Evaluation of Sunscreen Photostability Under Real-Time Irradiation Conditions

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Introduction

Accelerated irradiation conditions are widely used in the industry for *in vivo* SPF testing and the evaluation of sunscreen photostability *in vitro*. The MED in such testing is typically reached within 10-20 sec. In contrast, the MED under real sunlight is 10-20 min for fair-skinned individuals [1, 2], depending on geographical location and season. The emission of the accelerated test source differs from that of solar radiation in the UV region because it has more short UVB radiation than is present in sunlight and has less than half of the UVA-1 (340-400 nm) intensity available in sunlight [1]. At high intensities of light, the quantum yield of degradation, i.e., the amount of degradation per photon absorbed will often be less than at low intensities [3]. Sunscreens on skin are subjected to sunlight, elevated temperatures and certain humidity levels. The photostability of a sunscreen that is determined on the basis of irradiance levels only does not take into account these effects, which are present in real-life environment and can contribute to the photodegradation of sunscreen actives. Florida climate provides increased levels of three critical weathering variables simultaneously: higher solar radiant exposure, increased temperatures and more moisture. Arizona climate has higher UV, temperatures and lower humidity [3].

Method Description

The proposed *in vitro* method for evaluation of sunscreen photostability utilizes real-time exposure, solar irradiation, relative humidity and temperature similar to the conditions that are relevant to sunscreen users, for example Florida, Arizona, or other regions. It employs a Ci65A Xenon Weather-O-Meter, VITRO-SKIN® N-19 (IMS, Inc.) [4] or PMMA (DIN 8201-5) as the substrates and a transmittance analyzer with integrating sphere (PerkinElmer Lambda 35 with RSA-PE-20, Optometrics SPF-290S or Labsphere UV-1000S). When VITRO-SKIN was used, it was pre-cut into 4x4 cm pieces and hydrated according to the protocol described in [4]. A piece of hydrated substrate was mounted in a glassless slide, air-dried for 15 min and used as a reference. Test product was applied on the substrate according to [4], placed in a glassless slide mount and air-dried for 15 min. An application dose was 2 mg/sq. cm. Each product was tested at five repetitions. Initial absorbance spectra of tested sunscreens in UV region were obtained. The slides with references and test products were then placed in the sample holders and positioned in the Ci65A Xenon Weather-O-Meter. Irradiation time, humidity level and temperature were adjusted to resemble specific end-use conditions. After irradiation, the absorbance spectra were measured again and comparisons were made with the initial data.

Experimental Results

This method was used in the evaluation of numerous commercial and experimental sunscreen formulations and also for their optimization. For example, commercially available sunscreen SPF 45 (actives: 2% avobenzone, 15% homosalate, 5% octisalate, 7.5% octinoxate and 6% oxybenzone) and experimental formulation SPF 28 *in vivo* (actives: bis-ethylhexyloxyphenol methoxyphenol triazine and methylene bis-benzotriazolyl tetramethylbutylphenol) were evaluated at the following conditions: 1.1 W/m2 at 420 nm, which corresponds to approximately 50 W/m^2 at 300-400 nm and is close to an average irradiation in Florida; humidity level 65%; black panel temperature 60 °C; irradiation time 2 hrs. The results are presented in Fig. 1 and 2, respectively. We have also evaluated the photostability of a commercial sunscreen SPF 15 containing 3% avobenzone, 2% octisalate and 7.5 % octinoxate under conditions described above and found that the average UVA PF decreased by more than 80%. Several prototypes were created and tested. Prototype A contained 3% avobenzone, 2% octisalate and 7.5 % octinoxate; Prototype B contained the same actives as in A plus 2% bis-ethylhexyloxyphenol methoxyphenol triazine; Prototype C contained the same actives as in A plus 2% methylene bis-benzotriazolyl tetramethylbutylphenol (based on active level); and Prototype D contained the same actives as in A plus 1.5% methylene bis-benzotriazolyl tetramethylbutylphenol (based on active level) and 1.5 % bis-ethylhexyloxyphenol methoxyphenol triazine. Prototype A was not photostable. In contrast, Prototypes B, C and D were photostable, and a significant boost in SPF/PFA *in vitro* was achieved in these prototypes. Thus, we have found that methylene bis-benzotriazolyl tetramethylbutylphenol, bis-ethylhexyloxyphenol methoxyphenol triazine, or their combination effectively photostabilize avobenzone and our finding regarding prototype B are consistent with data presented in [5].

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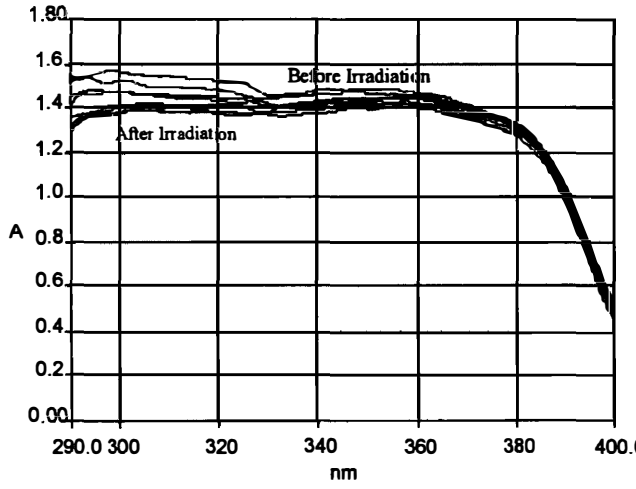
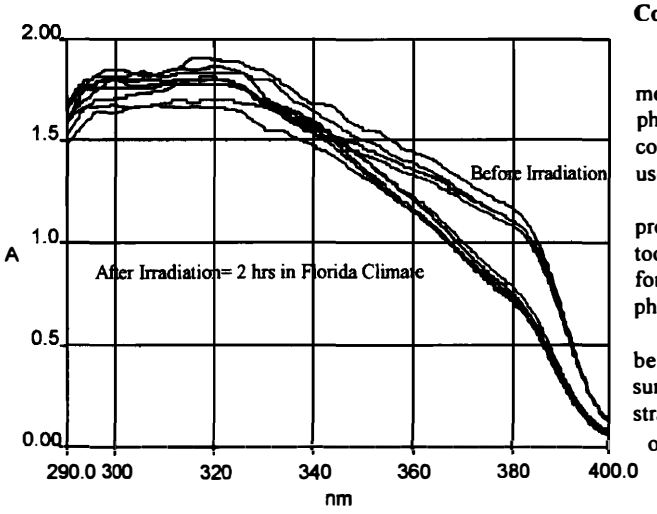


It was found that SPF 28 sunscreen has demonstrated excellent photostability. SPF 45 product was not photostable after just 2 hrs of real-time exposure or about 6 MED, which was a somewhat surprising discovery for a high SPF product. At the same time, the findings for SPF 45 are in agreement with Robert M. Sayre *et. al* data obtained in the specially designed set-up that spectrally resembled sunlight. In this study several SPF 15 and 30 products were evaluated, and the majority of loss of protection occurred by 2 -3 MED exposure for all products containing avobenzone [1].

Figure 1. Change in UV absorbance spectra of SPF 28 product with UV exposure



Conclusion

We have now developed an *in vitro* method for the evaluation of sunscreen photostability under real-time conditions that closely resemble an end-use outdoor environment.

The technique can be utilized as a product development and optimization tool to distinguish photostable formulations from those that are photolabile.

The results of such evaluations can be effectively communicated to the sunscreen consumer and can provide straightforward marketing claims based on familiar test conditions.

Figure 2. Change in UV spectra of SPF 45 product with UV exposure

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Acknowledgements

We are very grateful for the support of our colleagues at Ciba Specialty Chemicals, Inc., especially Dr. Joseph Lupia, Myriam Sohn, Shiela Loggins and Emily Bazemore. We would like to thank Dr. John Sottery and Peter Sottery of IMS, Inc. for valuable discussions.

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