OPTIMIZATION OF PARAMETERS OF THE PLASTIC-COATED SHEETS AT THE CORROSION TEST IN SALT SPRAY

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The paper deals with optimization of parameters obtained by corrosion test in a salt spray chamber at plastic-coated sheets, which are used in various areas of economy, e.g. automotive industry. The goal of the experiment was to find out the relation between input parameters (Zn layer, Top+primer, Backout) and output parameter – corroding time by means of Response Surface Methodology (RSM). The experiment was planned with the use of full factorial design. Evaluation of experimental results was processed by means of Minitab 16 statistical software.

KEYWORDS

plastic-coated sheets, test in salt spray chamber, corroding time, optimization of parameters, RSM - Response Surface Methodology

1 INTRODUCTION

In the area of technologies and utilization of construction materials the special emphasis is put on knowledge of mechanical, electric, chemical, but also magnetic properties of materials depending of their structure. Deterioration of materials by chemical or physical and chemical effects of environment is becoming more and more serious problem. Natural corrosion environments (atmosphere or water) are polluted by industrial activities and pollution causes that their aggression is rising. At the same time requirements for reliability and no-failure operation of materials are increasing, because disruption to their functions can have serious consequences in wide range of various areas of economy. To secure the longest possible durability life of products it is necessary to protect them from unwanted influences of environment, which become evident on the surface in the form of corrosion. Zinc coated sheets are long attested materials mainly at production of trapezoidal and corrugated sheets, folding roof covering, sheet covering of roofs, windows and last but not least in automotive industry. Undeniable advantage of zinc coated sheets is their low price. Big disadvantage is their low durability and currently low aesthetic value. Zinc coated sheets are laminated materials, in which the most favourable steel properties are connected with protective application of zinc layer against corrosion [Oppelt 2005]. Fluorescent zinc coating protects steel from corrosion in profitable and ecological ways and prolongs its durability – anti corrosion protection lasts for several decades [Cerny 1984], [Billy 1996]. The principle of the method of protection of material surface is connecting liquid zinc in 450 °C hot melting bath with steel, which needs to be protected. Massive metal zinc coating covers full area of the material in a perfect way with layer thickness from 50 to 150 µm. Zinc and steel react mutually. An alloy of iron and zinc is created on the steel surface. Protection, which markedly differs from other processes, develops from this inseparable connection of zinc with steel. Surfaces treated by fluorescent zinc coating are protected not only from the influence of wind and weather, but also optimally from mechanical load. Fluorescent zinc coating protects material from rusting. It is even able to close smaller damages with its cathodic protection.

Metal varnish of steel as well as the structure of steel surface remain in a good condition at the same time. However, zinc coated materials underlie corrosion over time. That is why it is recommended to apply coating on these sheets that need to be renewed, which ultimately increases the price. Reversible coating of zinc layer protects not only in mechanical, but also in chemical ways – it means in anticorrosive way [Tarhanicova 2008]. Another degree of surface treatment of cold-rolled zinc coated sheets is plastic-coated surface treatment. The layer of priming paint, so called primer [Tarhanicova 2005], which secures corrosion resistance of the entire system of coatings, is applied to bilateral hot zinc coated sheet with zinc layer and it is also applied by passivation. The final layer is usually formed by plastic-coated surface treatment on polyester basis with minimum thickness of 25 µm (shiny shade) and 35 µm (mat shade). Coated plastic is on the obverse and reverse at bilaterally painted plastic-coated sheets. At unilateral painted plastic-coated sheets there is only basic primer on the reverse. Top detachable foil as protection against scratching and depreciation of coloured layer of sheet is matter of course of supplied plastic-coated sheets (Fig. 1) [Evin 2016], [Tomkova 2004], [Kollarova 2002].

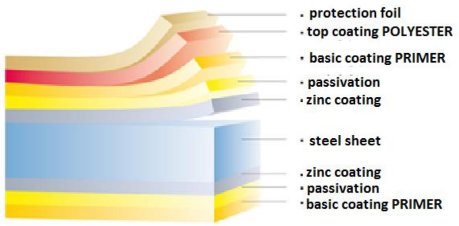

Figure 1. Structure of plastic-coated sheet.

Table 1 contains coating types, their terminology and thickness of their applied layers. Coloured shade of coatings is prescribed according to the International Colour Code RAL. Other coloured shades are possible after they are agreed.

Bottom coating - BACKOAT	thickness of 10 µm
Basic coating - PRIMER	thickness of 5 µm
Top coating - TOPCOAT	thickness of 25 µm
POLYESTER (PES)	applications inside and outside, industry [1]
POLYVINYLDENFLUORIDE (PVDF)	good resistance against corrosion, abrasion, chemicals

Table 1. Coating types.

2 EXPERIMENTAL

2.1 *Setting Up The Corrosion Test By Salt Spray And Principle Of The Method*

To set up the corrosion test by salt spray the international norm STN EN ISO 9227:2012 [STN EN ISO 9227:2012] was used. The norm specifies device, tannins and test procedures in neutral salt spray (NSS), salt spray acidified by acetic acid (AASS) and salt spray acidified by acetic acid that is accelerated by copper

(CASS) to assess the resistance against corrosion of metallic materials with permanent or temporal protection against corrosion and without it [6]. Tested sample is exposed to effects of sprayed salt spray for predetermined time. Possible occurrence of corrosion expressed by the degree of coating crack and degree of pustule formation is evaluated. The method describes possible process for testing resistance of materials with organic coating by effect of salt spray. Tests are carried out in a condensation chamber, where required environment can be prepared. Construction of the condensation chamber allows to change and keep conditions of the test, temperature and relative humidity by means of heater element and water bath of the corrosion chamber. Environment we used was created by sodium chloride (NaCl), distilled water and sodium carbonate (Na_2CO_3). The samples of plastic-coated sheets with dimensions of 200 mm x 150 mm were used to measure corroding time in the salt chamber. Dimension of 200 mm of panel sheet must be equal to the direction of rolling. Two cuts 40 mm long 90° angle-wise were made on the sample. Lower cut must head for horizontal central point and must be 50 mm distant from it. Vertical cut must be 60 mm distant from lower edge. 90° variable bend radius, which is parallel to the left edge (that is 200 mm long) and 25 mm distant from the left edge, was made. Variable bend radius is given by bend in the interval $\langle 1T, 3T \rangle$, where T is width of bent part of the sample.
Salt chamber CONSTASAL S 1000M was used for measurement. Its description, maintenance, calibration and its technical parameters are described in work regulations for operation of the device. Cutting tool with a solid steel spike was used to create a cut. The cut needs to have extension cross part, which reveals foundation metal that is approximately 1 mm wide (according to Clemen) (Fig. 2). Indentation must show the profile of V shape and must reveal at least 0,2 mm of metal back coat. The suitable shape of layout tools is a solid metal spike with radius approximately of 0,5 mm (van Laar). Clear self-adhesive tape was 25 mm wide with (10±1) N adhesion on 25 mm width according to IEC 60454-2.

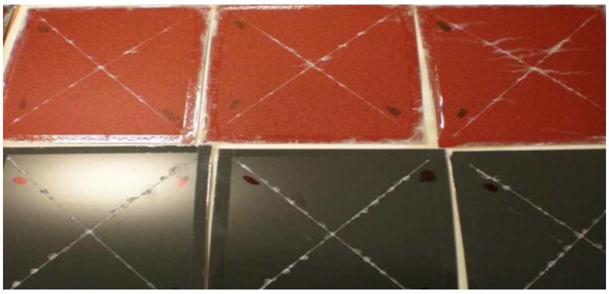

Figure 2. Stress-corrosion cracking.

2.2 Measuring Procedure In The Salt Chamber

1. 5 % salt solution was prepared (1 kg NaCl is dissolved in 20 litres of distilled water), pH of prepared solution must be in the interval 6 – 7.
2. Tank of the salt chamber was filled by salt solution.
3. Carriers with samples were placed into the salt chamber. The samples should not be placed above a jet and a diffusion cone.
4. Main switch was pressed to launch the test. Key shift with zero position was switched to spraying.
5. Indicator light of function of heating of the testing chamber SAL and indicator light of function of heating of humidifier were lit up. Indicator lights were switched off, when optimal temperatures (temperature of 35 °C in the salt chamber, humidification temperature of 50 °C) and pressure of 1bar were reached.
6. When the chamber was switched off, key shift with zero position was switched to ventilation. During measurement it was controlled if the jet works correctly. If the jet is clogged, it has to be cleaned. It is also necessary to control the amount of created condensate: 1 – 2 ml of condensate on the surface of 80 cm^2 should be created within 1 hour of the test and pH of the condensate should be between 6,5 and 7,2.

2.3 Processing Of Results

1. Corroding was measured in the following way: self-adhesive tape was stuck along the cut after drying out at laboratory temperature 24 hours after the chamber was switched off.
2. The tape was smoothed at the point of surface cut at least in the distance of 20 mm behind every cut end. The tape was toughly pressed by fingertips. It was removed within 5 minutes, seized for free end and gradually took off for 0,5 – 1 s approximately 60° angle-wise to the tested sample.

3 OBTAINED RESULTS AND THEIR DISCUSSION

3.1 Setting Up The Corrosion Test By Salt Spray And Principle Of The Method

Measurements of corrosion of plastic-coated samples in salt spray was carried out at U. S. Steel Košice – Labortest Ltd. in a testing laboratory of the Cold rolling mill. Measurement results are recorded in Table 2. Measured data were subsequently statistically processed.

Sample identification: zinc coated sheet with organic coating						
Required tests: norm used: corrosion test in salt spray (STN ISO 9227)						
Exp. No.	Std Ord.	Blocks	Zn-layer [μm]	Top+ primer [μm]	Backoat [μm]	Corroding Time [hrs]
1	7	1	139	25	10	1128
2	5	1	139	5	10	480
3	11	1	139	25	7	1128
4	16	1	144	25	10	1128
5	6	1	144	5	10	480
6	12	1	144	25	7	888
7	14	1	144	5	10	312
8	13	1	139	5	10	384
9	10	1	144	5	7	312
10	9	1	139	5	7	384
11	15	1	139	25	10	888
12	4	1	144	25	7	888
13	2	1	144	5	7	312
14	3	1	139	25	7	1128
15	1	1	139	5	7	480
16	8	1	144	25	10	888

Table 2. Measured values obtained from the experiment.

Software package Minitab 16 was used for statistical processing of results. In order to process measured results, full factorial design with 3 factors and 2 levels was created. It means that number of measurements should be 2^3. Since there are not any central points, the number of blocks is 1, the number of repeated measurements (for 8 coils – samples) at all non-central points of experimental plan is 2, so the entire number of measurements was $2x2^3$, i.e. 16. Randomization of experiment was created in order not to allow the influence of previous and following measurements. The analysis of factorial design in linear model, which includes elements up to degree 2 (element up to degree 3 presents scratch and was not taken into consideration), was carried out. Selected factors are indicated in the following way: factor A - Zn-layer, factor B - Top+primer, factor C - Backoat and interactions of factors A·B, A·C, B·C. Tables of factorial regression - Table 3, where analysis of variance for linear model was carried out and Tab. 4, where significance of factors and interactions is inscribed, were obtained at analysis of full factorial design. Software Minitab 16 creates considerable number of charts at analysis of factorial design. Pareto Chart of the Standardized Effects and Normal Plot of the Standardized Effects, which provide us sufficient amount of information about the influence of factors or their interactions on response, were chosen.

Analysis of Variance					
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	6	1583640	263940	27,30	0,000
Linear	3	1553868	517956	53,57	0,000
Zn-layer	1	39204	39204	4,06	0,075
Top+primer	1	1512900	1512900	156,49	0,000
Backoat	1	1764	1764	0,18	0,679
2-Way Interactions	3	29772	9924	1,03	0,426
Zn-layer*Top+primer	1	1764	1764	0,18	0,679
Zn-layer*Backoat	1	26244	26244	2,71	0,134
Top+primer*Backoat	1	1764	1764	0,18	0,679
Error	9	87012	9668		
Lack-of-Fit	1	6084	6084	0,60	0,460
Pure Error	8	80928	10116		
Total	15	1670652			

Table 3. Factorial regression: Corroding time on Zn-layer, Top+primer, Backout.

Estimated Effects and Coefficients for Corroding Time					
Term	Effect	Coef	SE Coef	T	P
Constant		700,50	24,58	28,50	0,000
Zn-layer	-99,00	-49,50	24,58	-2,01	0,075
Top-primer	615,00	307,50	24,58	12,51	0,000
Backoat	21,00	10,50	24,58	0,43	0,679
Zn-layer*Top-primer	-21,00	-10,50	24,58	-0,43	0,679
Zn-layer*Backoat	81,00	40,50	24,58	1,65	0,134
Top-primer*Backoat	-21,00	-10,50	24,58	-0,43	0,679

Table 4. Significance of factors and interactions.

Effect of factors or interaction of factors – response on change of the factor from value -1 to value 1 is calculated in the first column of Table 4. Regression coefficient can be found in the second column, which is a half of effect of factors or interaction of factors. Statistical significance of factor or interaction expressed by p can be found in the fifth column. The closer the value p of factor or interaction of factors to zero, the more statistically significant factor or interaction of factors. Table with calculated effects and coefficients serves us as a source of information in order to create mathematical model, which would have the following shape [Miller 2010].

Regression equation (1):

$$\text{Corroding time} = 700{,}5 - 49{,}5 \cdot A + 307{,}5 \cdot B + 10{,}5 \cdot C - 10{,}5 \cdot A \cdot B + 40{,}5 \cdot A \cdot C - 10{,}5 \cdot B \cdot C \quad (1)$$

where selected factors are indicated as it follows: factor A - Zn-layer, factor B - Top+primer, factor C - Backoat and interactions of factors A·B, A·C, B·C.

The next step is to separate "significant" factors or interactions from those that create only experimental scratch. Pareto Chart of the Standardized Effects and Normal Plot of the Standardized Effects are used for that. It is possible to conclude that only factor B - Top+primer would have real influence on corroding time, when corrosion occurs (Fig. 3).

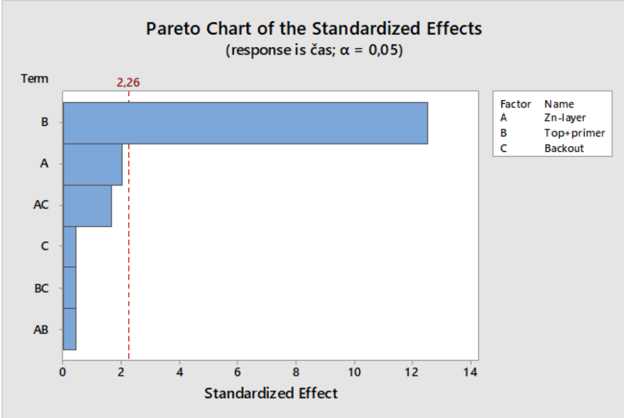

Figure 3. Pareto Chart of the Standardized Effects.

Points of effects of factors or interactions are described in the Normal Plot of the Standardized Effects (Fig. 4) and separated by a straight line. The points, which lie on the straight line, belong to scratch. The points, which lie beyond the straight line, belong to "real" factors. It is in accordance with the result obtained from Pareto Chart (Fig. 3).

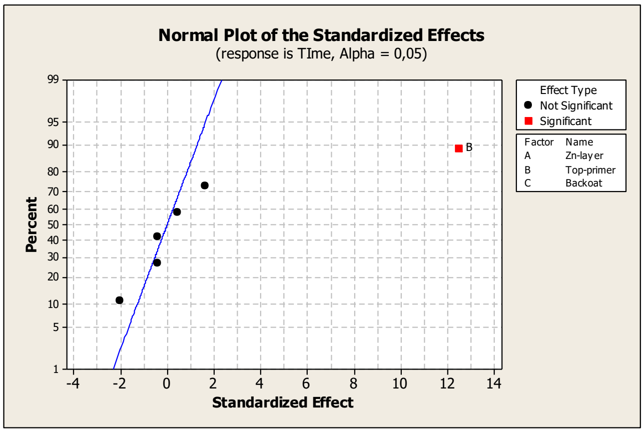

Figure 4. Normal Plot of the Standardized Effects.

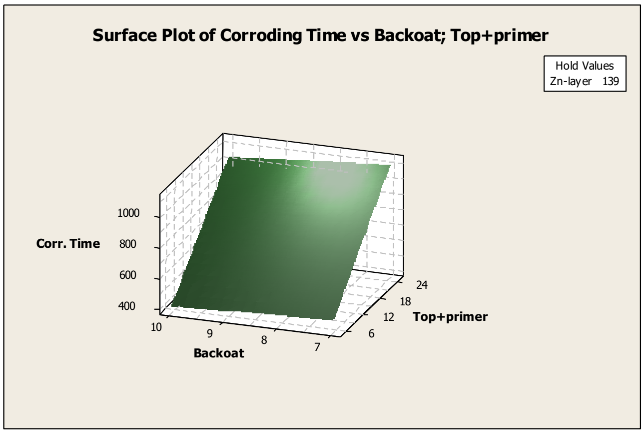

Figure 5. Surface Plot.

Three-dimensional Surface Plot, which displays shiny surface lighted from a suitable angle, provides even more demonstrative visualization of corroding time dependence on Top+primer and Backout at Zn-layer of 139 μm (Fig. 5). From Fig. 5 it is obvious that maximum value for parameter of corroding time is reached for Top+primer of 25 μm and Backoat of 7 μm and minimum value for corroding time is reached for Top+primer of 5 μm and Backaot of 10 μm. This display is available from Minitab16 - 3D graph tools program package.

3.2 Optimization Of Parameters

One of the main goals of the experiment was to define optimum values of parameters of samples in an effort to reach the longest corroding time of plastic-coated sheets. Optimization by using RSM method (Response Surface Methodology) helps to identify combinations of input parameters – factors: Zn-layer, Top+primer, Backout, which together influence corroding time of plastic-coated samples at the corrosion test in salt spray [Hadzima 2010], [Craig 1992], [Pierre 2012].

Mutual optimization must satisfy requirements for all observed variables. The result of optimization is measured by so called composite desirability, which presents weighted geometric mean of individual proprieties of responses within the interval scale ⟨0,1⟩. The unit represents ideal state. Zero indicates that one or more responses are beyond acceptable values. Table 5 displays results of RMS optimization for parameter of corroding time [Bohm 2006].

Parameters						
Response	Goal	Lower	Target	Upper	Weight	Importance
Corroding Time						
	Maximum	312	1128	1128	1	1
Variable Ranges						
Variable	Values					
Zn-layer	(139;144)					
Top+primer	(5; 25)					
Backoat	(7; 10)					
Starting Values						
Variable	Setting					
Zn-layer	139					
Top+primer	5					
Backout	7					
Solution						
Solution	1					
Zn-layer	Top+primer	Backout	Predi.Responce	Desirability		
139	25	7	1108,5	0,9761		
Multiple Response Prediction						
Variable	Setting					
Zn-layer	139					
Top+primer	25					
Backout	7					

Table 5. Response Optimization: Corroding Time.

4 CONCLUSION

Experiments were designed according to standard full factorial design to find out relations and creation of mathematical models between factors A - Zn-layer, B - Top+primer, C – Backcout and observed response – corroding time. Pareto Chart of the Standardized Effects and Normal Plot of the Standardized Effects were used to evaluate the influence of input factors on observed response. Optimization of input factors of plastic-coated sheets at the corrosion test in salt spray was carried out by using RSM method.

Based on experimental results and their statistical behaviour the following conclusions can be defined:

1. From above mentioned factors factor B -Top+primer coating [µm] compared to factor A - Zn-layer [µm] and factor C - Backout [µm] has the most significant influence on observed response of corroding time.
2. Optimum coatings were defined by means of RSM (Response Surface Methodology): Top+primer = 25 µm, Backout = 7 µm and Zn-layer = 139 µm to reach maximum value of observed variable – corroding time with significant statistical dependence of 97.61 %.
3. The life-time of plastic-coated sheets was compared with the life-time of a conventional zinc coating (hot deep galvanizing - HDG) - see Table 5 and article [Evin 2016], which show that the life-time of plastic-coated sheets is several times longer than the life–time of conventional zinc coating HDG, not to even mention the aesthetics of plastic-coated sheets used in construction building.
4. From economic point of view if optimal solution is enforced with the thickness of Zn-layer = 139 µm, and other non-optimal solution with the thickness of Zn-layer = 144 µm, several tens of euros would be saved at covering by plastic-coated sheets at the current price of zinc covering 100 m^2 (i.e. area of a family house) and keeping the longest possible durability.

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REFERENCES

[Billy 1996] Billy, J., Gaspar, V., Vargova, V., Pallagova, A., Vida, S. Zinc dipping of tensile newly designed IF construction and microalloyed steels. Final report of the partial task of research project, Licence No. P 5-12/3, pp. 61, October 1996

[Bohm 2006] Bohm, S. Evaluation of the effect of Chromate and Chromate free pretreatments on the durability of organic coatings on galvanised steel. In: Galvanised Steel Sheet Forum Dusseldorf, Deutchland, 2006

[Craig 1992] Craig, B. et al. ASM Handbook, 1992, Vol. 13, pp. 145

[Cerny 1984] Cerny, M. et al. Corrosion properties of metal construction materials. Praha: SNTL, 1984

[Evin 2016] Evin, E., Tomas, M., Graban, J., Toth, M. Analysis of the galvanised steel sheets resistance to corrosion after plastic deformation. Corrosion and Material Protection (in Czech: Koroze a ochrana materiálu), 2016, Vol. 60 (4), pp. 114-121

[Hadzima 2002] Hadzima, B., Liptakova, T., Palcek, P. Corrosion of magnesium and its alloys. In: Memorial volume of 17th international conference Corrosion in energetics 2002, HF TU Košice, Košice, 2002

[Kollarová 2002] Kollarova, M., Hrabcakova, L., Graban, M., Sohajova, M. Structural properties of Zinc and ZnMgAl coatings on steel sheets, Materials Science Forum, 2014, Vol. 782, pp. 623-626

[Miller 2010] Miller, I. DOE – Design and analysis of experiment by means of MINITAB. INTERQUALITY, spol s r. o., Praha, 2010. ISBN 9788090277052

[Oppelt 2005] Oppelt, J., Blahetova, M., Lasek, S., Cihal, V. Mechanics, 2005, Vol. 298, Politech. Opolska

[Pierrer 2012] Pierre, R Handbook of Corrosion Engineering, McGraw-Hill, 2012. ISBN 978-0-07-175037-0

[STN EN ISO 9227 2012] STN EN ISO 9227: 2012 (03 8132). Corrosion tests in artificial atmospheres - Salt spray tests (ISO 9227: 2012)

[Tarhanicova 2005] Tarhanicova, R. Metallographic analysis of organic-coated steels - white corrosion formation. Expertise No. MOP 606/05, December 2005

[Tarhanicova 2008] Tarhanicova, R. Metallographic analysis of organic-coated steels. Expertise No. MOP 99/08, February 2008

[Tomkova 2004] Tomkova, V., Graban, J. Evaluation of corrosion resistance of zinc-coated sheets with passivators from "A" producer. Expertise INOP – 43/2004

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