

## Cosmetic application in the development of an *in vitro* methodology for sunscreens analysis using mass spectrometry

Diogo N. de Oliveira (PG), Jeany Delafiori (IC), Mônica S. Ferreira (PG), Rodrigo R. Catharino (PQ)

### Abstract

Sunscreens are cosmetic formulations that present Ultraviolet (UV) filters, molecules that alone or in combination absorb, reflect or diffuse UV radiation, protecting the skin from harmful effects. Some filters may present low stability, raising questions about the quality, safety and efficacy of these products. Regulations enforce the conduction of tests that confirm labeled values of Sun Protection Factor (SPF) by *in vivo* testing using human subjects, and the UVA (Ultraviolet A) factor. Our study proposes the development of an *in vitro* methodology based on the analytical trend associating mass spectrometry (MALDI-MS Imaging) with a more direct and sample preparation-free approach, a novelty in the analysis of complex cosmetic matrices: Cosmetomics. Using this platform, our study was based on fingerprint analyses that allowed the evaluation of product quality through chemical stability, such as the assessment of changes in the chemical composition of the formulations through exposition time, the degradation profile of bemotrizinol ( $[M+H]^+$   $m/z$  628) and its relation with the label-declared SPF by qualitative and relative quantification method. The strategy provides interesting information on photostability, chemically proves that photoprotective effectiveness and photocleaved species do coexist in sunscreen formulations exposed to UV radiation and corroborates to a method that links UV filters concentration to SPF values. This technique shows viability to be part of quality control analyses as a new tool in the development of new molecules and sunscreens formulations.

*Keywords:* Sunscreen, Cosmetomics, Bemotrizinol

### Introduction

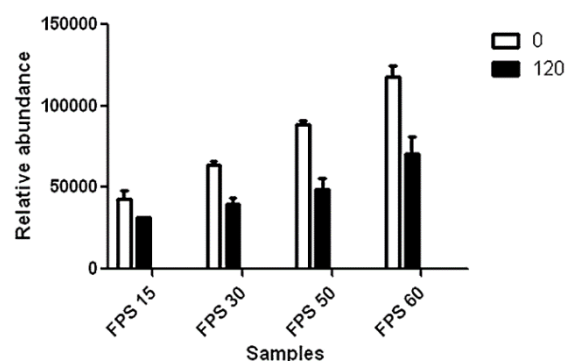
*In vitro* tests for UVB analysis have been developed, but are not yet accepted in any official resolution. With new trends on mass spectrometry (MS) applied to the assessment of cosmetic matrices<sup>1</sup>, this work aims to report an MS-based method, for fast *in vitro* sunscreen stability evaluation and SPF determination.

### Results and Discussion

The experiment was performed by exposing commercial sunscreens to UV radiation followed by direct analysis by MALDI-MS (matrix-free approach)<sup>2</sup>.

Using principal component analysis (PCA), significant differentiation of each SPF throughout time ( $t = 0, 30, 60$  and  $120$ ) was observed (except for SPF 15), suggesting chemical changes due to degradation markers ( $m/z$  516 and 404), which were identified as photocleaved products of the parent ion at  $m/z$  628.

Comparison of different SPFs grouped by time of exposure ( $t=0$  and  $t=120$ ), showed a more effective separation, explained by either the formation of degradation products, or, in a quantitative approach, the measuring of relative intensity of marker with the increase of SPF (Image 1). In  $t=0$  all SPFs were significantly different (One-way ANOVA), corroborating that SPF is directly linked to the concentration of the UV filter in the formulation.



**Image 1.** Relative abundance of the  $[M+H]^+$  ion  $m/z$  628 (MS-imaging data), grouped by SPF versus exposure time ( $t=0$  and  $t=120$  min).

### Conclusions

The method established a correlation between UV filter concentration and SPF values, enabling the assessment in a fast, cost-effective and straightforward way. Also, it provided information on byproducts, a feature that is not possible with the current *in vitro* spectrophotometric methods.

### Acknowledgement

CNPq, CAPES, INCT and FAPESP (2014/00084-2, 2011/50400-0 and 2013/20066-6).

<sup>1</sup> De Oliveira, D. N.; de Bona; Ferreira, M. S.; Catharino, R. R. *Materials*. **2013**, *6*, 1000.

<sup>2</sup> De Oliveira, D. N.; Delafiori, J.; Ferreira, M. S.; Catharino, R. R. *J. Chromatogr. B*. **2015**, *988*, 13..