SolarPACES 2022, 28th International Conference on Concentrating Solar Power and Chemical Energy Systems
Measurement Systems, Devices, and Procedures
https://doi.org/10.52825/solarpaces.v1i.629

Published: 14 Mar. 2024

Round Robin Test of Absorptance and Emittance of Particles for CSP

Florian Sutter[1[https://orcid.org/0000-0002-2981-5807]], Marco Montecchi[2[https://orcid.org/0000-0002-6942-8078]], Angel Morales Sabio[3[https://orcid.org/0000-0002-4690-1782]], Gema San Vicente[3[https://orcid.org/0000-0002-3939-1630]], Aránzazu Fernández-García[3[https://orcid.org/0000-0001-6044-4306]], Johannes Pernpeintner[4[https://orcid.org/0000-0003-4895-6135]], Tomás Reche-Navarro[1[https://orcid.org/0000-0002-9759-4735]], Simon Caron[1[https://orcid.org/0000-0002-7699-8108]], Lucía Marínez-Arcos[3[https://orcid.org/0000-0003-1696-6021]], Johannes Wette[3[https://orcid.org/0000-0003-1389-5176]], Alejandro Calderón[5,6[https://orcid.org/0000-0002-3746-2677]], Marc Majó[5[https://orcid.org/0000-0002-8373-4004]], Inés Fernández[5[https://orcid.org/0000-0001-9212-9714]], Patrick Davenport[7[https://orcid.org/0000-0003-1825-9697]], Tucker Farrell[7[https://orcid.org/0000-0002-7477-2068]], and Clifford K. Ho[8[https://orcid.org/0000-0002-2994-9450]]

[1] DLR, German Aerospace Center, Institute of Solar Research, Spain

[2] ENEA C.R. Casaccia, Italy

[3] CIEMAT-Plataforma Solar de Almería, Spain

[4] DLR, German Aerospace Center, Institute of Solar Research, Germany

[5] Universitat de Barcelona, Facultat de Química, Spain

[6] Departament d'Enginyeria Mecànica, Spain

[7] National Renewable Energy Laboratory, United States

[8] Sandia National Laboratories, United States

Abstract. Precise knowledge of particle optical properties is crucial to the advancement and success of directly-irradiated particle receiver technologies. This work presents the results of a Round Robin Test (RRT) that was conducted between seven laboratories, where each participant measured the absorptance and emittance for a set of five different particle types. This research was performed within the framework of the SolarPACES Task III group, and the results helped establish a guideline for evaluating the optical properties of particles. The guideline was published on the SolarPACES Task III website in May 2022.

Keywords: Particles, Absorptance, Emittance, Round Robin Test

1. Introduction and Methodology

Five types of particles have been included in the comparison: four different types of proppants were manufactured by Saint-Gobain, and one of them was additionally coated by CIEMAT with a spinel coating to improve optical properties (see Table 1). Since batches were very homogeneous, 100g of each particle type was sent to the participating institutes for absorptance and emittance measurement.

Typically utilized spectrophotometers (such as the PerkinElmer Lambda series) use integrating spheres, which require vertical sample positioning. In this case, the measurement of the particle film needs to be accomplished through a window. An exemplary sample holder for such a UV/VIS/NIR measurement through a quartz window is shown in Figure 1 (a) and [1].

Table 1. Particle types used for Round Robin Testing.

Sample ID	Trade Name / Description	Manufacturer	Diameter range (min. and max. mesh size)
BL 1630	BauxLite	Saint-Gobain	297-590 µm
BL 3050	BauxLite	Saint-Gobain	590-1190 µm
SB 3050	Sintered Bauxite	Saint-Gobain	297-590 µm
IP 3050	Interprop	Saint-Gobain	297-590 µm
Coated	BauxLite coated with black spinel	Saint-Gobain (bulk), CIEMAT (coating)	297-590 µm

To account for reflectance and absorption of the window, the particle measurement through the window needs to be corrected. Spectral measurements of particles through a window and a corresponding correction method were first described in [2]. The approach described in the citation is a first approximation, assuming a single reflection from the particle film (and not several back and forth reflections between particle film and window as depicted in Figure 1 b). Within the SolarPACES Task III group, several correction formulae have been discussed to improve the window correction method. A comprehensive summary of this analysis can be found in chapter 4 of the particle characterization guideline “Theoretical background of the window-particle optical model and correction functions” [3]. The method to correct the measurements in this RRT is described by Equation 1. It was selected because it is the easiest to apply among the six different correction methods discussed in [1], and because it shows good agreement with windowless measurements in the RRT and on the conducted validation experiments detailed in the section below.

In order to apply the window-correction, separate measurement of the spectral reflectance and transmittance of the window is required. The selected approach assumes negligible angular behavior of the window material. The reflectance of the particles can then be computed according to:

$$R_s = \frac{\rho_{w,s} - \rho_w}{\tau_w^2 + \rho_w\left(\rho_{w,s} - \rho_w\right)} \tag{1}$$

where R_s is the spectral reflectance of the particles, $\rho_{w,s}$ is the spectral reflectance of the particles measured through the window, ρ_w is the spectral reflectance of the window and τ_w is the spectral transmittance of the window.

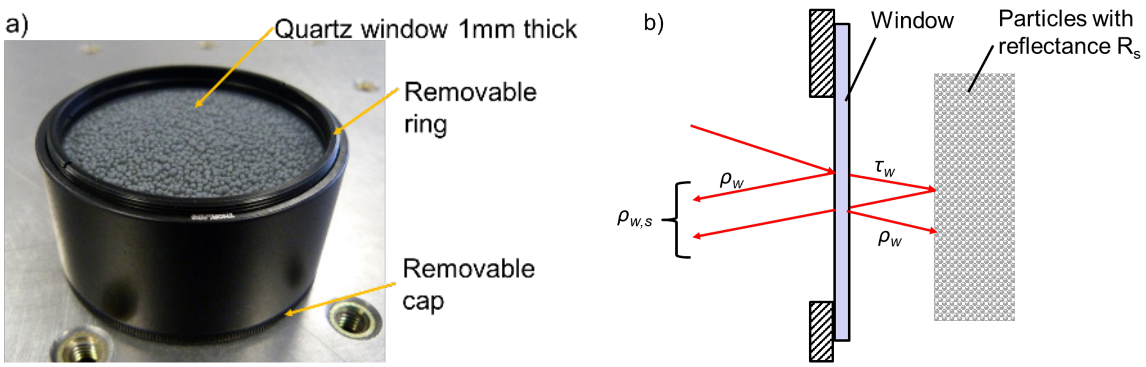

Figure 1. a) Exemplary sample holder to measure particles through a window in a spectrophotometer. b) Illustration of relevant optical parameters to apply the correction formula to eliminate the influence of the window.

Table 2 shows the equipment used by RRT evaluators for optical characterization of the samples. Some evaluators used beam-down configurations for the emittance measurement with

Fourier-transform infrared spectroscopy (FTIR) or handheld spectrometers. In this case, the measurement can be accomplished directly without need to measure through a window.

All emittance measurements in this RRT were conducted at room temperature and thermal emittance at 900°C was computed by weighting the measured spectrum with the black body emission curve at the same temperature. This approach is used frequently in the CSP community to characterize absorber materials. In [4] it was found that for Al_2O_3 and SiC particles, temperature has a negligible impact on emissivity from room temperature to around 1200 K. Nevertheless, measurements with high-temperature spectrophotometers or emissiometers (such as the one described in [5]), while out of scope for this work, remain of interest to check temperature dependence of the proppant types used here.

Table 2. Equipment utilized by each evaluator to measure absorptance (α) and emittance (ε) of the particles. PE stands for PerkinElmer Lambda, IS stands for integrating sphere, and AR stands for anti-reflective.

		Evaluator 1	Evaluator 2	Evaluator 3	Evaluator 4	Evaluator 5	Evaluator 6	Evaluator 1 / 6 / 7
α	Brand	PE1050	PE950	PE950	PE950	PE950	PE1050	Surface Optics 410
	Window	1mm quartz	4mm glass	2 mm SF4 glass	1mm sapphire	2mm quartz	1mm quartz	N/A
	Reference	Calibrated grey, diffuse refl. target, 10%R	Calibrated black, diffuse refl. target, 5-9%R	Certified spectralon, 99%R	Certified spectralon, 99%R	Calibrated grey, diffuse refl. target, 10%R	Certified spectralon, 99%R	Specular and diffuse calibration coupons provided by Surface Optics
ε	Brand	PE Frontier & Pike Mid-IR IntegratIR 76mm IS	-	PE FTIR 100 & 76mm Pike IS	-	PE Frontier & Pike Mid-IR IntegratIR 76mm IS	Thermo Nicolet 6700 FTIR & SOC-100	Surface Optics ET100
	Window	2mm ZnSe with AR-coating	-	2mm ZnSe with AR-coating	-	2mm ZnSe with AR-coating	None (beam-done)	N/A
	Reference	Diffuse gold reference RS-Au-02c	-	Diffuse gold reference	-	Diffuse gold reference	Diffuse gold reference, Avian Tech.	Specular gold calibration coupon provided by Surface Optics

2. Results

2.1 Validation of Window Correction Method

In order to evaluate if the window correction method introduces a systematic bias, all particle types have been measured with the Surface Optics 410 and ET100 devices directly and through quartz and ZnSe windows (see Figure 2). The resulting data before and after applying the window correction method are then compared to the direct (windowless) measurement. In order to properly apply Equation 1, ρ_w and τ_w were determined by means of a spectrophotometer and FTIR and weighted with the specific wavelength intensity distribution of each band of the Surface Optics instrument. Figure 3 shows that the measurement through the window and subsequent correction of the data according to Equation 1 is very well capable of reproducing the windowless measurement. In Figure 3 a) and b) this comparison is shown for different spectral bands of the BL1630 particles. The correction method fails to predict the directly measured value only in the band of 1.5-2 µm. Though not shown here, this artefact is also visible for all other particle types and is caused by the low transmittance of the utilized ZnSe window in the 1.5-2 µm range (see Figure 3 e). However, this effect is not problematic since typical FTIR equipment start measuring at 2 µm. In addition, the deviation in this specific wavelength range does not affect the predicted the solar-weighted absorptance or thermal emittance (see Figure 3 c and d).

The conclusion from this analysis is that the difference between direct (windowless) measurements and the corrected measurement through the windows is an average $\Delta = -0.013 \pm 0.011$ (omitting the artefact in the 1.5-2 µm band). The differences for the individual particle types are shown in Figure 3 f). It can be seen that the window correction method slightly overestimates the absorptance / emittance of the particles systematically. This bias is slightly above the uncertainty of windowless measurements among the three evaluators participating in the RRT with the Surface Optics device.

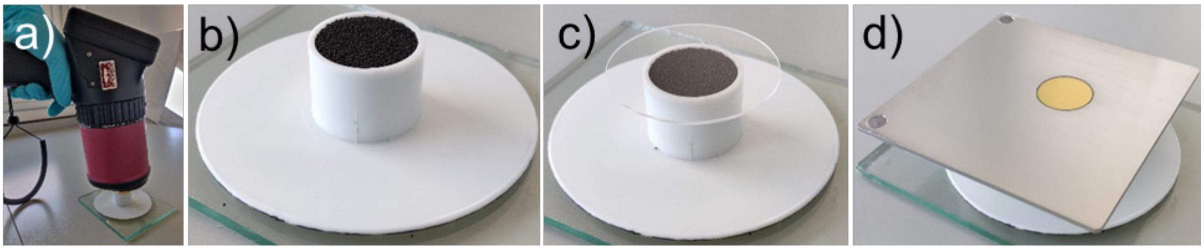

Figure 2. a) Measuring a particle sample with the Surface Optics handheld device. b) Direct measurement of particle sample (no window), c) Particle sample behind 1 mm quartz window, d) Particle sample behind 2 mm ZnSe window.

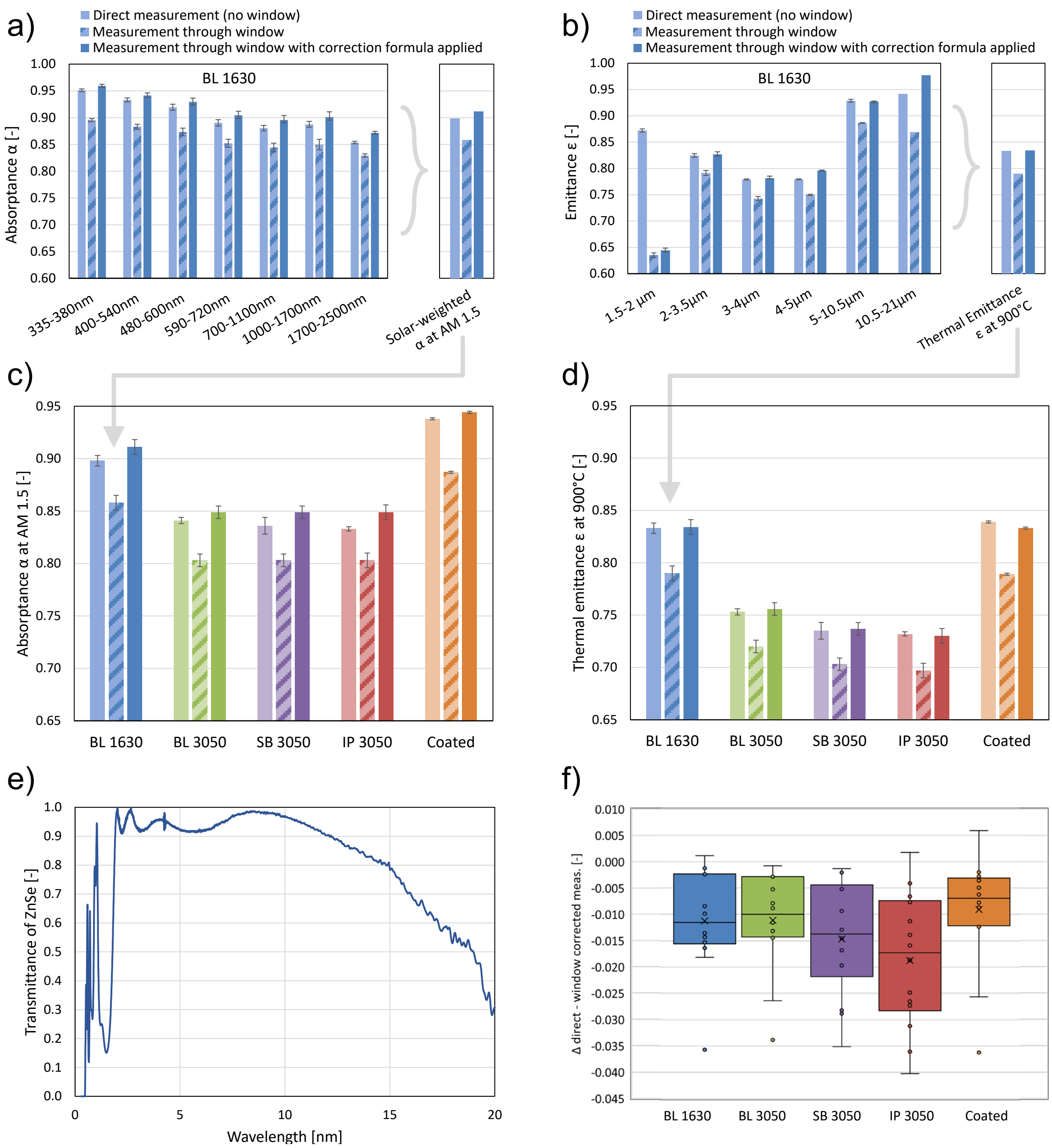

Figure 3. Comparison of direct (windowless) measurements with measurements through quartz (used for the bands up to 2500nm) and ZnSe (used from 1.5 – 21µm) windows with and without the window correction method for BL 1630 particles in the spectral bands of the a) SOC 410 (absorptance), b) the ET100 (emittance), c) for the solar absorptance of all particle types, d) for the thermal emittance at 900°C. e) Spectral transmittance of ZnSe window. f) Box-and-whisker plot representing the difference between direct (windowless) measurements and measurements through the quartz and ZnSe windows after applying the correction method (omitting data from band 1.5 – 2 µm).

2.2 Round Robin Test Results

Figure 4 shows the spectral and solar-weighted absorptance per evaluator for the five particle types included in the RRT. The corresponding spectral emittance and the computed thermal emittance at 900°C are shown in Figure 5.

An overview of the solar-weighted absorptance and thermal emittance data at 900°C is presented in Figure 6 a) and b). The overall trend of the differing absorptance properties of the particle samples is in line for all the evaluators. The average value per particle type and the standard deviation among all evaluators is displayed in Table 3. For the solar absorptance measurement, the maximum standard deviation is only σ=0.9% (σ=0.6% on average), which underlines the good agreement achieved by the evaluators in the RRT. For the thermal emittance measurement at 900°C higher standard deviations among the evaluators are recorded. Here the maximum standard deviation is σ=2.5% (σ=1.9% on average).

Table 3. Average solar absorptance and thermal emittance values determined by the evaluators per particle type.

Sample ID	Average α	Average ε (900°)
BL 1630	0.903 ± 0.009	0.835 ± 0.017
BL 3050	0.846 ± 0.007	0.760 ± 0.015
SB 3050	0.846 ± 0.005	0.747 ± 0.018
IP 3050	0.835 ± 0.006	0.732 ± 0.017
Coated	0.944 ± 0.004	0.844 ± 0.025

Figure 6 c) and d) show the deviation per evaluator from the average solar absorptance and thermal emittance values at 900°C determined in the RTT (as stated in Table 3). The plots help to detect systematic differences among the measurements of the evaluators. As stated before, in terms of solar absorptance, measurements are in good agreement, however small systematic differences are visible among the evaluators. The whiskers (representing the maximum and minimum values in the dataset of each evaluator) do not overlap for all evaluators with the average determined in the RRT: for evaluators 2 and 5 all measured values lie slightly above the average, while for evaluators 1 (for the windowless measurement) and 7 all values lie slightly below the average. In terms of thermal emittance at 900°C, the bias between the evaluators is more pronounced. A systematic difference between windowless measurements with the handheld Surface Optics ET100 device (evaluators 1, 6 and 7 all used the same device) and the FTIR measurements can be seen. Although the Surface Optics measurements among the 3 institutes and the FTIR measurements between the 4 institutes are in acceptable agreement (standard deviations of σ= 0.012 and σ= 0.011, respectively), the average bias between FTIR and Surface Optics is larger ($\Delta\varepsilon$= 0.029 ± 0.002). The bias could also be caused in part by the different incidence angle of the Surface Optics device (Θ=20°) and the FTIR measurements (Θ=8-12°).

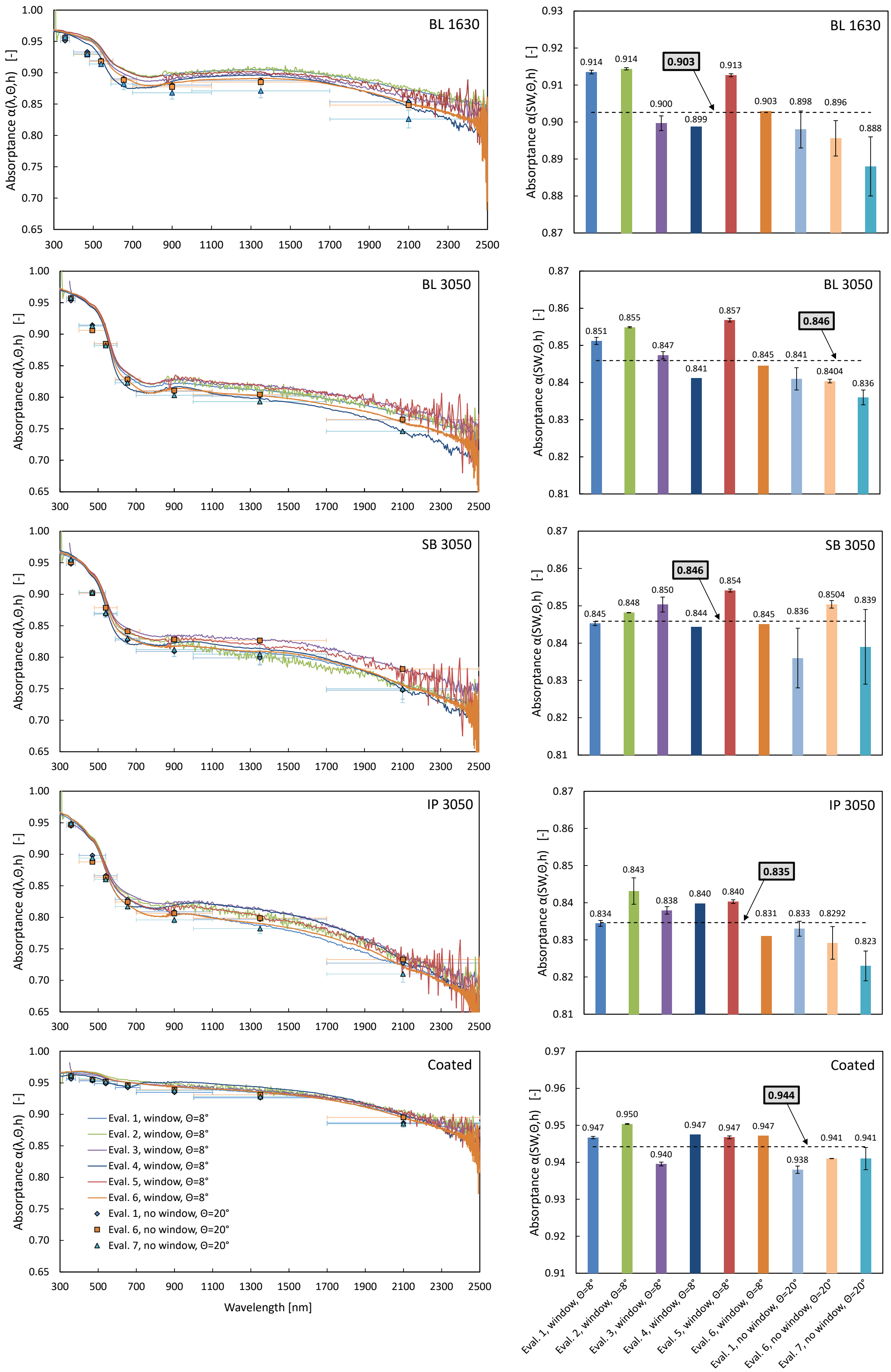

Figure 4. Spectral (left) and solar-weighted absorptance with ASTM G173 direct AM1.5 solar spectrum (right) per evaluator and particle type. Θ is the incidence angle of the measurement.

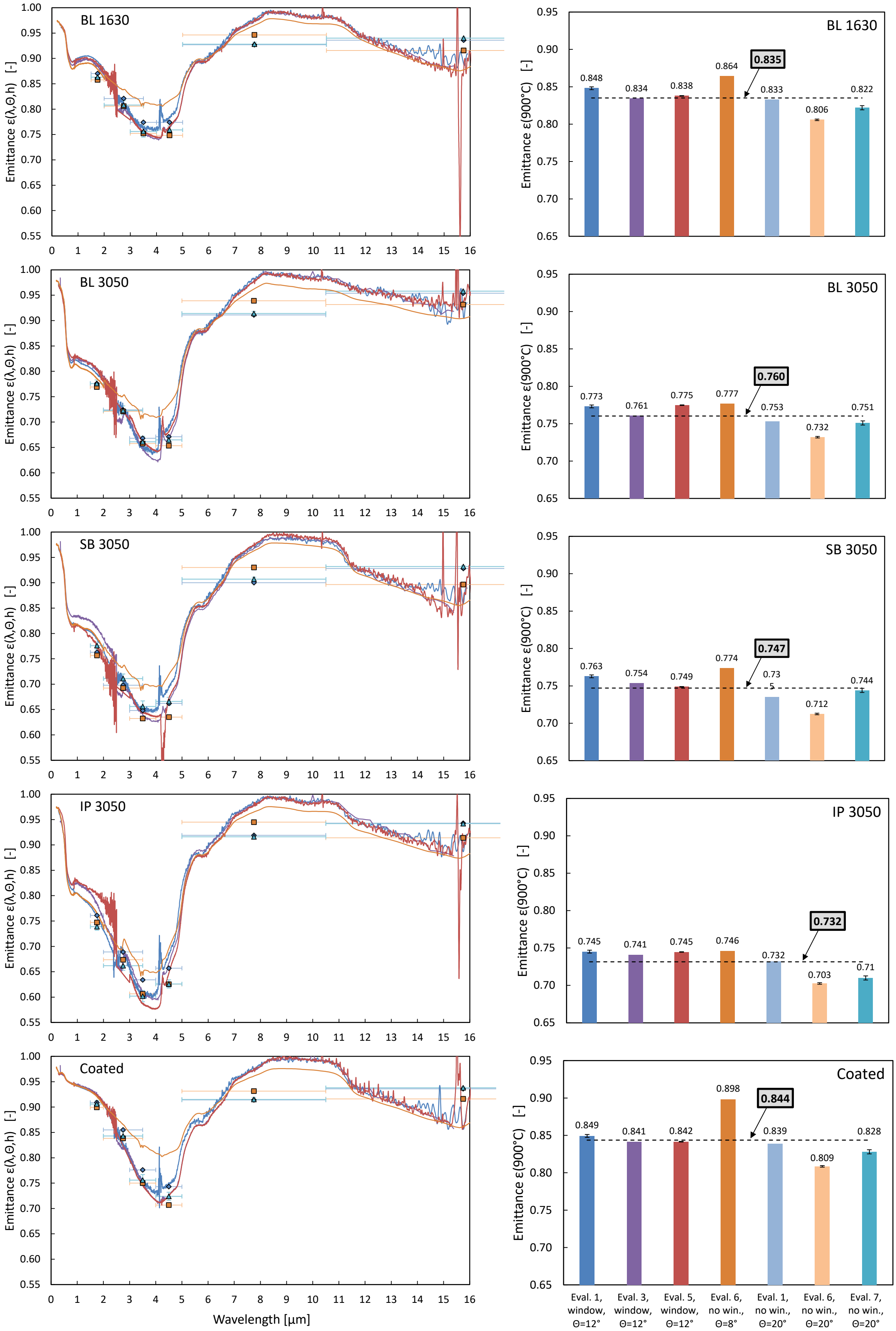

Figure 5. Spectral emittance (left) and computed thermal emittance at 900°C (right) per evaluator and particle type.

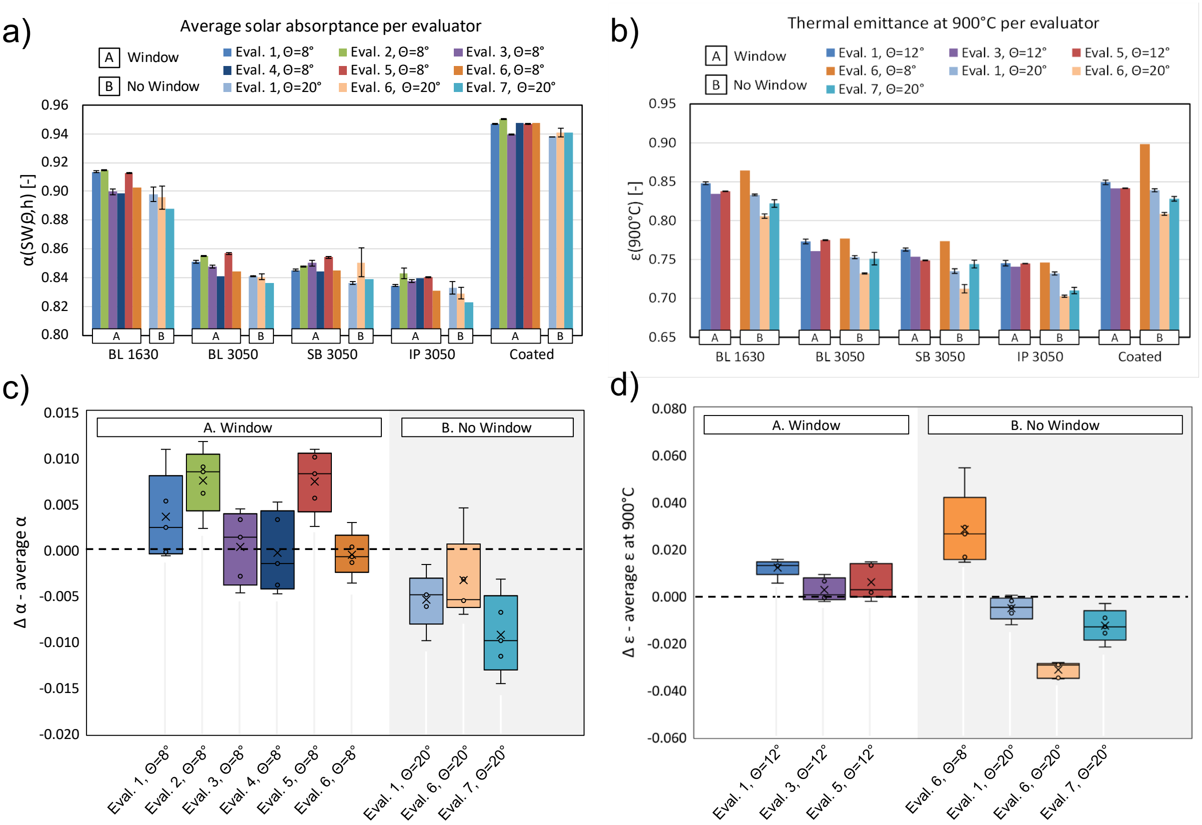

Figure 6. a) Overview of solar-weighted absorptance with ASTM G173 direct AM1.5 solar spectrum per evaluator and particle type, b) Overview of thermal emittance at 900°C per evaluator and particle type. c) Box-and-whisker plot representing the deviation from average solar-weighted absorptance of the RRT per evaluator, and d) the deviation from average thermal emittance at 900°C of the RRT per evaluator.

3. Summary and Conclusions

This paper presents a simple method to correct optical reflectance measurements of particle films through windows. The correction allows instrument users to collect optical measurements in spectrophotometers or spectrometers with their typical vertical sample port configuration. The correction method has been validated by measuring a set of particle types directly and through UV/VIS/NIR and IR windows and comparing the results after correcting the data. It was found that the measurement through the herein studied 1 mm quartz and 2 mm ZnSe windows with subsequent correction slightly overestimates the resulting absorptance / emittance by Δ= 0.013 ± 0.011. Since this difference is close to the uncertainty among different laboratories, we consider the window correction method a useful tool, capable of predicting direct (windowless) measurements.

The Round Robin Test presented in this paper was conducted on five different particle types with 16 different measurement devices among seven partner institutes. Data was collected from windowless measurements with handheld equipment and beam-down configured FTIR spectrometers, as well as from spectrophotometer and FTIR measurements through appropriate windows of different types. The agreement among the different measurement methods was very high for the solar-weighted absorptance (average standard deviation σ= 0.006 among the 9 investigated methods). For the measurement of the thermal emittance at 900°C the deviations were higher (average standard deviation σ= 0.019 among the 7 investigated methods) and a systematic of difference of *Δε*= 0.029 ± 0.002 was detected between the windowless measurements with the handheld Surface Optics and the FTIR spectrometer measurements.

The above-mentioned standard deviations will help end-users to estimate the associated measurement uncertainty of optical particle measurements. In addition, the successful conduction of this RRT has led to publication of a SolarPACES guideline detailing the measurement process, calibration procedure and window correction method (if applicable) for spectrophotometers, FTIRs and handheld devices.

4. Data availability statement

The data supporting the results of this article can be accessed upon request to the authors.

5. Author contributions

Florian Sutter: Conceptualization, Formal Analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing – original draft. **Marco Montecchi:** Methodology, Investigation, Software, Writing – review & editing. **Angel Morales Sabio & Gema San Vicente:** Investigation, Resources, Writing – review & editing. **Patrick Davenport:** Investigation, Visualization, Writing – review & editing. **All other authors:** Investigation, Writing – review & editing.

6. Competing interests

The authors declare no competing interests.

7. Funding

The authors want to thank the SolarPACES organization for funding the project “Characterization of optical properties of particles for CSP”. Part of this research has been funded by the European Union’s Horizon 2020 Research and Innovation Programme under grant agreement Nr. 958418 (CompassCO2) under which the company of Saint-Gobain to kindly provided the particles for this study and CIEMAT developed the coating for the particles.

8. References

1. A. Palacios, A. Calderón, C. Barreneche, J. Bertomeu, M. Segarra, A. I. Fernández: Study on solar absorptance and termal stability of solid particles materials used as TES at high temperature on different aging stages for CSP applications. Solar Energy Materials and Solar Cells 201 (2019) 110088, DOI: https://doi.org/10.1016/j.solmat.2019.110088
2. B. Gobereit, L. Amsbeck, C. Happich, M. Schmücker, M. Wolff: Assessment and improvement of optical properties of particles for solid particle receiver. Proceedings of the 11th International Conference on Energy Sustainability ES2017, Charlotte, 2017
3. SolarPACES Guideline “Method to evaluate the reflectance, absorptance and emittance of particles for concentrating solar power technology” Version 1, May 2022.Available online: https://www.solarpaces.org/csp-research-tasks/task-annexes-iea/task-iii-solar-technology-and-advanced-applications/
4. W. Zhao, Z. Sun, Z. Alwahabi: Emissivity and absorption function measurements of Al2O3 and SiC particles at elevated temperature for the utilization in concentrated solar receivers. Solar Energy 207 (2020) 183-191, DOI: https://doi.org/10.1016/j.solener.2020.06.079
5. P. Loutzenhiser, D. Ranjan, Z. Zhang: Advanced characterization of particulate flows for concentrating solar power applications. Georgia Tech, Gen3 CSP Summit 2021.