Physical Characteristic and SPF Value of Oxybenzone and Octyldimethyl PABA: Effect of Arbutin and Olive Oil in Carbomer 940 Gel Base

by Noorma Rosita

Physical Characteristic and SPF Value of Oxybenzone and Octyldimethyl PABA: Effect of Arbutin and Olive Oil in Carbomer 940 Gel Base

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ABSTRACT: The aim of this research was to investigate the influence of arbutin (3% w/w) and olive oil addition (3, 5, and 7% w/w) on characteristic and SPF values of sunscreen product containing oxybenzone and octyldimethyl PABA (3:7% w/w) in a carbomer 940 gel base. The characteristic test was conducted by observing the physical appearance, pH and spreading ability on the second day after the product was made; while the SPF values were measured through a spectrophotometric method. Based on the results of the characteristic test, it was evident that the addition of arbutin and olive oil affected the physical appearances i.e the organoleptic and consistency of sunscreen product as well as its spreading ability, but had no effect on its pH. However, the addition of arbutin and olive oil significantly improved the effectiveness of sunscreen product and increase in the concentration of olive oil resulted in a rise in the SPF values. Nonetheless, further study on the in vivo effectiveness using human subject is required to comprehend the SPF application.

Keywords: arbutin, olive oil, oxybenzone, octyldimethyl PABA, sun protection factor

1. INTRODUCTION

Exposure to sunlight could have both advantageous and harmful effects on the human body. According to Tranggono and Latifah (2007); Wilkinson et al. (1973), the adverse reaction to the UV sun rays includes erythema, tanning, sunburn, photo ageing, hyper pigmentation, photosensitivity and skin cancers. Hence, protecting the skin from these harmful effects of solar radiation could be achieved using sunscreens. Active ingredients present in sunscreens are divided to physical and chemical agents. The physical sunscreen agentsand not transparent, hence, reflect the UV rays from the skin and need high concentration to be effective. These make them not preferable for some people. The chemical sunscreen agents absorb UV-A radiation, such as oxybenzone, and UV-B radiation such as octyldimethyl PABA. According to Widianingsih and Lumintang (2002), a combination of an anti UV-A and anti UV-B sunscreen agents have been recently used in many sunscreen products in order to obtain high protection effect with broad spectrum sunscreen.

Normally, skin has its own protection mechanism against these harmful effect of UV rays, such as thickening of stratum corneum, sweating, and skin pigmentation. The abnormal increase of melanin as a result of this natural skins protection has the capacity to cause a non-homogenous skin color, usually disliked by most people. Hence, whitening agents are used to control the production and metabolism of this melanin in the epidermis. Arbutin is a hydroquinone derivate frequently used as whitening agent which inhibit melanin production. According to Mashood (2006), arbutin has a lower toxicity compared with other hydroquinone and its depigmentation effect is higher compared with kojic acid and vitamin C. This substance is used as whitening agent at various concentration, usually in the range of 0.5 - 3.0%. It has a low partition coefficient and penetration rate, hence, it needs the addition of a penetration enhancer in order to work optimally (Zulkarnain, 2003; Galilee, 2008; Mitsui, 1998).

A common penetration enhancer is olive oil. This is a pure oil obtained from Olea europaea Linn containing 83.5% oleic acid, a substance capable of interacting with and modifying the lipid bilayer of stratum corneum, thereby increasing its lipophilicity. Also, the ability of this substance as penetration enhancer in local anesthetic agent has been proven by Sharma and Fisher (1993). Olive oil has been widely used in sunscreen and other cosmocheutical preparation due to its emollient activity. Additionally, it is nonirritant and considered as a natural lipid with the highest compatibility with human skin (Rowe et al. 2003; O'Neil, 2006).

There are various sunscreen preparations available in the market such as cream, lotion and gel. However, among all, gel gives cool sensation, it is non-sticky, elegant, smooth and easy to be removed by washing. In addition, a synthetic gelling agent is usually required in a small quantity to produce a good gel which is consistent in its action. Hence, carbomer 940 is considered the most suitable gelling agent for this study.

In this study, a combination of oxybenzone 3% w/w (anti UV-A) and octyldimethyl PABA 7% w/w (anti UV-B) (Pratiwi, 2006) together with arbutin 3% w/w and various concentration of olive oil (3, 5, and 7% w/w) in carbomer 940 gel base are used in the formula, then the change in SPF value were observed through spectrophotometric method.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this study include Oxybenzone from Surya Dermato, Octyldimethyl PABA from Surya Dermato, Arbutin from Asia Visions Ltd., Olive oil from Brataco Chemicals, Carbomer 940 from Brataco Chemicals, Triethanolamine from Surya Dermato, EDTA Sodium from Surya Dermato, Methyl paraben from Surya Dermato, Propyl paraben from Surya Dermato, BHT from Brataco Chemicals, Tween 80 from Surya Dermato, Propylene glycol from Brataco Chemicals, and Isopropanol p.a. from Brataco Chemicals. All these ingredients were in pharmaceutical grade except isopropanol, which was in analytical grade.

Table 1. Formula used in experiment.

Composition	**Concentration in formula (% w/w)**				
	TS	**TS+A**	**TS+A+O 3%**	**TS+A+O 5%**	**TS+A+O 7%**
Oxybenzone	3	3	3	3	3
Octyldimethyl PABA	7	7	7	7	7
Arbutin	0	3	3	3	3
Tween 80	0.5	0.5	0.5	0.5	0.5
Olive oil	0	0	3	5	7
Carbomer 940	1	1	1	1	1
Distilled water up to	100	100	100	100	100

*TS = Sunscreen
TS+A = Sunscreen + Arbutin
TS+A+O 3% = Sunscreen + Arbutin + Olive oil 3%
TS+A+O 5% = Sunscreen + Arbutin + Olive oil 5%
TS+A+O 7% = Sunscreen + Arbutin + Olive oil 7%

2.2 Preparation of sunscreen gel containing arbutin and olive oil

Firstly, the EDTA Sodium was dissolved in distilled water, then an amount of carbomer was dispersed into it. This mixture was left for a night after the pH was adjusted to 6 with the addition of Triethanolamine (TEA). Next, Methyl-paraben and Propyl-paraben were dissolved in propylene glycol, then poured into a mixture of carbomer 940 and stirred to create a good gel base.

Also, Arbutin was dissolved in distilled water after which Tween 80 was added into it. The solution of BHT in olive oil was mixed together in it to form a good emulsion system. Next, Oxybenzone, Octyldimethyl PABA, and propylene glycol were mixed and added to the emulsion system, stirred well before being added into the gel base. This was then stirred well to form the sunscreen gel, which was kept in a tight container and stored well for further analysis. Other treatment formulas were similarly prepared with main composition as shown in Table 1.

2.3 Characteristics determination of sunscreens gel

The sunscreen gel was subjected to organoleptic test which was visually evaluated, however, the pH and spreading-capacity were determined at Day 2 after the formula was prepared, with the use of a Digital pH meter Schott CG 842, and a spreading-capacity measurer respectively.

To determine the pH, about 2 grams of the sunscreen gels was mixed thoroughly with 18 ml of water free-CO2 before measuring the pH with the pH meter.

Similarly, the spreading-capacity was determined by placing an approximately 1 gram of the gel on a glass plate with a millimeter scale. This was then covered with another glass plate, after which the change in gel-spreading was observed along with an increase of the given load.

2.4 Determination of SPF value of sunscreens gel

To observe the effect of the addition of arbutin and olive oil on SPF value, about 100.0 mg sunscreens was dissolved in 2.0 ml isopropanol, which was then centrifuged for 15 minutes at 50 rpm. Then, about 1.0 ml of the filtrate was poured into a 5.0 ml measuring flask and properly shaken till it was homogenous (10000 ppm).

Next, about 1.0 ml of the mixture was then pipetted into a 10.0 ml metered flask and diluted to make 1000 ppm solution. Then 1.0 ml of this solution was pipetted into another 10.0 ml flask after which isopropanol was added to dilute it and then properly shaken to obtain a concentration of 100 ppm, containing 10 ppm sunscreen's active ingredients. The UV spectrum of this solution was measured at 290 - 400 nm with the use of Double Beam UV-Vis Spectrophotometer Perkin Elmer Lambda EZ 201 at interval of 2 nm, with an absorption larger than 0.050.

Based on the method used by Petro (1981), the absorption data received were converted into the absorption value on 10 ppm concentration for each wavelength. The AUC of each formula from the shortest and longest wavelength were counted using the following equation:

$$AUC_{\lambda p-\alpha}^{\lambda p} = \frac{A_{p-\alpha} + A_p}{2} \lambda_p - \lambda_{p-\alpha}$$

Where:
AUC = Area under Curve
Ap = Absorption on p wavelength
Ap-α = Absorption on p-α wavelength

A total of AUC was obtained by adding each AUC between 2 wavelengths in series from 290 nm till 400 nm, with an absorption value above 0.050, and the formula for the SPF value was obtained by inserting the total AUC into the equation below:

$$log\, log\, SPF = \frac{Total\ area}{\lambda n - \lambda 1}\ x\ 2$$

Where:
λn = the longest wavelength above 290 nm that has an absorption value higher than 0.050
λ1 = 290 nm

The Log SPF value obtained from the equation was then converted into SPF value.

Table 2. Sunscreens category based on SPF value.

Category	SPF	Protection	
		sunburn	tanning
Minimum	2-<4	Minimum	-
Moderate	4-<8	Moderate	Small
High	8-<12	High	Limited
Very high	12-<20	Very high	Large
Ultra	20-30	Maximum	Maximum

*American Society of Health System Pharmacist, 2002

2.5 Statistical analysis

The coefficients variation of all data obtained from the experiment were calculated to ensure the homogeneity of the formula. Also, a One-way ANOVA test was used to assess the significance of differences and with regards to the multiple comparison of the F significant values, Tukey's test was used to compare the means of different treatment groups. Then results with the values of $p < 0.05$ were considered statistically significant.

3. RESULT AND DISCUSSION

The average data of organoleptic observation from all the sunscreen formula are shown in Table 3. Considering these results, there are differences in color and smell upon addition of olive oil to the formula. It was more yellowish and the smell was that of olive oil. An ascend viscosity of formula was observed as a result of an increase in the olive oil added. The viscosity of the formula increased with the addition of more olive oil.

Table 3. Organoleptic analysis of the sunscreens formula.

Formula	Color	Smell	Consistency
Gel base	Transparent	Octyldimethyl PABA	A bit viscous
TS	Yellowish white	Octyldimethyl PABA	Not very viscous
TS+A	Yellowish white	Octyldimethyl PABA	Viscous
TS+A+O 3%	Yellowish	Olive oil	Viscous
TS+A+O 5%	Yellowish	Olive oil	Very viscous
TS+A+O 7%	Yellowish	Olive oil	Very viscous

Table 4. The results of pH measurements of the sunscreen gels (n = 3).

Formula	pH (average)	% var. coefficient
Gel base	6.60 ± 0.08	1.29
TS	6.27 ± 0.05	0.79
TS+A	6.31 ± 0.05	0.74
TS+A+O 3%	6.31 ± 0.03	0.43
TS+A+O 5%	6.30 ± 0.05	0.86
TS+A+O 7%	6.29 ± 0.02	0.37

* The result were obtained from an average of 3 times replication

Table 5: Result of HSD test of the sunscreens pH value

Formula	N	pH value Classification (α=0.05)	
		1	2
TS	9	6.27	
TS+A+O 7%	9	6.29	
TS+A+O 5%	9	6.30	
TS+A	9	6.31	
TS+A+O 3%	9	6.31	
Gel base	3		6.60

The results of pH analysis showed no significant difference with the addition of arbutin or olive oil, as well as increase in the concentration of the olive oil. Also, the data in Table 4 and Table 5 show that the sunscreen formulas were all in a range of skin pH.

Additionally, the spreading capabilities of the formula were measured and the results are shown in Table 6 and Figure 1. Based on the data obtained, the spreading-capacity of the formula reduced with the addition of arbutin and increased with olive oil concentration as a result of an increase in its viscosity. The pH of sunscreen gels also play a major role in the sunscreen gels characteristic as the carbomer 940 consistencies were heavily affected by its acidity, a high acidity condition lowers the gels viscosity.

Table 6: Average slope of spread capability of sunscreens formula (n=3)

Formula	Average slope	% Var. coefficient
Gel base	0.2833 ± 0.0057	-
TS	0.3359 ± 0.0076	2.26
TS+A	0.3859 ± 0.0119	3.09
TS+A+O 3%	0.2713 ± 0.0094	3.47
TS+A+O 5%	0.2474 ± 0.0072	2.91
TS+A+O 7%	0.2238 ± 0.0028	1.25

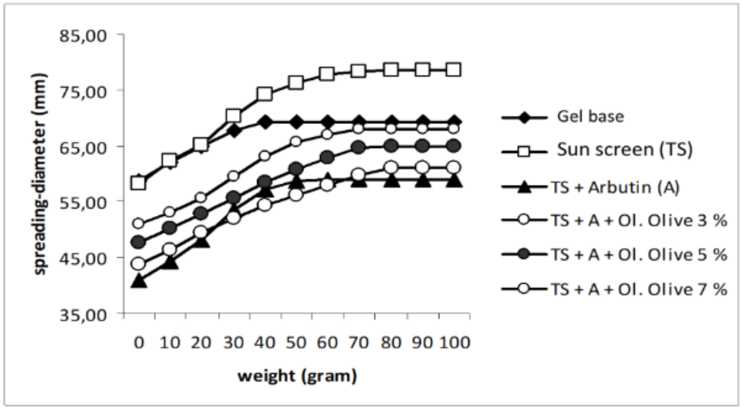

Figure 1. Spreading capability of sunscreen.

The SPF analysis was determined by extracting the sunscreen from its base with isopropanol. To make sure that the base gel carbomer 940 does not give any absorption in the UV spectrum, it was extracted using isopropanol and observed at a wavelength within the range of 290 - 400 nm. From the spectra it is known that the gel base did not give any absorption, therefore, it is assumed that any change within the observation of SPF value were caused by the active ingredient only.

To ensure the homogeneity in the formula, the % variation coefficient of each sampling in each replication as well as the % variation coefficient of each replication of each formula were calculated and the SPF value that resulted are shown in Table 7, which was then compared to the American Society of Health System Pharmacist standard shown in Table 2. This helps to know its protection capability. Also, this standard contains two different category of protection against UV rays as presented in Table 8.

Table 7: SPF data from the treatment formula

Formula	Replication	SPF (average)*	% var. coefficient *	SPF (average)**	% var. coefficient **
	1	6.73	2.08		
TS	2	6.56	2.36	6.66	4.09
	3	6.68	1.71		
	1	9.20	1.98		
TS+A	2	9.11	4.20	9.09	2.79
	3	8.97	2.23		
	1	11.91	3.63		
S+A+O 3%	2	11.84	1.73	12.02	3.14
	3	12.30	3.27		
	1	19.74	2.85		
S+A+O 5%	2	19.44	3.29	19.53	2.53
	3	19.41	1.96		
	1	26.30	2.18		
S+A+O 7%	2	25.21	3.08	25.92	3.44
	3	26.25	4.55		

*The data were obtained from 3 different samples in 1 product

**The data were obtained from an average of 3 different products in 1 formula

Table 8: The sunscreen's formula protection category

Formula	SPF (average)	Protection category
TS	6.66	Moderate
S+A	9.09	High
S+A+O 3%	12.02	Very high
S+A+O 5%	19.53	Very high
S+A+O 7%	25.92	Maximum

Considering the data in Table 7, it can be concluded that all of the sunscreen formulas during the experiment were homogeny and reproducible considering the fact that its variation coefficient were less than 6%.

These results were also supported from the amount of variation coefficient that was acquired from the pH value and the spreading-capability assessment that were also less than 6%.

Based on the comparisons above, the sunscreen's formula were categorized as shown in Table 8. In addition, the result of HSD test presented in Table 9 indicates that there was an increase in the SPF value of the sunscreen gels from moderate to maximum protection together with the addition of arbutin as well as an increase in olive oil concentration in the formula.

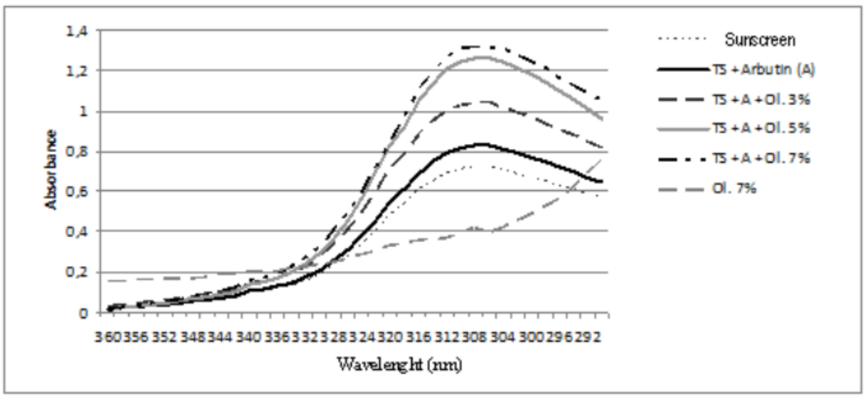

Figure 2. Absorption spectrum of sunscreen.

Table 9. Result of HSD test of the sunscreens SPF.

Formula	N	SPF category α=0.05				
		1	2	3	4	5
TS	9	6.60				
TS+A	9		9.09			
TS+A+O 3%	9			12.02		
TS+A+O 5%	9				19.53	
TS+A+O 7%	9					25.99

Table 9 shows that the addition of arbutin and olive oil significantly elevated the SPF value of sunscreen gels and the maximum protection was given by the formula which contains 7% concentration of olive oil. According to Chiari et al. (2014), synergistic effect of olive oil and synthetic sunscreen are shown by the green coffee oil.

Additionally, based on the screening above, as shown in Figure 2, it is obvious that the addition of arbutin and olive oil does not cause any movement on the maximum wavelength. However, an increase in the intensity of absorption was observed. Hence, it is predicted that an interaction occurred between the arbutin molecule and sunscreen agent which intensify the effect of auxochrome group and a decrease in polarity of sunscreen gels which affects the delocalization of the molecule. This resulted in a rise of energy demand needed for excitation to happen, thereby increasing the SPF value.

According to O'Neil (1984), other factors influencing the sunscreen SPF values include the pH, extinction coefficient and solvent polarity. Therefore, it is important to ensure that the reaction resulted to the SPF changes in the treatment formula.

4. CONCLUSION

The addition of arbutin and olive oil altered the physical appearance of sunscreen gel combination Oxybenzone and Octyldimethyl PABA, as well as the organoleptic and spreading ability of sunscreen product. However, this did not have any effect on its pH. Furthermore, the addition of arbutin and olive oil increased the SPF value of its sunscreen gel.

ACKNOWLEDGEMENT

The authors are grateful to the Project Grant Faculty of Pharmacy, Airlangga University, for funding this project work. The authors also like to thank Surya Dermato Medica Factory for providing the materials needed for the sunscreen gels preparation.

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