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DUPLEX ANTI-CORROSION PROTECTION OF STEEL USING A COMBINATION OF HOT-DIP GALVANISING AND WATER-SOLUBLE PAINTS

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The goal of this paper is an evaluation of research aimed at corrosion and mechanical resistance of the so-called duplex system applied to a steel sheet. This system consists of a metal coating applied by hot-dip galvanising, to which an acrylic, water-soluble paint is commonly applied (commonly available on the Czech market) using standard technology in an air stream. For the purposes of the corrosion resistance comparison, one set of test specimens is provided only with a hot-dip galvanised coating. The mechanical resistance of applied anti-corrosion protection was determined by means of a pull-off adhesion test (according to the ČSN EN ISO 4624 standard), as well as an indentation test (according to the ČSN EN ISO 1520 standard). The corrosion resistance tests were then conducted in a salt spray environment (according to the ČSN EN ISO 9227 standard) for the samples not damaged by mechanical tests. Based on the obtained results, in general, waterborne paints with high dry matter content, low specific gravity and high zinc phosphate content, which are applied in several thinner layers, can be recommended.

Keywords: duplex system; corrosion resistance; carbon steel; zinc coating; ecological coating

Protection of a steel substrate by means of coating involves a barrier creation preventing the given substrate from coming in contact with a corrosive environment. However, with increasing exposure time to a corrosive environment, this protective barrier degrades, which allows more and more air humidity to pass through. Moisture penetrates the pores in the coating and gets to the steel substrate, which is subsequently subjected to corrosion (Kuklík and Kudláček, 2014). Acceleration of the corrosion rate and subsequent degradation can be observed especially in thermally stressed coatings (Votava et al., 2020). As demonstrated also in Šmak et al. (2020), a cooling medium for the steel heat treatment has also a fundamental effect on subsequent corrosion protection of a machine part. In production of weld joints, the base material around welded metal can be also negatively affected (Poláková et al., 2018). Duplex systems are very suitable means in situations, where a coating of hot-dip zinc is applied to steel substrate prior to paint. In this case, the coating prevents erosion of passivation layer covering the zinc coating, and any penetrating moisture causes this passivation layer to be restored, which prevents the further zinc coating corrosion. The corrosion degradation of zinc coating occurs only when the coating is disintegrated. In this regard, the duplex system has a longer service life than both service lives of two coatings applied separately. Thus, in the duplex system, there is a mutual synergistic effect of the inorganic and organic coatings (Votava et al., 2016).

Emphasis has been placed not only on the anti-corrosion properties of paints but also on their environmental friendliness, which consists in replacing toxic inhibitory pigments (lead and chromium compounds) with phosphates and utilization of water instead of organic

solvents (Romagnoli and Vetere, 1995; Roselli et al., 2017). For ecological reasons, it is thus appropriate to use waterborne paints. However, the disadvantage of these coatings lies in their lower corrosion resistance in contrast to coatings containing organic solvents, which is caused by the formation of pores and cracks during their application. However, this disadvantage can be eliminated by applying more thinner coatings, as multilayer coatings have been found to be more corrosion resistant than a single coating of the same thickness (Spengler et al., 1997; Galliano and Landolt, 2002; Fernández-Pérez et al., 2014). Corrosion resistance can be further increased by the addition of solid particles (e.g., minerals, silicates or mica), the presence of which in the coating reduces the formation of pores and cracks. These components have a major impact on extending the coating life in corrosive environments (Votava et al., 2018). In this case, however, the shape of solid particles is also important, as their globular form shows lower corrosion resistance than the lamellar form (Gee, 1997; Wegmann, 1997). The current trend is the application of nanoparticles of chemical compounds based on zinc (e.g., ZnO or ZnCO₃), which increases the corrosion resistance of coating. Furthermore, zinc phosphate Zn₃(PO₄)₂ is frequently used. Zinc phosphate conversion coating is widely used in industry as a surface pre-treatment. It improves the corrosion resistance and increases the adhesion between metal surface and subsequent treatment, such as organic coatings. Zinc phosphate coatings on steel consist mainly of crystalline α -hopeite and orthorhombic α -Zn₃(PO₄)₂·4H₂O. These compounds are commonly used in industry and are mostly produced synthetically (Abdalla et al., 2020; Luptáková et al., 2012, 2015).

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Material and methods

For the experiment, five sets of samples were produced using a hot-dip galvanised sheet; the first set was used to determine the zinc coating weight by the gravimetric method, and the second was not treated with an anti-corrosion system (for comparison purposes). In each set, there were ten samples with dimension of $160 \times 65 \times 1$ mm. In each case, one sample was subjected to a pull-off adhesion test and five samples were always used for the indentation test. The remaining four samples were subjected to an intact corrosion resistance test in a salt spray environment. A commonly available hot-dip galvanised sheet – ferritic-pearlitic steel S235JRG1 – was selected for the base material.

Waterborne paints that are commonly available on the Czech market were tested. Instead of the trade name, the individual anti-corrosion systems were designated using Roman numerals I (application to sample set no. 3), II (application to sample set no. 4) and III (application to sample set no. 5). The main parameters of anti-corrosion systems provided by the manufacturer are given in Table 1.

The zinc coating thickness was measured by a non-destructive electromagnetic method in accordance with the procedure specified by the standard ČSN EN ISO 1461. Within one set, the measurement was performed 5 times on each sample, the resulting thickness is the arithmetic mean of 50 values (Table 2). Subsequently, the zinc coating weight was determined using the gravimetric method according to the ČSN EN ISO 1460. This method is based on weighing the sample before and after the decontamination of zinc coating in a solution of 500 ml of distilled water, 3.5 g of hexamethylenetetramine and 500 ml of hydrochloric acid. The exposure time required to decontaminate all the zinc coating was 20 minutes. The weight was determined using

a digital scale with an accuracy of 0.001 g. The ambient temperature was 22 °C. The results of these measurements are given in Table 3.

The anti-corrosion system coating was applied by air spraying technology at an ambient temperature of 21 ± 1 °C. The first set was left without the anti-corrosion system and the remaining sets of samples were first primed to a tolerance of 25 ± 5 μm (1 layer each). A top coat was applied in 3 layers, with an application interval of at least 24 hours and a tolerance of 20 ± 5 μm . The thickness of formed anti-corrosion system was thus approx. 85 μm in total. The coating thickness measurement was performed after its perfect drying using the magnetic induction method, which is non-destructive and can be used to measure the thickness of non-ferromagnetic coatings on a ferromagnetic substrate. The measurement was conducted using Elcometer 456 with a ferromagnetic and paramagnetic probe. The test was performed in accordance with the ČSN EN ISO 2808 standard. Within 1 set, the measurement was performed 3 times on each sample, therefore, the resulting thickness is the arithmetic mean of 30 values. The final coating thickness is given in Table 4. Furthermore, the weight of individual coating layers was determined using a digital scale with an accuracy of 0.001 g (the arithmetic means are, in this case, always calculated from 10 values). The results of this measurement are also given in Table 4. Figure 1 shows a graphical representation of the total thicknesses of coatings and, at the same time, the total weight of coatings.

After performing the aforementioned procedure, the formed anti-corrosion systems were subjected to mechanical tests. The pull-off adhesion test was performed first, immediately after the anti-corrosion system had dried completely. The test was performed in accordance with the ČSN EN ISO 4624 standard. Five measurements were always

Table 1 Main parameters of the anti-corrosion systems

Coating	Non-volatile solids (weight %)	Specific density ($\text{g}\cdot\text{cm}^{-3}$)	Emissions of volatile organic compounds ($\text{kg}\cdot\text{kg}^{-1}$)	$\text{Zn}_3(\text{PO}_4)_2$ (%)
Basic coating	55	1.25	0.130	1.0–2.0
Anti-corrosion system I	65	1.19	0.065	18.0–20.0
Anti-corrosion system II	44	1.26	0.062	–
Anti-corrosion system III	48	1.27	0.060	≤ 2.5

Table 2 Zinc coating thickness

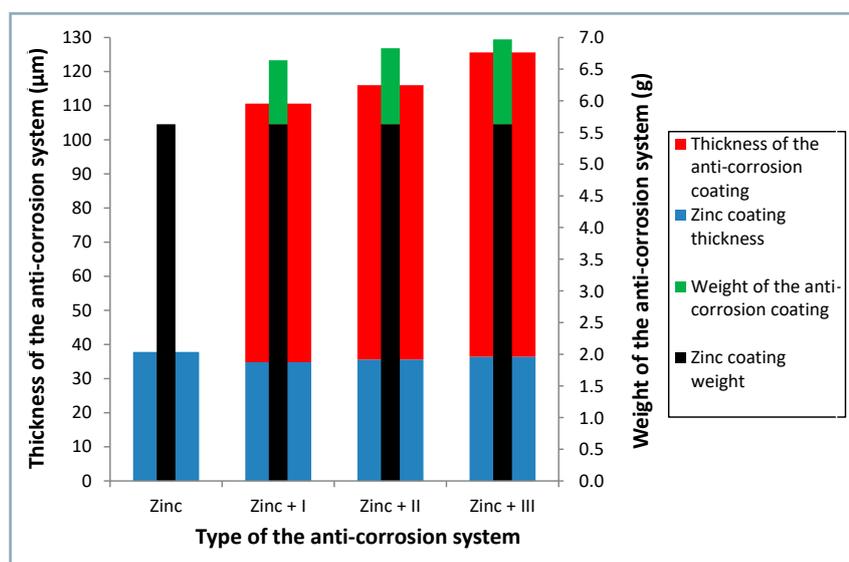
Set No.	Anti-corrosion protection No.	Arithmetic mean (μm)	Dispersion (μm^2)	Standard deviation (μm)	Coefficient of variation (%)
2	-	37.8	5.36	2.32	6.12
3	I	34.8	6.56	2.56	7.36
4	II	35.6	6.24	2.50	7.01
5	III	36.4	7.44	2.73	7.49

Table 3 Zinc coating weight (sample set no. 1)

No. of samples	Arithmetic mean (g)	Dispersion (g^2)	Standard deviation (g)	Coefficient of variation (%)
10	5.63	0.0057	0.0757	1.34

Table 4 Final thickness and weight of the anti-corrosion systems

Anti-corrosion protection no.		I	II	III
Arithmetic mean	thickness (μm)	75.8	80.4	89.2
	weight (g)	1.01	1.20	1.34
Dispersion	thickness (μm^2)	14.96	18.24	16.16
	weight (g^2)	0.0054	0.0036	0.0034
Standard deviation	thickness (μm)	3.87	4.27	4.02
	weight (g)	0.0733	0.0598	0.0581
Coefficient of variation	thickness (%)	5.10	5.31	4.51
	weight (%)	7.25	4.98	4.33

**Fig. 1** Graphical representation of the total coating thicknesses and total coating weight

performed on each anti-corrosion system (in each corner and in the middle of the sample), therefore, the arithmetic means are always calculated from five values.

Brief description of the preparation methodology of test specimens:

- degreasing of test specimens using "Perchlor";
- selection of a standard test cylinder with 20 mm diameter;
- application of two-component glue "Araldite";
- tensile stress analysis via Elcometer (measuring range 0–7 MPa).

Furthermore, a cupping test was performed in accordance with the ČSN EN ISO 1520 standard. Since the anti-corrosion systems showed considerable plasticity, they were subjected to observation at magnification of 50 \times using a Neophot

21 metallographic microscope. Five measurements were always performed on each anti-corrosion system, suggesting that the arithmetic means are always calculated from five values.

The experiment was completed with the corrosion resistance test in a salt spray environment in accordance with the standard ČSN EN ISO 9227. The experiment took place in a Liebisch chamber, type S400M-TR. In such a manner, four samples from each set were tested. The expected test time was determined at intervals of 5, 10, 15, 20, 25 and 30 days. However, daily visual observations were conducted during the test and, depending on the corrosion rate, the appropriate adjustment of the intervals was considered.

Test parameters:

- temperature in salt spray environment of 35 ± 2 $^{\circ}\text{C}$;
- concentration of sodium chloride in the spray medium 50 ± 5 $\text{g}\cdot\text{l}^{-1}$;
- pH of the saline solution 6.5–7.2.

Results and discussion

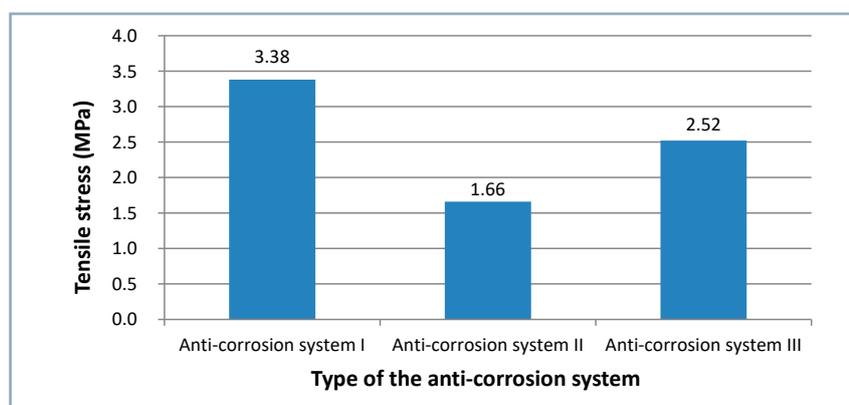
Pull-off adhesion test

The pull-off adhesion test was performed in accordance with the ČSN EN ISO 4624 standard, resulting in the tensile stress necessary for the failure of the weakest interface (adhesion failure) or the weakest component (cohesion failure) of produced anti-corrosion system. It was not possible to evaluate the adhesion of zinc coating to the steel substrate, as there was always an adhesion failure between the adhesive and the test specimen. The test results are given in Table 5 and Fig. 2.

Considering the Table 5, it is evident that the highest adhesion was recorded in anti-corrosion system I, which was caused by the highest dry matter content and, conversely, the lowest specific weight of the paints used (Table 1; Votava and Kumbár, 2017). The predominant adhesion failure between the substrate and the first layer further indicates a suitably selected base coat due to the good adhesion to the top coat. Anti-corrosion system III can then be described as a less suitable combination of coatings, since there was mainly an adhesion failure between the base coat and the top coat. Despite this fact, however, the adhesion between the base coat and the top coat was very good and the difference between it and anti-corrosion system I is not

Table 5 Results of the pull-off adhesion test of anti-corrosion systems

Anti-corrosion protection No.	Arithmetic mean (MPa)	Dispersion (MPa ²)	Standard deviation (MPa)	Coefficient of variation (%)
Zinc coating	–	–	–	–
	adhesion failure between the adhesive and the test specimen (100% of cases)			
I	3.38	0.0216	0.1470	4.35
	predominant adhesion failure between the substrate and the first layer (from 70%)			
II	1.66	0.0224	0.1497	9.02
	predominant adhesion failure between the substrate and the first layer (from 80%)			
III	2.52	0.0176	0.1327	5.26
	predominant adhesion failure between the first and second layer (from 80%)			

**Fig. 2** Graphical representation of the pull-off adhesion test of anti-corrosion systems

statistically significant. The fact that the passivation layer on the zinc coating did not reduce the adhesion of paint to substrate also contributes to the adhesion failure between the base coat and the top coat (Kuklík and Kudláček, 2014). The lowest adhesion was shown by anti-corrosion system II, approx. half of that compared to anti-corrosion system I. This phenomenon is caused by the significantly lower dry matter content and higher density compared to anti-corrosion system I. However, adhesion of all the three anti-corrosion

systems can be considered sufficient, with anti-corrosion system I being the most suitable. The measurement of the adhesion of waterborne coatings to the hot-dip galvanised substrate was also dealt with by e.g. Votava and Kumbár (2017) and their experiment yielded similar results, as the average values ranged from 2.2 MPa to 3.0 MPa.

Cupping test

The cupping test (ČSN EN ISO 1520) determines the ability of anti-corrosion system to plastically deform during the

formation of an indentation, i.e., the later the sample breakage, the higher the plasticity of coating. The test evaluation was performed based on the analysis of formation of capillary cracks (crack width up to 5 µm), as well as on the basis of the analysis of values up to layer failure (crack width over 5 µm). The test results are given in Tables 6 and 7 and Figs 3–5.

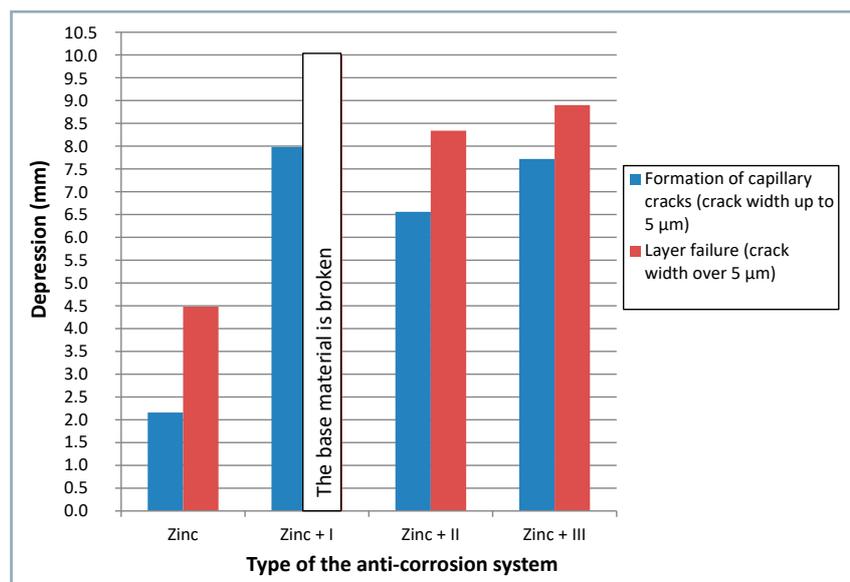
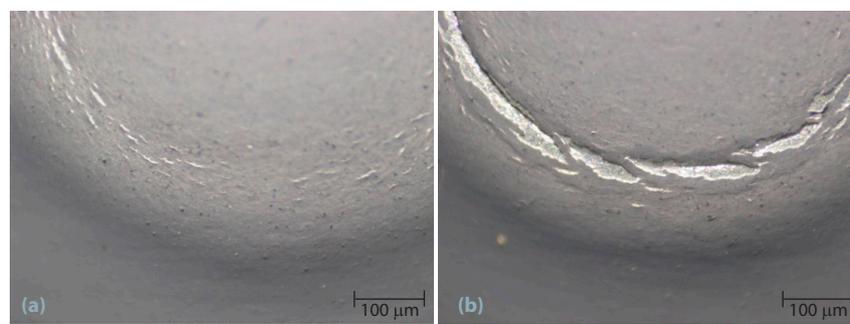
Based on this type of mechanical test, it was found that the onset of capillary cracking in the duplex system was approx. 3.5 times higher than in the zinc coating alone. Furthermore, the values up to the layer failure were almost 2 times higher in the case of a duplex system than in the case of zinc coating itself. Therefore, there is an obvious synergistic effect in duplex system. The use of anti-corrosion system I also seemed to be the most suitable in this case, since the onset of capillary cracking was the highest, with a mean of almost 8.0 mm. The values until the failure of layer could no longer be determined because the failure of base material (Fig. 5) occurred even before the failure of coating, indicating the highest plasticity of used anticorrosion protections. This

Table 6 Results of the cupping test – the beginning of formation of capillary cracks

Set No.	Anti-corrosion protection No.	Arithmetic mean (mm)	Dispersion (mm ²)	Standard deviation (mm)	Coefficient of variation (%)
2	zinc coating	2.16	0.0424	0.2059	9.53
3	I	7.98	0.1056	0.3250	4.07
4	II	6.56	0.1384	0.3720	5.67
5	III	7.72	0.0936	0.3059	3.96

Table 7 Cupping test results – values up to the layer failure

Set No.	Anti-corrosion protection No.	Arithmetic mean (mm)	Dispersion (mm ²)	Standard deviation (mm)	Coefficient of variation (%)
2	zinc coating	4.48	0.0416	0.2040	4.55
3	I	there is a failure of base material – values always ≥ 10 mm			
4	II	8.34	0.0744	0.2728	3.27
5	III	8.90	0.0600	0.2449	2.75

**Fig. 3** Graphical representation of the cupping test (arithmetic means)**Fig. 4** Anti-corrosion system II: a) depression 6.7 mm; b) depression 8.6 mm**Fig. 5** Anti-corrosion system I: failure of the base material (depression 10.5 mm)

was probably aided by the lowest overall average coating thickness (75.8 μm). The lowest values were reached in both cases in anti-corrosion system II. However, due to its relatively low adhesion found in the pull-off test, these values were not significantly lower compared to the other two duplex systems. Anti-corrosion system II can, therefore, be described as satisfactory in this respect. Barbato et al. (2002) justified the high toughness of waterborne paints containing acrylate resins. Furthermore, they also described the shortcomings of waterborne paints, which lie in their low abrasion resistance and low hardness.

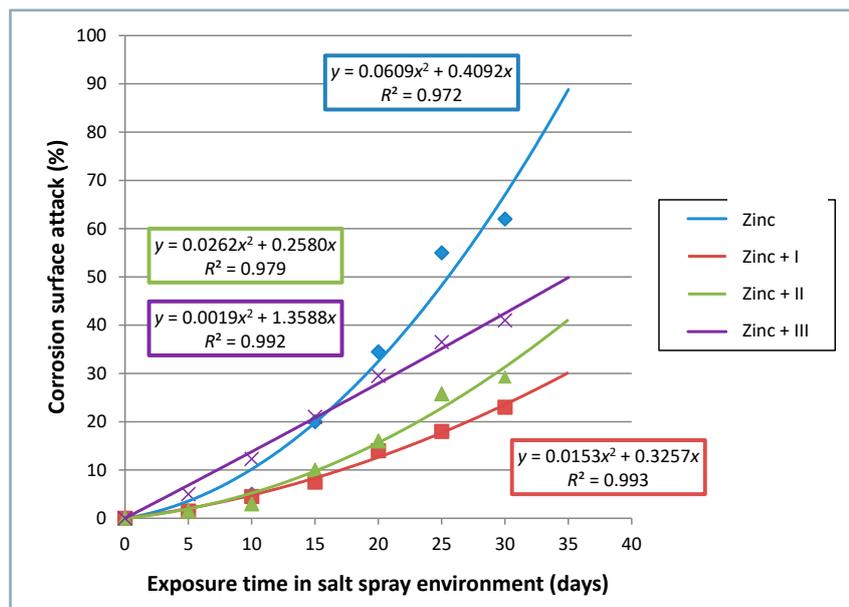
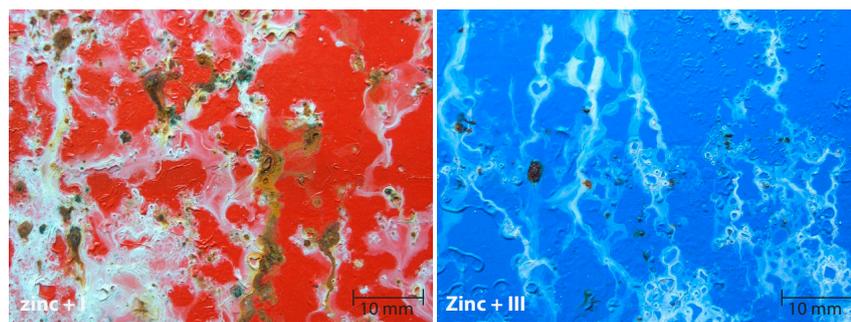
Corrosion resistance tests

The corrosion resistance tests were performed in a salt spray environment (ČSN EN ISO 9227), while only the samples undamaged by the mechanical tests were tested. The proposed 5-day intervals proved to be suitable for the evaluation of all samples and it was thus not necessary to adjust them continuously. The measured data were approximated by a polynomial of the 2nd degree, the suitability of which was confirmed by the high values of the coefficient of determination R^2 . The test results are given in Table 8, Figs 6 and 7.

Corrosion initially attacked the most by anti-corrosion system III and, after approx. 15 days of exposure, this attack was comparable to the zinc coating. From this point on, the zinc coating was most affected, which persisted until the end of corrosion test. Anti-corrosion system I seemed to be the most suitable in this case as well, as its corrosion attack was the lowest of all (despite the lowest overall average coating thickness) and even more than 2.5 times lower (after 30 days of exposure) compared to the zinc coating. The reason for the best

Table 8 Corrosion resistance test results

Set No.	Anti-corrosion protection No.	Exposure time in salt spray environment (days)					
		5	10	15	20	25	30
		corrosion surface attack (%) (arithmetic mean of 4 samples)					
2	zinc coating	1.5	5.0	20.0	34.5	55.0	62.0
3	I	1.5	4.5	7.5	14.0	18.0	23.0
4	II	1.5	3.0	10.0	16.0	25.8	29.3
5	III	5.0	12.3	21.0	29.5	36.5	41.0

**Fig. 6** Graph of the corrosion degradation of tested samples depending on the exposure time (30 days + forecast for 5 days)**Fig. 7** Comparison of the corrosion attack after 15 days of exposure

corrosion resistance of anticorrosion system I is the high content of zinc phosphate, which reduces the formation of pores and cracks in the applied coating (Fernández-Pérez et al., 2014). However, it was found that the low content of zinc phosphate (up to 3%) had no effect on a higher corrosion resistance (Votava and Kumbár, 2017). Despite the faster initial corrosion

degradation of anti-corrosion system III, the application of a duplex system is clearly more advantageous in the long run.

Conclusion

Based on the obtained results, it can be stated that the formation of a duplex system, using a combination of zinc coating and waterborne paints, can

be clearly recommended, as there was a statistically significant increase in the resistance in both mechanical and corrosion tests. The improvement in the mechanical properties of duplex systems is due to the high toughness of the waterborne paints and the increase in corrosion resistance confirms the synergistic effect of both coatings. The anti-corrosion systems produced in this way can then be advantageously used not only in industrially polluted areas but also in agriculture – both in plant and animal production – e.g. in order to increase the anti-corrosion protection of zinc coatings in load-bearing structures of agricultural buildings. The application of waterborne paints is advantageous in this case despite their low abrasion resistance, as the risk of this wear is minimal.

The most suitable duplex system is the combination of a zinc coating and anti-corrosion system I. This anti-corrosion system excels in its high dry matter content and, conversely, its low specific gravity, which contributes to the highest adhesion to the zinc coating. Furthermore, this anti-corrosion system also excels in its high content of zinc phosphate, which together with the multilayer coating also contributes to the highest corrosion resistance. Therefore, in general, waterborne paints with high dry matter content, low specific gravity and high zinc phosphate content, which are applied in several thinner layers, can be recommended.

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