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## **NANOCOMPOSITE WOOD COATING FILM PARAMETER STABILITY AFTER SHORT TERM INTENSIVE UV+IR IRRADIATION**

*A study of the colour parameters, gloss retention, illuminance under glass plate and surface hardness of clear waterborne acrylic coating films containing inorganic nanoparticles during a UV+IR ageing test was carried out. Two different coating mixtures with CeO<sub>2</sub> and ZnO nanoparticles (5%) were prepared and applied on clear glass plates. Intensive UV+IR irradiation of samples of film coatings was divided into 6 cycles and performed for 30 hours in total. It was found that inorganic nanoparticles had a significant impact on the overall colour stability of the coating films as well as on the coating gloss retention. Inorganic nanoparticles influenced the transparency properties and the coating film surface hardness.*

**Keywords:** clear acrylic coating film, inorganic nanoparticles, UV+IR irradiation, colour stability, gloss retention, hardness

### **Introduction**

In order to reduce the surface discolouration of outdoor wood products, inorganic UV (ultraviolet light) absorbers can be incorporated into the structure of various clear organic wood coatings [Rowell 2012]. It is known that up to 5% of inorganic nanoparticle additives can significantly increase the photostability of wood coating parameters [Cristea et al. 2010].

Waterborne wood coatings are resistant to colour changes and are more environmentally friendly to use [Talbert 2008]. Despite that, waterborne coatings are denoted by higher surface stress compared to solvent-based composition coatings [Wicks et al. 2007]. With the objection to the reduction of the UV impact on the stability of acrylic coatings, metal oxide nanoparticle additives can

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be used [Cristea et al. 2010; Schaller et al. 2012]. This way, resistance against UV+IR irradiation (which causes yellowing and total discolouration) can also be improved [Krystofiak et al. 2012].

The size of nanoparticles is important regarding the dissipation of short-wave radiation and absorbance of a wider range of UV spectrum [Saadat-Monfareda et al. 2012]. The smaller size yields better intensity, transparency, higher surface energy and photocatalytic activity of the nanoparticles [Faure et al. 2013; Khan et al. 2013]. The most widely used compounds are CeO<sub>2</sub>, ZnO and TiO<sub>2</sub> nanoparticles absorbing UV rays by virtue of their electronic structure [Kubacka et al. 2012], e.g., ZnO nanoparticles absorb the 250-400 nm part of the UV spectrum [Hong et al 2009; Talam et al. 2012]. When UV radiation energy increases, electron-hole pairs start to form. This photo-oxidation reaction causes the formation of free radicals which influence the decomposition of organic molecules [Wang et al. 2009]. CeO<sub>2</sub> nanoparticles also absorb UV radiation and have lower photocatalytic activity than ZnO [Lima et al. 2012; Herrling et al. 2013], but this type of nanoparticle results in discolouration (yellowing) of the coating [Cui et al. 2007; Tessier et al. 2008]. Cristea et al. [2012] observed that ZnO nanoparticles retain the colour and the gloss stability for a longer UV radiation period. Meanwhile, Cayton et al. [2010] discovered that an increase in the concentration of ZnO nanoparticles also improves the absorbance of UV rays at 375 nm wavelength. Adding 1% of ZnO nanoparticles to the coating structure increases the UV light absorbance up to 81%; meanwhile, with the use of 3% of ZnO nanoparticles, the absorbance increases 1.2 times as well – yet, as a result, the white undertone for the coating dramatically increases too (up to 4 times), whereas the transparency of the coating film highly reduces. It was confirmed by Lowry et al. [2008] that upon increasing the number of inorganic nanoparticles into the coating structure, resistance against UV rays also improves. With the change of the concentration of the nanoparticles in acrylic wood coating films, the elasticity of the nanocomposite coating also changes [Forsthuber et al. 2013].

There is little scholarly literature available which considers the ageing method, focusing on the significance of the additional thermal influence of infrared radiation on coating films. Due to this, the recommended high concentration (5%) of CeO<sub>2</sub> and ZnO nanoparticles was chosen. The aim of this work is to evaluate such properties of nanocomposite waterborne clear acrylic wood coating films as colour and gloss stability. To determine the changes in the coating transparency and hardness during intensive UV+IR irradiation.

## Materials and methods

For our research, the following commercial clear waterborne acrylic wood coating was chosen: viscosity 107-113 KU (by Stormer), solid content 38% ±1% by weight, specific gravity 1020 kg/m<sup>3</sup> ±30 kg/m<sup>3</sup>, gloss 27-33 by Gardner 60°.

Two different coating mixtures containing inorganic nanoparticles were produced by using the mechanical mixing method. Coating samples without inorganic nanoparticles (AK) were selected as a reference group. The following types of coating mixtures were used: AKZ – an AK coating containing 5% of ZnO nanoparticle dispersion (particle size: 20 nm); AKC – an AK coating containing 5% of CeO<sub>2</sub> nanoparticle dispersion (particle size: 10 nm).

In advance, prepared mixtures of the coatings were applied onto clear glass plates with dimensions of 45 × 45 × 5 mm by using a special film applicator frame. The wet film thickness did not exceed 200 µm. After the application of the coatings, the samples were conditioned at a temperature of 23°C ±2°C and a relative humidity of 50% ±5% for 72 hours. In total, five samples for each coating system were prepared.

### Ultraviolet light combined with infrared radiation (UV+IR) ageing test

For the irradiation test, an ultraviolet light (UV) and infrared radiation (IR) quartz lamp (UV+IR) system was used according to the Standard of the Republic of Poland No. [PN-88/F-06100/07:1988]. The radiation energy of the quartz lamp (model *Famed*, type *VT-4*) did not exceed 740 W. The distance between the coating samples and the quartz lamp was 400 mm. The irradiation process was divided into 6 cycles of 0, 1, 3, 6, 10, 20 and 30 hours.

### Colour measurements

Colour measurements were performed in five constant spots per sample according to the CIE  $L^*a^*b^*$  system. In order to evaluate the colour change, a *DATACOLOR 600* reflection spectrophotometer and the white background were used. For the measurements,  $L^*$ ,  $a^*$ ,  $b^*$  coordinates were chosen, where  $L^*$  represents the lightness parameter within the range from white (100) to black (0), parameters  $a^*$  and  $b^*$  being  $a^*$  red (+) to green (–) and  $b^*$  yellow (+) to blue (–).  $L^*$ ,  $a^*$ ,  $b^*$  values were used so that to calculate colour change  $\Delta E^*$  as a function of UV+IR irradiation duration with the following equation (1):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  are the differences between the initial and the final values after UV+IR irradiation of  $L^*$ ,  $a^*$ ,  $b^*$  parameters, respectively.

### Gloss measurements

The gloss values of the coating films were determined in five sectors by using a portable photoelectric gloss meter *PICO GLOSS 503* by Gardner [ISO 2813:2014] at 60° incidence angle. For the measurements, a special template and the white background were used. Gloss retention was calculated as the percentage change in the gloss values during the UV+IR ageing test respective to the initial values.

## Illuminance measurements and transparency evaluation

The illuminance under the glass plate parameter, which describes the change of the property of transparency in accordance to the size of illumination was evaluated in five measurement spots per sample by using a special measurement stand consisting of a light source and a light flow meter *Luksomierz* type *L-02*.

## Hardness measurements

The surface hardness gradient of the coating films was determined by using a *Persoz* pendulum damping tester (model *299/300*) by taking into account the numerical values of oscillation in terms of the relevant ISO standard [ISO 1522:2006]. For each sample, two measurement sectors were selected.

## Results and discussion

The type of inorganic nanoparticles in the coating mixture influenced  $L^*$ ,  $a^*$ ,  $b^*$  colour parameters of the initial coating film as well as  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  parameters during intensive UV+IR irradiation (see table 1 and table 2). The sample group containing 5% ZnO nanoparticles yielded a slightly light undertone to waterborne clear acrylic coating films; whereas CeO<sub>2</sub> (5%) delivered a yellow undertone. Similar results of nanoparticle impact on the initial colour of 2K polyurethane clear coating were observed by Schaller et al. [2012]. During intensive 30 h of UV+IR irradiation, the smallest change of  $\Delta L^*$  was found in the AKZ group, where the lightness parameter was 1.5-1.9 times more stable in comparison to other examined sample groups. Intensive UV+IR irradiation caused the trend of the rise of the green colour ( $\Delta a^*$ ) parameter. After 6 hours of irradiation,  $\Delta a^*$  parameter started to increase up to constant values, yet, no change to the red shift was observed. Higher stability of  $\Delta a^*$  was determined in the AKZ group (up to 5.6 times). It was noticed that during the first 6 hours of irradiation, the coating films featured an intensive peak to the yellow shift ( $\Delta b^*$ ), while only ZnO nanoparticles exerted significant influence on the control of the stability of the coating trend shift to the yellowish hue (up to 3.9 times). From the 6<sup>th</sup> hour of the UV+IR irradiation test onwards, the  $\Delta b^*$  parameter started to stabilize across all the sample groups.

Intensive UV+IR irradiation time and inorganic nanoparticle additives had an impact on the overall colour change of the coating films (fig. 1). The highest values of the  $\Delta E^*$  parameter after 30 hours of irradiation were observed in the AK group where the difference between the overall colour change of the clear coating film samples group and AKZ was 2.6 times higher, and, in comparison with the AKC sample group, it was 1.3 times higher. Despite that, both coating groups with inorganic nanoparticles showed promising colour protection against

**Table 1. Colour parameter  $L^*$ ,  $a^*$ ,  $b^*$  mean values before UV+IR irradiation test**

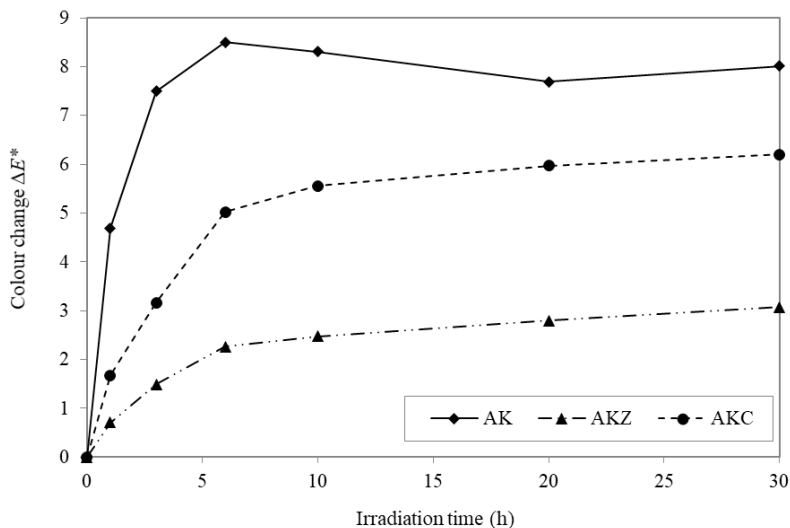
Sample group	Additive	Parameter	Non-irradiated
AK	–	$L^*$	80.89 (0.73)
		$a^*$	-0.85 (0.25)
		$b^*$	-5.99 (0.97)
AKZ	ZnO (5%)	$L^*$	80.19 (0.57)
		$a^*$	-1.15 (0.12)
		$b^*$	-3.91 (0.94)
AKC	CeO <sub>2</sub> (5%)	$L^*$	69.95 (1.58)
		$a^*$	-0.14 (0.27)
		$b^*$	4.89 (1.01)

Standard deviation (SD) values of  $L^*$ ,  $a^*$ ,  $b^*$  parameters are shown in parentheses.

**Table 2. Impact of UV+IR irradiation on the colour parameters  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  mean values**

Sample group	Parameter	1 hour	3 hour	6 hour	10 hour	20 hour	30 hour
AK	$\Delta L^*$	-0.95 (0.27)	-0.62 (0.49)	-1.42 (0.49)	-1.39 (0.49)	-1.45 (0.41)	-2.40 (1.42)
	$\Delta a^*$	-1.43 (0.31)	-2.26 (0.82)	-2.27 (1.16)	-2.05 (1.38)	-1.79 (1.37)	-1.73 (1.44)
	$\Delta b^*$	4.26 (0.95)	6.99 (2.52)	7.83 (3.48)	7.61 (4.23)	7.04 (4.25)	6.97 (4.43)
	$\Delta E^*$	4.60 (1.00)	7.39 (2.66)	8.33 (3.57)	8.06 (4.37)	7.52 (4.29)	7.86 (4.37)
AKZ	$\Delta L^*$	-0.43 (0.40)	-0.55 (0.36)	-1.00 (0.52)	-1.09 (0.49)	-1.13 (0.43)	-1.34 (0.81)
	$\Delta a^*$	-0.02 (0.14)	-0.22 (0.12)	-0.26 (0.14)	-0.23 (0.13)	-0.27 (0.28)	-0.31 (0.17)
	$\Delta b^*$	0.39 (0.28)	1.30 (0.29)	1.92 (0.34)	2.14 (0.35)	2.48 (0.30)	2.58 (0.62)
	$\Delta E^*$	0.68 (0.35)	1.48 (0.25)	2.25 (0.26)	2.47 (0.25)	2.79 (0.25)	3.07 (0.41)
AKC	$\Delta L^*$	-0.71 (0.31)	-0.83 (0.27)	-1.39 (0.59)	-1.35 (0.40)	-1.49 (0.48)	-0.99 (0.82)
	$\Delta a^*$	-0.19 (0.05)	-0.63 (0.08)	-0.97 (0.12)	-1.02 (0.21)	-1.05 (0.27)	-0.68 (1.08)
	$\Delta b^*$	1.20 (0.29)	2.70 (0.52)	4.42 (0.61)	5.00 (0.86)	5.39 (1.07)	5.41 (1.08)
	$\Delta E^*$	1.45 (0.26)	2.91 (0.45)	4.77 (0.62)	5.29 (0.88)	5.71 (1.09)	5.95 (1.14)

SD values are shown in parentheses.

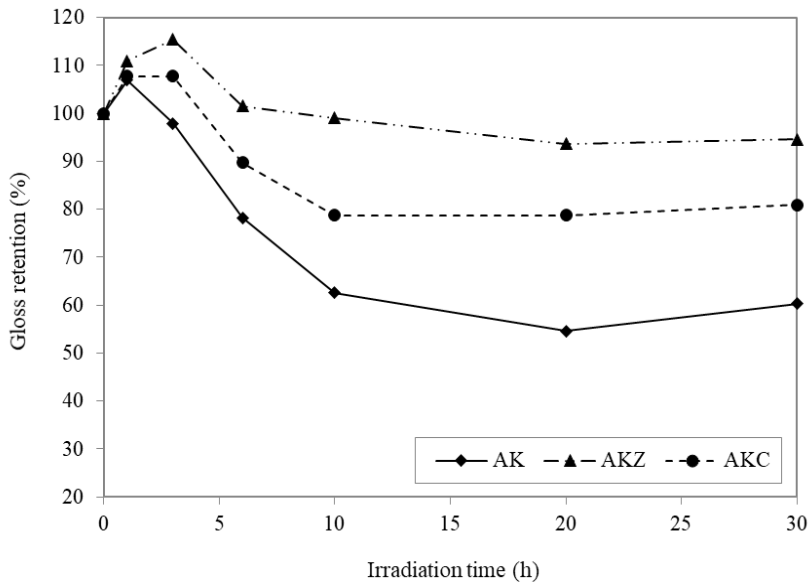


**Fig. 1. Coating films overall colour change  $\Delta E^*$  during UV+IR irradiation**

a short and intensive period of UV+IR irradiation. Comparable results were presented in a study by Nowaczyk-Organista [2009], where the overall colour change  $\Delta E^*$  protection against UV irradiation was investigated in the acrylic lacquer product group containing 4% of ZnO nanoparticles. A similar tendency was also observed in this work, but in our case, 1 percentage point more of the same type ZnO nanoparticle dispersion compound in the AK coating composition was used. The addition of ZnO nanoparticles provided stability to the most promising colour of the coating film in comparison to the other sample groups. As mentioned above, CeO<sub>2</sub> nanoparticles exerted influence on the initial coating film colour due to the yellow undertone alone [Schaller et al. 2012; Khan et al. 2013]. Yet, the AKC samples group maintained the high coating colour deviation stability while undergoing UV+IR irradiation.

Figure 2 illustrates coating film gloss retention performance during the 30 h of intensive UV+IR irradiation. The retention of the sample gloss parameter was different when compared to the colour measurements, during which, the intensive rising  $\Delta E^*$  peak was mainly maintained up to the 6 hours of irradiation. During the first phase of the test (0-3 h), the UV+IR irradiation intensity and the high surface temperature had an apparent impact on the different gloss performance and expedited the decrease of glossiness. This may be caused by the interchain crosslinking formatting between the adjacent chain of free radicals as well as by the subsequent loss of the surface gloss, which, after 3 hours, started to increase due to the polymer chain breakdown [Pospíšil and Nešpurek 2010; Hang et al. 2015]. The difference between the gloss retention values after 30 h of UV+IR irradiation was 1.3 (AKC) – 1.6 (AKZ) times higher when compared to

the AK sample group. It is assumed that inorganic nanoparticles exerted a positive influence on waterborne clear acrylic coating film gloss retention.

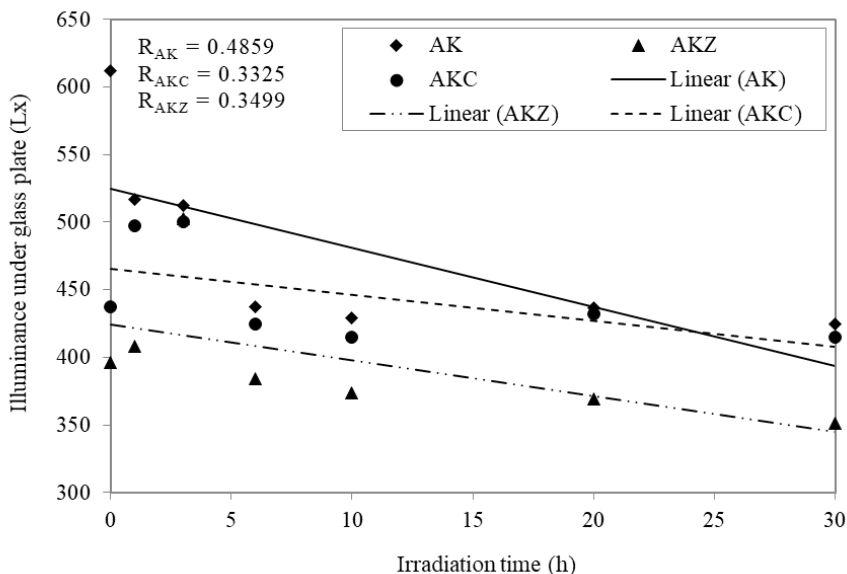


**Fig. 2.** Effect of UV+IR irradiation time on the gloss retention of coating films

Hu et al. [2009] and Mirabedini et al. [2011] remarked that various methods of sample ageing affect the differences of the loss of the coating film gloss. Nevertheless, comparable results were observed in the study by Miklečić et al. [2017], where the highest stability level regarding the loss of the gloss of nanocomposite waterborne polyacrylate coating films after longterm artificial and natural weathering were observed in the coating film groups containing ZnO (2% and 4%) nanoparticles. The tested coating systems showed a significantly higher resistance to UV+IR irradiation than furniture lacquer coatings (1 h normative requirements in accordance with PN-88/F-06100/07:1988 standard).

Schaller et al. [2012] and Faure et al. [2013] note that inorganic nanoparticles show a significant impact on the initial transparency of coating films. It was also observed that ZnO (5%) nanoparticles reduced the initial coating film illuminance up to 1.5 times whereas CeO<sub>2</sub> (5%) nanoparticle additives decreased this number by 1.4 times, respectively (fig. 3). A similar trend of the initial transparency loss due to the impact of ZnO nanoparticle additives was found in the experimental study by Grüneberger et al. [2015], where the opaqueness of acrylic coatings increased along with the concentration of ZnO nanoparticles (from 0.7% to 9.1%) in the structure of the coating film. In this study, the coating film illuminance under the glass plate parameter and the visual transparency were mostly reduced in the clear coating group

(by 1.4 times). Meanwhile, the reduction of the visual transparency of the AKZ and AKC coatings was significantly slower during the UV+IR ageing test. Despite the overall thermal impact, the loss of the nanocomposite coating film illuminance was only up to 1.1 times. During UV+IR irradiation, the most promising visual transparency levels were observed in the coating group containing  $\text{CeO}_2$  nanoparticles.

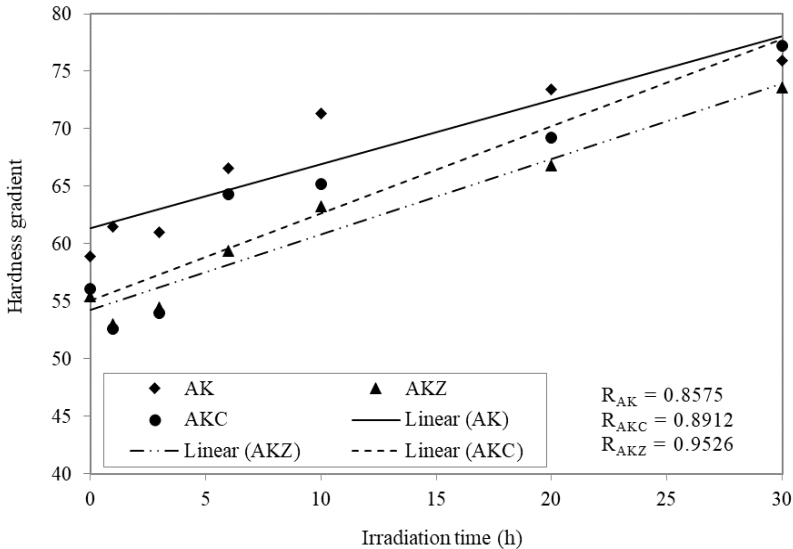


**Fig. 3. Dependence of the coating film illuminance parameter on UV+IR irradiation**

With the increase of the time of UV+IR irradiation, the coating film surface hardness gradient also increased (fig. 4). During the UV+IR irradiation test, inorganic nanoparticles had a significant impact on the initial coating hardness gradient (according to *ANOVA* statistical analysis); also, an influence was observed on the performance of coating films. A similar trend was detected in the study of Vaja et al. [2012], where the addition of ZnO nanoparticles increased the hardness of the acrylic coating by 5%. In this work, the change between the initial hardness gradient of clear coating films and the nanocomposite coatings was 4.8% (for AKC) and 5.9% (for AKZ). Meanwhile, the coating film hardness gradient after 30 h of UV+IR irradiation for the AK sample group increased by 1.3 times, whereas for the coatings containing ZnO or  $\text{CeO}_2$  nanoparticles, it went up by 1.3-1.4 times, respectively. Due to the inhomogeneity of the coating films, which is a consequence of the degradation mechanism, the hardness gradient during the ageing process can increase [Wicks et al. 2007; Cristea et al. 2012]. On the other hand, during the degradation of nanocomposite coatings, inorganic nanoparticles can shift and produce a higher



nano additive concentration near the coating surface [Cristea et al. 2011]. This can theoretically explain the increased hardness gradient in nanocomposite coatings after 30 h of UV+IR irradiation in comparison with the clear AK coating group.



**Fig. 4. Influence of UV+IR irradiation time on the hardness of a coating film**

## Conclusions

1. The combined ultraviolet light (UV) and infrared radiation (IR) ageing method can be used with the intention to evaluate the physical and mechanical properties of the resistance of nanocomposite wood coatings.
2. Additives of inorganic (5% of ZnO or CeO<sub>2</sub>) nanoparticles exerted influence on the initial clear colour of a waterborne acrylic coating film and also made a significant impact (up to 2.6 times) on the overall colour stability  $\Delta E^*$  of coating films during 30 h of intensive UV+IR irradiation.
3. Surface gloss retention was obtained in the coating sample groups containing CeO<sub>2</sub> and, especially, ZnO nanoparticles; the gloss retention parameter changed only by 5.4% in the AKZ group.
4. Inorganic nanoparticle additives had an impact on the acrylic coating film illuminance parameter and its performance during the UV+IR test. In order to preferably understand the impact of UV+IR irradiation on the wood coating film transparency, more profound analysis should be carried out and novel methodologies should be developed, e.g. UV-VIS transmission spectroscopy should be devised.

5. A significant influence of inorganic nanoparticles on the hardness gradient of the initial coating films as well as on their performance was observed during the 30 h of UV+IR irradiation test. A closer linear dependence on the irradiation time was observed in the AKZ group ( $R = 0.95$ ).

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### List of standards

- PN-88/F-06100/07:1988** Meble. Metody badań właściwości powłok lakierowych i laminowanych. Oznaczanie odporności na działanie zmiennych temperatur (Testing methods for the properties of lacquers and decorative laminated surfaces. Variable temperature test)

**ISO 2813:2014** Paints and varnishes. Determination of gloss value at 20 degrees, 60 degrees and 85 degrees

**ISO 1522:2006** Paint and varnishes. Pendulum damping test

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