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Influence of an Increased Controlled Dose of Irradiance on the Change of Selected Physicochemical Properties of Paint Systems Used in Rolling Stock

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Summary
The article presents the results of ageing tests with the use of laboratory light radiation tested according to the EN ISO 16474-2 standard for painting systems used in rolling stock. The influence of aging on such properties as gloss, colour and hardness, was determined using two different irradiance settings of the order of 60 W/m^2 and 120 W/m^2 for the wavelength 300÷400 nm and the same levels of radiant exposure. In addition, this study describes the most important information about laboratory aging tests with simulation of sunlight, temperature, moisture including rain, which are included in ISO standardised test methods. The influence of weather parameters has been described, in particular the influence of an increased dose of irradiance on the degradation of selected physicochemical properties of coatings and the possibility of the predicted ageing progress (changes in selected properties). The obtained results of laboratory tests allow to make certain assumptions regarding the increase of the dose of irradiance and thus the shortening of the laboratory testing time in the context of the assessment of the most desirable properties of the coating for a given application with the use of laboratory ageing tests.

Keywords: accelerated weathering, xenon-arc radiation, paint coatings, irradiance, radiant exposure, hardness, gloss, colour, rolling stock

1. Introduction

Accelerated ageing of materials due to light radiation is currently simulated in instruments equipped with xenon arc lamps or fluorescent UV lamps. Other types of light sources, such as metal-halide or mercury-vapour lamps, are also used, but much less frequently and in a different research context or type of tested objects. The most versatile instruments are those equipped with xenon lamps, to which a suitable filter system is applied, thus enabling light with a stable energy distribution to be produced, most faithfully reproducing the natural light of the sun across the entire spectral range, i.e. UV + VIS + IR. Different types of filters can be applied to such lamps, but the most common are those that simulate natural solar radiation outdoors (from 290 nm) or indoors through window glass (cutting off the shortest wavelength range and shifting the radiation away from the ≥ 310 nm range). The second type of instrument is equipped with fluorescent UV lamps, which do not require optical filters and only simulate radiation in the ultraviolet range, not the entire light spectrum as xenon lamps do. There are three types of fluorescent lamps: UVA-340 (used to simulate light outdoors), UVA-351 (used to simulate light indoors after passing through window glass) and UVB-313 lamps (which emit radiation that is not present in natural sunlight – causing the most destructive effects). For ageing tests with light simulation, ultraviolet radiation in the 290÷400 nm range is of greatest importance, as the shorter the wavelength, the more energy it carries, causing more efficient photodestruction of the material. Nevertheless, some materials can also show absorption of radiation in the visible light range, i.e. 400÷800 nm (dyes), and light in the infrared range (> 800 nm) can cause a large amount of heat to build up in the tested material and thus accelerate the rate of the reactions taking place. Using instruments equipped with xenon lamps, it is possible to test a greater variety of material types than with instruments equipped with fluorescent UV lamps [1, 2, 3, 4].

The diagram (Fig. 1) illustrates the comparison between solar and laboratory radiation using xenon

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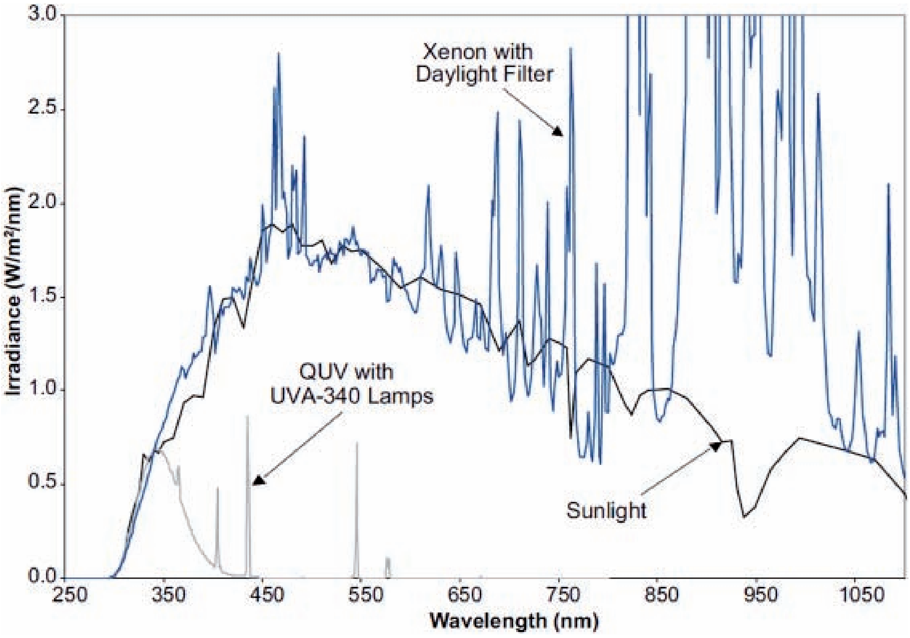

Fig. 1. Comparison of solar radiation with simulated laboratory radiation [5]

lamps with a daylight filter and a UVA-340 fluorescent lamp (simulating daylight).

To date, a number of standardised laboratory tests have been developed worldwide to correspond as closely as possible to the results obtained naturally. Some of these tests are described in the EN ISO 4892 series (4 parts on ageing of plastics) and EN ISO 16474 (4 parts on ageing of paints and varnishes). These standards are virtually the same and differ only in their assignment to a material group. The first part of the aforementioned standards provides precise information on the requirements that must be met for manufacturers of ageing instruments to simulate the accelerated effects of artificial weathering and artificial irradiation (deviations of selected parameters and design solutions are permitted). Part I of the standard also provides information on the interpretation of data obtained from artificial accelerated weathering or accelerated irradiation and classifies factors that reduce the degree of correspondence between exposure to artificial accelerated ageing (or artificial accelerated irradiation) and actual natural exposure (Annex b).

The second part of the aforementioned standards establishes methods for exposing samples to xenon arc lamps in the presence of moisture in order to reproduce the effects of atmospheric conditions on products exposed to daylight or daylight filtered through window glass in actual end-use environments. Part 3 deals with tests using instruments fitted with fluorescent UV lamps and Part 4 with instruments with carbon arc lamps, which were the first types of lamps to be used in laboratory practice and are now rarely encountered.

An important issue to consider when conducting ageing tests in the laboratory, which is not mentioned in the listed standards, is the duration of the laboratory test in relation to the natural exposure of the material to external conditions and the required "lifetime" of the material. It is theoretically impossible to determine the exact number of laboratory test hours proportionally translating exposure time under natural conditions. This is due to the inherent variability and complexity of outdoor conditions. These variables mainly include: the geographical location of the exposure site, local geographical features (such as wind causing drying of samples or proximity to water bodies causing dew or higher salinity), random annual weather variations or seasonal variations (exposure of samples in winter may only be 1/7 as strong as in summer, for example). The reproducibility of the results is also affected by the variability of laboratory test conditions. Variables include: the cycle of the laboratory instrument (light hours and humidity hours), the temperature of the instrument (warmer means faster), the properties of the tested material, the light source (spectral range) [2].

Given the issues mentioned, some assumptions and simplifications are required in order to determine the optimum time for conducting accelerated laboratory tests in relation to the time exposure outdoors and the required minimum durability, but in this way it is possible to obtain practical durability data for the tested items from the accelerated laboratory tests carried out. The best answer that can be obtained from ageing laboratory tests is a ranking of the material's strength compared to another material [2].

In the case of photochemical reactions and relationships, the so-called Bunsen-Roscoe law (1859) law is most often invoked. It states that the mass of the product of a photochemically initiated reaction is proportional to the product of the irradiance (E) and the exposure time (t). In other words, the "photoresponse" of a material depends only on the total energy involved (radiant exposure[2] – H) and the same amount of radiant exposure causes the same level of photochemical changes (or changes in the given material properties). It does not matter at what time it is applied or what level of irradiance is applied ($E \cdot t$ = constants for the response of the material in question). However, it was found that in the case of very short or very long exposures (tests done on photographic film), this law failed, with the effect always being reproducible, and the newly defined mathematical equation was named the Schwartzschild effect [6].

Nowadays, there are many ageing instruments available on the market that allow a wide range of options in terms of control of selected parameters independently, including the setting and control of high irradiance values reaching up to 200 W/m^2 with respect to the bandwidth 300÷400 nm. The use of increased levels of irradiance may reduce the testing time proportionally, but it may also significantly affect the test results, which will have no significance when translated into the results obtained during natural ageing of the material. By increasing the irradiance level, we can, for example, achieve the glass transition temperature of the polymer as a result of an indirect increase in the surface temperature of the sample, as well as proportionally reduce the exposure periods for the tested materials with regard to parameters such as simulated temperature, humidity or rain. Some materials may be sensitive to a factor other than just the photodegradation mechanism, e.g. polyamides are strongly sensitive to moisture [7]. The ageing process is also intensified by increasing the values of parameters such as the ambient temperature of the specimens or the surface temperature of the specimens, or by varying the moisture load cycle. However, the most common practice is to intensify the process by increasing the dose of irradiance or combining it with an increased temperature value only.

When tests are carried out at higher irradiance levels, it may not be possible to achieve constancy in parameters such as the ambient or surface temperature of the samples or the relative humidity of dry periods, and even the slightest change in these parameters can affect the final test result. There are some approaches that describe the effect of changing the above-mentioned parameters on the rate of reactions taking place, such as the Arrhenius equation linking the kinetics of chemical reactions to the rate constant of the reaction, the activation energy and the temperature at which the reaction takes place. This equation gives an estimate of the effect of increasing temperature on the rate of reaction, where it is assumed that a 10°C increase in temperature, on average, causes the reaction to accelerate by as much as two times (in practice, this theory does not hold true and is strongly dependent on the type of material being aged) [8]. In the case of moisture, alternating cycles of wet and dry periods, causing stresses in the coating and consequently the formation of cracks, are important. In this case, the amplitude of variation is important, as too rapid changes result in water not being able to penetrate the coating and no significant changes being observed, while longer periods of alternating wet and dry cycles are capable of causing much greater damage to the coating due to deeper water penetration [9].

If the irradiance level (E) is increased, a validation procedure must first be carried out for the test material to ensure that the test results are not significantly affected by the increase in irradiance. The process of such validation is described, for example, in ISO/TS 19022 – Plastics – Controlled acceleration of laboratory weathering by increased irradiance [10], where the following procedure is assumed:

1. test at the lowest irradiance level (e.g. with irradiance[3] E = 60 W/m^2);
2. test at increased irradiances (with higher irradiances, e.g. E = 80 W/m^2, 100 W/m^2, 120 W/m^2 etc.);
3. the other process parameters (temperature, humidity) remain constant, although it is not always possible, as the ambient temperature of the sample or the surface temperature of the sample also increase as a result of irradiation, and the technical capabilities of the instrument may make it impossible to achieve the set parameters due to, for example, an inefficient cooling system or a change in the spectral radiation distribution during lamp use (higher intensity in the IR range);
4. creating a graph as a function of the total radiant exposure ($H = E \cdot t$ = constant) with regard to the selected property under study, e.g. colour change ΔE;
5. calculation of linear correlation coefficients, e.g. Pearson's or Spearman's rho (non-parametric method).

[2] Radiant exposure – received by a surface per unit area, or equivalently irradiance of a surface integrated over time of irradiation. This is sometimes also called "radiant fluence". The unit of radiant exposure is the watt-second (a.k.a. joule with the symbol J) per square metre [W·s/m^2].
[3] Irradiance – radiant flux per unit area. The unit of irradiance is watts per square metre [W/m^2].

Literature data show that, for coating/painting systems, radiation can be intensified up to the energy limit of the so-called three suns (180 W/m^2), where such an applied radiation dose gives a very good correlation, usually above 90% for coating systems. It has been conventionally assumed (it is not standardised) that the energy of one sun for the broad measurement band 300÷400 nm is about 60 W/m^2, with an identical irradiance setting required by EN ISO 16474-2 [7, 11, 12], among others.

The ability to increase the irradiance dose and thus proportionally reduce the test time (achieving the target radiant exposure faster) is becoming an invaluable and crucial tool when conducting accelerated laboratory ageing tests. This makes it possible to obtain the results of ageing tests for the materials in question in a much quicker time and frees up the test apparatus (which unfortunately has very limited sample capacity, and the purchase of such equipment and its operation is very expensive) for further tests. Optimisation in the form of reduced test duration can also reduce test costs (in this case, much depends on the ageing apparatus and type of apparatus cooling).

As part of the research, it was decided to check whether conducting the ageing process according to EN ISO 16474-2 [11], using two different irradiance levels doses while keeping other process parameters such as ambient temperature, surface temperature and humidity at the same level, would produce similar changes depending on the total radiant exposure. Parameters such as gloss, colour and hardness were determined and the irradiance used in the tests was 60 W/m^2 and 120 W/m^2 for the wavelength range 300÷400 nm, respectively. The value of 120 W/m^2 is the maximum instrument setting for irradiance at the wavelength range 300÷400 nm for Xenotest 440 used in the tests. A total of five radiant exposure levels were considered: 108 MJ/m^2, 216 MJ/m^2, 324 MJ/m^2, 432 MJ/m^2 and 540 MJ/m^2, measured at wavelengths of 300÷400 nm. Translated into natural ageing, they correspond to ageing times of approximately 8, 17, 25, 34 and 42 months, respectively. Data were estimated assuming an annual average radiant exposure of 154 MJ/m^2 for the measurement band of 290÷400 nm for Poland in 1997-2001 [13].

2. Research material, methodology and apparatus used

The selected research material included 11 complete paint systems used, among others, in Polish rail

Table 1

Summary of tested systems

System No.	Type of substrate (steel)	Full system composition
I	ST3S	• epoxy primer • polyester putty • acrylic filler primer • acrylic-polyurethane topcoat
II	ST3S	• epoxy primer • polyester putty • acrylic filler primer • acrylic-polyurethane topcoat
III	DC01	• epoxy primer • polyester putty • polyurethane filler primer • acrylic basecoat • polyurethane clearcoat
IV	DC01	• epoxy primer • polyurethane topcoat
V	ST3S	• epoxy primer • polyester putty • acrylic filler primer • polyurethane basecoat • acrylic clearcoat 1 • acrylic clearcoat 2
VI	ST3S	• epoxy primer • acrylic-polyurethane topcoat
VII	DC04	• epoxy primer • polyester putty • polyurethane filler primer • polyurethane topcoat
VIII	DC04	• epoxy primer • polyester putty • polyurethane filler primer • polyurethane filling and colouring primer • polyurethane clearcoat
IX	ST3S	• epoxy primer • polyester putty • acrylic filler primer • acrylic basecoat • acrylic clearcoat
X	ST3S	• epoxy primer • epoxy putty • acrylic primer • acrylic basecoat • polyurethane clearcoat
XI	ST3S	• epoxy primer • polyurethane topcoat

[Authors' own elaboration].

transport and tested by the Railway Research Institute over many years. A general summary of the composition of the systems tested is shown in Table 1. The substrate was a variety of structural steels.

The study used the scheme shown in Table 2. Tests were carried out using the standard settings recommended by EN ISO 16474-2 [11], and it was decided to double the irradiance level to 120 W/m^2 while maintaining the other ageing parameter settings consistent with Table 2. Test results for different irradiance levels were compared for selected physicochemical properties of the coatings, such as gloss, colour and hardness (pendulum damping method), at fixed doses of total radiant exposure. The exact methodology and apparatus used for the measurements are summarised in Table 3.

Table 2

Values of applied settings including tolerances in the ageing tests

Daylight filter method						
Test No.	**Radiant exposure at which the assessment was carried out, *H* [MJ/m^2] / approximate exposure time [h]**	**Test duration**	**Irradiance 300÷400 nm, *E* [W/m^2]**	**Black Standard Temperature BST, [°C]**	**Temperature in the chamber, CH [°C]**	**Relative humidity, RH [%]**
1	108 / 500 216 / 1000 324 / 1500	102 min dry	60 ±2	65 ±3	38 ±3	50 ±10$^{3)}$
		18 min rain	60 ±2	–	38 ±3	–
2	108 / 250 216 / 500 324 / 750 432 / 1000 540 / 1250	102 min dry	120 ±10$^{1)}$	65 ± 10$^{2)}$	38 ±3	50 ±10$^{3)}$
		18 min rain	120 ±10$^{1)}$	65 ± 10$^{2)}$	38 ±3	–

$^{1)}$ At higher set values, as a result of the ageing of the xenon lamps over time, there were increasing difficulties in maintaining the stability of the irradiance parameter, which had an average fluctuation of about 5 W/m^2, and at the end of the test it was necessary to reduce the irradiance dose to a level of 110 W/m^2, as the Xenotest 440 was unable to achieve the set irradiance level.
$^{2)}$ As a result of the ageing of the lamps during the test, an increasing difficulty in maintaining the surface temperature of the specimen was noted, which may be indicative of the increased proportion of infrared radiation due to the ageing of the xenon lamps, thus increasing the test temperature. The highest measured surface temperature value for the set irradiance level of 120 W/m^2 was 75°C, and the temperature was on average about 5°C higher than the set temperature.
$^{3)}$ Stabilisation time after raining cycle – approximately 30÷40 minutes.
[Authors' own elaboration].

Table 3

Testing methodology including the apparatus used

No.	**Test type**	**Reference document (method)**	**Apparatus**	**Notes**
1	Radiation resistance (UV+VIS+R)	EN ISO 16474-1 [14] EN ISO 16474-2 [11]	Xenon weathering instrument, Atlas GmbH Xenotest 440, XENOSENSIV RC 34 BST (Fig. 2)	Methodology: EN ISO 16474-2, section 7.3, table 3, method A; filters simulating daylight with BST surface temperature control (65°C) and radiation control in the wavelength range (300÷400) nm; irradiance: 60 W/m^2 and 120 W/m^2; temperature 38°C, relative humidity 50%, test periods: 102 min dry, 18 min rain; total radiant exposure: Table 2.
2	Evaluation of coating defects after ageing test (visual evaluation)	EN ISO 4628-1 [15] EN ISO 4628-2 [16] EN ISO 4628-3 [17] EN ISO 4628-4 [18] EN ISO 4628-5 [19] EN ISO 4628-6 [20]	Not applicable	Evaluation with unaided eye.

Table 3 cont.

No.	Test type	Reference document (method)	Apparatus	Notes
3	Determination of colour coordinates	ISO 7724-1 [21] ISO 7724-2 [22] ISO 7724-3 [23]	Konica Minolta CM2600D, CCSII spectrophotometric standards Lucideon Ltd (Fig. 3)	D/8° sphere spectrophotometer with diffuse illumination; measurements were made with and without gloss trap (SCI/SCE), using D65 illuminant, 10° observer (measurement range 360÷740 nm, sampling every 10 nm); colour coordinate results were determined using the CIELAB 1976 system for the parameters L* (brightness), a* (red-green), b* (yellow-blue) while colour differences were determined using the parameter ΔE*ab.
4	Determination of gloss	EN ISO 2813 [24]	Rhopoint IQ-S glossmeter (Fig. 4)	The instrument simultaneously measures in three gloss geometries 20°, 60°, 85°. The results for the universal 60° geometry were analysed.
5	Determination of hardness	EN ISO 1522 [25] PN-79/C-81530 [26]	TQC Sheen TI SP 0500 (Fig. 5)	A König and Persoz pendulum was used for the measurements and the measured hardness was related to the hardness against glass (calibration).

[Authors' own elaboration].

Fig. 2. Test stand for ageing samples according to EN 16474-2 [11]; xenon weathering instrument, Atlas GmbH Xenotest 440 [Pic. M. Garbacz]

Fig. 3. Test stand for determining colour according to EN ISO 7224-2 [22]; Konica Minolta CM2600D spectrophotometer, CCSII Lucideon Ltd spectrophotometric standards [Pic. M. Garbacz]

Fig. 4. Test stand for determining gloss in accordance with EN ISO 2813 [24]; Rhopoint IQ-S glossmeter [Pic. M. Garbacz]

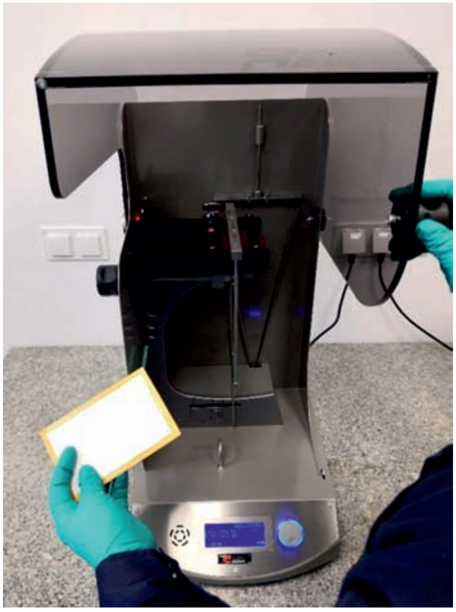

Fig. 5. Test stand for determining hardness according to EN ISO 1522 [25]; pendulum hardness tester TQC Sheen TI SP 0500 [Pic. M. Garbacz]

3. Test results and their interpretation

For the test, the Xenotest 440 ageing instrument, manufactured by Atlas GmbH, was used, equipped with two xenon lamps with a rotating sample drum to ensure uniform radiation distribution and full automation of the test (no need for manual sample rotation). An integrated BST 300÷400 nm radiometer was used to monitor radiation and surface temperature. In addition, checks and/or calibrations were made with an additional calibrated sensor during the measurement. The manufacturer of the instrument declares the life of the lamps (ensuring correct radiation distribution) at a setting of 60 W/m^2 to be 4,000 h (approx. 6 months), while at a setting of 120 W/m^2, the life of the lamps is almost shorten three times to 1,500 h (approx. 2 months). During the tests carried out at a setting of 60 W/m^2, no problems in achieving the dose of the set irradiance level or reduced lamp life were observed. Unfortunately, with an irradiance setting of 120 W/m^2, a relatively rapid decrease in lamp wear was noticeable and it was necessary to increase the tolerance of allowable deviations from the instrument's declared settings (for irradiance this was ±10 W/m^2, while for surface temperature it was ±10°C). No major difficulty was observed in maintaining the ambient temperature in the chamber for the set temperature of 38°C, but it was necessary to increase the airflow (cooling) to a maximum level of 2,500 RPM (rotations per minute of the fan). New lamps achieved the set parameters for several hundred hours (500 h on average), and a successive decrease in the stability of the irradiance level was observed during the progressive ageing test. At the same time, there was a noticeable increase in the surface temperature of the samples on average over the entire test period to a temperature 5°C higher than the set temperature (at the very end of the life of the lamps, the temperature reached a level around 10°C higher than the set temperature). This was probably due to the increase in IR radiation levels due to the ageing of the lamps. In each case, the completion of the conducted test was determined by the declared total radiant exposure (MJ/m^2) rather than the total length of the test. The test results presented in the article are the average of at least 3 samples from each system tested. All tests carried out according to the standards listed in Table 3 were performed within the scope of the laboratory's accreditation (AB 369) granted by the Polish Centre for Accreditation for compliance with the EN ISO 17025 standard [27].

3.1. Assessment of defects resulting from ageing in the laboratory

Table 4 summarises the results of the evaluation tests for the coating defects, assessed in accordance with the EN ISO 4628 series of standards. The specified expanded uncertainty for the results of the evaluation of the defects, after ageing, for an assumed significance level of α = 5% and a coverage factor of k = 2.0, is ±1 on the evaluation scale for the size and number of the defects evaluated according to the recommendations provided in the EN ISO 4628 series of standards [28].

Table 4

Summary of all tested paint systems for the evaluation of degradation of coatings according to the EN ISO 4628 series of standards

System No.	Irradiance 60 W/m² (radiant exposure: 324 MJ/m², *T* = 1500 h)					Irradiance 120 W/m² (radiant exposure: 324 MJ/m², *T* = 750 h)				
	Blistering EN ISO 4628-2	Rusting EN ISO 4628-3	Cracking EN ISO 4628-4	Flaking EN ISO 4628-5	Chalking EN ISO 4628-6	Blistering EN ISO 4628-2	Rusting EN ISO 4628-3	Cracking EN ISO 4628-4	Flaking EN ISO 4628-5	Chalking EN ISO 4628-6
I	0(S0)	Ri0	0(S0)	0(S0)	2	0(S0)	Ri0	0(S0)	0(S0)	2
II	0(S0)	Ri0	0(S0)	0(S0)	2	0(S0)	Ri0	0(S0)	0(S0)	2
III	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
IV	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
V	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
VI	0(S0)	Ri0	0(S0)	0(S0)	0 [1)]	0(S0)	Ri0	0(S0)	0(S0)	0 [1]
VII	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
VIII	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
IX	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
X	0(S0)	Ri0	0(S0)	0(S0)	0	0(S0)	Ri0	0(S0)	0(S0)	0
XI	0(S0)	Ri0	0(S0)	0(S0)	4	0(S0)	Ri0	0(S0)	0(S0)	4

[1)] Intense colour change (more matt, but no chalk marks on the tape).
[Authors' own elaboration].

Out of all 11 tested systems aged according to EN ISO 16474-2 [11] for radiant exposure of 324 MJ/m² (for irradiance levels of 60 W/m² and 120 W/m²), none showed coating changes in the form of blistering, rusting, cracking or flaking. System I, II and XI (chalking) and VI (intense colour change) showed the same changes in relation to the set radiant exposure for different irradiance values.

3.2. Change in gloss and colour after ageing tests using different irradiance values

Before ageing, the average gloss and colour values were determined. The results are summarised in Table 5. Table 6, however, shows the mean values of the gloss difference and the percentage of gloss retention with respect to the baseline value before laboratory ageing for an angle of gloss measurement of 60° determined according to EN ISO 2813 [24]. The same table presents the average colour differences ΔE* (CIELAB 1976) determined according to EN ISO 7724-3 [23] using a spherical spectrometer with a measuring geometry of d:8°. Differences for gloss and colour values were determined against the values summarised in Table 5.

Figures 6, 7, 8 illustrate a collective, specular graphical summary of the results (for a given tested property according to Table 6) of the two different irradiance values of 60 W/m² and 120 W/m² for all 11 paint systems tested as a function of the constant radiant exposure values at which the measurements were taken. For the mean results of the change in the selected property as a function of radian exposure level, the equations of the lines and the corresponding coefficients of determination were determined, assuming that the intercept was not significant (the equations of the lines were derived from the origin of the coordinate system). In the case of a simple regression line for the entire range of radiant exposures tested (0 MJ/m², 108 MJ/m², 216 MJ/m², 324 MJ/m², 432 MJ/m², 540 MJ/m²), the line was drawn through the mean results without including System XI in the calculations, as it was only tested for the first 3 radiant exposure levels.

The specified uncertainty of the ageing test for an assumed confidence level of 95% and a coverage factor of $k = 2$ is 8% [28] (determined based on literature data).

The estimated expanded uncertainties of the gloss and colour measurements at the same 95% confidence level and for a coverage factor of $k = 2$ are:

- for gloss measurements ±4 [GU] for all 3 measurement geometries [29],
- for colour measurements of achromatic samples: ±0.64 for L*, ±0.19 for a*, ±0.32 for b* and ±0.43 for ΔE* [30],
- for colour measurements of chromatic samples: ±0.80 for L*, ±0.80 for a*, ±0.89 for b* and ±0.83 for ΔE* [30],

whereby the expanded uncertainty values for the gloss test were determined based on measurements with a single instrument for 3 different operators and for the same samples, and the expanded uncertainty for the colour test was determined with reference to ceramic reference standards certified by an accredited body.

Table 5

Gloss values (20°, 60°, 85°) and colour coordinates (CIELAB 1976) determined before the ageing test (reference values) for the given paint system

System No.	Average specified gloss values (EN ISO 2813) [24]				Average specified colour coordinates CIELAB 1976 (EN ISO 7724-2 [22])						
	Type of coating	20° [GU][3)]	60° [GU][3)]	85° [GU][3)]	Colour	SCI[1)]			SCE[2)]		
						L*[4)]	a*[4)]	b*[4)]	L*[4)]	a*[4)]	b*[4)]
I	glossy	89	93	98	blue	37.9	–5.3	–28.3	28.6	–7.9	–35.4
II	glossy	87	92	98	blue	38.0	–5.6	–28.0	28.8	–8.4	–35.1
III	glossy	89	94	97	light grey / white	82.2	–1.1	–2.8	79.6	–1.2	–2.7
IV	glossy	89	92	100	dark grey	30.1	–0.2	–2.0	15.5	–0.4	–3.1
V	glossy	85	91	97	blue	37.0	–3.8	–27.7	27.5	–5.8	–35.0
VI	glossy	74	86	95	red	44.4	49.1	29.3	38.0	57.6	47.7
VII	glossy	72	92	96	dark grey	34.6	–1.4	–2.5	23.5	–2.3	–3.2
VIII	glossy	84	92	95	light grey	58.1	–3.2	–3.6	53.7	–3.6	–3.7
IX	glossy	86	90	95	light grey / white	65.8	–1.3	–0.2	62.2	–1.4	0.0
X	glossy	71	94	95	light purple	71.7	1.2	–10.7	68.4	1.2	–11.2
XI	Medium glossy	13	52	58	light purple	72.9	1.1	–9.9	70.2	1.2	–10.2

1) SCI (Specular Component Included) – it includes the specular and diffused reflected light.
2) SCE (Specular Component Excluded) – it excludes specular reflected light.
3) Refers to the measuring geometry at which the measurement is performed (incident beam axis and optical axis of the receiver perpendicular to the tested surface);
4) The colour system co-ordinates L*, a*, b* mathematically describe a given colour in the CIELAB 1976 colour space. The scales of the a-axis (green to magenta) and b-axis (blue to yellow) extend between –150 and +100 and –100 and +150 values, while the L* axis describes the brightness of the colour within the 0 to 100 value.
[Authors' own elaboration].

Table 6

Mean values of gloss difference and % gloss retention in 60° geometry and mean colour change ΔE* (CIELAB 1976) for tests conducted in an ageing chamber with xenon lamps using two different irradiance levels

System No.	H [MJ/m²]	Gloss – EN ISO 2813 [24]		Colour – EN ISO 7724-3 (ΔE* – CIELAB 1976) [23]			
		Irradiance 60 W/m² for the 300÷400 nm range	Irradiance 120 W/m² for the 300÷400 nm range	Irradiance 60 W/m² for the 300÷400 nm range		Irradiance 120 W/m² for the 300÷400 nm range	
				SCI	SCE	SCI	SCE
		Δ60°; change [GU] / % in gloss retention with respect to the initial value		$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$, where ΔL^*, Δa^*, Δb^* the difference of the measured colorimetric indicators before and after a set level of radiant exposure			
I	108	–1 / 98.9	–2 / 97.8	1.6	2.2	1.5	1.8
	216	–44 / 52.7	–16 / 82.8	1.3	8.3	1.9	4.1
	324	–82 / 11.8	–78 / 16.1	1.6	12.2	1.3	11.6
	432	–	–88 / 5.4	–	–	2.1	13.3
	540	–	–91 / 2.2	–	–	5.0	15.7
II	108	0 / 100.0	–2 / 97.8	1.6	2.1	1.4	1.7
	216	–40 / 56.5	–16 / 82.6	1.4	7.9	1.6	4.4
	324	–79 / 14.1	–71 / 22.8	1.7	11.8	1.3	11.5
	432	–	–85 / 7.6	–	–	2.1	13.0
	540	–	–90 / 2.2	–	–	4.3	14.8
III	108	0 / 100.0	–1 / 98.9	0.5	0.5	0.3	0.3
	216	1 / 101.1	–1 / 98.9	0.6	0.7	0.4	0.4
	324	1 / 101.1	–2 / 97.9	0.7	0.8	0.5	0.5
	432	–	–1 / 98.9	–	–	0.6	0.6
	540	–	–8 / 91.5	–	–	0.7	0.7
IV	108	1 / 101.1	–1 / 98.9	0.2	0.3	0.2	0.5
	216	1 / 101.1	0 / 100.0	0.3	0.4	0.2	0.4
	324	–1 / 98.9	–2 / 97.8	0.3	0.2	0.3	0.5
	432	–	–3 / 96.7	–	–	0.2	0.2
	540	–	–8 / 91.3	–	–	0.9	0.5
V	108	–1 / 98.9	–1 / 98.9	0.6	0.7	0.4	0.8
	216	–3 / 96.7	0 / 100.0	0.9	0.9	0.7	0.9
	324	–3 / 96.7	–1 / 98.9	1.0	1.2	1.0	1.1
	432	–	–2 / 97.8	–	–	1.1	1.4
	540	–	–6 / 93.4	–	–	1.3	1.5
VI	108	–6 / 93.0	–4 / 95.3	2.6	7.5	2.1	4.2
	216	–14 / 83.7	–8 / 90.7	3.6	11.9	3.4	8.6
	324	–17 / 80.2	–11 / 87.2	5.3	15.6	4.6	11.8
	432	–	–12 / 86.0	–	–	5.6	14.3
	540	–	–18 / 79.1	–	–	9.1	22.4
VII	108	–4 / 95.7	–2 / 97.8	0.1	0.2	0.0	0.5
	216	–5 / 94.6	–1 / 98.9	0.1	0.6	0.1	0.4
	324	–6 / 93.5	–2 / 97.8	0.0	1.0	0.1	0.5
	432	–	–5 / 94.6	–	–	0.2	0.8
	540	–	–10 / 89,1	–	–	0.7	1.3

Table 6 cont.

VIII	108	–1 / 98.9	–2 / 97.8	0.7	0.9	0.8	0.9
	216	–2 / 97.8	–1 / 98.9	1.0	1.3	1.1	1.2
	324	–2 / 97.8	–2 / 97.8	1.3	1.5	1.3	1.4
	432	–	–4 / 95.7	–	–	1.5	1.6
	540	–	–5 / 94.6	–	–	1.8	1.7
IX	108	–1 / 98.9	–1 / 98.9	0.5	0.6	0.4	0.4
	216	–2 / 97.8	0 / 100	0.6	0.7	0.6	0.7
	324	–1 / 98.9	–1 / 98.9	0.9	1.0	0.7	0.8
	432	–	–2 / 97.8	–	–	0.8	0.8
	540	–	–7 / 92.2	–	–	1.1	0.9
X	108	–2 / 97.9	–1 / 98.9	1.4	1.5	1.5	1.6
	216	–5 / 94.7	0 / 100.0	2.4	2.6	2.4	2.6
	324	–13 / 86.2	–5 / 94.7	3.9	4.3	3.8	4.1
	432	–	–10 / 89.4	–	–	5.4	5.7
	540	–	–6 / 93.6	–	–	8.0	8.3
XI	108	–5 / 90.4	0 / 100.0	0.6	0.6	0.6	0.5
	216	–25 / 51.9	–9 / 82.7	0.8	2.3	0.9	2.1
	324	–39 / 25.0	–34 / 34.6	2.2	4.1	2.0	3.7

[Authors' own elaboration].

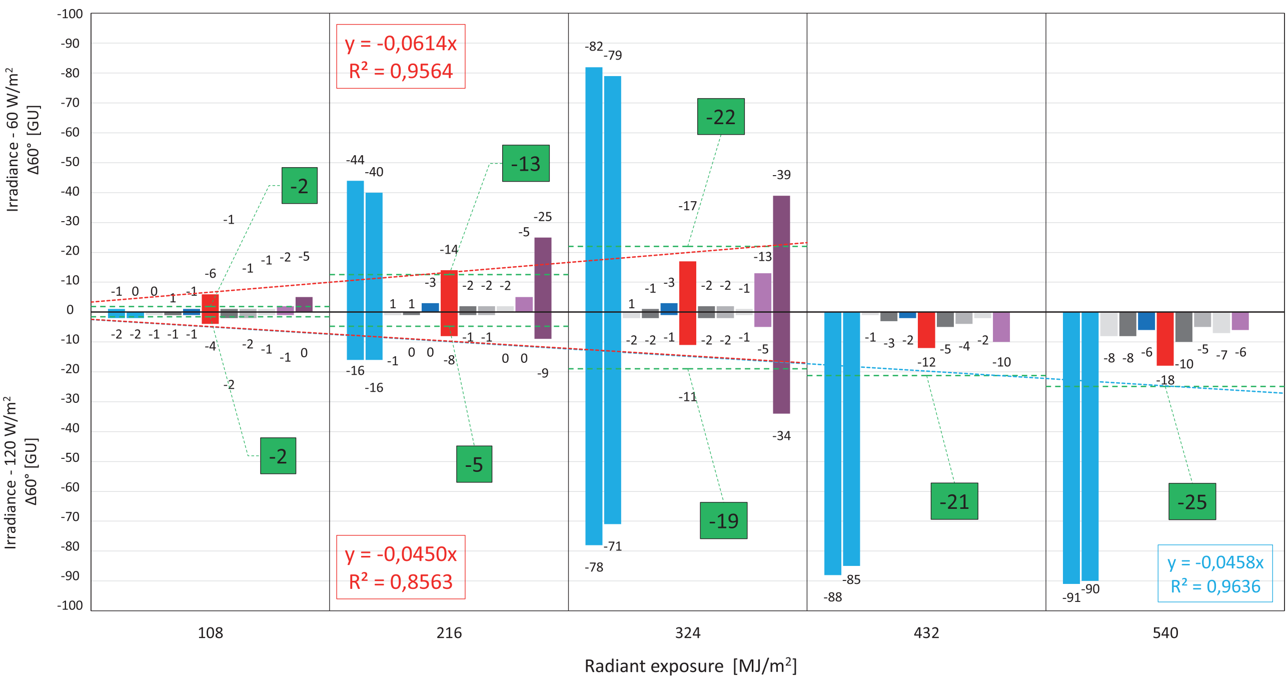

Fig. 6. Graphical presentation of average gloss changes in the 60° geometry, determined according to EN ISO 2813 [24] for two different doses of irradiance (60 W/m² and 120 W/m²) and different radiant exposures (108 MJ/m², 216 MJ/m², 324 MJ/m², 432 MJ/m², 540 MJ/m²) for the tested paint systems [own elaboration]

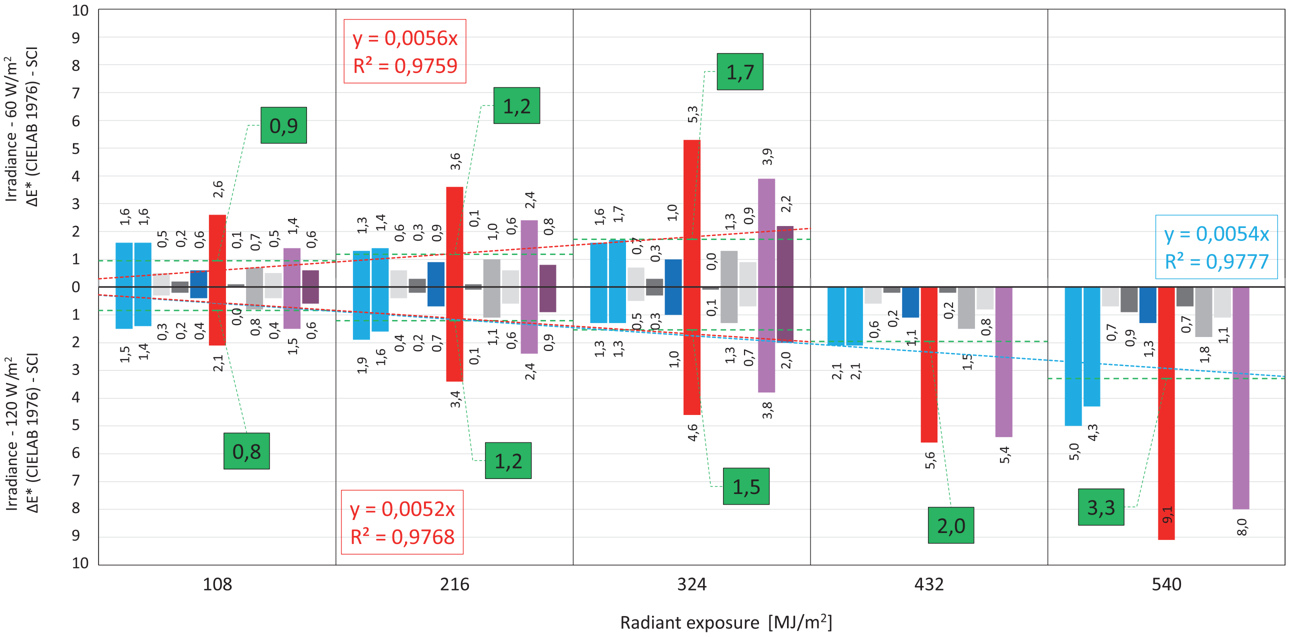

Fig. 7. Graphical presentation of average color changes ΔE* (CIELAB 1976) determined according to EN ISO 7724-3 [23] for two different doses of irradiance (60 W/m² and 120 W/m²) and different radiant exposures (108 MJ/m², 216 MJ/m², 324 MJ/m², 432 MJ/m², 540 MJ/m²) for the tested paint systems using the SCI mode (Specular Component Included) [own elaboration]

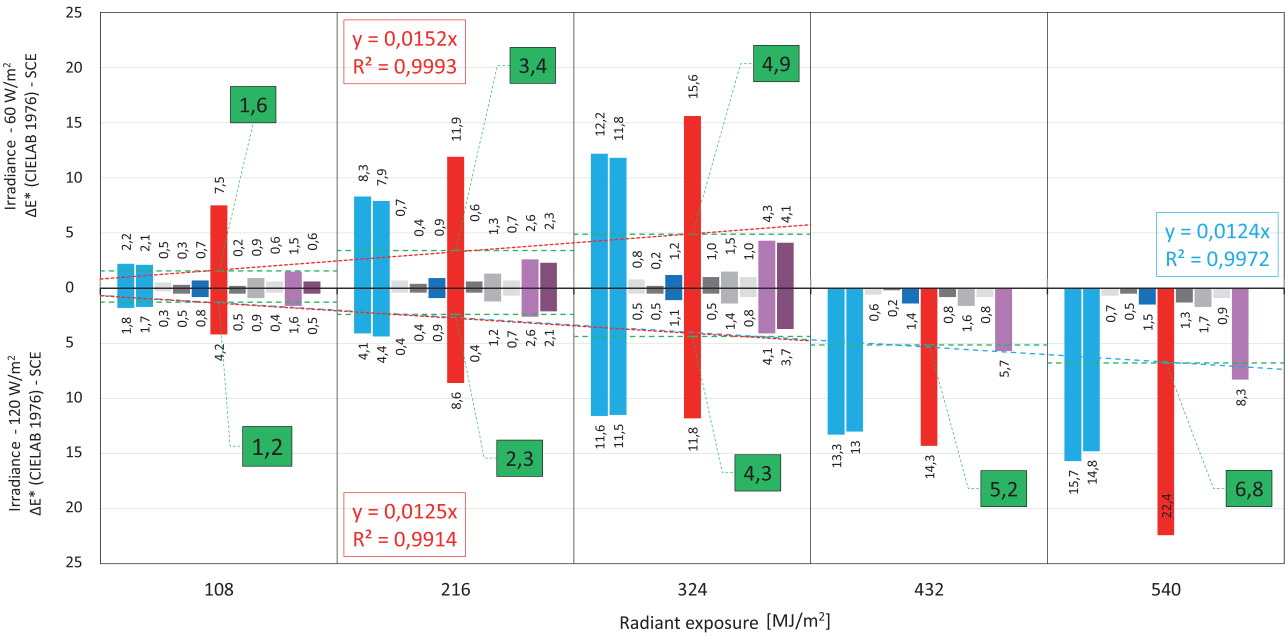

Fig. 8. Graphical presentation of average color changes ΔE* (CIELAB 1976) determined according to EN ISO 7724-3 [23] for two different doses of irradiance (60 W/m² and 120 W/m²) and different radiant exposures (108 MJ/m², 216 MJ/m², 324 MJ/m², 432 MJ/m², 540 MJ/m_2) for the tested paint systems using the SCE mode (Specular Component Excluded) [own elaboration]

From the data derived from the graphs (Figs. 6, 7, 8), it is possible to estimate the average percentage agreement of the results (or the percentage of difference) of the coating property tested in the context of the different irradiance doses applied. The information obtained in this way is the easiest indicator to interpret, making it possible to assess in a general way whether an increased irradiance dose has a significant effect on the final results of the selected changes in gloss or colour properties of the paint coatings. Knowing that the slope of a linear equation tells you how fast one variable follows another, you can estimate the percentage agreement of the results (or the percentage of difference) of curve sketching, i.e. the percentage agreement of the average rate of change occurring as a function of radiant exposure for the selected coating property. Theoretically, at best, the lines should have the same curve and the same rate of change, i.e. the same slope. In typical mathematical terms, a measure of the rate of change in the value of any function is described by the derivative of the function at a given point, and in the case of a linear function, this is a constant value (the slope of the line) equal to the tangent of the angle of inclination of the line that is the graph of that function to the OX axis. Table 7 summarises the slopes of the determined lines (derivation from the origin of the coordinate system, intercept b = 0) and shows the mean percentage fit of the function (fit of the mean rate of change) for an increased irradiance dose of 120 W/m^2 relative to the results determined up to a range of 324 MJ/m^2 for an irradiance of 60 W/m^2.

On the basis of the calculation data in Table 7 (columns 5 and 6), it was found that a relatively high average percentage agreement was achieved for the function of selected changes in paint system properties, i.e. gloss and colour, as a function of irradiance at two different value for fixed radiant exposure levels (60 W/m^2 vs. 120 W/m^2). A much more accurate fit of the mean results was achieved for colour measurements in SCI mode which includes both the specular and diffused reflected light, where a mean fit of 95% was achieved. The situation is much worse for gloss measurements (measurements carried out in 60° geometry) and colour measurements, which excludes specular reflected light (SCE mode) into account, where in the first case an average match of 74% was achieved, while in the second case the value averaged 82%. This result shows that the exposure at a level of 60 W/m^2 was more destructive than at a level of 120 W/m^2 for the same radiant exposure level of 324 MJ/m^2. Continuing the test up to radiant exposure of 540 MJ/m^2 for an irradiance of 120 W/m^2 hardly changed the fit of the linear function (and thus the slope of the line) determined from the larger number of measurement points (retained nature of the linear change).

3.2. Change in hardness (pendulum damping method) after ageing tests using different irradiance values

After the ageing test with two different irradiance settings, in addition to a visual assessment in terms of properties such as gloss and colour, an analysis was made of the change in the physical and mechanical property of hardness. Table 8 summarises the results of hardness tests on the tested paint systems evaluated in accordance with EN ISO 1522 [25] using the pendulum damping method with Konig and Persoz pendulums (hardness against a glass constant). The table contains the experimental results before and after ageing using two different irradiance values (60 W/m^2 and 120 W/m^2) and for different levels of total radiant exposure (324 MJ/m^2 and 540 MJ/m^2), while Figure 9 graphically summarises the data from Table 8 for easier interpretation.

Table 7

Summary of the mean percentage fit of the linear function in the y = ax relationship determined for the dose-dependent changes in selected properties of the tested paint systems in the context of the different irradiance levels tested

1	2	3	4	5	6
Tested property	**Slope of line a determined to a range of 324 MJ/m^2 for irradiance of 60 W/m^2**	**Slope of line a determined to a range of 324 MJ/m^2 for irradiance of 120 W/m^2**	**Slope of line a determined to a range of 540 MJ/m^2 for irradiance of 120 W/m^2**	**Average percentage fit of the function (rate of change) relative to the results specified up to a range of 324 MJ/m^2 for an irradiance of 60 W/m^2 (standard setting – column 2)[1)]**	
				3 vs. 2	**4 vs. 2**
Gloss / Δ60° [GU]	–0.0614	–0.0450	–0.0458	73%	75%
Colour / ΔE* SCI	0.0056	0.0052	0.0054	93%	96%
Colour / ΔE* SCE	0.0152	0.0125	0.0124	82%	82%

[1)] Calculated from the dependence of the quotient of the slope of the line for an increased irradiance dose of 120 W/m^2 versus the slope of the line determined for a standard irradiance setting of 60 W/m^2 multiplied by 100% [own elaboration].

Table 8

Average hardness values of the tested paint systems determined according to EN ISO 1522 [25] before and after ageing tests

System No.	Relative hardness (against glass) according to ISO 1522 [25]							
	König pendulum				Persoz pendulum			
	No ageing	Samples aged using Xenotest according to EN ISO 16474-2 [11]			No ageing	Samples aged using Xenotest according to EN ISO 16474-2 [11]		
		Irradiance 60 W/m^2 for the 300÷400 nm range 324 MJ/m^2	Irradiance 120 W/m^2 for the 300÷400 nm range 324 MJ/m^2	Irradiance 120 W/m^2 for the 300÷400 nm range 540 MJ/m^2		Irradiance 60 W/m^2 for the 300÷400 nm range 324 MJ/m^2	Irradiance 120 W/m^2 for the 300÷400 nm range 324 MJ/m^2	Irradiance 120 W/m^2 for the 300÷400 nm range 540 MJ/m^2
I	0.33	0.44	0.40	0.38	0.40	0.45	0.43	0.41
II	0.35	0.45	0.46	0.44	0.43	0.46	0.50	0.45
III	0.33	0.58	0.56	0.54	0.37	pendulum sliding	pendulum sliding	0.51
IV	0.55	0.73	0.73	0.76	pendulum sliding	pendulum sliding	0.72	0.70
V	0.29	0.59	0.60	0.58	0.32	0.58	0.57	0.56
VI	0.41	0.48	0.55	0.47	pendulum sliding	0.51	0.54	0.43
VII	0.08	0.12	0.16	0.11	0.08	0.12	0.17	0.14
VIII	0.07	0.08	0.12	0.10	0.07	0.09	0.11	0.11
IX	0.17	0.45	0.53	0.51	0.19	0.45	0.48	0.43
X	0.41	0.53	0.55	0.55	0.40	pendulum sliding	0.48	0.45
XI	0.52	0.53	0.56	–	0.47	0.49	0.49	–
Average:	**0.32**	**0.45**	**0.47**	–	–			
Average[1]:	**0.30**	**0.45**	**0.47**	**0.44**	–			

[1] Average without including System XI, which was not assessed at an irradiance setting of 120 W/m^2 for the 300÷400 nm range and after a radiant exposure of 540 MJ/m^2 [own elaboration].

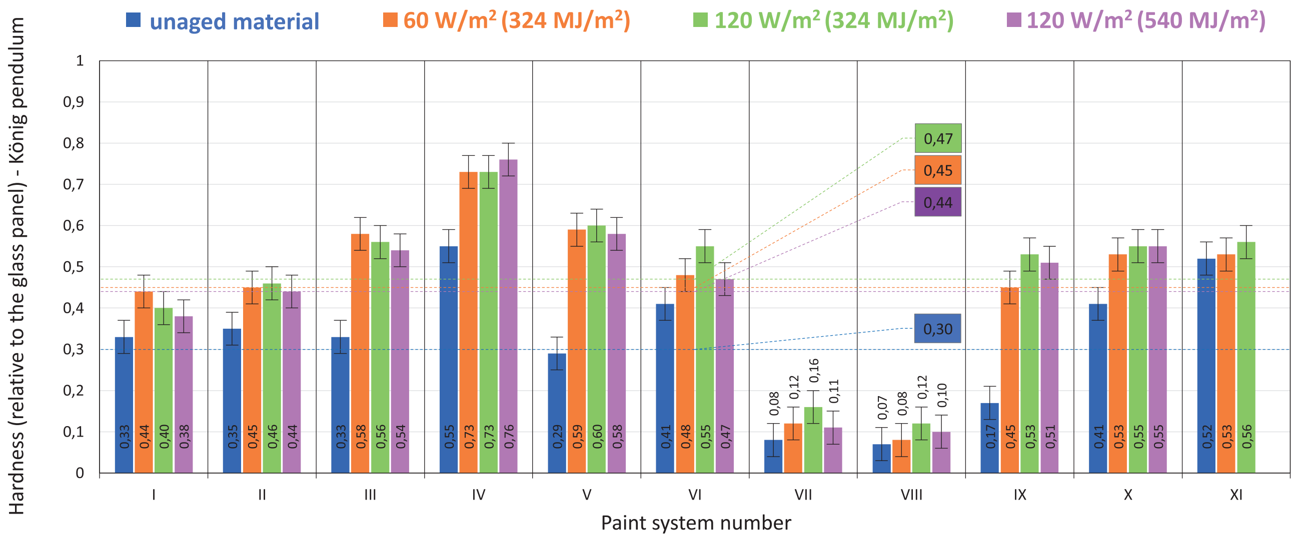

Fig. 9. Graphical presentation of the results presented in Table 8 for the average hardness values of the tested paint systems tested according to EN ISO 1522 [22] for tests carried out in the aging chamber with xenon lamps according to EN ISO 16474-2 [9] with the use of irradiance values of 60 W/m^2 and 120 W/m^2 for the wavelength range 300÷400 nm [own elaboration]

The specified expanded uncertainty at a confidence level of 95% and a coverage factor of $k = 2.0$ for the pendulum hardness method according to EN ISO 1522 [25] is ±0.04 of the hardness relative to the glass constant for the König pendulum and 6% of the mean value relative to the glass constant for the Persoz pendulum [31].

Due to the occurrence of the Persoz "pendulum sliding" phenomenon for many of the cases tested, the analysis of the experimental results for the hardness parameter of the paint systems tested and the effect of ageing on this parameter was based on the results obtained using a König pendulum (weight 200 g).

An important finding is that, following the ageing test of the paint systems, an increase in hardness was observed for practically all the systems tested. The average hardness value for tests carried out using Xenotest according to EN ISO 16474-2 [11] increased on average from 0.32 to 0.45 (a 41% increase) for samples aged at an irradiance level of 60 W/m^2 and a total radiant exposure of 324 MJ/m^2. A similar level of hardness increase to 0.47 (a 47% increase) was achieved for samples aged at an irradiance level of 120 W/m^2 with the same total radiant exposure. Further irradiation of the samples had no significant effect on the subsequent increase in hardness of the systems, where a similar average result of 0.44 of glass hardness was obtained as an average for the 10 systems (system XI not included) for a total radiant exposure of 540 MJ/m^2 measured for a wavelength of 300÷400 nm.

4. Summary and conclusion

This article presents and analyses the results of laboratory tests to evaluate the laboratory light resistance of paint systems used in the railway industry. A comparative study of coating properties such as gloss, colour and hardness was carried out in the context of applying identical total radiant exposures, but for two different irradiance level (60 W/m^2 and 120 W/m^2). Mainly 3 irradiance values of 108 MJ/m^2, 216 MJ/m^2 and 324 MJ/m^2 were considered, for which complete results were obtained.

The lowest conformity of results for the two different irradiance values used at the different radiant exposure levels was obtained for the coating property of gloss. The agreement in this case for the average rate of change of the 11 systems tested was 74%, but it should be noted that this may be an underestimation or overestimation (cannot be clearly stated), due to the fact that a large proportion of the paint systems tested were resistant to artificial laboratory radiation (for the range of radiant exposure level achieved) and the loss of this coating feature. The direction of the observed changes was negative, i.e. the values achieved for an irradiance of 120 W/m^2 obtained lower gloss loss values than at the standard irradiance setting derived and suggested in EN ISO 16474-2 [11] of 60 W/m^2. The reason for this phenomenon may be a reduction in test time and thus a lower number of cycles and a lower temporal contribution of secondary destructive factors in the form of temperature and humidity (the susceptibility of the material to these components). The reason may also be that xenon lamps wear out faster with increased irradiance and shift the radiation contribution towards longer wavelengths and thus wavelengths carrying less energy necessary and used to destroy chemical bonds. Commercially available radiation detectors for ageing tests involving xenon lamps measure the total radiation level mostly in the long wavelength bandwidth range, e.g. 300÷400 nm or 300÷800 nm, and do not provide information on the exact radiation distribution.

In the case of colour measurements carried out in two different configurations, i.e. with the specular component included and excluded (gloss trap), an average agreement of the results (rate of change) of 95% was achieved in the first case, while an average agreement of only 82% was achieved in the second case, which takes into account the influence of gloss on the perceived colour impression. Again, as in the case of gloss evaluation, the average rate of change of the tested paint systems was lower for a higher irradiance level than for the standard settings proposed in the standard.

The hardness of the aged paint systems, measured using the pendulum method, showed very high compliance of results for the two irradiance levels used (60 W/m^2 vs. 120 W/m^2) for the same radiant exposure of 324 MJ/m^2 where the average difference for the 11 tested systems was only 0.02 hardness, representing an average match of the results of 95% (a result included in the specified uncertainty of the method). Further ageing of the samples to 540 MJ/m^2 did not significantly affect the further increase in hardness.

Based on the data obtained, it can be concluded that in the case of polyurethane or acrylic coatings (top protection of the tested systems), an irradiance dose increased to at least 120 W/m^2 at a wavelength of 300÷400 nm results in similar values of coating changes, such as gloss, colour or hardness, depending on the total irradiation dose and can be successfully implicated in the testing of such materials in order to reduce the time of the ageing test. However, it must be kept in mind that compliance with regard to measurements, especially gloss, can be significantly underestimated.

When conducting studies with increased irradiance levels, it should be noted that intensifying the radiation in xenon lamp instruments proportionally shortens their service life. The operating costs of the instrument are then higher or at most remain similar (much depends on the type of instrument used) compared to tests conducted at lower irradiance settings, but over a longer period of time. In this case, however, test time is saved and there is the possibility of making the apparatus available more quickly, which is often invaluable and more important than financial considerations alone. When deciding to increase the dose of the irradiance level, a very important step is to control the temperature conditions of the sample environment and the surface temperature or humidity. This is done indirectly by replacing the lamps with new ones more frequently when too large a deviation from the specified set values and recorded parameters is reached by the laboratory ageing instrument. An increasing number of instruments on the market allow for complete control of the ageing process in the form of a built-in controller with the possibility of programming specific parameter settings, permitted deviations, as well as maximum exposure time, so that there is no need to constantly monitor the operation of the instrument (the Xenotest 440 used in this study also had these functions, so control of the ageing process was greatly facilitated). In the event of failure to achieve the set parameters within the set time and range – the instrument stops the test (all data and measurement errors are recorded in the instrument's memory or on a memory card), which allows full repeatability of this type of testing and reduces the impact of the variability of the degradation mechanisms involved in the ageing of the samples.

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