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화장품에서 UV 차단제의 피부 자극성과 SPF 측정

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SPF Measurement and Cytotoxicity of Sunscreen Agents in Cosmetic

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요 약: 최근의 소비자들은 waterproof 기능을 가진 광범위한 UV 차단 제품을 선호하며, 피부 안전성에도 많은 관심을 가지고 있다. NR method로 세포 자극성(*in-vitro*)을 실험한 결과, UV-B의 자외선 차단제들은 0.08 w/v% 이상에서 세포의 생존율이 떨어졌고, UV-A의 자외선 차단제들은 0.06 w/v% 이상에서 세포의 생존율이 떨어졌다. 무기 자외선 차단제들의 patch-test는 각각 10.5와 11.25 이하로 피부에 자극성은 없었다. UV 흡수 곡선, UV-B는 octyl methoxycinnamate(OMC), UV-A는 butyl methoxy dibenzoylmethane(BMDM) 가장 적합하였다. 따라서, 유기 자외선 차단제는 OMC와 BMDM이 nylon과 polyethylene에 코팅된 원료인 Nylonpoly™ UVA/UVB을 사용하였다. 무기 자외선 차단제는 zinc oxide (ZnO)와 titanium dioxide(TiO_2)를 사용하였다. ZnO와 TiO_2의 혼합 비율은 6 : 4가 적당하였다. 총 UV차단제의 함량은 ZnO 6%, TiO_2 4%와 Nylonpoly™ UVA/UVB 5%를 썬 스크린 크림에 혼합하였다. *In-vitro*의 SPF값은 38.9이었다. 응용으로, 동일 함량을 넣은 O/W type과 W/S type의 썬 스크린 크림을 만들어 시간 경과에 따른 SPF를 측정한 결과, SPF가 O/W type보다 W/S type이 5배 이상 오래 지속되었다. 이것은 해수욕, 등산, 스키를 탈 경우에 효과적이다. 본 연구는 자외선 차단제의 피부 트러블을 최소화하고자 하였으며, 매끄러움성, 피부 안전성과 자외선 차단 효과가 우수한 화장품이라고 생각되어진다.

Abstract: Consumers have recently preferred to purchase extensive UV intercepting products, which are waterproof and free from side effects on skin. During the testing of cytotoxicity (*in-vitro*) in neutral red (NR) method, cell survival ratio of UV-B interceptors decreased to just above 0.08 w/v%, and it was observed that the UV-A interceptors the ratio also decreased to just above 0.06 w/v%. In addition patch-tests of inorganic UV interceptors resulted in no skin irritation even below 10.0 and 11.25. In absorption curves, UV-B was most suitable for octyl methoxycinnamate (OMC) and UV-A for butyl methoxy dibenzoylmethane (BMDM). For this reason, Nylonpoly™ UVA/UVB the material of OMC and BMDM coated with Nylon & polyethylene, was used as the organic UV interceptor. Zinc oxide (ZnO) and titanium dioxide (TiO_2) was used as inorganic UV interceptors. The appropriate mixture ratio of ZnO and TiO_2 was 6 to 4 : 6% of ZnO, 4% of TiO_2 and 5% of Nylonpoly™ UVA/UVB were all combined and added to our sunscreen cream. The SPF value of *in-vitro* was 38.9. In practical application, each sun protection factor (SPF) duration of oil-in-water (O/W) emulsion and water-in-silicone (W/S) emulsion containing sunscreen cream of the same content showed that W/S type of sunscreen cream was 5 times as durable as the other. Therefore, this product is fit for use in swimming, climbing or skiing. This research is to minimize skin trouble caused by UV interceptors and to make one with proper softness, skin safety

and UV intercepting efficiency.

Key words: sun protection factor(SPF), cytotoxicity, oil-in-water, water-in-oil, titanium dioxide, zinc oxide, Nylonpoly™ UVA/UVB, Skin irritation

1. INTRODUCTION

The method of sun protection factor (SPF) in UV intercepting products has varied previously from nation to nation and company to company. But in 1978, The food and drug administration (FDA) of the U.S.A. established a standard of SPF measurement in 1983 and 1984. Australia and Germany have also begun to establish their own standards.[1,2] Materials of basic cosmetic products have been extracted from natural objects such as animals and plants. Ultraviolet ray is defined as the wave length of 200~400 nm, and divided into UV-B of 280~320 nm and UV-A of 320~400 nm. It is known that the formation of skin cancer begins with the damaged DNA of skin cells and mutation of cells by deformed DNA is the primary factor in accelerated skin aging.[3,4] Thus, the direct irradiation of UV-B of ultraviolet ray to skin should be prevented because it causes skin irritation and furthermore skin cancer.

UV-A has higher skin permeability which causes more troubles to skin oxidation than UV-B. To solve this, ultraviolet ray should be completely shut off to skin. Although, UV-B and UV-A interceptors have been developed, most of the those products are only allowed to use within a limited amount of sunscreen cream due to their toxicity and safety of UV ray.[5~7]

In addition, UV interceptors are mainly composed of organic synthetic materials, and their cytotoxicity are believed to be a primary assignment that cosmetic researchers have to deal with. For this reason, there have been many trials on high values of SPF by applying an inorganic UV protector. Also, natural synthetic UV interceptors have been developed to use a group of amino acids stabilizing emulsification. In order to capsulate Suncaps (Sunsmart Co. USA) with octyl methoxycinnamate (OMC) in matrix with zinc oxide (ZnO), many sun creams are capsulated with polymethylmathacrylate (PMMA).[8~10]

The most of companies are devoting their efforts to make safe products from cytotoxicity. More efficient UV interceptors are especially needed to enjoy sports such as swimming, skiing and mountain climbing. For this reason, the durability and waterproofing properties of the UV interceptors have become very important.[11~13]

In this study, we have tested the cytotoxicity of UV interceptors and have tried to find the materials with the least irritation on skin. With the recent importance of cytotoxicity of UV interceptors, we tried to minimize cytotoxicity by using some raw materials that contact skin indirectly by coating it with a measuring the range of UV extinction of organic and inorganic UV interceptors. SPF effect was evaluated by the method of O/W and W/S emulsification, and the intercepting effect was measured by comparing SPF in *in-vitro*.

2. MATERIALS AND METHOD

2.1. Materials

In order to measure the skin irritation factors of UV interceptors, we employed organic and inorganic UV interceptors in UV-B and UV-A ranges. For emulsifiers used in UV intercepting cream, W/O emulsifier of cetyl dimethicone copolyol (Goldshmidt, Germany) and sorbitan sesquioleate (ICI, America) were used. The silicone oil was used to improve a feel touch, and magnesium-aluminum silicate copolyol (ECC, America) and magnesium stearate(Witco Organic) were used as a steady increaser. Also, to remove the irritation of an organic UV interceptor, Nylonpoly™ UVA/UVB(Creations Couleurs™, USA) coated with nylon and polyethylene were used for OMC and BMDM. Lastly, we used ZnO and TiO_2 to obtain a high SPF value.

In this experiment, reagents were used without refining exclusive cosmetic materials. Cosmetic in-

gredients used were vitamin-E and sodium hyaluronate. Positive/negative deionized water passaged ion exchange resin top was used.

2.2. The measurement method of skin irritation of sunscreen agents

For a cell, normal human fibroblast was cultured by combining 5% of CO_2 at 37℃ with DEME containing 5% of fetal bovine serum. The cell collected with neutral red assay was diluted by a culture medium containing 2% of serum and inoculated 90 μL of cell suspension (2500~3000 cells/well) in each well of 96 well tissue culture plates and then, cultured at 37℃ with 5% CO_2 for 2 days. After culture, it was exchanged with 90 μL of a new culture medium and treated with 10 μL of a testing material, and then cultured again for 2 days. Into each well padding culture of the tissue, 100 μL of neutral red solution (50 μg/μl) was added and kept for 3 hours. We extracted neutral red in a cell using 1.0% of acetic acid : ethanol(1 : 50=v : v) solution after treating with 100 μL of a solution of 1.0% of $CaCl_2$ in 10% of formalin condensed in lysosome of a cell which was not damaged or alive when neutral red passed complete plasma membrane. Extracted neutral red was directly measured for its cytotoxicity in 540 nm with ELISA reader.[14,15]

2.3. *In-vitro* SPF measurement

To measure UV intercepting value of UV-B and UV-A, we made the most stable W/O emulsion cream containing a fixed amount of UV intercepting material. We then measured the SPF with SPF-290 analyzer (*Fig.* 1). The amount of the sample for the measurement was 2.0 μL/cm^2.

The test method is a modification of the *in-vitro* SPF testing method of Diffey and Roboson.[16] Therefore, in this theoretical study SPF value was calculated as the following:

$$SPF = \frac{\sum_{290}^{400} E_\lambda \cdot B_\lambda}{\sum_{290}^{400} (E_\lambda \cdot B_\lambda / MPF_\lambda)}$$

$$MPF_\lambda = 1/T_\lambda$$

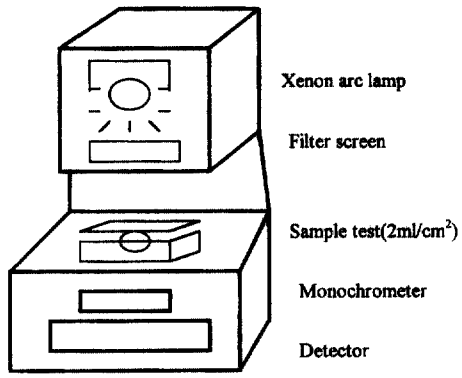

Fig. 1. Schematic diagram of the SPF 290-analyzer.

$$MPF_\lambda = \sum_{i=1}^{n} (MPF_\lambda)\, i/n \ (n=18)$$

λ: wavelength of monochromatic light

T_λ: transmittance at λ

MPF_λ: an indicator of the protection property of a sunscreen product at λ

B_λ: a measurement of the effectiveness of a given λ to produce erythema

E_λ: spectral irradiance of terrestrial sunlight at middy

3. RESULTS AND DISCUSSION

3.1. UV-protector

Nowadays, the harmfulness of ultraviolet rays on skin is percepted as seriously and has become not only a problem with cosmetic but an earthwide environmental pollution. Therefore, products with high SPF also need safety to skin and stability of components. To enhance the effects of intercepting ultraviolet rays, it need to be combine with the materials possessing the effect of intercepting ultraviolet ray. UV interceptors can be classified as organic UV absorbers and inorganic UV interceptors (*Table* 1). Inorganic UV dispersers are composed of metal oxide. A UV intercepting mechanism made up as ultrafine particles of metal oxide acts in complex condition of absorption and dispersion, is called a general interceptor. This UV interceptor has a wide intercepting range of ultraviolet rays and is widely applied to various products. Today, the main characteristics of UV interceptors of UV-B and UV-A are as follows:

Table 1. Ingredients of major sunscreen used in Korea

Ingredients	λ_{max} (nm)	Max (conc.%)	Trade name
UV-B abosrbers			
Octylmethoxy cinnamate	311	7.5	Parsol MCX
(DEA)-Methoxy cinnamate	290	8.0	Bernel Hydro
Octyldimethyl-PABA	311	9.0	Eusolex 6007
2-Phenylbenzimidazole-5-sulphonic acid	310	4.0	Eusolex 232
Homosalate	306	10.0	Kemester HMS
Cctocryene	303	10.0	Uvinul N-539
UV-A absorbers			
Butylmethoxydibenzoylmethane	343~365	5.0	Parsol 1789
Benzophenone-1	292~332	-	Uvinul 400
Benzophenone-2	345~354	-	Uvinul D-50
Benzophenone-3	295~330	2~6	Uvinul M-40
Benzophenone-4	289~332	5~10	Uvinul MS-40
Benzophenone-12	280~342	-	Uvinul 408
Physical pigments			
Titanium dioxide	320	2~25	Ultrafine TiO_2
Zinc oxide	380	2~25	Z-COTE HP-1
Ferrous oxide	280~	-	-
Talc 1623	280~	-	-

3.1.1. UV-B intercepting effect

UV-B causes acute inflammation or irritation of skin, increases melanin pigment, and becomes a cause of spots and freckles. A lot of products with high SPF value are appearing on the market.

UV intercepting mechanism of ultrafine TiO_2 is according to combining action of absorption and dispersion. It has been reported that the smaller the diameter of the particle, the higher absorption and the bigger diameter, the higher dispersion.[17] Ultraviolet TiO_2 has different reflective index according to its crystallization. A TiO_2 with small reflective index is now being developed.[18,19] It also has a high transparency to a visible ray. The case of getting loose in white on the skin is rare. The reflective index of TiO_2 is about 1.9. This value is a little higher than the reflective index of oil (1.5). When it is applied onto the skin, it shows a transparent color. It may cause raging fire, discoloration, condensation and division of combining ingredients. For these reasons, silica, alumina and silicone are used to treat with the surface and they contribute to dispersion and waterproofing factors.[20~24]

3.1.2. UV-A intercepting effect

UV-A is define as the range of 320~400 nm and corresponded about 90% of ultraviolet rays which reach the surface of the earth. Though the effect of UV-A is less than that of UV-B, UV-A reaches down to deep skin (*Fig.* 2) to the inner skin and cause a chronic skin trouble to lead skin aging. On the other hand, UV-B is absorbed or dispered only on the outer skin. Main effects of UV-A on skin is

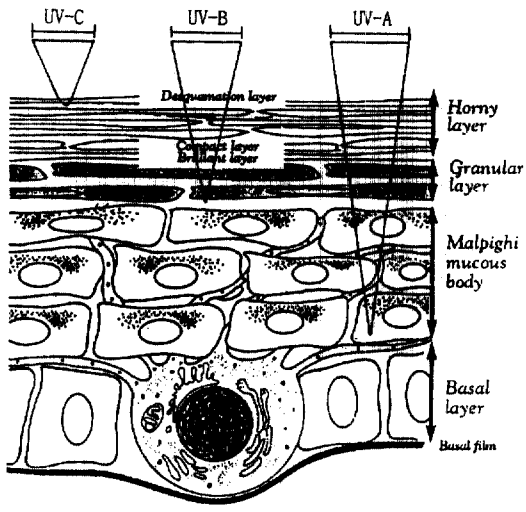

Fig. 2. Schematic diagram of UV transmittance on the skin.

as follows:

(a) changed immediately melanin by the temporal oxide of melanin,

(b) causes delay-type melanin though it is weak to cause inflammation or irritation.

(c) increases of a bad effect of UV-B.

(d) increases of pigment formation.

(e) reaches the inner skin and changes the fiber of the inner skin.

(f) causes aging by promoting wrinkle formation.

The cytotoxicity of UV-A are now in the course of research by many researchers. Established UV interceptors are mainly benzophenone or BMDM, but discoloration of those materials are a problem. Therefore, ZnO is widely used as an up to date UV-A interceptor and an inorganic system UV interceptor.

Recently, ZnO is one of the noticed materials because of its high safety and excellent ray stability. UV absorber has the largest absorption wave length around 320 nm and can cover relatively large ranges from UV-B and UV-A. Comparing with ultrafine TiO_2, ZnO has no cloudiness in high concentration and sensuous property.

3.2. Absorption spectrum of UV interceptor

We measured UV absorption spectrum of organic UV interceptors which is widely used nowadays (*Table* 1). UV was measured within the range of 200~400 nm (*Fig.* 3a). UV-B interceptors showed the absorption maximum curve in the range of 280~320 nm and UV-A interceptors showed its the absorption maximum curve in the range of 280~360 nm (*Fig.* 3b). For absorption wave length of interceptors ZnO (particle size: 0.03 μm) had a high extinction from 280 to 400 nm and TiO_2 (particle size: 0.03 μm) showed the largest absorption curves at 318 nm and 382 nm (*Fig.* 3c).

Ferrous oxide and Talc are regarded as a materials having UV intercepting effect because they showed a absorption curve in the range of UV.

3.3. The stability of organic UV interceptor

We observed UV interceptors in *Table* 1. after making the concentration of each sample to 3% in

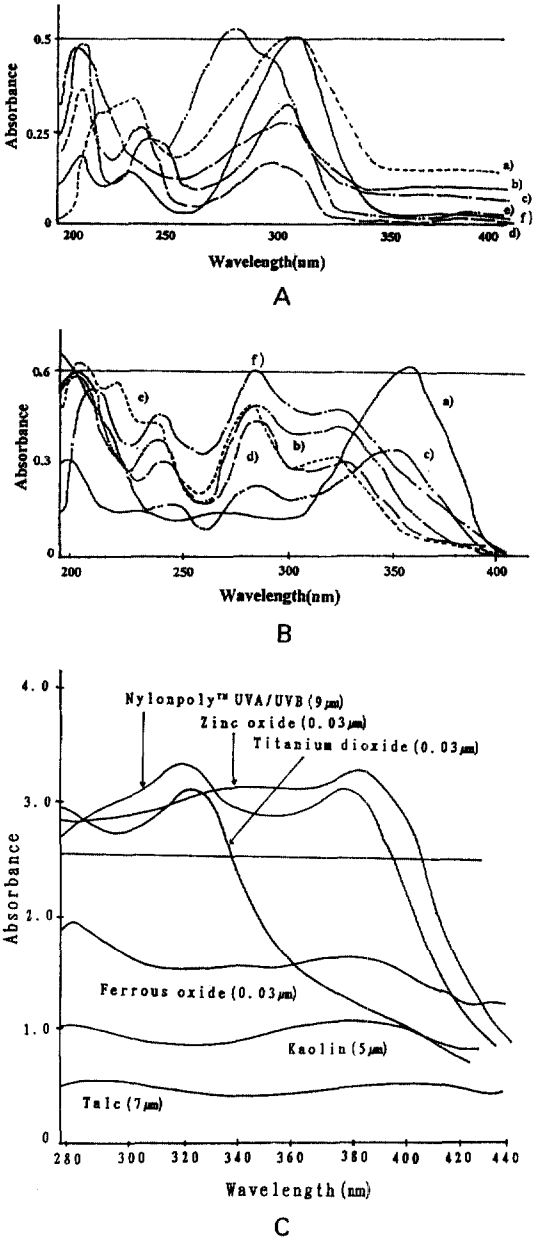

Fig. 3A. Manifold UV-B zone spectra in chemicals. a) OMC (5.12 ppm), b) DEA-methoxycinnamate (5.35 ppm), c) Octyldimethyl-PABA (5.20 ppm), d) 2-phenylbenzimidazole-5-sulphonic acid (5.66 ppm), e) Homosalate (5.45 ppm), f) Octocrylene (5.10 ppm). B. Manifold UV-A zone spectra in chemicals. a) BMDM (5.18 ppm), b) Benzophenone-1 (5.10 ppm), c) Benzophenone-2 (5.10 ppm), d) Benzophenone-3 (5.12 pm), e) Benzophenone-4 (5.18 ppm) f) Benzophenone-5 (5.12 ppm). C. Manifold of UV-B, A zone spectra for UV scattering pigments.

mineral oil as solvent for 2 weeks at sunlight. As a result of the observation, all materials of UV-B became discolored, but among them, OMC and BMDM were the least discolored.

3.4. Skin irritation test of UV agents

The experiment on skin irritation (*in-vitro*) was performed in the method of section 2.2. Survival ratio (%) of a cell was measured by increasing the concentration as 0.001-0.8 w/v%. As a result of the experiment an UV-B interceptor starts reducing its survival ratio above 0.08 w/v% (*Fig.* 4). As a result of the experiment, an UV-A interceptor starts reducing its survival ratio above 0.06 w/v% (*Fig.* 5). Nylonpoly™ UVA/UVB showed 88% of survival ratio even up to 0.8 w/v%. With the result, we could find that Nylonpoly™ UVA/UVB (*Fig.* 6) is safer than organic UV interceptors in the respect of skin irritation. The skin safety of ZnO and TiO_2 can be proved with a general material, but in this experiment, used without experiment because of the difficulty combining a culture medium. Also, the results of patch-test of inorganic UV interceptors exhibited no skin irritation even below 10 and 11.25 (*Table* 2).

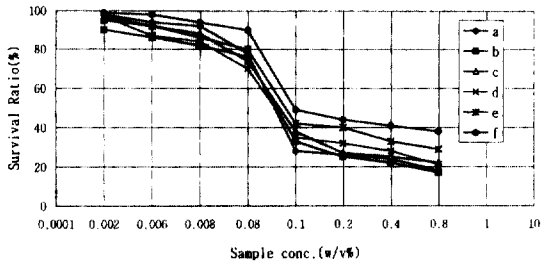

Fig. 4. Cytotoxicity measurement of UV-B agents; a) Octocrylene, b) Octyldimethyl PABA, c) 2-PBISA, d) DEA-methoxycinnamate, e) Homosalate, f) OMC.

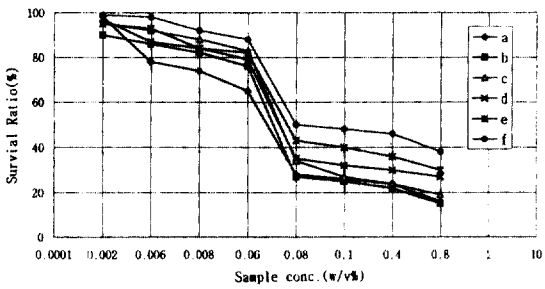

Fig. 5. Cytotoxicity measurement of UV-A agents; a) B-1, b) B-12, c) B-2, d) B-3, e_ B-4, f) BMDM.

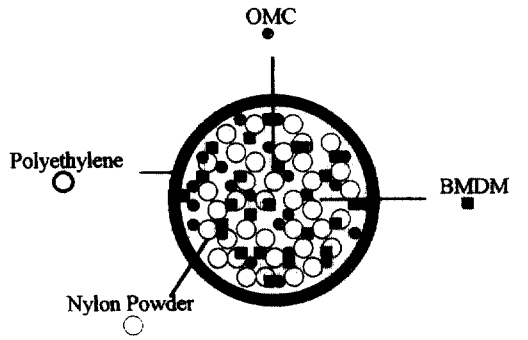

Fig. 6. Schematic diagram of a Nylonpoly™ UVA/UVB.

3.5. Mixing condition of ZnO and TiO_2

We measured SPF by changing mixing ratio to blank, 10 : 0, 0 : 10, 5 : 5 and 6 : 4 based on total 10% of ZnO and TiO_2. When ZnO : TiO_2 was mixed in 5 : 5, the SPF value was 16.5, and when it was changed to 6 : 4, the SPF value was 19.8 (*Fig.* 7). The ratio of ZnO and TiO_2, therefore, was decided as 6 : 4 in this experiment. It is considered that the reason is a rising effect of tow raw materials, because ZnO shows extensive UV absorption in the range of 200~400 nm.

3.6. SPF *in-vitro* measurement

Up to now, we measured SPF as *in-vitro* after mixed ZnO, TiO_2 and Nylonpoly™ UVA/UVB properly in consideration of the stability and safety of UV interceptors. W/S type sunscreen cream was made by mixing 6% of ZnO and 4% of TiO_2 and 5% of Nylonpoly™ UVA/UVB. And its SPF was 38.9 (*Fig.* 8).

3.7. Water-in-silicone (W/S) emulsion system

Water-in-oil system has various defects such as heavy feel touch and oily. To solve the problems, Silicone emulsion system with a square or pentagonal ring is widely used and cyclomethicone and dimethicone are also generally used.

Cyclomethicone is light oil with low volatilization and has a good common use with other solvents. For its emulsion, a surface active agent with a sil-

Table 2. Cytotoxicity measurement by patch-test

Samples	Result of evaluation (No. of subjects)					Average skin irritation range (%)	Remarks
	+++	++	+	±	-		
UV-B sunscreen agents							
Octylmethoxycinnamate	-	1	2	7	9	18.75	Parsol MCX
DEA-methoxycinnamate	-	1	3	8	8	21.25	Bernel Hydro
Octyldimethyl PABA	-	1	5	7	7	25.00	Eusolex 6007
2-phenylbenzimidazol-5-sulphonic acid	1	1	4	6	8	26.25	Eusolex 232
Homosalate	-	2	3	10	5	27.50	Kemester HMS
Octocrylene	-	1	6	4	9	23.75	Uvinul N539
UV-A sunscreen agents							
Butyl methoxydibenzoylmethane	-	1	3	8	8	21.25	Parsol 1789
Benzophenone-1	2	3	4	5	6	37.50	Uvinul-400
Benzophenone-2	-	2	6	8	4	32.50	Uvinul-D50
Benzophenone-3	-	1	5	9	5	27.50	Uvinul-M40
Benzophenone-4	1	1	3	8	7	26.25	Uvinul-MS40
Benzophenone-12	2	1	4	9	4	35.00	Uvinul-408
Pigments							
Titanium dioxide (TiO_2)	-	-	2	4	14	10.00	UV-Titane M160
Zincoxide (ZnO)	-		2	5	13	11.25	

$$\text{Average skin irritation range} = \frac{\sum(\text{Results of evaluation} \times \text{Standard numbers of person})}{\sum(\text{total persons} \times \text{numbers of sincere marked irritation})} \times 100$$

#. Standard of evaluation

Irritation range		Values of standard valuation	Decision of skin irritation	Range
Sincere marked irritation	+++	2.0	Sincere marked irritation	above 30
Marked irritation	++	1.5	Moderate marked irritation	20~29
Moderate marked irritation	+	1.0	Slight marked irritation	10~19
Slight marked irritation	±	0.5	No irritation	0~9
No irritation	-	0		

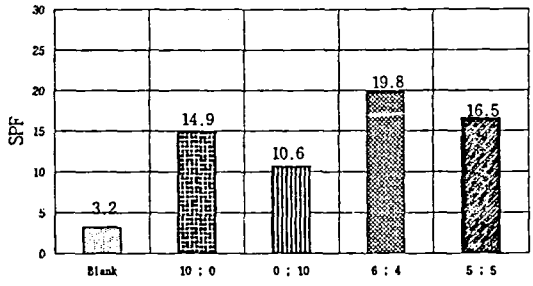

Fig. 7. Optimum mixture ratio of ZnO and TiO_2 for the sunscreen cream (SPF value).

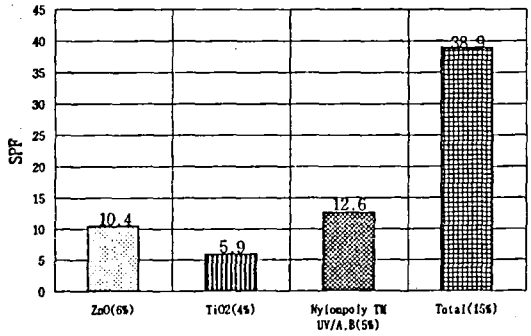

Fig. 8. SPF value of ZnO : TiO_2 : Nylonpoly^{TM} UV-A, B for W/S type sunscreen cream.

icone ring is usually used.

The emulsifiers used in W/S were cetyldimethicone cocpolyol and sorbitan sesquioleate and as stabilizer, $MgSO_4 \cdot 7H_2O$, Mg-stearate, Mg-Al-stearate copolymer were used. The durability of SPF was measured with W/S (*Table* 3) type products and O/W-type (*Table* 4). In a general O/W type sunscreen cream, its decreased to 6.5 in 20

Table 3. Formula of water-in-silicone emulsion system for sunscreen cream

Phase	Ingredients	Contents (%)	Remarks
Oil phase	A) Dimethicone (6cs)	7.0	Emollient
	Cyclomethicone	8.0	Emollient
	Al-Mg silicate copolyol	3.0	Stabilizer
	Octyl dodecanol	5.0	Emollient
	Mkagnesium stearate	0.5	Stabilizer (W/O)
	Zinc stearate	0.3	Stabilizer (W/O)
	Cetyl dimethicone copolyol	1.5	W/O Emulsifier
	Sorbitan sesquioleate	0.5	W/O Emulsifier
	Butylhydroxy toluene	0.01	
	Propyl paraben	0.15	Preservatives
	Tocopheryl acetate	0.4	
Water phase	B) Xanthan gum (2% soln.)	5.0	Thickener
	Glycerin	3.0	Moisturizer
	1,3-Butylene glycol	2.0	Moisturizer
	Methyl paraben	0.2	Preservatives
	$MgSO_4 \cdot 7H_2O$	0.5	Stabilizer
	Deionized water	Q.S	
UV base	C) Zinc oxide	6.0	UV screen agent
	Titanium dioxide	4.0	UV screen agent
	Nylonpoly™ UVA/UVB	5.0	
	D) Perfume	0.2	

Preparation in the laboratory: 1) melt components of water phase (A) at 80~82℃ and mix intimately. 2) Heat components of oil phase (B) to 82~84℃ and add to water phase (A) and UV base (C) while stirring. Stir for 15 minutes at this temperature (H/M speed: 3,000 rpm). 3) start cooling and continue mixing with propeller stirrer speed 15 rpm. 4) When temperature drops below 45℃ added additives (D) mix after each addition to get uniform mixture. 5) at 30℃ stop mixing, take sample for quality control and when approved, pack the product.

minutes after 1 application and in W/S type Sunscreen cream, its SPF value kept the same 25.7 after 100 minutes (*Fig.* 9).

For waterproof testing, 0.3 g of the sample cream was applied to the skin in an area of 30 cm^2. After that, the skin was immersed in water for 20 min. then stay out of the water for 20 min. This procedure was repeated three times and then the skin was irradiated.

The sunscreen cream prepared with water-in-silicone system was recognized higher SPF value than that of O/W system under both static and wat-

Table 4. Formula of oil-in-water emulsion system sunscreen cream

Type	Ingredients	Contents (%)	Remarks
Water phase	A) Magnesium silicate (10%soln.)	5.0	Thickener
	Xanthan gum (2%soln.)	5.0	Stabilizer
	TEA-stearate (10%soln.)	8.0	Consistency agent
	EDTA-2Na	0.02	Chelating agent
	Glycerin	3.0	Moisturizer
	1,3-Butylene glycol	2.0	Moisturizer
	Methyl paraben	0.2	Preservatives
	Deionized water	Q.S	
Oil phase	B) Dimethicone (6cs)	7.0	Emollient
	Cyclomethicone	8.0	Emollient
	Al-Mg silicate copolyol	3.0	Stabilizer
	Octyl dodecanol	5.0	Emollient
	Magnesium stearate	0.5	Stabilizer
	Zinc stearate	0.3	Stabilizer
	Glyceryl stearate PEG-100 stearate	1.5	O/W Emulsifier
	Polysorbate-60	1.0	O/W Emulsifier
	Butylhydroxy toluene	0.01	
	Propyl paraben	0.15	Preservatives
	Tocopheryl acetate	0.4	
UV base	C) Zinc oxide	6.0	UV screen agent
	Titanium dioxide	4.0	"
	Nylonpoly™ UVA/UVB	5.0	"
ad-ditives	D) Polyacrylamide/ C13-14isoparaffin/ Laureth-7 (SEFIGEL #305)	0.3	Thickening agent
	Perfume	0.2	

Preparation in the laboratory: 1) mix ingredients of oil phase (A) and start heating up (70~72℃) to melt oil phase. 2) mix ingredients of water phase (B) and start heating up (72~74℃). Start mixing oil phase at 1500~2000 rpm while adding water phase slowly. depending on sample size, the addition of water phase should be 10~15 min. 3) homogenize, start cooling and continue mixing with propeller stirrer speed 15 rpm. 4) when temperature drops below 45℃ added additives (D) mix after each addition to get uniform mixture. 5) at 30℃ stop mixing, take sample for quality control and when approved, pack the product.

erproof testing.

4. CONCLUSION

This study was performed to find least irritating

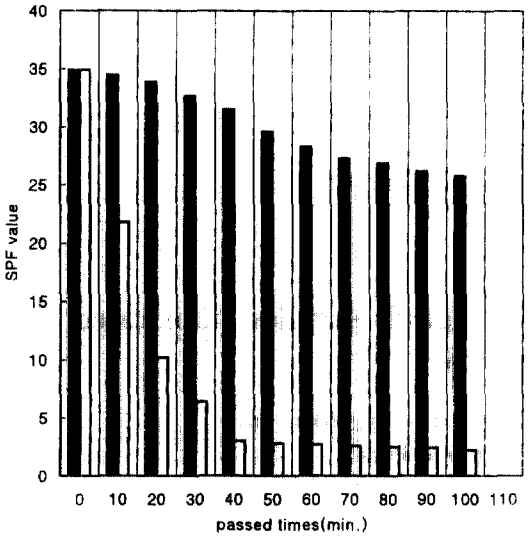

Fig. 9. SPF of water-in-silicone system and oil-in-water system by pased times.

the raw materials to skin by testing the skin toxicity, safety and the stability of a synthetic UV interceptor and pigments. Nowadays, because skin irritation of an organic UV interceptor is on the rise, this study have tired to indirectly contact skin by coating the synthetic raw materials.

1. In testing cytotoxicity (*in-vitro*) in NR method, cell survival ratio of UV-B interceptors decreased above 0.08 w/v%, and so did that of UV-A interceptors above 0.06 w/v%. Also, In addition patch-tests of inorganic UV interceptors resulted in no skin irritation even below 10.0 and 11.25.

2. Zinc oxide and titanium dioxide were used as inorganic UV interceptors. The appropriate mixture ratio of ZnO and TiO_2 was 6 to 4. 6% of ZnO, 4% of TiO_2 and 5% of Nylonpoly™ UVA/UVB were all combined at our sunscreen cream. The SPF value of *in-vitro* was 38.9.

3. In practical application, each SPF duration of O/W type and W/S type containing sunscreen cream of the same content showed that W/S type of sunscreen cream was 5 times as durable as the other. This product is fit in swimming, climbing or skiing. This research is to minimize skin trouble caused by UV interceptors and to make one with proper softness, skin safety and UV intercepting efficiency.

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