

Selective Absorber Coatings Qualification - ISO 22975-3:2014 Full Application

T. C. Diamantino¹, A. Nunes¹, R. Gonçalves¹, S. Páscoa¹, T. Chambino¹ and M. J. Carvalho¹

¹ LNEG - Laboratório Nacional de Energia e Geologia. Estrada do Paço do Lumiar, 1649-038 Lisboa, Portugal

Abstract

The main R&D challenges in the development of Solar Thermal Collectors are focused on increasing reliability and long-term performance stability. Absorber coatings have a major role in conversion of solar radiation into thermal energy. For this reason, testing procedures for qualification were developed and are described in ISO 22975-3:2014. Full application of the test methodologies proposed in ISO 22975-3:2014 is seldom referred for commercial absorber coatings commercially available. This work reports the results of the full tests to five absorber coatings (commercially available), two obtained by physical vapor deposition (PVD) and three by air spray with selective paints. Additional results, obtained with different accelerated aging tests including salt spray and/or sulfur dioxide and nitrogen dioxide as contaminants, are presented. The advantages and limitations of the methodologies proposed in ISO 22975-3:2014 will be discussed, as will be analyzed and proposed new methods of accelerated aging tests.

Keywords: *Solar thermal collectors, Absorber coatings, Qualification tests*

1. Introduction

Countries with high solar resource and a significant amount of coastal regions are the places with the highest population density, constituting a large and prospective market for solar thermal technology and a challenge in terms of reliability and durability for solar thermal technology. Solar thermal collectors (STC) have to withstand stress conditions like high temperatures, high humidity, ultraviolet irradiance or wind and snow loads depending on the geographic location. Literature points out to dependence on other influences like prevailing wind conditions, contaminants like chlorides, SO₂ and NO_x, global solar irradiation, wetness time and precipitation (Köhl, M., et al, 2004; Slamova, K et al, 2016).

Most of the absorbers materials are metallic, copper or aluminium, with different types of coatings obtained by different application methods (Mihelčić M. et al, 2015). Metallic materials are particularly susceptible to corrosion due to combined effect of the different atmospheric parameters namely chloride ions, sulphur dioxide, temperature and relative humidity.

For absorber coatings, due to its major role in conversion of solar radiation into thermal energy, testing procedures for qualification were developed (ISO 22975-3:2014). This international standard specifies a scheme of accelerated ageing tests for qualification of absorber surfaces. Three types of degradation processes are considered: high temperature degradation, degradation by the action of condensed water and degradation caused by high humidity in air containing a small concentration of sulphur dioxide (SO₂). Although SO₂ is one of the dominating airborne pollutants for atmospheric corrosion, the consideration of SO₂ as main corrosion factor for absorber surfaces may be disputable for collectors placed in marine environments as chloride ion from sodium chloride is considered to be major corrosion agent in these regions (Slamova, K et al, 2016; Diamantino, T et al, 2017). Additionally, nitrogen oxides levels have remained constant or even increased with urban NO_x levels higher than SO₂ levels in most cities (Castãno et al, 2007).

In this work, commercially available absorbers with different substrate materials and coatings, PVD - Physical Vapor Deposition and PC - Paint Coatings, were submitted to the full set of tests of ISO 22975-3:2014 and to additional accelerated aging tests with different atmospheric contaminants. The objective of this study allowed the test laboratories involved to gain experience in the application of the tests, but also to identify the advantages and limitations of the methodologies proposed in ISO 22975-3:2014.

2. Experimental

2.1. Selective solar absorber coatings

In this study five selective absorber coatings applied on aluminum were considered, 2 coatings obtained by PVD are commercially available and were acquired from STC manufacturers (PVD 1 - commercial name *mirotherm*[®] and PVD 2 - commercial name *eta plus*[®]); 3 selective paint coatings (two PCs thickness sensitive spectrally selective (TSSS) with silicone binders (PC1 and PC2) which commercial names are SUNCOLOR TS S Black and SOLKOTE HI/SORB-II[™], respectively; 1 PC thickness insensitive spectrally selective (TISS) with a poly(urethane) binder (PC3) which commercial name is SUNCOLOR PUR Black. All the paints used were acquired from the selective paints commercially available. The paints were applied according with the technical instructions of the products on aluminum substrates. Before the application of the paints, all the aluminum samples were subjected to a pre-treatment without chromium. Firstly the panels were acid degreased/deoxidized with Gardacid P 4307 and Gardoclean 450B for 10 min at ambient temperature. Then the specimens followed to a passivation stage with Gardobond X 4707 for 45-60s at ambient temperature. For application by air spray it was used a gravity fed spray gun with a 1.5 mm nozzle and air compressor settled to 2 bar pressure.

The samples evaluated have a dimension of 50 X 50 mm for tests under ISO 22975-3:2014 (section 2.2) and 65 x 140 mm for tests describe in 2.3 and 2.4..

2.2. Methodologies for Solar Absorber Surfaces Qualification (ISO 22975:2014)

2.2.1. Optical properties

All the selective solar absorber samples used specifically in this study were optically characterized by measurement of solar absorption (α_s) and thermal emittance (ϵ_t). The solar absorption (α_s) was determined based on the solar absorption hemispherical solar spectral irradiance (incident on a 37° tilted plane, equator-facing) and the reflectance measurements were done on Perkin Elmer's Spectrophotometer Lambda 950 UV/VIS/NIR with a 150 mm integrating sphere. The thermal emittance (ϵ_t) was determined with a portable emissometer, Devices & Service Company model AE-AD3, which measure a surface emittance at 80 °C of temperature and compared later with measurements using a Spectrophotometer Frontier IR/NIR of Perkin Elmer, with integrating sphere For measurement of the spectral reflectance between 2,5 and 20 μm a diffuse gold coating reference calibrated by Avian Technologies LLC was used.

Evaluation of absorber coatings according to ISO 22975-3:2014 is based on a performance criterion (Pc) dependent directly on the optical properties of the collector and which is defined according to equation (1).

$$Pc = -\Delta\alpha_s + 0,5 \Delta\epsilon_t \quad (1)$$

Where $\Delta\alpha_s$ is the change of solar absorptance during an aging test and $\Delta\epsilon_t$ the change in thermal emittance in the same test. In order to an absorber coating be qualified the value of Pc shall be lower or equal to 0.05.

Optical properties, like solar absorptance and thermal emittance are also the main quantitative values for comparison of absorber coatings and its selection for use in STC. Roberts, D. E., 2013 introduces a figure of merit (FM) based on these optical properties with the purpose of making the selection of solar absorber coatings easier. This figure of merit (FM) is given by equation (2).

$$FM = \alpha_s - 0,44\epsilon_t \quad (2)$$

2.2.2. Coating Adhesion

The coating adhesion of all absorber surfaces were evaluated by pull-off method according to ISO 4624:2002 for deformable substrates. A tensile tester INSTRON 4467 with a load cell of $\pm 5\text{KN}$ was used. Steel test dollies used have a nominal diameter of 20 mm. The adhesive selected was a cyanoacrylate glue.

2.2.3. Assessment of thermal stability

The assessment of thermal stability of the different solar absorber coatings was performed according ISO 22975-3:2014. The equipments used were an HERAEUS LUT 6050 until temperatures of 250°C and a Heratherm Oven OMH100 for higher temperatures, until 330°C. The temperature in the chambers is uniform within the range of ± 1 °C.

2.2.4. Resistance to condensed water

The qualification scheme and the conditions for resistance to condensed water were according to ISO 22975-3:2014. Binder MKF 240 was used as test chamber with sample holders and cooling block as shown in Figure 1.

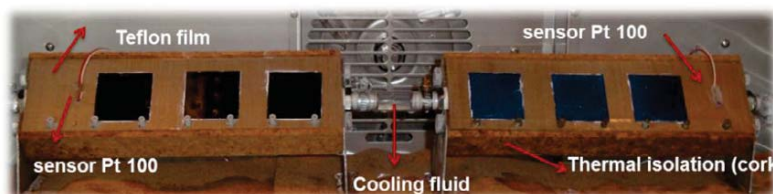


Fig. 1 - Combined sample holder and cooling block for condensation test

2.2.5. Resistance to high humidity air containing sulfur dioxide

The test procedure involves exposure in circulating air of a relative humidity of 95% RH, temperature of 20°C, a sulfur dioxide volume fraction of 10^{-6} and a linear flow rate of air in the range between 1mm/s and 5mm/s. This test is performed according to ISO 10062:2006. According to this standard, the corrosion rate of zinc is considered as a measure for severity of environmental stress on absorber surfaces. During this test, 6 samples of zinc with known initial mass were used. The mass loss was determined for specific time periods, after 90h, 180h and 360h while measuring 2 samples for each time period. Assuming that mass loss rate is linear with testing time, it is possible to calculate the zinc corrosion rate (r_{Zn}), in $mg/m^2/h$, by linear fitting (Least squares method). The average corrosion rate of zinc obtained ($0.014g/m^2/h$) is used to determine the shortest acceptable failure times in hours:

- For a type A solar collector: Shortest acceptable failure time, $t_{f,A} = 2.5/r_{Zn}$
- For a type B solar collector: Shortest acceptable failure time, $t_{f,B} = 7.5/r_{Zn}$

Since the solar absorber surfaces studied could be use in either type of solar collectors, it was decided to submit them to both failure times. According to the determined chamber corrosivity for Zinc, for a type A solar collectors, 180h, and for a type B solar collector, 540h, were respectively considered. A WEISS WK3-340/0-BSB chamber was used.

2.3. Methodologies of accelerated aging tests with continuous exposure of contaminants

2.3.1 Neutral salt spray test

A continuous neutral salt mist spray with a concentration of $50g/l \pm 5g/l$ of sodium chloride in demineralized water, pH value of the solution between 6.5-7.2 and test chamber temperature between $35^{\circ}\pm 2^{\circ}C$ was used (ISO 9227:2012). The inclination of test samples was $20^{\circ}\pm 5^{\circ}$ from the vertical. The duration of the test was 120 hours with observations and evaluation of optical properties after 24, 48, 72, 96 and 120 hours. 6 replicates were used. The chamber used is an Erichsen Model 606/400L. This test was chosen because it is the oldest and most widely used method for laboratory accelerated corrosion testing.

2.3.2 Saturated atmosphere in the presence of sulfur dioxide

A test in a saturated atmosphere in the presence of sulfur dioxide according with ISO 3231:1993 during 5 cycles (24 hours each cycle) was performed. Each cycle consists of 8 h at $40^{\circ}C\pm 3^{\circ}C$ and 100% of RH ($SO_2 - 0.2L$) and 16 h at $18-28^{\circ}C$ and RH max 75% (test chamber vented). 6 replicates were used. The chamber used is a VLM Model CCT 400-Air CTD-A. This test was selected because is widely used to evaluate and qualify metallic and organic coatings for anticorrosive protection to industrial atmospheres with a higher concentration of sulfur dioxide than the test proposed by ISO 22975-3:2014.

2.4 Methodologies of accelerated aging tests with cyclic variation of corrosion promoting gases, higher humidity, salt spraying and drying

This study began applying a methodology of ISO 21207:2015 (Method A) that involves exposure of the test objects to the following test cycle: a) neutral salt spray testing (ISO 9227:2012 for 2 h in a mist of a salt solution containing a mass fraction of 5 % of sodium chloride at $35^{\circ}C$, followed by drying for 22 h in a

standard laboratory climate; b) exposure for 120 h in a test atmosphere containing a mixture of corrosion-promoting gases, volume fraction of NO₂ equal to 1.5×10^{-6} and of SO₂ equal to 0.5×10^{-6} , at a relative humidity of 95 % and at a temperature of 25 °C, followed by drying for 24 h in a standard laboratory climate. The climatic chambers used were a WEISS WK3-340/0-BSB for mixture of corrosion-promoting gases and a Erichsen (Model 600-400L) for salt spray environment. All the tests performed with solar absorber surfaces were simultaneously monitored with reference materials to evaluate atmosphere corrosivity. The optical properties were evaluated after 2, 3 and 4 test cycles. This method was chosen because is recommended for the qualification of coatings, as well as, to predict the lifetime of electronic equipments and is also suitable for comparative testing (ISO/TR 16335:2013).

2.5 Evaluation the corrosivity of accelerated aging tests chambers

The corrosivity of the different accelerated aging test chambers were performed with Zinc standard samples (reference material 99.95%) simultaneously with absorber coatings. Two replicates (section 2.2.5) or three replicates were used for each test period (section 2.3 and 2.4). The samples are rectangular plates with dimensions 100 mm x 150 mm, and a thickness of 1 mm. After exposure, corrosion products were removed in accordance with ISO 8407:2009. The corrosion rates were obtained by weight loss per unit area during exposure periods of different accelerated aging tests.

3. Results and Discussion

3.1. Solar Absorber Surfaces Qualification (ISO 22975-3:2014)

Table 1 resumes the initial optical properties, figure of merit (FM) and adhesion of coatings. These values of optical properties are the average value of thirty samples (50 x 50 mm) and the corresponding standard deviation is also indicated (σ_α for solar absorptance and σ_ϵ for thermal emittance). PVDs have similar values of solar absorptance and thermal emittance. The Paint Coatings PC1 and PC2 have also similar values of solar absorptance and thermal emittance values, but with lower solar absorptance and higher thermal emittance than PVDs. PC3 has the lower solar absorptance and the higher thermal emittance. Except for PVD1 that has a lower adhesion, all other coatings have very similar rupture resistance values.

All absorber coatings were submitted to the full tests according to ISO 22975-3:2014. In Table 2 a summary of results obtained is shown. Figure 2 shows the optical properties converted in results of performance criteria (Pc) and Figure of Merit (FM) of the different selective coatings during the different accelerated aging tests indicated in ISO 22975-3:2014 for surfaces qualification.

All the absorber coatings were qualified for the test in saturated atmosphere in the presence of sulfur dioxide, in terms of optical properties and adhesion of coatings. In terms of thermal stability PVD1 did not qualify due to the changes in optical properties (performance criteria - $P_c > 0,05$). PC3 also did not qualify but due to low adhesion of coating (ruptures resistance $< 0,15$ MPa).

Tab. 1 – Initial optical properties, adhesion and figure of merit (FM) of PVDs and PC coatings

Absorber reference	Optical initial values		Coating Adhesion (rupture resistance – Mpa) (σ)	FM
	Solar absorptance $\alpha \pm \sigma_\alpha$	thermal emittance $\epsilon \pm \sigma_\epsilon$		
PVD1	0.96±0.00(1)	0.11±0.00(2)	0.9 (0.3)	0.91
PVD2	0.96±0.00 (1)	0.11±0.00(3)	3.9 (2.1)	0.91
PC1	0.94±0.00(2)	0.46±0.04	4.0 (0.6)	0.74
PC2	0.94±0.00(1)	0.50±0.03	3.7 (1.7)	0.72
PC3	0.92±0.01	0.58±0.03	4.8 (1.4)	0.66

Tab. 2 – Overall durability results of PVDs coatings and PCs according to ISO 22975-3:2014

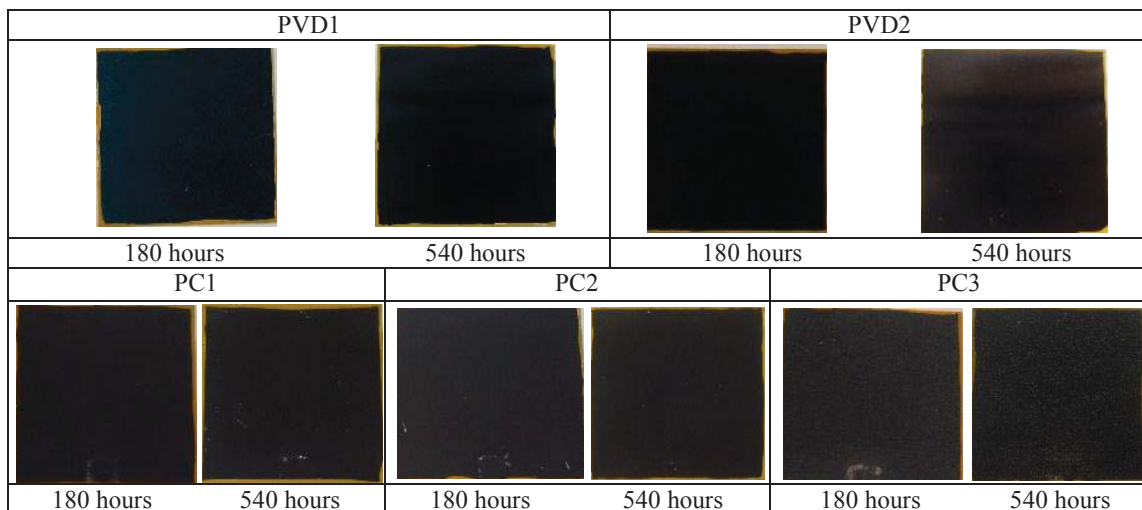
	Thermal Stability	Condensed Water	High humidity air containing sulfur dioxide
PVD1	NQ (<i>optical properties</i>)	Q*	Q
PVD2	Q	Q*	Q
PC1	Q	Q*	Q
PC2	Q	Q*	Q
PC3	NQ (<i>adhesion loss</i>)	Q*	Q

Q – qualified; NQ – not qualified; *require confirmation with thermal conditioning

In terms of resistance to condensed water all the coatings were qualify. However, the results require confirmation because the tests were performed with samples without conditioning. This conditioning considered in ISO 22975-3:2014 consists in submitting each test the sample for at least 5 hours at maximum absorber temperature. The thermal stability tests were performed with samples without and with conditioning. The test of resistance to high humidity air containing sulfur dioxide was performed with all samples conditioned.

Table 3 shows the photographic records of selective solar absorber coatings after exposure to 180 hours and 540 hours to high humidity air with sulfur dioxide. In all coatings (PVDs and PCs) no visual degradation (Table 3) nor significant changes in the optical properties were observed (Figure 2 – e) and f).

Tab. 3 – Photographic records of PVDs (PVD1 and PVD2) and PCs (PC1, PC2 and PC3) after 180h and 540h of exposure to high humidity air containing sulfur dioxide, according to ISO 22975-3:2014



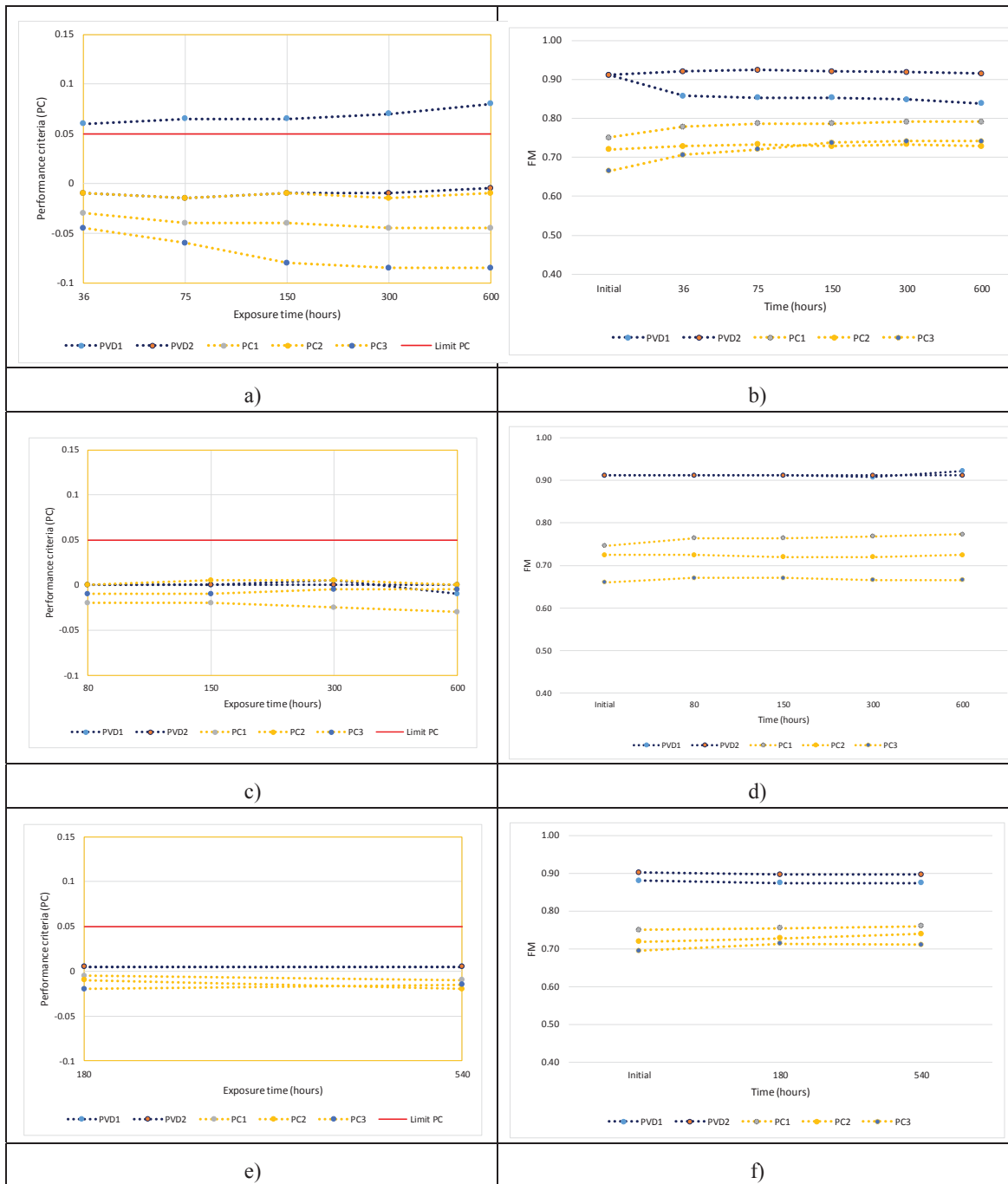


Fig. 2 – Evolution of optical properties in terms of Performance Criteria (Pc) and Figure of Merit (FM) of selective solar absorber coatings after thermal stability test (a) and b)), condensed water test (c) and d)) and test of resistance to high humidity air containing sulfur dioxide (e) and f))

3.2. Accelerated aging tests with continuous exposure of contaminants

Figure 3 and Table 4 show, respectively the results of the optical properties and visual degradation of the different selective solar absorber surfaces after the exposure to accelerated aging tests with continuous exposure of contaminants (salt spray and sulfur dioxide). Neutral salt spray led to a higher degradation in PVDs than in PCs. Considering only PVDs, PVD2 showed higher degradation than PVD1. For PCs, PC3 showed the best behaviour in terms of anticorrosive protection and optical properties (lower Pc and higher FM). For the sulfur dioxide resistance test opposite results are observed, i.e., the PCs showed higher degradation than PVDs. No significant changes were observed between the 2 PVDs and the 3 PCs.

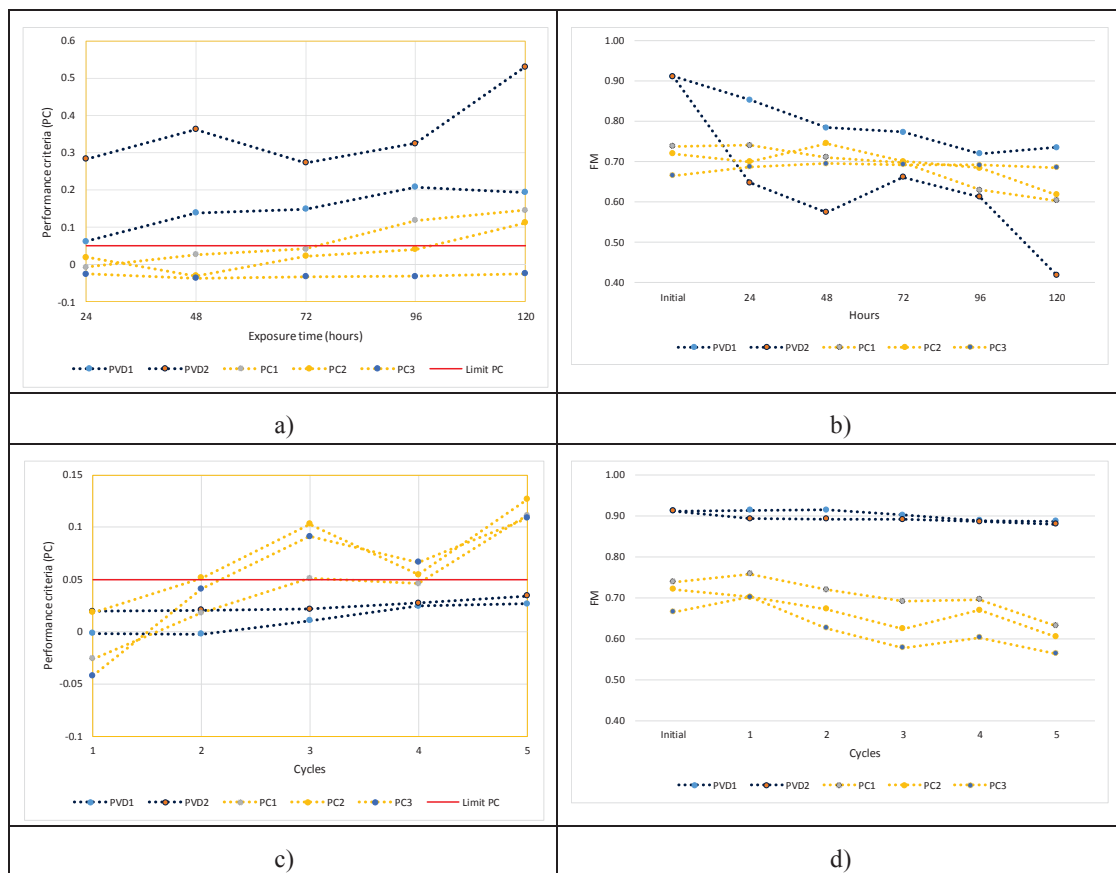

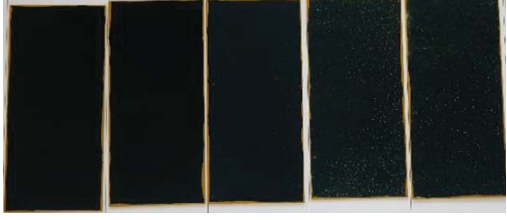






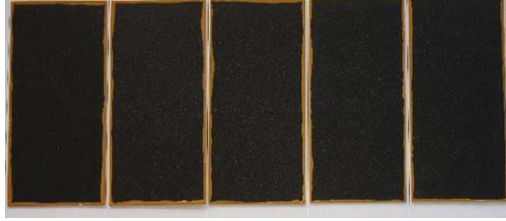



Fig. 3 – Evolution of optical properties in terms of Performance Criteria (Pc) and Figure of Merit (FM) of selective solar absorber coatings after neutral salt spray test (a) and b)) and to humid atmospheres containing sulfur dioxide (c) and d))

Tab. 4 – Photographic records of PVDs (PVD1 and PVD2) and PCs (PC1, PC2 and PC3) of exposure to neutral salt spray during 120 hours (ISO 9227:2012) and to humid atmospheres containing sulfur dioxide during 5 cycles (ISO 3231:1993)

Solar absorber coating	Neutral salt spray (24h, 48h, 72, 96 and 120h)	Sulfur dioxide (0.2L) (1, 2, 3, 4 and 5 cycles)
PVD1		
PVD2		
PC1		
PC2		
PC3		

3.3. Accelerated aging tests with cyclic variation of corrosion promoting gases, higher humidity, salt spraying and drying

Figure 4 and Table 5 show, respectively the results of the optical properties and visual degradation of the different selective solar absorber surfaces during the 4 weeks (4 cycles – Method A) of exposure to accelerated aging tests with cyclic variation of corrosion promoting gases, higher humidity, salt spraying and drying.

PVD2 showed higher corrosion which is accompanied with the higher reduction of FM (decrease of absorbance and increase of emittance) and a P_c higher than 0.05. PCs showed also higher degradation, namely PC1 and PC2 reaching values of FM similar to PVD2.

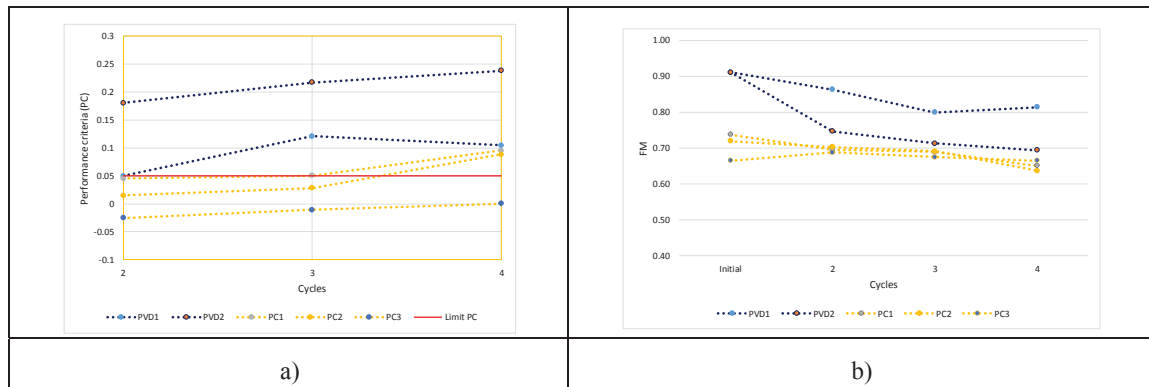
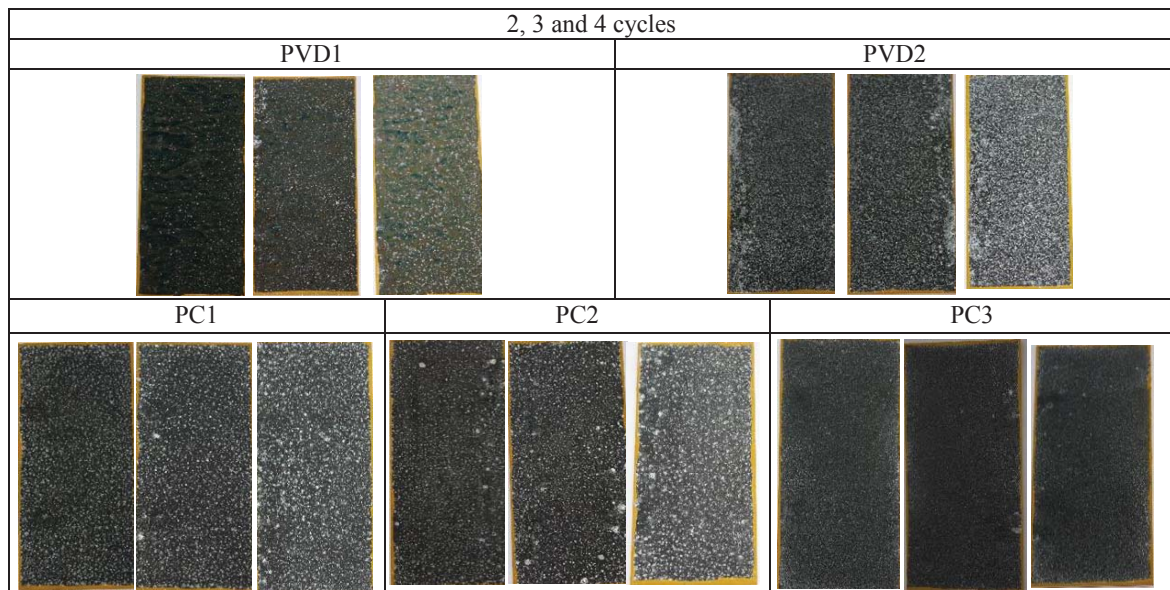


Fig. 4 – Evolution of optical properties in terms of Performance Criteria (Pc) and Figure of Merit (FM) of selective solar absorber coatings during exposure (4 cycles – 4 weeks) to accelerated aging test with cyclic variation of corrosion promoting gases, higher humidity, salt spraying and drying

Tab. 5 – Photographic records of PVDs (PVD1 and PVD2) and PCs (PC1, PC2 and PC3) during exposure (4 cycles – 4 weeks) to accelerated aging test with cyclic variation of corrosion promoting gases, higher humidity, salt spraying and drying (ISO 21207:2015)



The results obtained with this methodology show results similar to those presented by Diamantino et al, 2017 with the same solar absorber coatings exposed to outdoor exposure testing sites with different corrosivity (urban test site with marine influence – medium corrosivity and an industrial/marine test site with very high /extreme corrosivity) in terms of the ranking of Pc, FM and anticorrosive protection. PVD2 showed the higher Pc and PVD1 the higher FM in both OET sites as seen in this accelerated aging test. PC3 showed the best anticorrosive protection and PVD2 the lower in both OET sites, as well as, from results obtained by electrochemical impedance spectroscopy (Fernandes et al, 2017), as well as in this test method.

3.4. Zinc corrosivity in accelerated aging tests chambers

In order to compare the different corrosivity of accelerated aging tests presented, Table 6 resumes the values obtained experimentally and from literature, assuming that mass loss rate is linear with testing time. Neutral salt spray is clearly the higher corrosive test followed the SO₂ resistance test based on ISO 3231:1993.

According to ISO 22975-3:2014 the atmospheric corrosivity at the bottom part of the solar collector under service conditions may typically correspond to a corrosion rate of zinc of 0.1 or 0.3 g/m² per year depending of the type of collector. Other authors (Slamova et al, 2016) evaluated collectors under stagnation conditions obtaining a zinc corrosion rate of 19.72g/m² per year. Carvalho, M. J. et al, 2017 obtained zinc corrosion rates, in different type of collectors with different solar absorber surfaces under stagnation exposed in same atmosphere, of 4.5 g/m² and 99.9 g/m² per year. So, the corrosivity of the SO₂ resistance test proposed in ISO 22975-3:2014 seems to be insufficient to guarantee durability of 25 years for solar selective absorber surfaces.

Tab. 6 – Zinc corrosion rate (g/m²/day) in the different accelerated aging tests

Accelerated aging tests	Corrosion rate (g/m ² /day) (range)
SO ₂ (ISO 22975-3:2014)	0.34 (0.26-0.39)
Neutral salt spray (ISO 9227:2012)	25 (12.5-50) ⁽¹⁾
SO ₂ (ISO 3231:1993)	1.25 (1.13-1.37)
Cycles (ISO 21207 - Method A:2015)	0.81 (-) ⁽²⁾

⁽¹⁾ Value assuming that mass loss rate is linear with testing time. The value indicated in ISO 9227:2012 is 50±25g/m² (48 hours)

⁽²⁾ Value assuming that mass loss rate is linear with testing time. The value indicated in ISO 21207:2015 (Method A) is 40g/m² (4 weeks or 4 cycles)

4. Conclusions

It was the main objective of this work the implementation of standardized test methods for qualification of different solar absorber coating surfaces. The tests according to ISO 22975-3:2014 were implemented and applied to 5 absorber coatings that were commercially available (two PVDs and three PCs).

All absorber coatings were qualified for resistance to sulfur dioxide. PVD1 was not qualified in terms of thermal resistance due to optical properties and PC3 was equally not qualified but due to loss of coating adhesion. All surfaces are qualified relatively to resistance to condensed water. However the paint and PVD coatings require confirmation because the study was performed only with samples without previous thermal conditioning. Temperature can lead to some adhesion and optical weakness in the organic and PVDs coatings respectively during the exposure to condensed water.

The methodologies of accelerated aging tests with continuous exposure to contaminants (salt spray and sulfur dioxide) revealed that PVDs are more susceptible to degradation induced by chloride rather than sulfur dioxide and PCs suffer further degradation with sulfur dioxide than chlorides. Countries with high solar resource and amount of coastal regions with high population density constitute a large and prospective market for STC and a challenge in terms of reliability and durability. The results obtained with the methodology describe in ISO 21207: 2015 considering an accelerated aging test with cyclic variation of corrosion promoting gases (SO₂ and NO₂), higher humidity, salt spraying and drying reveal similar results to those presented by Diamantino et al, 2017 with the same solar absorber coatings exposed to outdoor exposure testing sites with different corrosivity (urban test site with marine influence – *medium corrosivity* and an industrial/marine test site with *very high /extreme corrosivity*) in terms of the ranking of Pc, FM and anticorrosive protection. PVD2 obtained the higher Pc and PVD1 the higher FM in both OET sites and in accelerated aging according to ISO 21207:2015 (Method A). PC3 showed the best anticorrosive protection and PVD2 the lower in both OET sites, in accelerated aging test according to ISO 21207:2015 and from results obtained by electrochemical impedance spectroscopy (Fernandes et al, 2017).

The methodologies proposed in ISO 22975-3:2014 are an important tool for qualification of selective absorber

coatings. However these tests require many hours and don't reflect the environments where the collectors are exposed. These results showed that the accelerated aging test for qualification of selective solar absorber coatings must include at least sulfur dioxide and chloride as contaminants, beyond the resistance to temperature and humidity condensation. Additional studies are ongoing, namely to study the degradation mechanism of solar selective surfaces under the influence, separately and combined, of SO₂, NO₂ and chlorides in a defined temperature and humidity, with and without dry cycles and UV radiation under the cover. The procedures of accelerated aging tests to qualified coatings guaranteeing high durability should have gas with low concentrations together with a proper choice of temperature and relative humidity, salt spray, radiation and air flow to generate the degradation that have been observed in well characterized field environments with a specific atmospheric corrosivity.

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