol	0.5	5
Stearic acid	3	-
Lanolin	1	1.5
Butyl hydroxyl toluene	0.02	0.1
Cera alba	-	2
Span 80	-	6.7
Tween 80	-	1.3
Glycerine	2	-
TEA	0.76	-
Nipagin	0.1	0.1
Parfum	q.s	q.s
Aquadest	92.595	39.675

Chemicals

Germanicol cinnamate was isolated previously from Tabat barito (Ficus deltoidea).[8]

Instruments

UV-Visible double beam spectrophotometer (UV-1800, Shimadzu limited, Japan) with 1 cm matched quartz cells, micropipette of variable volume 10-1000 µL (eppendorf.) and digital electronic balance (Kern.) were used in this study.

Sunscreen Cream Formulation[10,11]

Table 2: Physical Parameters of *germanicol cinnamate* sunscreen cream

Parameter	Observation	
	Formula A	Formula B
Color	White	White
Odor	Aromatic	aromatic
Spreadability	Good and uniform	Good and uniform
pH	7.35	5.80
Flow properties	Plastic	Thixotropic
Viscosity	49500-99000	49500-99000
Total microbial count	Nil	Nil
Patch test for irritancy	No irritation reaction persists	No irritation reaction persists

Step I

Aqueous Phase Preparation

Formula A

Glycerine (2% w/w), triethanolamine (0.72% w/w) and nipagin (0.1% w/w) were dissolved in deinosed water and heated up to 80°C.

Formula B

Tween (1.3% w/w) and nipagin (0.1% w/w) were dissolved in deionised water and heated up to 80^{0}C

Step II

Oil phase preparation

Formula A

Lanoline (1% w/w), stearic acid (3% w/w), cetyl alcohol (0.5% w/w), butyl hydroxytoluene (0.02%w/w) were mixed meanwhile, germanicol cinnamate (0.025% w/w) was added to swell using a homogenizer (8000 rpm) and heated up to 80 0C.

Formula B

Paraffin liquidum (43.6% w/w), cetyl alcohol (5% w/w), lanoline (1.5% w/w), butyl hydroxytoluene (0.1%w/w), cera alba (2% w/w), span 80 (6.7% w/w) and tween (1.3% w/w) were mixed meanwhile, germanicol cinnamate (0.025% w/w) was added to swell using a homogenizer (8000 rpm) and heated up to 80 °C.

Step III

Mixing phase

Oil phase was added to water phase at 80 °C with continuous stirring for 20-25 min and then it was homogenized (8000 rpm) till a homogenous emulsion was formed. It was then poured into the wide mouthed container and stored at temperature not exceeding 37°C.

Determination of Physical Parameters of Cream[12]

Preparation of herbal cream has always been a challenging task and the cream is accepted only if it is tested appropriately for various physical parameters like ease of spreadability, appearance as specified in Table 2.

Determination of *in-vitro* SPF of Sunscreen Cream

1.0 g of all samples was weighed, transferred to a 100 mL volumetric flask, diluted to volume with ethanol, followed by ultrasonication for 5 min and then filtered through cotton, rejecting the ten first mL. A 5.0 mL aliquot was transferred to 50 mL volumetric flask and diluted to volume with ethanol.

Then a 5.0 mL aliquot was transferred to a 25 mL volumetric flask and the volume completed with ethanol.

The absorption spectra of samples in solution were obtained in the range of 290 to 450 nm using 1 cm quartz cell, and ethanol as a blank.

The absorption data were obtained in the range of 290 to 320, every 5 nm, and 3 determinations were made at each point, followed by the application of Mansur equation.[13]

$$\text{SPF} = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times Abs(\lambda)$$

Where EE(I)- erythemal effect spectrum; I(I)-solar intensity spectrum; Abs-Absorbance of sunscreen product; CF-correction factor(=10).

The value of EE x I are constant and predetermined (Table 3).[14]

Table 3: Normalized product function used in the calculation of SPF

λ (nm)	A1	A2	A3	EE x I	EE x I x Â
290	0.726	0.856	0.739	0.0150	0.0116
295	0.820	0.801	0.888	0.0817	0.0683
300	0.889	0.820	0.808	0.2874	0.2411
305	0.851	0.873	0.870	0.3278	0.2835
310	0.840	0.873	0.868	0.1864	0.1603
315	0.756	0.844	0.743	0.0839	0.0655
320	0.615	0.834	0.652	0.0180	0.0126
		Σ			0.8429

$$\text{SPF} = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times Abs(\lambda)$$

30 = CF x 0.8429

CF = 30 / 0.8429 = 35.59

RESULTS AND DISCUSSION

In the present study germanicol cinnamate which was previously isolated from Ficus deltoidea was formulated into two types of sunscreen creams (FA as oil in water and FB as water in oil cream type).

The finished products have white colour and gel like consistency. Creams were evaluated by UV spectrophotometry applying Mansur mathematical equation.[13] The SPF labeled values were in the range of 8 to 30. These products and SPF values of samples obtained using the UV spectrophotometric method were shown in Table 3. It indicates that formulated sunscreen cream was found near the range of good sunscreen activity and hence germanicol cinnamate may be considered as good candidate for sunscreen or cosmaceutical purposes. Further this cream was evaluated for spreadability, viscosity and rheology, physical stability etc.

Observations of the protection power of the sunscreen preparations containing germanicol cinnamate in FA and FB in vitro were done without irradiation and with irradiation under ultraviolet light.

Results showed that germanicol cinnamate was able to provide maximum protection to absorb UV rays, both formulations FA and FB had the SPF values of 7.15 and 14.25 respectively (Table 4).

Table 4: SPF value with length of radiation

Formula	SPF value/length of radiation (hours)			
	0	1	3	5
FA	3.48	6.90	7.54	7.15
FB	8.98	14.25	9.62	8.67

There are many factors affecting the determination of SPF values, as for example, the use of different solvents in which the sunscreen agent was dissolved; the concentration of the sunscreen agent; the addition of other active ingredients; the type of emulsion; the effects and interactions of vehicle components, such as esters, emollients and emulsifiers used in the formulation; the interaction of the vehicle with the skin; the pH system and the emulsion rheological properties, among other factors, which can increase or decrease UV absorption of each sunscreen agent.

The effect that different solvents and emollients have upon the wavelength of maximum absorbance and upon the UV absorbance of several sunscreens chemical, alone or in combination is well known and documented.[15,16]

Excipients and other active ingredients can also produce UV absorption bands, thus interfering with those of UVA and UVB sunscreen. This effect is reflected in a finished formulation, especially for lotions with an SPF greater than 15. The effect of a solvent is only realized at high percentages.

Therefore, to develop sunscreens with better safety and high SPF, the formulator must understand the physico-chemical principle, not only the UV absorbance of the actives, but also vehicle components, such as esters, emollients and emulsifiers used in the formulation, since sunscreens can interact with other components of the vehicle, and these interactions can affect sunscreens efficacy.

The effectiveness of germanicol cinnamate as sunscreen active agent was measured by determining the value of SPF (Sun Protection Factor).[14] The test showed that germanicol cinnamic solution in ethanol at a concentration of <60 mg / mL have a minimal protective effect (SPF 2 - <4).

Meanwhile at a concentration of 120 ug / ml and 150-200 mg / mL, cinnamic germanikol compounds each having an extra protective effect (SPF 6 - <8) and maximum protection (SPF 8 - <15) respectively. Effects of ultra protection (SPF ≥ 15) is shown by germanicol cinnamic at a concentration of 250 mg / mL.

Based on its SPF value, it is known that germanicol cinnamate able to provide protection to the skin against UV-B radiation and can be used as an active ingredient in the preparation of cosmetic creams are effective as a sunscreen with a minimum value of protection to the ultra protection.

The results of the examination of all the additional materials (cetyl alcohol, stearic acid, lanolin, cera alba, glycerin, liquid paraffin, Tween 80, Span 80, and nipagin) used has met the requirements of the Indonesian Pharmacopoeia.[12]

Formulation of germanicol cinnamate in a semisolid dosage form was then conducted to observe whether the basis influence the SPF value of the active ingredient.

Two cream bases were made, the oil in water (FA) and the water in oil (FB), germanicol cinnamate at concentration of 0.025% was added (Table 1).

Results showed that cream FA and FB were physically stable during storage with the pH value of 7.35 (FA) and 5.80 (FB) which were well tolerated by the skin. The washable values were 6.6 and 16.7 mL of water for FA and FB respectively.

The rheograms of FA and FB (Figure 1 and 2) showed a pseudo plastic-viscoelastic combination of flow properties which was good for topical preparation.

The particle size ranged from 0 - 133 μm and distributed normally which confirmed that the active ingredient had been distributed homogenously into the cream base. The SPF values of FA were 3.48; 6.90; 7.54 and 7.15 after UV radiation exposure for 0, 1, 3 and 5 hours respectively which showed a maximum protection.

FB demonstrated a better protection with the SPF values of 8.98; 14.25; 9.62 and 8.67 after UV exposure for 0, 1, 3 and 5 hours respectively, which was a maximum protection.

There was a decrease in the SPF value of germanicol cinnamate in the creams (the highest SPF of FA was 7.54 and FB was 14.25) in comparison to germanicol cinnamate alone ($\geq$ 15) at the same concentration. Germanicol cinnamate in a water in oil cream base showed a better UV protection.

CONCLUSION

The proposed UV spectrophotometric method is simple, rapid, employs low cost reagents and can be used in the *in vitro* determination of SPF values in many cosmetic formulations. The proposed methodology may be useful as a rapid quality control method. It can be used during the production process, in the analysis of the final product, and can give important information before proceeding to the *in vivo* tests.

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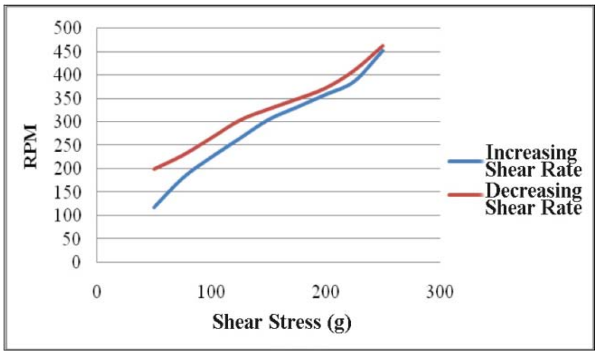

Figure 1: Rheological properties of FA cream

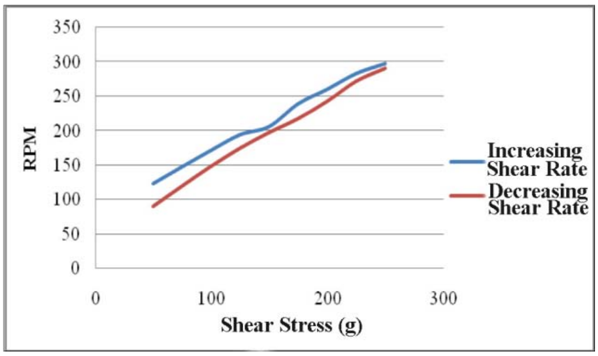

Figure 2: Rheological properties of FB cream

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