Synergy of mica and inorganic UV filters maximizes Blue Light Protection as first defense line

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Introduction – Blue light and its effects on skin

Protection strategies – First & Second defense line

Method to assess blue light protection as first defense line

Results & Interpretation

Conclusion & Outlook
Agenda

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02 Protection strategies – First & Second defense line

03 Method to assess blue light protection as first defense line

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Solar radiation spectrum (CIE 85)

Composition of sunlight*

- **5% Ultraviolet light**
  (100-400 nm)
- **50% Visible light**
  (400-700 nm)
- **25% Blue light**
  (400-500 nm)
- **45% Infrared light**
  (700 nm - 1 mm)

Penetration of radiation and impact in skin

- **UVB** (280-320 nm): sunburn, DNA dimer formation
- **UVA** (320-400 nm): oxidative reactions affecting DNA, proteins, lipids; immune suppression; pigmentation
- **VIS** (400-700 nm): oxidative stress; (hyper)pigmentation
- **IR-A** (700-1450 nm): oxidative damage, major impact on extracellular matrix integrity; heat damage

All solar radiations lead to formation of ROS and excess free radicals in skin and contribute to skin aging and wrinkling.

Source: SCENIHR, Health Effects of Artificial Light, 2012
Effect of *visible light/HEVL* on skin

**Radical formation**


Radical formation in human skin (ex vivo) – Determination by ESR in range 280 – 690 nm:

- **UVB**: 4 % of radicals
- **UVA**: 46 %
- **VIS**: 50 %

~ 35 % in HEV domain!
Effect of visible light on skin Pigmentation


- Pigmentation induced by VIS was darker and more sustained than with UVA1 in skin types IV–VI subjects (n=20, lower back)
- No pigmentation observed in skin type II, even with max. dose VIS (n=2)

Light sources:
- **UVA1**: 340–400 nm (99.7% UVA1, 0.12% UVA2, 0.17% VIS, < 0.00001% UVB)
- **VIS**: 400–700 nm (98.3% VIS, 0.19% UVA1, 1.5% IR)

Irradiation doses:
- UVA1: 1-60 J/cm² (20 J/cm² UVA ~ 1h sun exposure)
- VIS: 8-480 J/cm² (300 J/cm² VIS ~ 1h sun exposure)
Effect of visible light on skin
Radical formation – Photoaging


**VIS induced significant production of ROS, pro-inflammatory cytokines (IL-1α, IL-6, GM-CSF, IL-8) and MMP (MMP-1, MMP-9) in RHE**

• 180 J/cm² VIS had similar effects as 6 J/cm² UVA/B

**VIS induced significant free-radical production in vivo**
(n=40, forehead, Chemiluminescence)

Light sources:
• **UV**: 290–400 nm (nearly devoid of VIS and IR)
• **VIS**: 400–700 nm (98.3% VIS, 0.14% UVA, 2% IR)

Irradiation doses:
• UV: 6 J/cm² (~ 20 min. sun exposure)
• VIS: 40-240 J/cm² (~ 15-90 min. sun exposure in Texas)
Effect of high energy visible light on skin ROS formation


- Dose-dependent significant degradation of cutaneous carotenoids directly after blue/violet light irradiation (n=9, skin type II-III, forearms, resonance Raman spectroscopy)
- Can be explained by generation of free radicals including ROS due to the blue-violet light activation of mitochondrial activity.
- In correlation with previous results obtained for UV and NIR irradiation of skin in vivo.

Light source:
- Blue/violet light: max at 440 nm (80% in range 380–495 nm)

Irradiation doses:
- 50-100 J/cm² (57 J/cm² of blue/violet light ~ 1h sun exposure in southern Europe)
Effect of high energy visible light on skin
ROS formation

Y Nakashima et al. Blue light-induced oxidative stress in live skin.
Free Radical Biology and Medicine 108 (2017) 300–310

- Blue light induces the formation of ROS in human skin, most probably superoxide via flavin as the photosensitizer.
  (n=5, hands, decrease in autofluorescence of skin)

- Blue light (and UVA) induced mitochondrial oxidative stress in the skin of live mice.
  No oxidative stress induced by green, red, far red or infrared light.

Light source and irradiation dose (humans):
- Blue LED: max at 460 nm, 6.6 J/cm²

Light sources and irradiation doses (hairless mice):
- UVA LED: max at 365 nm, 2.6-26 J/cm²
- Blue LED: max at 460 nm, 2.6-26 J/cm²
- Green LED: max at 523 nm
- Red LED: max at 623 nm
- IR LED: max at 850 nm  up to 80 J/cm²
**Effect of high energy visible light on skin Pigmentation**

**L Duteil et al.** Differences in visible light-induced pigmentation according to wavelengths: a clinical and histological study in comparison with UVB exposure. *Pigment Cell Melanoma Res* (2014) 27, 822–826

- **Blue/violet irradiation** induced a dose-correlated hyperpigmentation in skin type III/IV subjects, significantly more pronounced and lasting than with UVB (n=12, back)

- **Red light** had no impact on pigmentation in vivo.

Light sources and irradiation doses:
- **UVB**: max at 306 nm, 1.5 MED (~ 25/45 min. sun exposure for skin type III/IV)
- **Blue LED**: 415 ±5 nm, 10-150 J/cm² (43.8 J/cm² 415 nm-light ~ 83 min. sun exposure)
- **Red LED**: 630 ±5 nm, 10-150 J/cm²
Effect of high energy visible light on skin Pigmentation – Pathway

C Regazzetti et al. Melanocytes Sense Blue Light and Regulate Pigmentation through Opsin-3.

• Blue light directly stimulates the melanogenesis pathway in melanocytes
  – Phototype-dependent: Up-regulation of TYR and DCT in dark NHMs (IV-VI); no impact on TYR activity in light NHMs (I-II)

• Blue light induced dose-dependent immediate pigment darkening ex vivo (skin IV)
  – Up-regulation of TYR and DCT, MITF increase

• Proposed mechanism: OPN3 functions as sensor for blue light in melanocytes and activates melanogenesis

Light source and irradiation doses (cells):
• Blue/violet LED: 415 ±5 nm, 50 J/cm²
• Blue LED: 465 ±5 nm, 62.5 J/cm²
  (~ 90 min. sun exposure in summer)

Light source and irradiation doses (ex vivo):
• Blue/violet LED: 415 ±5 nm, 5-90 J/cm²
Effect of visible light/ HEVL on skin

Summary of short literature review

• VIS (400-700 nm) **penetrates deeply** into the skin (dermis)

• VIS induces **free radicals and ROS** formation
  – Main impact from HEVL (400-500 nm)

• VIS and especially HEVL has a major impact on **skin tone**
  – VIS, especially HEVL can produce a darker and more sustained pigmentation than UV(A) in skin phototypes III and higher

• VIS/HEVL pathway for melanogenesis is specific
  – Direct impact on **melanocytes**

**Clear recommendation to extend sun protection measures into the VIS range**
– especially blue light/HEVL range
Introduction –
Blue light and its effects on skin

Protection strategies –
First & Second defense line

Method to assess blue light protection as first defense line

Results & Interpretation

Conclusion & Outlook
Light protection strategies
First & Second defense line

Shielding film with absorption, scattering, reflection properties. Radiations should not enter the skin.
PREVENTIVE action

Antioxidants help reduce the overall radical burden reaching skin.
RESPONSIVE action

Sunlight induces free radicals formation and oxidative stress in skin

Combine both lines of defense
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HEVL/ Blue light protection – First defense line

Concept

**AIM**
Reduce the amount of HEVL radiation reaching skin i.e. **reduce the transmission of HEVL** through a protective cosmetic formulation

**HOW?**
Passing of light through the formulation is reduced when light is reflected, scattered or absorbed by particulate matter within the formulation, e.g. inorganic materials like TiO$_2$ UV filters, functional fillers, etc.

**ASSESSMENT**
Measure the transmission rate (T) of HEVL through test emulsions with/without active materials with spectrophotometer – **The lower the transmission, the higher the protection!**
HEVL protection – First defense line
Absorbing/ Scattering/ Reflecting substances

Micronized titanium dioxide UV filters

Global approved UV-A/UV-B filters
Transparent on skin
Different coatings and particle sizes
3 – 10 % use level

**INCI:** Titanium Dioxide (nano), Alumina or Silica, (other)

Skin color correcting functional fillers

White pigments with **gold/ red/ blue/ green interference** in µm range
High chroma, low coverage, low luster for immediate skin color correction
3 - 5 % use level

**INCI:** Titanium dioxide, Mica, Tin oxide
HEVL protection – First defense line

Test emulsions

- 2 standard emulsion systems: o/w and w/si
- Base emulsions serving as placebo (without any scattering nor absorbing nor antioxidative ingredient) may already impact HEVL transmission level (T) – depending on film thickness
- To assess the real protection provided by the test materials in a specific formulation, the HEVL transmission should be compared to that of the placebo emulsion

Protection (%) =

\[1 - \frac{T(\text{emulsion with TiO}_2/\text{Filler})}{T(\text{base emulsion})}\] x 100

Example of % protection calculation:
- Placebo o/w: \(T = 55\%\)
- T-ASA (5 %): \(T = 38\%\); \(38/55 = 69\%\) of the transmission of basis emulsion

\[\boxed{31\% \text{ protection}}\]
HEVL protection – First defense line

Assessment methods

1 Spectroscopic measurements in short cut cuvettes

- **Device**: Lambda 900 UV-VIS-NIR spectrometer (Perkin Elmer)
- **Cuvette**: consisting of 2 glass plates forming a recess in the middle
- **Application**: sample applied with a syringe, defined film thickness of 0.1 mm
- **Baseline**: short cut cuvette filled with glycerol
- **Measurement of % transmission T (400-500 nm)**: 1 cuvette/ sample
- **HEVL transmission of placebo emulsions**: o/w: T = 55 ± 1%; w/si: T = 73 ± 2% (n=5)

**pro:**
- Fast
- Easy to implement

**contra:**
- Layer thickness ~ 5x topical application on skin
- Artefacts Up to 50 % contribution of emulsion chassis on HEVL transmission
HEVL protection – First defense line
Assessment methods

Spectroscopic measurements on PMMA plates

- **Device:** Cary 300 Bio UV-VIS spectrometer (Agilent) with integrated sphere
- **Plates:** PMMA molded plates (HD6, HelioScreen)
- **Application:** as described by ISO 24443 method (in vitro UVA-PF) with an amount of 1.3 mg/cm²
- **Baseline:** plate coated with glycerol (0.58 mg/cm²)
- **Measurement of % transmission T (400-500 nm):** 4 plates/s sample (1 point/plate)
- **HEVL transmission of placebo emulsions:** o/w: T = 92 ± 1% ; w/si: T = 90 ± 0% (n=4)

**pro:**
- Close-to-reality application conditions on skin
- Negligible influence of emulsion chassis on HEVL transmission

**contra:**
- Spreading procedure needs to be trained for reproducible measurements
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**Results – o/w emulsion**

**Transmission of HEVL through TiO₂ UV filters (3 – 10 %)**

- **Dose-dependent performance**
- **Coating plays a role**
- **Most efficient grades:**
  - **T-Si** (Silica coating): up to **44 % protection**
  - **T-AMn** (Al₂O₃/MnO₂ coating): up to **38 % protection**

![Normalized transmission [%] (PMMA plate)](chart)

<table>
<thead>
<tr>
<th>Placebo (o/w)</th>
<th>T-AS</th>
<th>T-ASA</th>
<th>T-AMn</th>
<th>T-Si</th>
<th>T-SiCP</th>
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<tr>
<td>0%</td>
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<td>83%</td>
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T (o/w placebo): 92 %
Results – w/si emulsion

Transmission of HEVL through TiO₂ UV filters (5 %)

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<tr>
<th>Normalized transmission [%]</th>
<th>Placebo (w/si)</th>
<th>T-AS</th>
<th>T-ASA</th>
<th>T-Si</th>
<th>T-SiCP</th>
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<tbody>
<tr>
<td>(PMMA plate)</td>
<td>100%</td>
<td>81%</td>
<td>77%</td>
<td>80%</td>
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</tbody>
</table>

- Low influence of coating
- Similar protection of ~20 % at 5 % use level

T (w/si placebo): 90 %
Results – o/w emulsion

Transmission of HEVL through functional fillers (3 – 5 %)

- Dose-dependent, however moderate effect
- Thickness of TiO₂ coating on the mica platelet plays a role: blue > red > gold
- Best is **FF-BI** (blue interference): up to **15 % reduction** of HEVL transmission

<table>
<thead>
<tr>
<th>Placebo (o/w)</th>
<th>FF-Go</th>
<th>FF-Re</th>
<th>FF-BI</th>
<th>FF-Gr</th>
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<tr>
<td>96%</td>
<td>94%</td>
<td>93%</td>
<td>91%</td>
<td>95%</td>
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</tbody>
</table>

Normalized transmission [%] (PMMA plate)

T (o/w placebo): 92 %
Results – Combination approach
Additive protective effects!

10 % T-AMn (Al₂O₃/MnO₂) + 5 % FF-BI (o/w emulsion)

5 % T-ASA (Al₂O₃/Stearic Acid) + 3 % FF-BI (o/w emulsion)

Normalized Transmisssion [%]
(PMMA plates)

Placebo (o/w)  
T-AMn  
FF-BI  
T-AMn + FF-BI

Normalized Transmisssion [%]
(PMMA plates)

Placebo (o/w)  
T-ASA  
FF-BI  
T-ASA + FF-BI

46 % protection
38 % protection
Results – Combination approach

Additive protective effects – Impact of different fillers (3 %)

- Addition of **3 % filler** improved protection given by **5 % T-AS** (Al₂O₃/ Simethicone coating): from **17 %** up to **24 – 28 %**

- Real additive effects: Measured values in line with expected calculated HEVL transmissions (or even slightly lower!)

> Such market relevant combinations maximize good HEVL protection provided by TiO₂ as first defense line.
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Summary & Conclusion

1. **Titanium dioxides** have been successfully used to protect against UV radiation since years.
   We proved in this study an excellent protection extended to HEVL.
   → Risk of radical formation in skin is minimized.

2. Combination with other type of inorganic materials – e.g. appropriate functional fillers – results in improved first defense against HEVL.

3. Main benefits of this smart combination: PROTECT and CORRECT skin
   - **TiO$_2$ UV filters:** UV + HEVL protection
   - **Functional Fillers:** Additional HEVL protection, skin tone correction, galenic/haptic aspects
Conclusion & Outlook

The blue light protection efficacy of cosmetic formulations can be assessed *in vitro* by means of transmission measurements on PMMA plates – simple & convenient method.

Is it possible to apply this method for the evaluation/quantification of the blue light protection level of market products?
HEVL protection – First defense line

Screening of market sunscreens with various SPF

- 15 sunscreens, SPF 15 to SPF 50+
- Mass-market/ middle-price/ premium segment
- Representative of EU market (top sellers)
- Market availability in Germany (spring 2018)

<table>
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<tr>
<th>UV filters</th>
<th>(1) SPF 15</th>
<th>(2) SPF 15</th>
<th>(3) SPF 20</th>
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HEVL protection – First defense line

Screening of market sunscreens with various SPF

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<th>HEVL transmission [%]</th>
<th>Glycerol (1) SPF15</th>
<th>(2) SPF15</th>
<th>(9) SPF30</th>
<th>(10) SPF30</th>
<th>(11) SPF30</th>
<th>(14) SPF50+</th>
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<tr>
<td>Blue Light Protection - First defense line</td>
<td>100,0</td>
<td>94,5</td>
<td>87,1</td>
<td>89,6</td>
<td>90,1</td>
<td>86,4</td>
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Organic UVF only
HEVL protection – First defense line

Screening of market sunscreens with various SPF

- HEVL transmission [%]

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<th>Glycerol</th>
<th>SPF15</th>
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- SPF15

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<td>85,5</td>
<td>89,6</td>
<td>90,1</td>
<td>86,4</td>
</tr>
</tbody>
</table>

- SPF30

<table>
<thead>
<tr>
<th>SPF50+</th>
<th>SPF50+</th>
</tr>
</thead>
<tbody>
<tr>
<td>72,6</td>
<td>86,0</td>
</tr>
</tbody>
</table>

Organic UVF only

VIS protection claim, no TiO₂

Blue Light Protection - First defense line
HEVL protection – First defense line

Screening of market sunscreens with various SPF

<table>
<thead>
<tr>
<th>Glycerol</th>
<th>SPF15</th>
<th>SPF15</th>
<th>SPF20</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF50</th>
<th>SPF50+</th>
<th>SPF50+</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPF15</td>
<td>100.0</td>
<td>94.5</td>
<td>87.1</td>
<td>75.2</td>
<td>69.9</td>
<td>72.5</td>
<td>80.3</td>
<td>85.5</td>
<td>89.6</td>
<td>90.1</td>
<td>86.4</td>
<td>76.2</td>
<td>86.0</td>
</tr>
</tbody>
</table>

- **Organic UVF only**
- **VIS protection claim, no TiO₂**
- **Contain TiO₂**
- **TiO₂ UVF only**
HEVL protection – First defense line
Performance of market sunscreens with various SPF

<table>
<thead>
<tr>
<th>Glycerol</th>
<th>SPF15</th>
<th>SPF15</th>
<th>SPF20</th>
<th>SPF20</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF30</th>
<th>SPF50</th>
<th>SPF50+</th>
<th>SPF50+</th>
</tr>
</thead>
<tbody>
<tr>
<td>HEVL transmission [%]</td>
<td>100.0</td>
<td>94.5</td>
<td>87.1</td>
<td>87.1</td>
<td>75.2</td>
<td>75.2</td>
<td>72.5</td>
<td>72.5</td>
<td>80.3</td>
<td>80.3</td>
<td>85.5</td>
<td>85.5</td>
<td>89.6</td>
<td>89.6</td>
<td>90.1</td>
<td>90.1</td>
</tr>
<tr>
<td>HEVL transmission [%]</td>
<td>100.0</td>
<td>94.5</td>
<td>87.1</td>
<td>87.1</td>
<td>75.2</td>
<td>75.2</td>
<td>72.5</td>
<td>72.5</td>
<td>80.3</td>
<td>80.3</td>
<td>85.5</td>
<td>85.5</td>
<td>89.6</td>
<td>89.6</td>
<td>90.1</td>
<td>90.1</td>
</tr>
</tbody>
</table>

**Proposed classification:**

- **MODERATE**
  - < 20 % protection
- **GOOD**
  - 20 – 50 % protection
- **EXCELLENT**
  - > 50 % protection

Sunscreens containing TiO₂ UV filters show good to excellent first defense from blue light.
Sarah Kögler, Application
Anett Moschner, Application
Michael Termier, Application
Alexander Kielbassa, Bus. Dev.
Silke Hornung, Global Marketing

Jutta zur Lage, Application
Dr. Lilia Heider, Global Marketing
Dr. Frank Pflücker, Bus. Development
Marina Lefort, Technical Marketing
## Test materials – INCI

### Broad spectrum inorganic UV-A/UV-B filters

<table>
<thead>
<tr>
<th>Code</th>
<th>INCI</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-AS</td>
<td>Titanium Dioxide (EU: nano), Alumina, Simethicone</td>
<td>Amphiphilic, versatile and high SPF efficient</td>
</tr>
<tr>
<td>T-ASA</td>
<td>Titanium Dioxide (EU: nano), Alumina, Stearic Acid</td>
<td>Hydrophobic, vegetable derived coating</td>
</tr>
<tr>
<td>T-AMn</td>
<td>Titanium Dioxide (EU: nano), Alumina, Manganese Dioxide</td>
<td>Amphiphilic, with anti-radical properties</td>
</tr>
<tr>
<td>T-Si</td>
<td>Titanium Dioxide (EU: nano), Silica</td>
<td>Hydrophilic, alumina free, increased compatibility and synergy with Avobenzone</td>
</tr>
<tr>
<td>T-SiCP</td>
<td>Titanium Dioxide (EU: nano), Silica, Cetyl Phosphate</td>
<td>Amphiphilic, best compatibility with challenging ingredients</td>
</tr>
</tbody>
</table>

### Skin color correcting functional fillers

<table>
<thead>
<tr>
<th>Code</th>
<th>INCI</th>
<th>Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>FF-Go</td>
<td>Titanium Dioxide, Mica, Tin oxide</td>
<td>White pigment, low coverage, with gold interference</td>
</tr>
<tr>
<td>FF-Re</td>
<td>Titanium Dioxide, Mica, Tin oxide</td>
<td>White pigment, low coverage, with red interference</td>
</tr>
<tr>
<td>FF-Bl</td>
<td>Titanium Dioxide, Mica, Tin oxide</td>
<td>White pigment, low coverage, with blue interference</td>
</tr>
<tr>
<td>FF-Gr</td>
<td>Titanium Dioxide, Mica, Tin oxide</td>
<td>White pigment, low coverage, with green interference</td>
</tr>
</tbody>
</table>
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