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A new in vitro screening method to assess water resistancy

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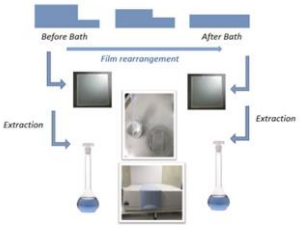
Introduction:

Recently, an increased number of methods for determining the ultraviolet (UV) protection factor have been proposed. But *in vitro* water resistance is yet not validated.
A previous studies demonstrated that the absorbance value of sunscreen depends on the uniformity/homogeneity of spreading on the test substrate [1]. This uniformity of spreading can be modified by different factors such as heat or ventilation [2-3], but also by the interaction with water which can modify the film homogeneity of the sunscreen. This is one reason why spectroscopic methods based only on UV transmission to measure the water-resistance *in vitro* can lead to reproducibility issues.

Here, is described a new method for determining the water-resistance *in vitro* of sunscreen products based on:
- The SPF *in vitro* determination as recommended by Cosmetics Europe for the sunscreen application and spreading [4-6]
- And on a previous publication for the immersion aspect and the absorption of sunscreens in dilute solution [7]

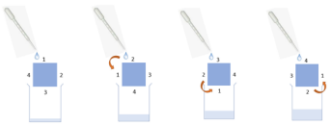


Materials & Methods:

This 4 steps protocol consists in studying the absorbance of sunscreens extracted from a product before and after rinsing to determine a water resistance index correlated to the *in vivo* ISO 18861 & ISO 16217 [8-9].

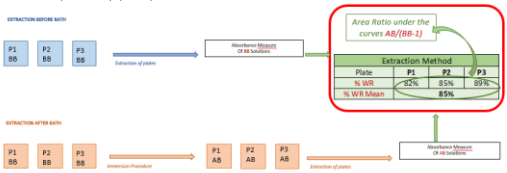
Step 1: Sunscreen application: Weigh 26,5 mg of studied sunscreen on SB6 PMMA plates and spread it thanks to a spreading protocol used to determine the SPF recommended by Cosmetics Europe. Spread 3 plates for the "Before Bath" (BB) measurement and 3 plates for the "After Bath" (AB) measurement.

Step 2: Plates rinsing: Rinse the 3 plates spread for the "After Bath" measurement with a pipette over its entire surface turning it methodically with 40mL of demineralized water then let dry it for 30 minutes in an oven at 30°C (figure 2).



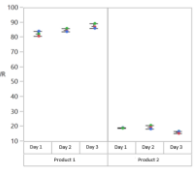
Step 3: Sunscreen filters extraction: Immerse each plate in a 250ml beaker using approximately 50mL of Isopronol and extract the filters from the sunscreen thanks to 15 minutes of ultrasound.

Step 4: Absorbance measurements of the extracted solutions: Blank with Isopropanol then analyze each solution thanks to a UV spectrophotometer, from 290 to 400nm using a 1mm cuvette. The area under the absorbance curve before bath (BB) and after bath (AB) are calculated.
The percentage of water resistance is then calculated using the following formula:
%WR = (ABx100)/(BB-1)



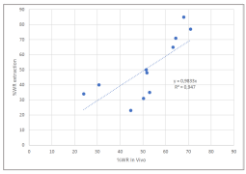
Results & Discussion:

The statistical analysis of the obtained results demonstrated that the method is repeatable and reproducible



The correlation with the results of the *in vivo* water-resistance determination (ISO methods) was satisfactory whatever the SPF level or the % of water-resistance of the product

SPF Claim	Base	%WR Vivo	%WR Extraction
30	O/W	53	35
50+	O/W	51,8	48
30	W/O	64,5	71
30	W/O	71	77
30	O/W	50,3	31
11,5	O/W	68	85
50	O/W	30,7	40
30	O/W	63,3	65
50+	O/W	24	34
30	O/W	51,5	50
30	O/W	44,7	23



The even spreading of the sunscreen onto the substrate is key to the performance of transmission spectroscopy on a thin film [10-13]. Until this is mastered, it is not possible to obtain reproducible intra or interlaboratory results. The rearrangement of the product can lead to a SPF value after bath over or under-evaluated depending on the nature of the product measured.

Simple regression analysis revealed a strong correlation between *in vitro* and *in vivo* values, with a r2=0.98, a slope of 0,947.
These results encourage us to test this new *in vitro* water resistance method on a larger scale by the industry in order to validate it on a larger number of products and laboratories.

Thanks to this new method, it is possible to obtain *in vitro* water resistance percentage values which are not biased by the possible rearrangements of the thin film since the product is dissolved in a solvent. These results give the actual *in vitro* percentages of water resistance affecting the products.

Conclusions:

A method for determining *in vitro* the water resistance percentage based solely on UV transmission is often inaccurate since it involves taking the absorbance measurement twice in the calculations - once before and once after immersion. The resulting ratio will therefore show significant variations.

The protocol was thus changed to involve a spectroscopic technique in dilute solution in order to avoid absorbance measurements of thin films.

The *in vitro* water resistance percentages obtained for the 11 products tested were then found to be reproducible and in line with the results obtained *in vivo*. This new *in vitro* water-resistance method seems to provide a more accurate water resistance percentage from the product tested, since they are not/less influenced by the redistribution of the thin film.

We concluded that this new technical approach seems promising and might become a good candidate for validation in a ring test,

Acknowledgements:

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