TESTING THE INFLUENCE OF THE COMPOSITION OF ANTI-CORROSION PIGMENTS, FILLERS AND CONDUCTIVE POLYMERS ON THE ANTI-CORROSION EFFICIENCY AND PHYSICAL-MECHANICAL PROPERTIES OF THE POLYASPARTIC COATING SYSTEM AND COMPARISON OF TWO TYPES OF POLYISOCYANATE HARDENERS

TESTOVÁNÍ VLIVU SLOŽENÍ ANTIKOROZNÍCH PIGMENTŮ, PLNIV A VODIVÝCH POLYMERŮ NA ANTIKOROZNÍ ÚČINNOST A FYZIKÁLNĚ-MECHANICKÉ VLASTNOSTI POLYASPARTÁTOVÉHO NÁTĚROVÉHO SYSTÉMU A POROVNÁNÍ DVOU TYPŮ POLYISOKYANÁTOVÝCH TUŽIDEL

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Summary

For the corrosion protection of metal materials, a number of paint binders are used in conjunction with suitable anti-corrosion pigments, the formulations of these paint materials are also optimized using a number of fillers or various additives. Chemically drying binders based on 2K epoxies and 2K polyurethanes are often used for the formulation of anti-corrosion coatings and entire protective systems. Currently, coatings and systems based on so-called polyaspartic binders appear to be very promising, in terms of low energy requirements for achieving final curing and very good technical and mechanical parameters. The systems can cure quickly without the need for additional heating, both at low temperatures and below freezing point down to 10 °C.

To optimize these coating systems, coatings based on polyaspartic binders containing inorganic anti-corrosion pigments, conductive polymers and inorganic fillers at different concentrations were formulated and prepared on a laboratory scale. The anti-corrosion properties of the coatings were tested in an atmosphere of neutral salt fog and continuous condensation, and the physical properties and mechanical resistance of the tested coatings were also determined. Based on the evaluation of all testing, the optimal composition of the formulation for the polyaspartate coating system was selected in terms of corrosion protection and high mechanical resistance.

Key words

Polyaspartic coatings, inorganic fillers, anticorrosive pigments, conductive polymers, polyaniline.

INTRODUCTION

Corrosion protection of industrial metallic structures is of vital importance and intensive research work is carried in this field since decade. Economical loss due to corrosion globally is around 2.5 trillion USD [1]. By applying corrosion protection methods, it is estimated that around 15–35 % loss from corrosion could be recovered. Coating the metallic structure is an effective method to prevent corrosion and increase the working life of structure. Anticorrosive property of coatings is prime requirement when it comes to critical metallic structures like pipelines, ship hulls, bridges, statues, wagons. Polyaspartic system combines the best properties of a two-coat or three-coat protection system for steelwork, while taking production throughput to the next level.

Polyaspartic coating is well known as one of popular eco-friendly coatings because of non VOC and excellent mechanical properties such as fast curing, abrasion resistance, chemical resistance, light resistance and so on. Despite of a number of advantages applying polyaspartic coatings to industries, there are limitations to replace conventional polyurea and waterborne coatings in particularly continuous processing lines because of its very short pot life and relatively high price. The development of polyaspartic esters is fairly recent, with the initial work being reported in 1990. Zwiener, et al., first showed the applicability of polyaspartic esters as co-reactants for polyisocyanates. This patented technology was initially used in two-component polyurethane solvent-borne coating formulations because the polyaspartic esters are excellent reactive diluents for high-solids polyurethane coatings. They can be blended with hydroxyl functional polyester and polyacrylate co-reactants, thus allowing for reduction of volatile organic compounds (VOC) in relatively high solvent-containing coating systems. More recent developments have concentrated on achieving low or near-zero VOC-containing polyurea coatings where the polyaspartic ester is the main component of the co-reactant for reaction with a polyisocyanate. Because of the moderately fast curing feature, these coatings can offer productivity improvements, along with highbuild, low-temperature curing, and abrasion and corrosion resistence [2].

The polyaspartic direct-to-metal coating also features durable UV stability and low VOC content and long-lasting anti-corrosive protection for up to 25 years [3].

The goles of study

Protective coating as per current standard, which is a three-coat system of zinc-rich primer/epoxy/polyurethane paint is widely used. However, a new class of coating systems consisting of a zinc-rich primer topcoated with fast-dry, high-build (thick film).

Polyaspartics promises anti-corrosive results that are comparable in some situations with the three-coat systems. These two-coat systems eliminate the intermediate epoxy layer, so painting a steel overpass can be completed overnight. When application specifications are followed, two-coat systems can reduce labor as well, increasing worker productivity and decreasing the overall cost of coating applications [4]. The curing reaction between -NH of aspartic and -NCO of blocked isocyanate and properties of coatings were improved by modifications in formulation of paints. Therefore, this article discusses about analyzing performance of polyaspartic coatings formulated with different kinds of Inorganic fillers, Anticorrosive pigments and conductive polymers. The mechanical resistance of the prepared films was evaluated using the results of mechanical tests. The anticorrosive efficiency of the prepared films was evaluated using the results of corrosion tests SST and Humidity.

Therefore, study of polyaspartic coatings by formulation it with incorporation of different types of commercially available inorganic fillers and anticorrosive pigments to get the optimum corrosion efficiency is studied. Overall whole study can be classified in three parts.

Part 1 – Evaluation of anticorrosive property of polyaspartic coatings by incorporation of different inorganic fillers.

Part 2 – Evaluation of anticorrosive property of polyaspartic coatings by incorporation of different anticorrosive pigments and varying its concentration.

Part 3 – Evaluation of anticorrosive property of polyaspartic coatings by incorporation of different conductive polymers and varying its concentration.

Raw materials used were as follows.

Tab. 1: Description of raw materials used to formulate and testing of polyaspartic coatings

Type	Material
Resin	Polyaspartic resin 1 (med. reactivity)
	Polyaspartic resin 2 (low. reactivity)
Inorganic Filler	Filler 1 (natural baryte 1)
	Filler 2 (natural baryte 2)
	Filler 3 talc / dolomite
	Filler 4 litopone 30% ($ZnS/BaSO_4$)
	Filler 5 magnesium oxide - nano (MgO)
Anticorrosive Pigments	AC pigment 1 (zinc phosphate)
	AC pigment 2 (zinc free)
Conductive Polymers	CP1 (polyaniline – H_3PO_4)
	CP2 (polyaniline – PTSA)
Hardener	Hardener 1 (silane –functional aliphatic HDI)
	Hardener 2 (mixture of flexible aliphatic HDI – 9:1)

EXPERIMENTAL PART

Coatings were prepared in dissolver at 1300–1500 rpm, glass beads were used as grinding medium. Usuall time for grinding paint was 40 mins. Coatings were applied to steel and glass panels and further investigated for mechanical and corrosion properties.

Preparation of polyaspartic coatings by only inorganic fillers

In part 1 of the study, coatings were formulated by using inorganic fillers. There was total six batches were prepared which are as follows:

P1B1- Filler 1 (natural baryte 1) – 20%
P1B3- Filler 3 (talc / dolomite) – 20%
P1B5- Filler 5 (MgO) – 5% + Filler 3 (talc / dolomite) – 15%
P1B2- Filler 2 (natural baryte 2) – 20%
P1B4- Filler 4 (litopon) – 20%
P1B6- Filler 5 (MgO) – 10% + Filler 3 (talc / dolomite) – 10%

Preparation of polyaspartic coatings by anticorrosive pigments

In part 2 of the study, coatings were formulated by using anticorrosive pigments and inorganic fillers. Anticorrosive pigments were added by reducing the concentration of filler 3. There was total six batches were prepared which are as follows:

P2B1- AC Pigment 1 (zinc phosphate) – 3%
P2B4- AC Pigment 2 (zinc free) – 1%
P2B2- AC Pigment 1 (zinc phosphate) – 5%
P2B5- AC Pigment 2 (zinc free) – 2%
P2B3- AC Pigment 1 (zinc phosphate) – 7%
P2B5- AC Pigment 2 (zinc free) – 2%

Preparation of polyaspartic coatings by conductive polymer

In part 3 of the study, coatings were formulated by using conductive polymers and inorganic fillers. Conductive polymers were added by reducing the concentration of filler 3 (talc / dolomite). Two types of conductive polymers were used CP1 (polyaniline-H_3PO_4) and CP2 (polyaniline-PTSA). There was total six batches were prepared which are as follows:

P3B1- CP1 (polyaniline-H_3PO_4) – 1%
P3B4- CP2 (polyaniline-PTSA) – 1%
P3B2- CP1 (polyaniline-H_3PO_4) – 2%
P3B5- CP2 (polyaniline-PTSA) – 2%
P3B3- CP1 (polyaniline-H_3PO_4) – 3%
P3B6- CP2 (polyaniline-PTSA) – 3%

Application of coatings

2K Polyaspartic coatings were applied by using two different types of hardeners (Hardener 1 and Hardener 2). Steel and glass panels were applied by use of applicators having varying WFT. Usually coating on steel panels were applied with WFT 300 µm and on glass with WFT 150 µm.

Mechanical properties of the coatings

Several performance tests were carried out to check the mechanical properties of different coating films. The physical–mechanical quantities indicate the flexibility, the elasticity and the strength of the paint film. These tests provide a basis for studying the mechanism of action of protective organic coatings. The physico-mechanical evaluation was carried out after application on steel panels – size 100 mm × 50 mm × 0.5 mm (Standard low-carbon steel panels QD-24, Q-Lab Corporation). The dry film thickness (DFT) was measured with a magnetic gauge according to ISO 2808.

The degree of the adhesion of the coatings by a lattice method (ISO 2409)

Determination was made by means of a special cutting blade with cutting edges 2 mm apart and involved the degree of adhesion of the created 2 mm × 2 mm squares to a base substrate.

Corrosion test procedures

Environmental influence on the paints is studied by various corrosion tests where the atmospheric conditions are intensified. Film degradation, and primarily the extent of corrosion under the paint film on a protected base are studied. The accelerated corrosion test was carried out in an NaCl atmosphere with water steam condensation. The first test paints were applied on the steel panels - size 152 mm × 102 mm × 0.8 mm (Standard low-carbon steel panels S-46, Q-Lab Corporation) by an applicator with a 300-µm slit. The dry film thickness (DFT) was measured with a magnetic gauge according to ISO 2808.

The degree of blistering on the surface of the coatings (ASTM D 714-78), the degree of corrosion at the test scribe (ASTM D 1654-92) and the degree of steel surface corrosion (ASTM D 610-85) were evaluated after the exposure in the corrosive environments after 120, 480 and 720 hours.

Condensation test - Humidity chamber exposure (ISO 6270)

The exposure of the samples in a testing chamber was performed, the machine is used to evaluate the moisture resistance of the coating system in the high humidity environment of continuous condensation. The condensation test machine simulates rain and dew damage to the coating by producing continuous condensation on the surface of the sample under test.

RESULTS AND DISCUSSION

Results of the mechanical properties of coating systems

The results of physico-mechanical tests are given in Table 2 (a–c). Results of hardeness and degree of adhesion is mentioned. Adhesion is excellent of almost all the coatings whereas the hardeness is different.

Tab. 2a: Mechanical properties of the polyaspartic coatings (part 1) WFT = 150 ± 5 µm

Batch	Hardener	Cross cut test [dg.]	Hardness König 7 Days [swing count]
Filler 1 (natural baryte 1) – 20%	Hardener 1	0/5B	58
Filler 1 (natural baryte 1) – 20%	Hardener 2	1/4B	39
Filler 2 (natural baryte 2) – 20%	Hardener 1	0/5B	57
Filler 2 (natural baryte 2) – 20%	Hardener 2	1/4B	45
Filler 3 (talc / dolomite) – 20%	Hardener 1	0/5B	47
Filler 3 (talc / dolomite) – 20%	Hardener 2	4/1B	39
Filler 4 (litopone) – 20%	Hardener 1	0/5B	50
Filler 4 (litopone) – 20%	Hardener 2	1/4B	47
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 1	0/5B	49
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 2	0/5B	54
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 1	0/5B	54
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 2	2/3B	57

Tab. 2b: Mechanical properties of the Polyaspartic coatings (part 2) WFT = 150 ± 5 µm

Batch	Hardener	Cross cut test [dg.]	Hardness König 7 Days [swing count]
AC pigment 1 – 3%	Hardener 1	0/5B	39
AC pigment 1 – 3%	Hardener 2	0/5B	45
AC pigment 1 – 5%	Hardener 1	0/5B	51
AC pigment 1 – 5%	Hardener 2	0/5B	49
AC pigment 1 – 7%	Hardener 1	0/5B	44
AC pigment 1 – 7%	Hardener 2	0/5B	42
AC pigment 2 – 1%	Hardener 1	0/5B	52
AC pigment 2 – 1%	Hardener 2	5/0B	47
AC pigment 2 – 2%	Hardener 1	1/4B	63
AC pigment 2 – 2%	Hardener 2	2/3B	48
AC pigment 2 – 3%	Hardener 1	0/5B	65
AC pigment 2 – 3%	Hardener 2	0/5B	51

Tab. 2c: Mechanical properties of the Polyaspartic coatings (part 3) WFT = 150 ± 5 µm

Batch	Hardener	Cross cut test [dg.]	Hardness König 7 Days [swing count]
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 1	0/5B	56
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 2	0/5B	46
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 1	0/5B	65
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 2	0/5B	46
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 1	3/2B	68
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 2	0/5B	46
CP 2 (polyaniline–PTSA) – 1%	Hardener 1	0/5B	66
CP 2 (polyaniline–PTSA) – 1%	Hardener 2	4/1B	45
CP 2 (polyaniline–PTSA) – 2%	Hardener 1	0/5B	62
CP 2 (polyaniline–PTSA) – 2%	Hardener 2	0/5B	50
CP 2 (polyaniline–PTSA) – 3%	Hardener 1	1/4B	76
CP 2 (polyaniline–PTSA) – 3%	Hardener 2	4/1B	51

Assessment of the accelerated corrosion tests

The results of this corrosion test performed for the individual paints in a chamber with salt mist after 480h of exposure are given in Table 3 (a-c). From the results it is clear that occurrence of blistering was slightly observed on the organic coating. Another evaluated phenomenon was the corrosion in the cut. The last corrosion phenomenon to be evaluated was the corrosion on the steel panel.

Tab. 3a: Results of the corrosion tests performed in a salt mist chamber for coatings after 480 h of exposure (part 1), DFT = 100 ± 5 µm

Batch	Hardener	Blisters In cut [dg.]	Blisters In Surface [dg.]	Corrosion In Surface [%]
Filler 1 (natural baryte 1) – 20%	Hardener 1	2MD	6MD	3
Filler 1 (natural baryte 1) – 20%	Hardener 2	2MD	8MD	33
Filler 2 (natural baryte 2) – 20%	Hardener 1	4D	6MD	1
Filler 2 (natural baryte 2) – 20%	Hardener 2	2MD	6MD	16
Filler 3 (talc / dolomite) – 20%	Hardener 1	2F	4M	1
Filler 3 (talc / dolomite) –20%	Hardener 2	6D	6MD	33
Filler 4 (litopone) – 20%	Hardener 1	4F	6MD	0,3
Filler 4 (litopone) – 20%	Hardener 2	4D	8D	50
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 1	4F	6M	0.1
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 2	4M	8M	0.01
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 1	2M	6M	0.03
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 2	4D	6MD	16

Tab. 3b: Results of the corrosion tests performed in a salt mist chamber for coatings after 480 h of exposure (part 2), DFT = 100 ± 5 µm.

Batch	Hardener	Blisters In cut [dg.]	Blisters In Surface [dg.]	Corrosion In Surface [%]
AC pigment 1 – 3%	Hardener 1	4D	-	0.03
AC pigment 1 – 3%	Hardener 2	4D	-	0.1
AC pigment 1 – 5%	Hardener 1	6F	-	0.03
AC pigment 1 – 5%	Hardener 2	4M	-	0.03
AC pigment 1 – 7%	Hardener 1	4MD	-	0.03
AC pigment 1 – 7%	Hardener 2	4MD	-	0.03
AC pigment 2 – 1%	Hardener 1	2M	4F	0.03
AC pigment 2 – 1%	Hardener 2	4D	4MD	10
AC pigment 2 – 2%	Hardener 1	2MD	4F	0.01
AC pigment 2 – 2%	Hardener 2	4D	6M	10
AC pigment 2 – 3%	Hardener 1	4MD	4F	0.01
AC pigment 2 – 3%	Hardener 2	2MD	8M	3

Tab. 3c: Results of the corrosion tests performed in a salt mist chamber for coatings after 480 h of exposure (part 3), DFT = 100 ± 5 μm

Batch	**Hardener**	**Blisters In cut [dg.]**	**Blisters In Surface [dg.]**	**Corrosion In Surface [%]**
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 1	2MD	4M	0.3
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 2	4MD	6M	33
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 1	2F	6F	1
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 2	2MD	6D	50
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 1	2MD	8M	10
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 2	4F	8MD	16
CP 2 (polyaniline–PTSA) – 1%	Hardener 1	4F	8F	0.3
CP 2 (polyaniline–PTSA) – 1%	Hardener 2	2MD	8MD	33
CP 2 (polyaniline–PTSA) – 2%	Hardener 1	2MD	8M	10
CP 2 (polyaniline–PTSA) – 2%	Hardener 2	2D	8D	33
CP 2 (polyaniline–PTSA) – 3%	Hardener 1	2F	8F	10
CP 2 (polyaniline–PTSA) – 3%	Hardener 2	2MD	8MD	16

The results of this corrosion test performed for the individual paints in a chamber with condensation after 480h of exposure are given in Table 4 (a–c).

Tab. 4a: Results of the corrosion tests performed in a Humidity chamber for coatings after 480 h of exposure (part 1), DFT = 100 ± 5 μm

Batch	**Hardener**	**Blisters In Surface [dg.]**	**Corrosion In Surface [%]**
Filler 1 (natural baryte 1) – 20%	Hardener 1	8F	0.3
Filler 1 (natural baryte 1) – 20%	Hardener 2	8MD	10
Filler 2 (natural baryte 2) – 20%	Hardener 1	8F	0.03
Filler 2 (natural baryte 2) – 20%	Hardener 2	8MD	10
Filler 3 (talc / dolomite) – 20%	Hardener 1	X	0.01
Filler 3 (talc / dolomite) – 20%	Hardener 2	4MD	33
Filler 4 (litopone) – 20%	Hardener 1	-	0.03
Filler 4 (litopone) – 20%	Hardener 2	8MD	16
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 1	–	0.01
Filler 5 MgO 5% + Filler 3 (talc / dolomite) – 15%	Hardener 2	8F	0.03
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 1	–	0.01
Filler 5 MgO 10% + Filler 3 (talc / dolomite) – 10%	Hardener 2	8MD	16

Tab. 4b: Results of the corrosion tests performed in a Humidity chamber for coatings after 480 h of exposure (part 1), DFT = 100 ± 5 µm

Batch	Hardener	Blisters In Surface [dg.]	Corrosion In Surface [%]
AC pigment 1 – 3%	Hardener 1	–	0.01
AC pigment 1 – 3%	Hardener 2	8F	0.1
AC pigment 1 – 5%	Hardener 1	–	0.01
AC pigment 1 – 5%	Hardener 2	4M	16
AC pigment 1 – 7%	Hardener 1	–	0.01
AC pigment 1 – 7%	Hardener 2	8F	0.01
AC pigment 2 – 1%	Hardener 1	–	0.01
AC pigment 2 – 1%	Hardener 2	4MD	1
AC pigment 2 – 2%	Hardener 1	–	0.01
AC pigment 2 – 2%	Hardener 2	6D	0.3
AC pigment 2 – 3%	Hardener 1	8F	0.01
AC pigment 2 – 3%	Hardener 2	6D	0.03

Tab. 4c: Results of the corrosion tests performed in a Humidity chamber for coatings after 480 h of exposure (part 1), DFT = 100 ± 5 µm

Batch	Hardener	Blisters In Surface [dg.]	Corrosion In Surface [%]
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 1	8F	0.3
CP 1 (polyaniline–H_3PO_4) – 1%	Hardener 2	6D	33
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 1	–	0.1
CP 1 (polyaniline–H_3PO_4) – 2%	Hardener 2	8D	33
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 1	8F	0.3
CP 1 (polyaniline–H_3PO_4) – 3%	Hardener 2	8D	33
CP 2 (polyaniline–PTSA) – 1%	Hardener 1	–	0.01
CP 2 (polyaniline–PTSA) – 1%	Hardener 2	4MD	50
CP 2 (polyaniline–PTSA) – 2%	Hardener 1	8F	0.3
CP 2 (polyaniline–PTSA) – 2%	Hardener 2	6D	50
CP 2 (polyaniline–PTSA) – 3%	Hardener 1	–	0.01
CP 2 (polyaniline–PTSA) – 3%	Hardener 2	4MD	50

CONCLUSIONS

This study focuses on developing and analyzing polyaspartic coatings with use of different commercially available inorganic fillers, anticorrosive pigments and conductive polymers. The results of accelerated corrosion tests Salt Spray and Humidity demonstrate the following.

Comparing performance of both the hardener No.1 (silane –functional aliphatic HDI) and hardener No. 2 (mixture of flexible aliphatic HDI – 9:1) it is evident from the results of SST and Humidity that hardener 1 is way better in anticorrosion performance than hardener 2.

From Part 1 of experiments, observing the results of different batches it can be concluded that P1B5 = (MgO 5% + Filler 3 (talc / dolomite 15%) and P1B6 = (MgO 10% + Filler 3 (talc/ dolomite) 10% showed excellent corrosion efficiency both in SST and Humidity chambers after 480 hrs as compared to several other fillers.

From Part 2 of experiments, observing the results of different batches it can be concluded that P2B1 = AC pigment 1 (zinc phosphate) 3% and P2B2 = AC pigment 1 (zinc phosphate) 5 % and P2B3 = AC pigment 1 (zinc phosphate) (7%) showed excellent corrosion efficiency both in SST and Humidity chambers after 480 hrs. So it can be concluded that performance of AP pigment 1 is better as compared to anticorrosive pigment AP pigment 2.

From Part 3 of experiments, observing the results of different batches it can be concluded that P3B2 = CP1 (polyaniline - H_3PO_4) 2% and P3B4 = CP2 (polyaniline - PTSA) 1% showed excellent corrosion efficiency both in SST and Humidity chambers after 480 hrs. conductive polymer CP2 (polyaniline PTSA) showed better overall performance than CP1 polyaniline - H_3PO_4.

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