



Hyperspectral survey method to detect the titanium dioxide percentage in the coatings applied to the Cultural Heritage

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DIPARTIMENTO DI
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E SCIENZE DELLA TERRA
DIBEST

Motivation and strategy

The challenge is to provide a quick and non-invasive survey method able to evaluate the titanium dioxide amount in the coatings applied on Cultural Heritage. In fact, the titanium dioxide (TiO_2) weight percentage (w%) incorporate into the coating depends on both application phase and, over time, environmental biological and chemical conditions.

The specific objective provides the use of a field hyperspectral sensor, in order to assess influence of amount of TiO_2 on the spectral signature of material

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1. Method (sample preparation and acquisition of the spectral signatures)

2. Results and Discussion (data analysis and modeling)

3. Conclusions and future developments

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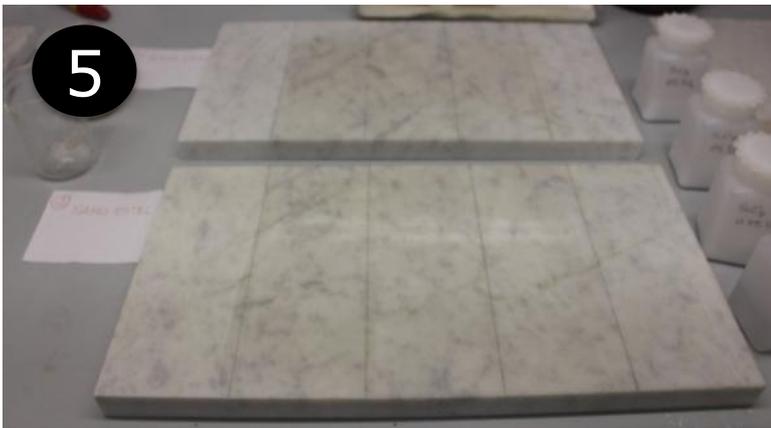
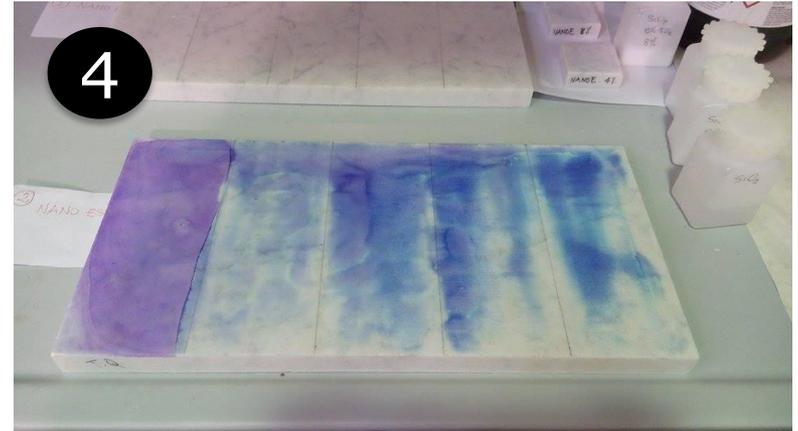
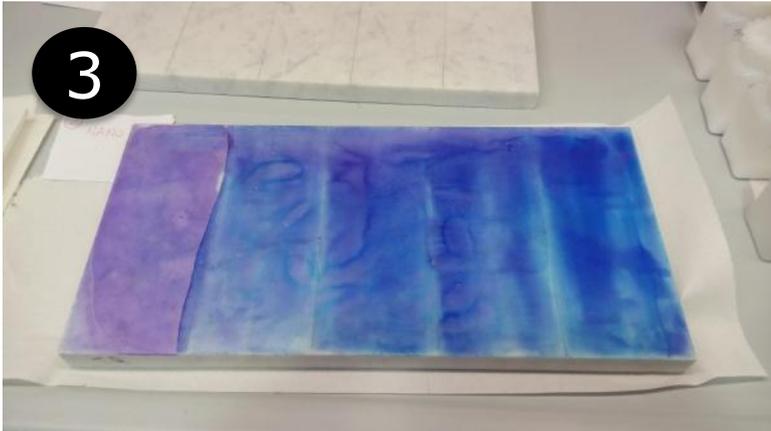
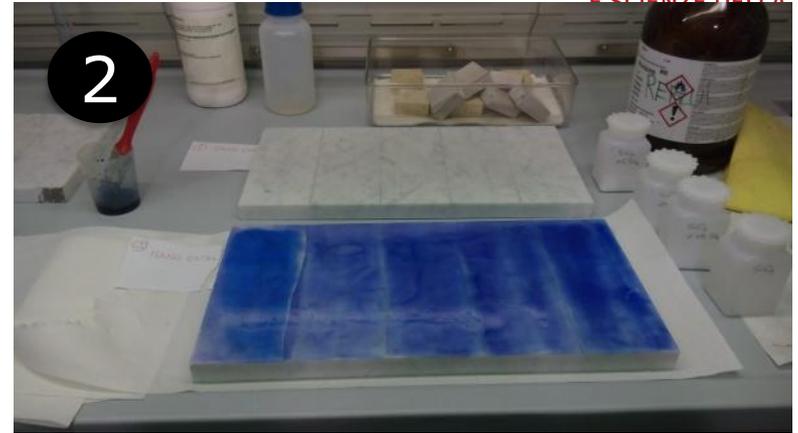
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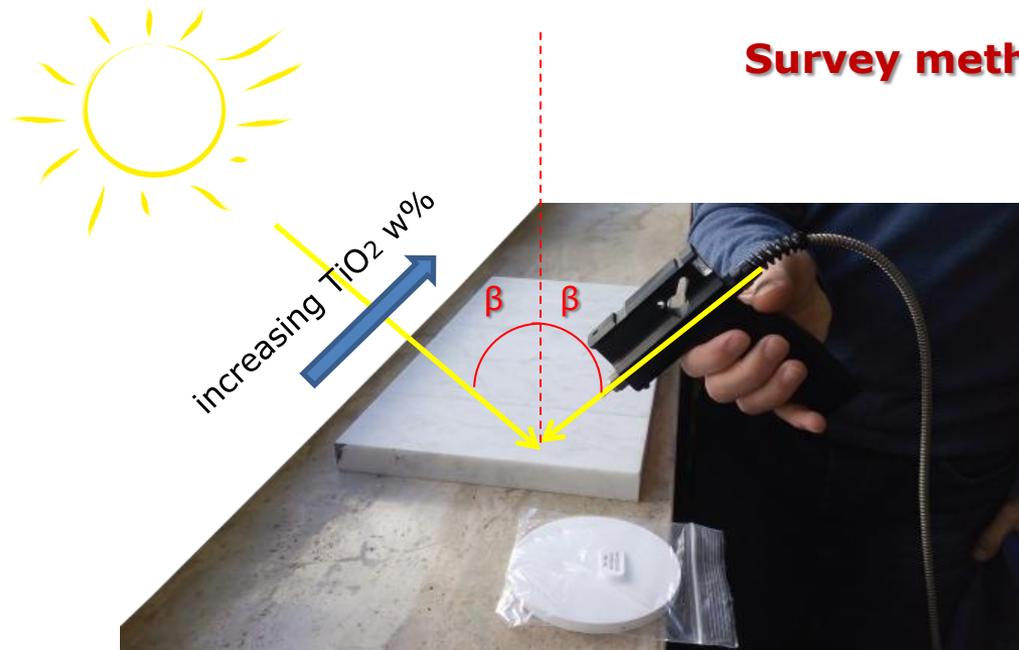
Preparation of the marble samples



- 1 Untreated marble
- 2 Methylene blue application
- 3 Drying phase
- 4 TiO_2 self-cleaning activity
- 5 Samples coated with NANOESTEL blended to increasing TiO_2 w%

FieldSpec 4 Field Spectroradiometer (ASD Inc.) – Technical characteristics

Spectral Range	350-2500 nm	
Spectral Resolution	3 nm @ 700 nm 10 nm @ 1400/2100 nm	
Spectral sampling (bandwidth)	1.4 nm @ 350-1000 nm 1.1 nm @ 1001-2500 nm	
Scanning Time	100 milliseconds	
Stray light specification	VNIR 0.02%, SWIR 1 & 2 0.01%	
Wavelength reproducibility	0.1 nm	
Wavelength accuracy	0.5 nm	
Maximum radiance	VNIR 2X Solar, SWIR 10X Solar	
Channels	2151	
Detectors	VNIR detector (350-1000 nm): 512 element silicon array SWIR 1 detector (1001-1800 nm): Graded Index InGaAs Photodiode, Two Stage TE Cooled SWIR 2 detector (1801-2500 nm): Graded Index InGaAs Photodiode, Two Stage TE Cooled	



Survey method

For each measurement 25 spectral signatures were collected. Furthermore, for each sample 10 measurements were performed, in order to take into account variability of the local conditions due to the coating application and the base stone.

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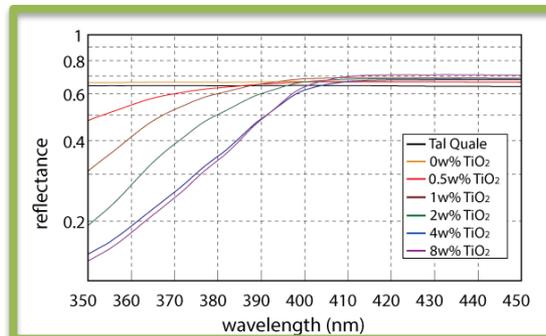
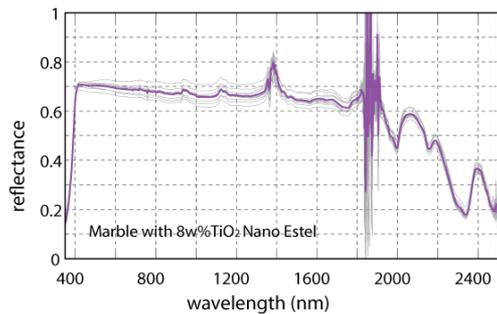
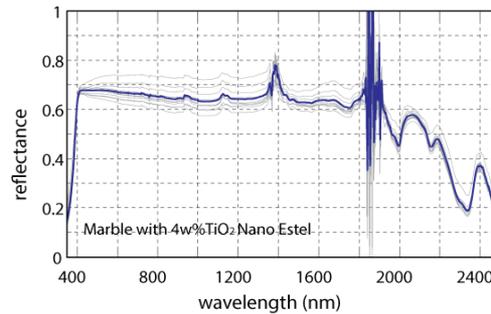
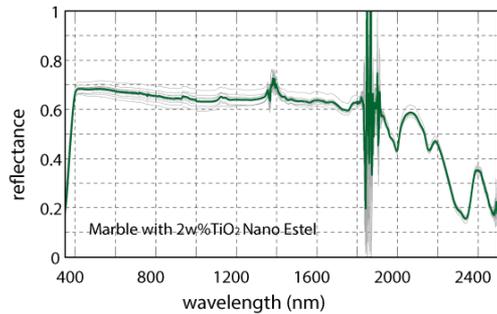
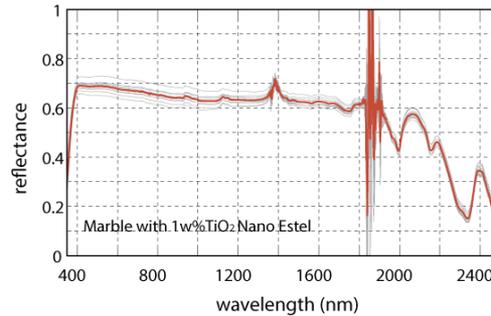
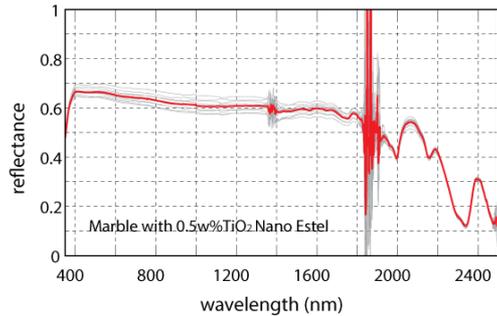
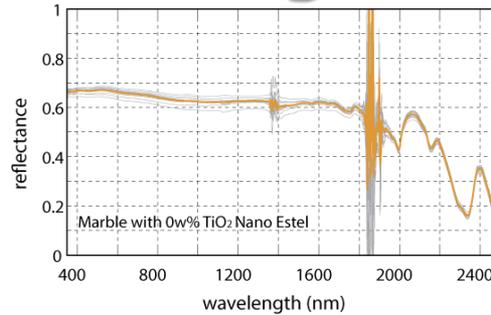
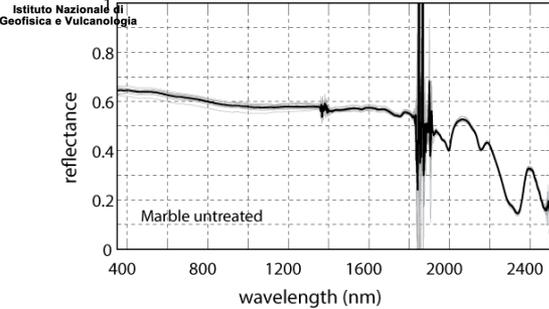
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Spectral signatures



Spectral signatures obtained on marble samples covered by NANOESTEL, whit increasing weight percentage of titanium dioxide.

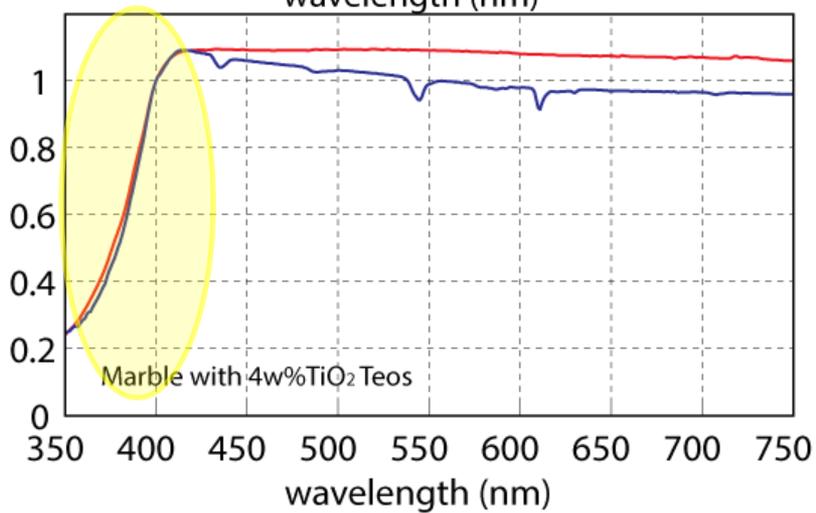
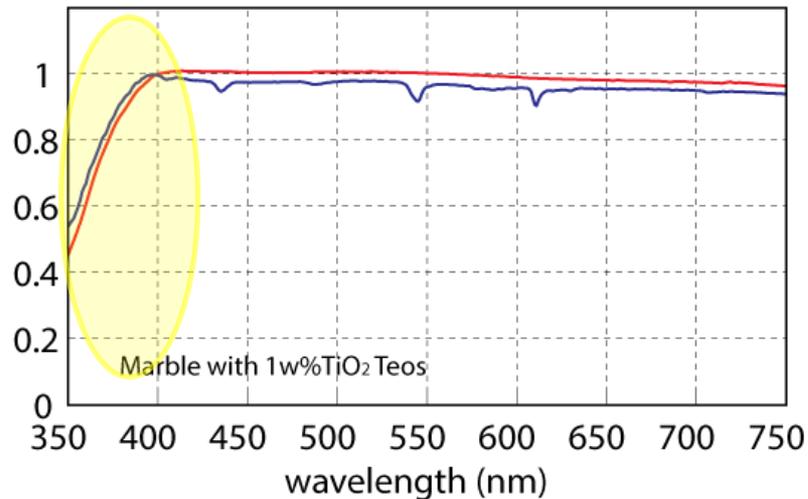
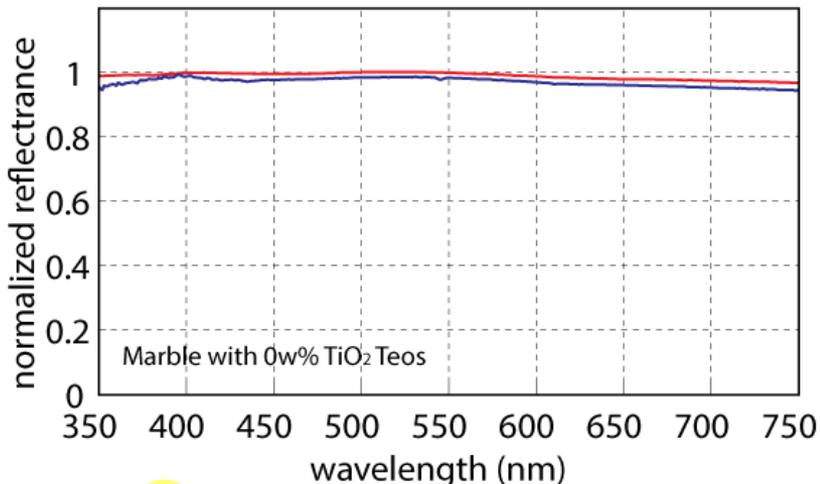
Gray curves are average values obtained by 25 acquisitions on the same measurement point, for each sample are selected 10 measurement points.

Colored curves are average of the 10 measurement points for each sample.

Comparison among spectral signatures in the range 350-450 nm are shown in this graph. These measures were collected on June 15, 2016.



Comparison of spectral signatures



Comparison between average spectral signatures obtained by data collected on March 31 (blue lines) and June 15 (red lines), 2016. For each signature, reflectance values are normalized respect to that obtained at 400 nm.

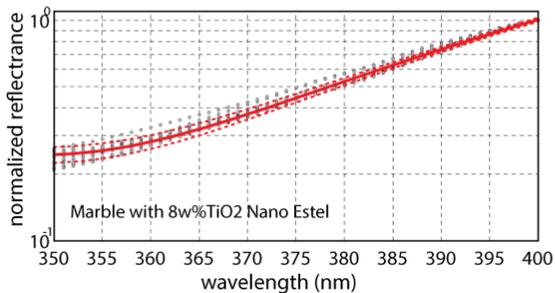
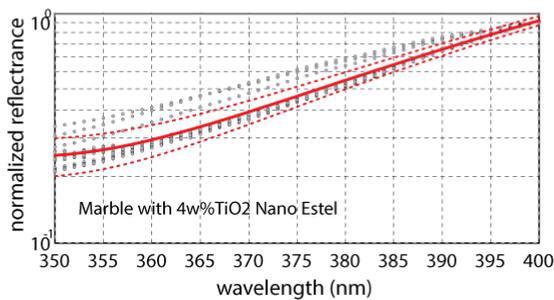
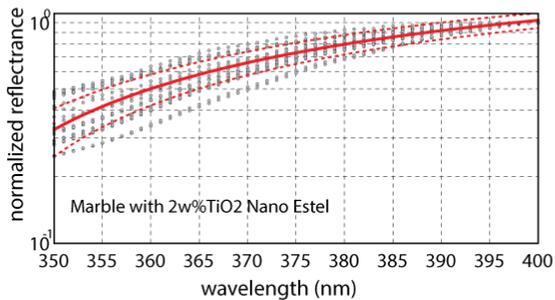
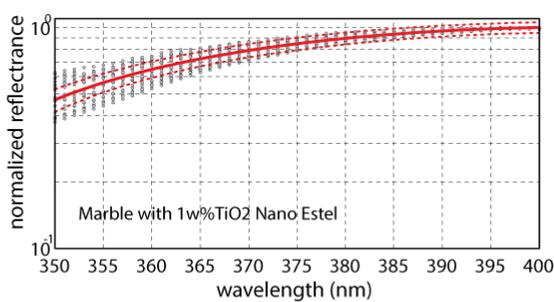
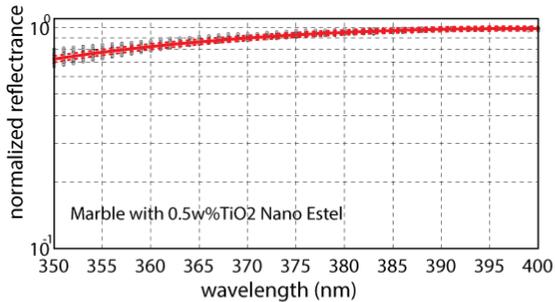
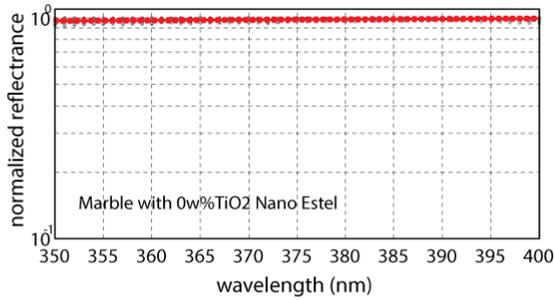
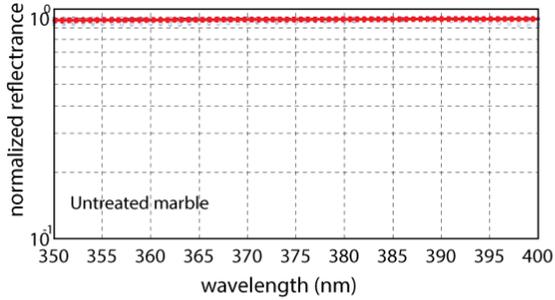
The two measurements are comparable in the wavelength range 350-400nm, especially.



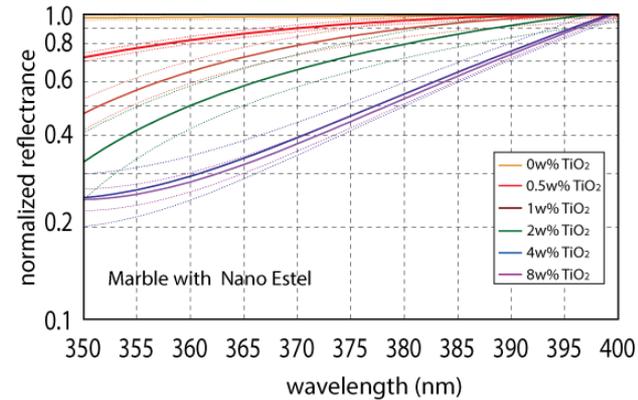
Fitting curves (quadratic polynomials)

Fitting curves obtained by data regression through quadratic polynomials.

- Great reliability is obtained for TiO₂ lower than 1w% and higher than 4w%
- Also, for intermediate values of TiO₂ w%, a good reliability are encountered
- Curves with TiO₂ 4w% and 8w% are not statistically different

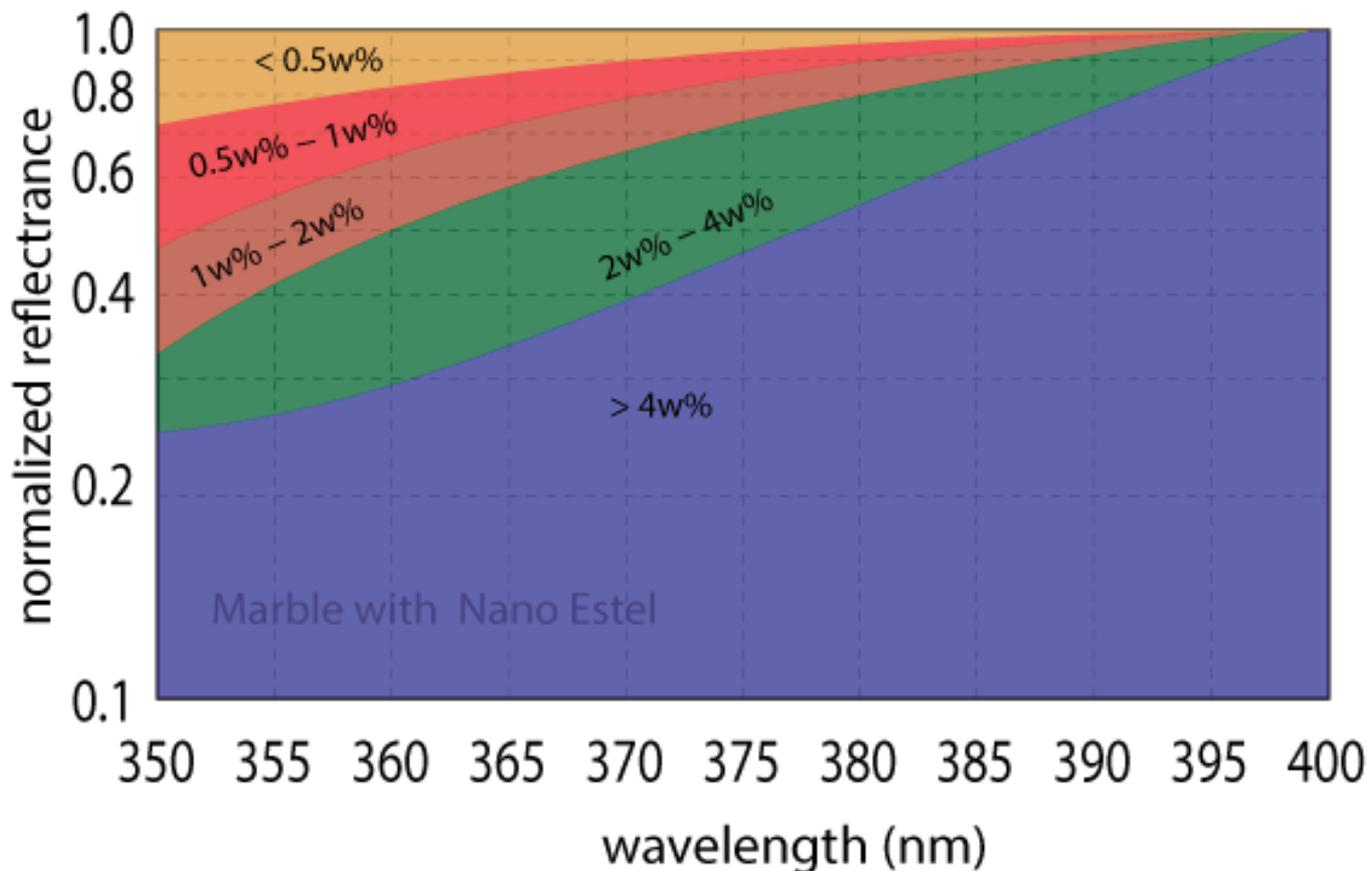


● normalized reflectance data
 — fitting model (quadratic polynomial)
 - - - 75% confidence bounds





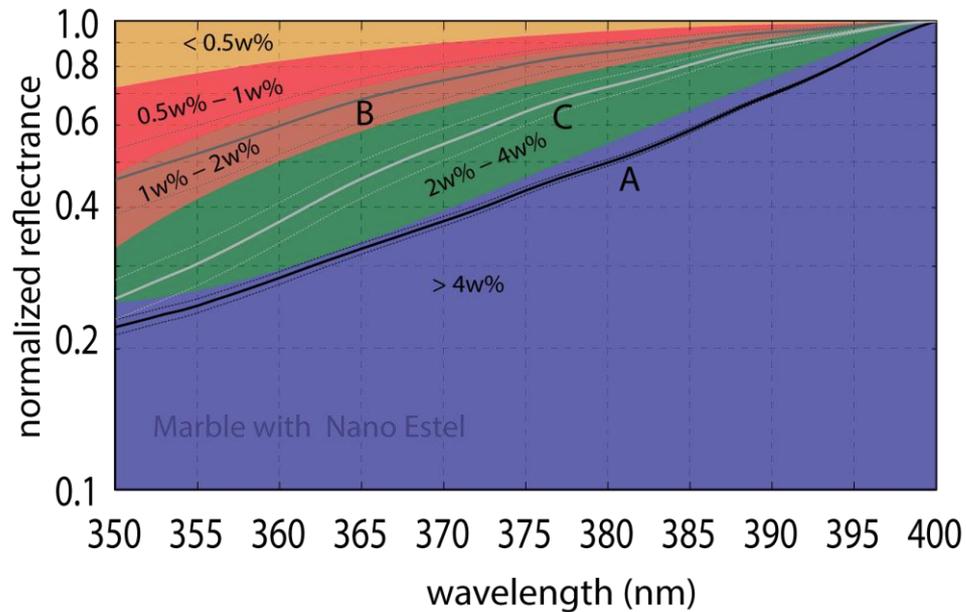
Control chart



Control chart was obtained from the fitting curves, in order to assess the TiO₂ w% from field spectroradiometric survey in the range 350-400 nm.

Blind tests

Three marble samples superficial covered by NANOESTEL with unknown TiO₂ weight percentage (named A, B, C)



Sample	TiO ₂ w% from control chart	TiO ₂ w% from laboratory
A	>4w%	10w%
B	between 1w% and 2w%	1w%
C	between 2w% and 4w%	4w%

Proposed procedure seems to give good results in terms of detected TiO₂ w%; in fact, these are comparable with those declared by laboratory.

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Conclusions

- Coatings with NANOESTEL, blended to different weight percentages of the TiO₂, were applied on marble samples and tested through field spectroradiometric survey.
- The results allowed to develop a procedure to check the state of the coating applied on marble of Cultural Heritage by its spectral signature.

Future developments

- To investigate with more attention the spectral range between 450 and 1800nm, in order to increase reliability of detection
- To compare results obtained by marble and travertine stones coated with different nanoparticle product (NANOESTEL and TEOS) blended to increasing TiO₂ w%

Thank you for the attention !