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DEGRADATION PROCESSES IN ANTICORROSIVE COATINGS FOR MACHINERY DESIGNED FOR FERTILISER APPLICATION

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Abstract

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In agricultural practice, anticorrosion protection of steel components is utterly indispensable. It is of particular importance for machines and devices working in an environment with increased corrosion activity. The aim of the present publication is the analysis of the use of 5 types of anticorrosion protection in machines with increased corrosive stress. These are coating systems based on alkyd and acrylic resins. Anorganic anticorrosion system is represented by a coating created by hot-dip galvanising. A system of duplex protection created by hot-dip galvanising and organic alkyd-urethane coating system will also be examined. The experimental section of the publication can be divided into two sub-domains. The first is focused on the analysis of the mechanical properties of anticorrosion coating. The analysis is performed via the Erichsen elasticity test according to EN ISO 1520:2001 and a pull-off test according to EN ISO 4624:2003. The individual results allow the estimation of the resistance against delamination of the surface layers. The second domain is focused on the corrosion resistance of individual coatings in an environment with increased corrosion stress. The tests were performed in salt mist environment in accordance with EN ISO 9227:2012. A cyclic test was also performed with the use of DAM 390 industrial fertiliser. It is a long-term test where significant correlation with real conditions can be assumed.

Keywords: corrosion, anticorrosion protection, organic coating systems, galvanising, mechanical properties of the coating

INTRODUCTION

The use of anticorrosion protection requires the knowledge of the basic principles of corrosion processes and the mechanics of the individual types of corrosion as well as anticorrosion protection. A chemical reaction with the elements of the corrosive environment turns metal into a solution identical or similar to the one it was originally produced from. For this reason, anticorrosion protection is indispensable (Trethewey, Chamberlain, 1995, Tulka, 2005).

Environments which could be characterised as highly aggressive are encountered quite commonly in agricultural practice. These environments include especially those where industrial or farm fertilisers are used. The corrosive influences affecting the machines operating in this sector are enormous. For this reason, anticorrosion protection must be designed not only for long-term protection of machine components, but must also fulfil specific mechanical-physical criteria to guarantee reliable adhesion and elasticity of the coating system.

Tests have shown that water-soluble coating systems have excellent mechanical properties (Votava, 2013a). One of their main advantages are the ecological aspects of their use. The volatile organic compound (VOC) content must be limited in coating systems in order to reduce the subsequent release of these pollutants into the environment (Funke, 1997, Dobson, 1996). However, reduction in the content of organic soluble substances partially also reduces the anticorrosive properties of the coatings. Therefore, inhibitors based on zinc phosphate are being used. The free zinc particles give rise to a barrier effect as well as to passivation of zinc plates (Cappari *et al.*, 2000). The decisive factor, however, is the chemical composition (acrylic, alkyd polyamide) of the coating system itself (Almeida *et al.*, 1999).

The anticorrosion mechanism of coating systems consists mainly of the barrier effect of the coating. The essence is thus the prevention of air humidity permeating through to the base material. However, the aging of the coating leads to its internal degeneration and loss of ability to isolate the steel substrate. It is thus necessary to choose an appropriate combination of additives and pigments to preserve the toughness of the coating – and not just in alkyd and acrylic paint systems (Johnson, 1994, Chico *et al.*, 2008, Chico, 2012). The quality of the anticorrosion systems is then checked via tests assessing not only the elastic parameters of the new coating but also the decrease in elasticity at a given corrosive stress. The most common method is corrosive stress testing in salt mist. However, the corrosion activity can be partially distorted during this artificial corrosion process when compared to e.g. atmospheric corrosion (Deyá *et al.*, 2010).

Since the production of anticorrosion coatings based on hot-dip galvanisation is currently constantly increasing, the NaCl environment can be evaluated as too aggressive for the testing of these coatings (Ambrož, Kašpar, 1990). A significant advantage of zinc coatings is the option of passivation and the ability to protect even damaged sections against corrosion. This aspect is typical of metals which are less noble that the base foundation material which is being protected (Morishita *et al.*, 1997, Kending *et al.*, 2003, Bexell *et al.*, 2007). Another option for increasing anticorrosion protection is also the combination of organic and anorganic (metal) coating. This alternative is used mainly in environments where the different anticorrosion systems complement each other (Votava, 2013b). Coatings created by hot-dip galvanisation can then be covered by coating systems containing other metals such as Al or Cr (Sumon *et al.*, 2013).

The elimination of corrosive influences from the steel surfaces of components is a key issue not only in engineering, but also in agricultural primary production. Specific anticorrosion protection is also represented by PVD and CVD methods, alternatively by bare refined steel dedicated to environments with a higher abrasive stress (Čičo *et al.*, 2011, Viňaš *et al.*, 2013, Paulíček *et al.*, 2013). For this reason, anticorrosion protection that is adequate for the environment is essential.

MATERIALS AND METHODS

Based on reasearch of the issue, five types of anticorrosion protection of steel components were selected for environments with increased corrosion stress. The use of these anticorrosion systems is expected in machines for the application of both industrial and farm fertilisers.

Steel S235JRG1 was used as a base material; surface roughness *Ra* did not exceed 0.8 µm, it is a value valid for cold-rolled material.

Alkyd-urethane coating system: An anticorrosion coating system created by the dispersion of pigments and fillers in an alkyd-urethane bitumen solution with additives. A variant that dries in air was selected for the individual tests. The pigments of the given anticorrosion systems are of mostly alkaline character. This option is used as a self-founding (foundation and top layer in one) coating system. The coating is characterised by high elasticity, firmness and resistance to weathering. This anticorrosion coating is suitable for environments with increased corrosion activity. The main parameters of the coating system are listed in Tab. I.

Acrylic coating system (diluted by organic solvent): This anticorrosion system is based on acrylic polymers. The paint has a thixotropic character, which along with the special composition allows the creation of a thick layer and good protective properties in one painting. It is a dispersion of pigments, fillers and anticorrosion substances in a solution of modified resins and organic solvents with the addition of drying agents and additives. The product is characteristic by excellent adhesion to steel surfaces and rapid drying even in thick layers. It is also suitable for the coating of light metals such a galvanised sheet metal.

Acrylic coating system (diluted with water): An anticorrosion system created by the polymerisation of acrylic and metacrylic acid. The basis is a polymer diffused in water in presence of other substances (pigments and additives). The principle of the creation of a covering film is based on water evaporation. For this reason, a longer time delay must be maintained between the individual coatings. The anticorrosion system contains minimum amount of emissive volatile organic substances; it is a highly ecological anticorrosion system It also features excellent elasticity and elastic deformation capability. It is suitable for indoor and outdoor use.

Hot-dip galvanising: The tested samples were hot-dip galvanised in laboratory zinc plating conditions. The samples were dipped for 120 s. The temperature of the galvanising bath was 450 ± 5 °C. In order to improve foundering performance, the galvanising zinc bath was alloyed by 2 % of pure aluminium. After removal from the galvanising bath, a gradual cooling in air followed. The thickness of the zinc coating substrate is highly dependent on the time the component remained in the zinc bath. According to experimental measurements, the period of 120 s results in coating thickness in the sample of approximately 65 µm.

Duplex system: To compare the speed of corrosion attack, the zinc coating was painted with an alkyd-urethane resin layer. The coating was applied onto the base material by air spraying at ambient temperature of 22 °C in two layers with a minimum of 4 hours between the application of the

I: *Main parameters of anticorrosion systems*

	Non-volatile substances in dry matter [%wp]	Density [g/cm3]	Emissive organic volatile substances [kg/kg]	Organic solvent content [kg/kg]
Alkyd-urethane film	65	1.20	0.42	0.31
Acrylic organic dispersion	59	1.37	0.45	0.38
Acrylic water dispersion	54	1.25	0.13	0.03
Hot-dip galvanising	-	-	-	-
Duplex system:	65	-	0.42	0.31

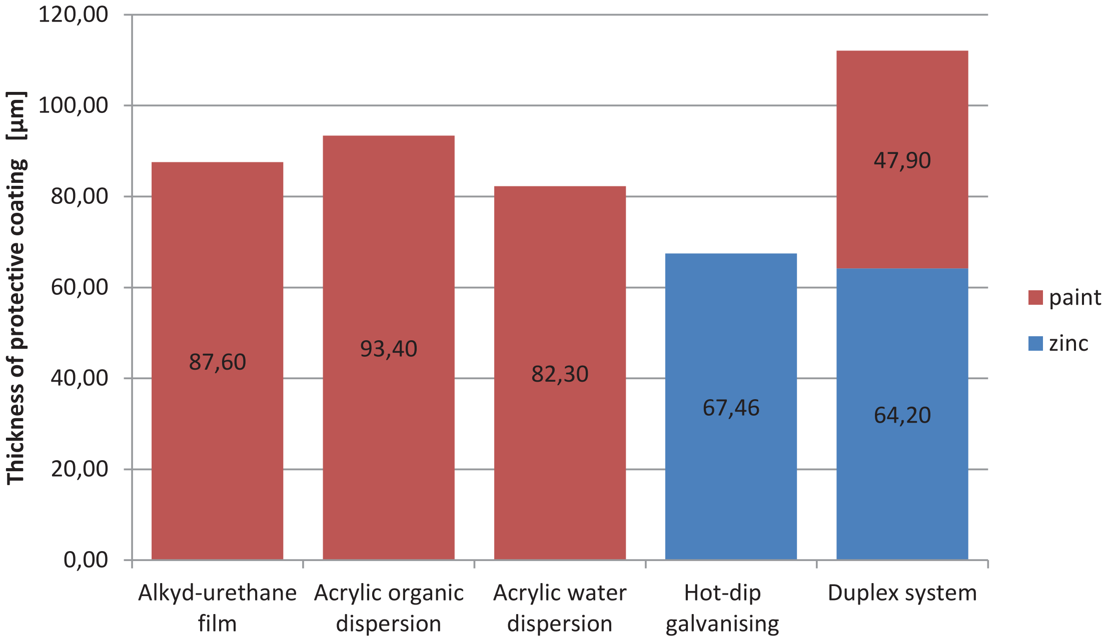
second layer. The main advantage of this method is the sealing of the pores of the zinc layer and also the insulation of the metal layer itself from the outside environment.

Determination of the thickness of the passivation coating

The anticorrosion systems tested were applied onto steel as well as galvanised base in three to four layers. The first and the subsequent layers were made of the same underlying substrate (in the anticorrosion substrates selected, there is no need for a primer). The thickness of the first layer of the coating was 30 ± 5 µm. This was followed by the application of a top coating with thickness in the range of 25 ± 5 µm. The total thickness values of the anticorrosion system are thus ca 90 µm, see Fig. 1. Measurement of the thickness of the coating was performed on a dry film via the magnetic induction method. It is a non-destructive test which can be used for ferromagnetic bases. There is also an alternative method to measure coating thickness: metallographic scratch pattern, however it is a destructive and a time consuming method.

Evaluation of the porosity of anticorrosive coatings

Porosity of an anticorrosive coating, which may be associated with the nature of the base material, leads to premature formation of red rust. For this reason, a detection test for the presence of pores and cracks in the coating was performed after each application of the anticorrosion system. The analysis was performed using Elcometer 270. The low voltage porosimeter (pinhole detector) operates on the principle of closed electrical circuit. This occurs if moisture passes through a defect in the coating and reaches the metal base.



1: *Thickness of the coating as scanned by an Elcometer ferromagnetic probe*

The largest number of pores was recorded in the first anticorrosion layer. However, synthetic coatings have shown a much lower occurrence of defects when compared to water soluble coating. The recorded values of the occurrence of pores are listed in Tab. II.

Pull-off test according to EN ISO 4624:2003 standard

The ability of the anticorrosion system to withstand dynamic and static stress is one of the criteria for the evaluation of the quality of protective coatings. One of the most important characteristics in this respect is the adhesion between the anchoring profile of the base material and the coating substrate.

The adhesion of the anticorrosion system was determined according to EN ISO 4624:2003 standard. This standard establishes the procedure for pull-off testing in single-layered and multilayered coating systems. The result of the test is the determination of the tension in pulling required to damage the weakest boundary (adhesive failure) or the weakest component (cohesive failure) of the tested body. The analysis of the adhesion of individual anticorrosion systems was preformed immediately after complete drying of the anticorrosion coating. The delay was set at 48 hours. The test results are shown in Tab. III.

Procedure for the preparation of test bodies:

- degreasing of test samples by Perchloroethylene,
- application of standardised test roller for pull-off test Ø 20 mm,
- application of two-component adhesive Araldite,
- analysis of pull-off force – Elcometr (measuring range 0–7 MPa).

Cupping test according to Erichsen

The international standard EN ISO 1520:2001 defines the standard testing method for determining the ability of anticorrosion coatings from 0.1 to 2 mm in thickness to plastically deform when the substrate is shaped via cupping. The accuracy of the Erichsen device is ensured by a digital deviation indicator whose contact areas are precisely calibrated with a two-arm lever for the transmission of motion.

In combined coatings, a synergistic effect of the individual layers of the protective system occurs. Since the tested systems have different degree of coating delamination, the collapsed zinc foundation disrupts the top anticorrosion layer, see Fig. 2. However, the delamination value was more than 3 mm, which is 1.5× more than in common synthetic coatings on steel substrate. The individual cupping values are recorded in Tab. IV.

Evaluation of corrosion resistance via cyclic testing in DAM liquid fertiliser

The cyclic test of corrosion was performed using liquid fertiliser DAM 390, undiluted. It is a nitrogen fertiliser with 30 % nitrogen content: a quarter of the nitrogen is in the form of ammonium, a quarter in the form of nitrate and one half in the form of amide. The fertiliser consists of a solution of ammonium nitrate and urea. 100 litres contain 39 kg of nitrogen. It has a density of 1300 kg/m^3 at 25 °C, and salting out temperature of −10 °C.

The cyclic test was performed on all anticorrosion systems in laboratory conditions. The prepared samples were soaked for 60 s and then placed onto drip stands with a 48 hour delay. The cycle was repeated regularly throughout the test, which lasted for 60 days.

The corrosion processes were visually assessed based on percentage invasion of the surface by red rust. There was not noted a higher percentage of red corrosion depending on different cross-section of intermetallic phases. The presence of red rust indicates the failure of the protective anticorrosion layer which results in the damaging of the foundation material. The speed of corrosion degradation is listed in Tab. V.

In atmospheric conditions, zinc coatings have the ability to create passivation layers which are corrosively much more noble than zinc itself (Almeida *et al.*, 1989).

There are chemical compounds ZnO, $ZnCO_3$, etc. Many of these compounds are produced synthetically and industrially used. Current trend is to apply nanoparticles of these compounds in order to increase anticorrosion resistance of the anticorrosion coating (Luptáková *et al.*, 2012, Luptáková *et al.*, 2015). As a result of chemical action of nitrogen fertilisers, however, a significant degradation speed was detected in the intermetallic stages of the zinc coating, see Fig. 3, which were unable to passivate the zinc coating itself. Metallographic preparations were prepared from samples (benchmarks before testing) and samples tested during the cyclic test.

Overall evaluation of corrosion resistance of coatings used according to EN ISO 9227:2012 (salt spray test)

Corrosion resistance testing of individual anticorrosion layers was performed as standardised by EN ISO 9227:2012 – *Salt spray test.* Since water soluble coating systems have higher corrosion resistance, this method is suitable especially for the analysis of pores and non-homogeneity of the given anticorrosive system. The experiment was performed in Liebisch chamber, type S400M-TR.

Test parameters:

- test sample ambient temperature 35 ± 2 °C,
- sodium chloride concentration in the spray medium 50 ± 5 g/l,
- pH value of the saline solution 6.5–7.2,
- the test duration was in the following intervals: 1, 2, 3, 5, 10, 15, 20 and 30 days. Based on daily visual observation, the intervals can differ depending on corrosion speed.

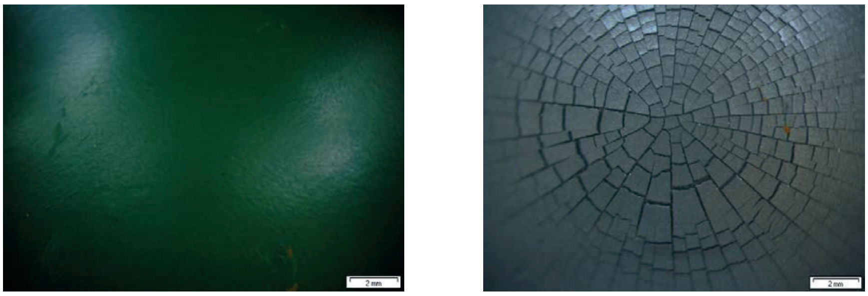
II: *Main parameters of anticorrosion systems*

Tested coatings		Sample 1	Sample 2	Sample 3	Sample 4
Alkyd-urethane film	Layer 1	×	×	o	×
	Layer 2	×	×	×	×
	Layer 3	×	×	×	×
Acrylic organic dispersion	Layer 1	o	o	×	×
	Layer 2	×	×	×	×
	Layer 3	×	×	×	×
Acrylic water dispersion	Layer 1	o	o	o	o
	Layer 2	o	o	o	o
	Layer3	×	o	×	×
	Layer 4	×	×	×	×
Hot-dip galvanising	Since the coating is conductive, the method cannot be used for the detection of defects.				
Duplex system:	Layer 1	×	o	×	×
	Layer 2	×	×	×	×

Legend: o: presence of cavity on the surface of the sample, ×: no pores or cavities detected on the surface of the sample.

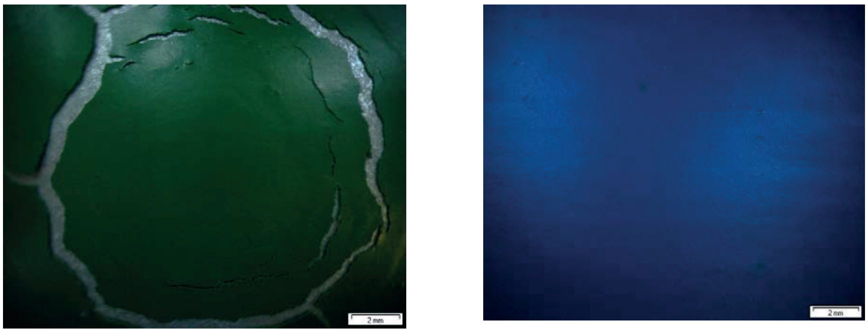
III: *Adhesion values of individual anticorrosion systems*

Application	Anticorrosion protection	Pull-off force [MPa]					Arithmetic average [MPa]
		1	2	3	4	5	
Steel plate + anticorrosion system	Alkyd-urethane film	5.2	5.8	5.3	5.3	5.6	5.40
	Acrylic organic dispersion	6.1	5.9	6.3	6.0	5.8	6.02
	Acrylic water dispersion	3.0	3.5	3.2	3.1	3.4	3.20
Hot-dip galvanising + anticorrosion system	Hot-dip galvanising	Delamination occurs only in the adhesive					
	Duplex system	4.9	5.1	5.2	5.5	5.2	5.10



Alkyd-urethane film

Acrylic organic dispersion



Duplex system:

Acrylic water dispersion

2: *Indentation from a spherical cap according to EN ISO 1520:2001, 10times magnification.*

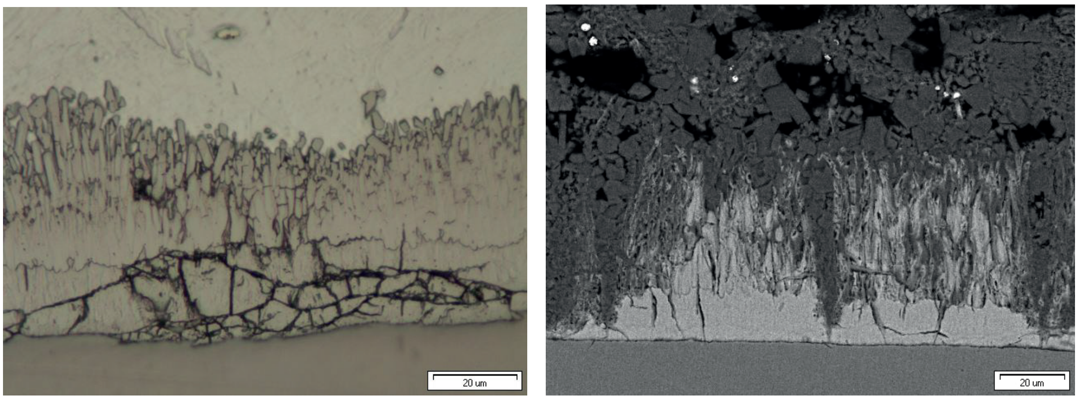
IV: *Cupping values according to EN ISO 1520:2001*

Application	Anticorrosion protection	Pull-off force [MPa]					Diameter [mm]
		No. 1 [mm]	No. 2 [mm]	No. 3 [mm]	No. 4 [mm]	No. 5 [mm]	
Steel plate + anticorrosion system	Alkyd-urethane film	7.2	*	7.1	7.3	6.9	7.10
	Acrylic organic dispersion	3.1	3.8	3.7	3.5	3.9	3.60
	Acrylic water dispersion	*	*	*	*	*	*
Hot-dip galvanising + anticorrosion system	Hot-dip galvanising	1.9	2.3	2.0	2.0	1.7	1.98
	Duplex system	4.2	4.5	4.1	4.2	4.5	4.30

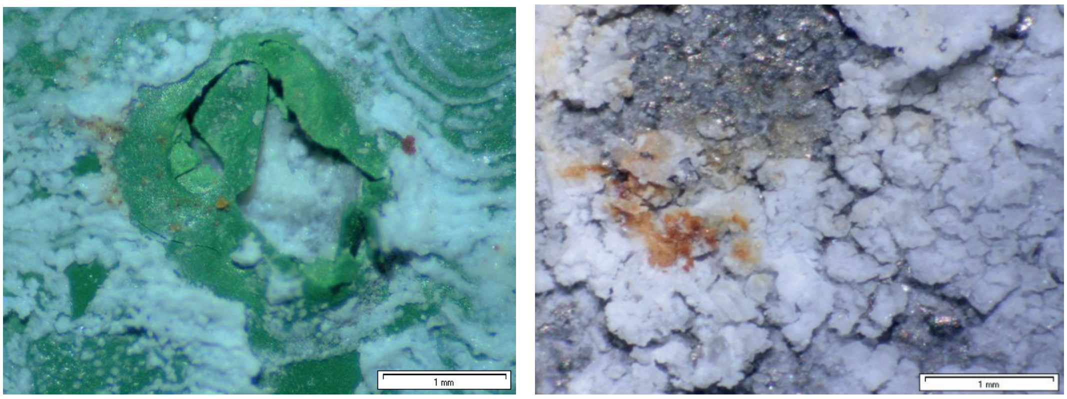
Legend: * the indentation of a steel ball reached 8 mm in the tested samples without breaking the anticorrosion coating (limit ductility value of steel plate)

V: *Evaluation of corrosion degradation in [%]*

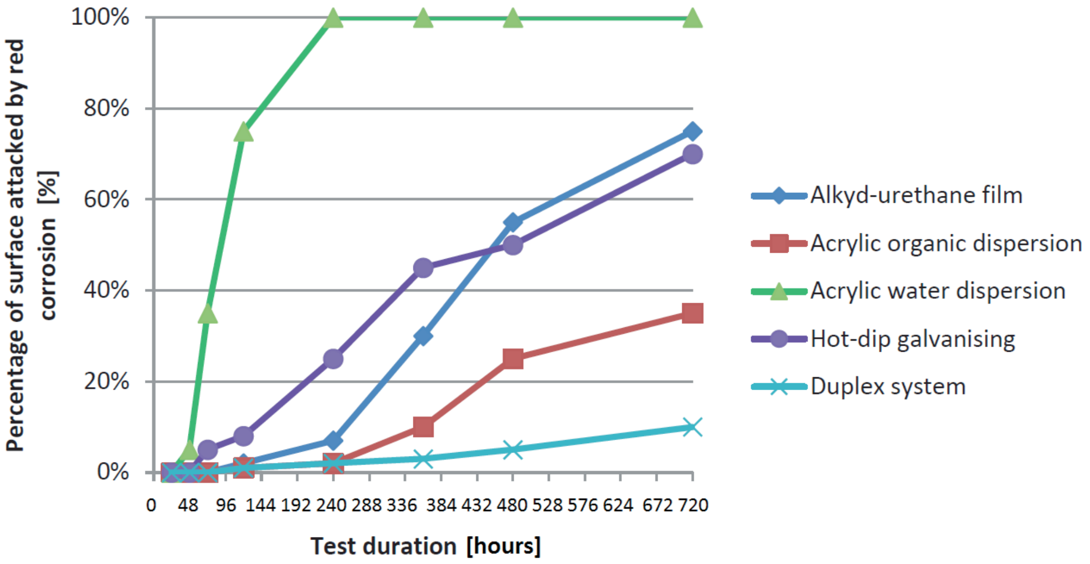
Anticorrosion system	Test duration [days]					
	10	20	30	40	50	60
Alkyd-urethane film	0	1	1.5	1.5	2	3
Acrylic organic dispersion	0	0	0	1	2	2.5
Acrylic water dispersion	0	2	5	12	18	27
Hot-dip galvanising	0	15	20	42	63	75
Duplex system	0	0	0	0	Less than 0.5	



3: *Transformation of zinc coating into corrosion products during the cyclic test*



4: *Blistering and point corrosion of duplex (left) and zinc coating (right)*



5: *Thickness of the coating as scanned by an Elcometer ferromagnetic probe*

Blistering on the protective coating as shown in Fig. 4 is evidence of depreciation of the barrier effect of the anticorrosion system. The blisters indicate points where the coating lost adhesion to the substrate or the individual layers lost adhesion to each other. The number of blisters in the tested samples indicates the overall quality of the anticorrosion film. The occurence of blisters in water soluble acrylate coatings was evident already on the second day after exposure to salt spray. In the duplex anticorrosion system, the first occurrences were recorded after 15 days of exposure to NaCl.

The overall evaluation of corrosion degradation is shown in Fig. 5. Since organic coatings absorb water, there is a higher occurrence of blisters in water soluble coating systems. This negative effect clearly contributes to the reduction in the anticorrosion ability of the given system.

DISCUSSION

All the applications of anticorrosion coating were performed in identical laboratory conditions. The thickness of the anticorrosion coatings was fixed to up to 100 µm. The duplex system, which consists of zinc + alkyd-urethane film, showed average value of 65 µm of zinc substrate + 45 µm of coating. However, the issue with organic anticorrosion system may lie in the porosity of the substrate. Cavities and pores in the anticorrosion system also decrease the adhesion between the anchoring profile and the anticorrosion layer. This was also detected in the water soluble coating system, where the values obtained from pull-off testing were approximately 3 MPa. The values obtained for other systems were approximately 1.9 times higher. However, cupping tests have shown high toughness and elasticity of water soluble and alkyd-urethane anticorrosion systems.

The main specification for the application of these anticorrosion systems in environments containing industrial and farm fertilisers are the results of corrosion tests. The best results in cyclic testing in DAM 390 liquid fertiliser were achieved by the duplex system. Corrosion attack in the tested samples after 60 days of testing did not exceed more than 1 % of the sample surface. The duplex system also achieved lowest attack rate in salt spray testing. Alkyd-urethane coating alone on steel substrate was corrosively attacked already after 10 days of salt spray testing as evidenced by the formation of blisters from the edge of the sample onwards. The cause may be the lower proportion of additives in the coating. Good results were achieved by the synthetic acrylic coating, where only local point corrosion appeared during the test. The laboratory test also confirmed the unsuitability of water soluble coating systems for environments under chemical reactivity.

CONCLUSION

The degradation processes in all anticorrosion systems can be divided into two categories: mechanical degradation, where the flexibility, adhesion, elasticity etc. of the coating changes, and degradation of the coating substrate itself, which is no longer capable of creating a 100 % barrier between the underlying material and the surrounding environment.

The tests have clearly confirmed the influence of thickness and above all the chemical composition of protective coating on the anticorrosion capabilities of the system. Based on the mechanical and corrosion tests performed, it is clear that duplex systems are well suited for environments with high corrosion activity. It is an adequate combination of anticorrosion protection created by both a barrier and a passivation layer. Based on cyclic and constant corrosion testing, anticorrosion coating based on acrylic organic dispersion is recommended for duplex systems. However, it is important to note the increased risk of coating delamination, as confirmed by cupping tests according to Erichsen. The higher purchase cost of duplex anticorrosion system is clearly balanced out by the increase in the longevity of the entire machine.

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