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THE INFLUENCE OF SURFACE PRETREATMENT ON COATING ADHESION IN CATAPHORETIC PAINTING OF ALUMINUM PARTS

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Abstract: *This study examines the impact of surface pretreatment on coating adhesion during cataphoretic painting of aluminum parts. Different degreasing agents were applied to aluminum test samples under varying experimental conditions determined through a planned design. Following the pretreatment stage, the samples were coated using cataphoretic deposition technology. Adhesion performance was evaluated by means of the grid-cut test in accordance with ISO 2409:2020. The experimental results revealed that the average coating thickness exerted the most significant influence on the adhesion of the cataphoretic coating.*

Key words: *Aluminum parts, cataphoretic painting, degreasing agent, grid test, adhesion.*

1. INTRODUCTION

Metal corrosion represents not only an economic issue but also an environmental and safety concern. Hence, effective corrosion prevention methods are essential. Organic coatings applied to metallic surfaces have been proven to efficiently slow corrosion by isolating the substrate from aggressive environments. However, the effectiveness of such coatings is affected by several factors, including coating composition, uniformity, and thickness. [1-4]

Studies investigating the behavior of cataphoretic coatings during aging have demonstrated that internal stresses accumulate over time, leading to degradation such as adhesion loss and increased porosity, particularly under fluctuating humidity and temperature. [5-7]

Other authors found that aging increases stress, leading to coating degradation such as loss of adhesion and increased porosity, especially when exposed to moisture and temperature changes. [6-9]

Recent research employing electrochemical impedance spectroscopy (EIS) and AC/DC/AC testing has provided rapid and reliable

assessments of coating durability. For example, investigations into lead-free epoxy coatings on aluminum substrates subjected to environmental aging revealed deterioration in barrier performance. Aluminum surfaces, due to their susceptibility to filiform corrosion, often require additional conversion treatments to enhance adhesion and corrosion resistance [8–11].

Several works have explored how process parameters such as deposition time, electrolyte immersion duration, solution pH, and coating thickness influence delamination and corrosion behavior of cataphoretic layers. These processes are often modeled using equivalent electrical circuits to interpret electrochemical responses and degradation mechanisms [12–14].

Further studies concerning aluminum alloys and filiform corrosion suggest that conventional pretreatments do not always yield satisfactory adhesion. Damage to the conversion layer during coating can compromise protection efficiency, although promising results under sodium chloride exposure imply potential benefits when alternative painting techniques are applied [15–17].

Plasma electrolytic oxidation (PEO) has also emerged as a sustainable surface engineering

method, forming ceramic oxide layers on aluminum and other light metals. This process employs plasma discharges at high voltages to generate dense, wear- and corrosion-resistant oxide coatings. Numerous studies have examined aspects of PEO such as anodic mechanisms in alkaline media [18–19], discharge properties [20], anodizing pretreatments [21], and coating durability on composites [22]. Recent research also highlights advances in eco-friendly polymer nanocomposite coatings for sustainable protection systems [23].

Building upon these insights, the present work focuses on assessing how specific surface pretreatment parameters—particularly degreasing variables—affect the adhesion quality of cataphoretic coatings on aluminum components.

2. MATERIAL AND METHODS

2.1 Cataphoresis painting

Cataphoretic painting, or electrophoretic deposition, is an electrochemical method for applying uniform, corrosion-resistant coatings onto conductive surfaces. In this process, paint particles suspended in an aqueous medium migrate under an electric field toward the metal substrate, which serves as the cathode. This results in an even, compact, and well-adhered coating layer. Cataphoretic painting offers several advantages over conventional methods, including high transfer efficiency, uniform coverage of complex geometries, excellent corrosion resistance, and reduced environmental impact due to its water-based nature and low solvent content [28-30].

2.2 Cross-cut test

The quality of the applied coatings was evaluated using the Cross-Cut Test according to ISO 2409:2020 [31]. An Elcometer 1542 device was employed to determine coating adhesion. The procedure involved making six parallel and six perpendicular cuts with 1 mm spacing, suitable for coatings up to 60 μm thick. Each sample was tested at five different sites, followed by gentle brushing and adhesive tape removal to eliminate loose particles. Testing was

conducted under controlled conditions at 23 °C to ensure repeatability.



Fig. 1. Procedure of Cross-cut test.

The evaluation was performed using a tenfold magnifier and a Keyence microscope (Figure 1), which allowed for accurate analysis of the adhesion quality according to the standard. The grid test is important for evaluating the adhesion of the coating, which is one of the main parameters of the quality of the surface treatment. Through this test, it is possible to identify how firmly the coating adheres to the substrate and to detect any defects caused by poor-quality degreasing.

A total of 48 separate experiments were performed. The type of degreasing solution (SOL_01 to SOL_03) was determined as the determining factor. For each of the degreasing solutions, a separate experiment was performed according to the DoE. The levels of the independently monitored variables were the same for each of the three experiments.

3. RESULTS AND DISCUSSION

Technological processes of surface treatments represent multifactorial, stochastic processes where individual factors, input variables act on the resulting properties of the layer either as main effects or in mutual interactions. The research and analysis of the properties of the formed layers is complicated in this case by the necessary technological operations (degreasing, pickling, phosphating, etc.) before the layer formation process itself. The adhesion of the cataphoretic layer to the base material was chosen as the dependent variable for the study. To simplify the

experimental procedure and minimize the necessary operations, aluminum (EN AW 1050-H24) was chosen as the base material, which does not require additional pickling, activation and phosphating processes.

The key research question was: How does the degreasing process affect the adhesion of the formed layer? Since the layer adhesion expressed by the ISO 2409 standard represents an ordinal variable expressed in degrees Gt0 to Gt5, we transformed this response into a nominal, binary scale using the technological criterion:

- if the adhesion of the layer reaches a value \leq Gt1, the response is assigned a value of 1 (passes),
- if the adhesion of the layer reaches a value $>$ Gt1, the response is assigned a value of 0 (fails).

Automotive industry standards were used for the evaluation. For example, the M3018: 2020-06 standard (Man Truck & Bus SE) defines in Table 5 the maximum permitted adhesion value according to DIN EN ISO 2409 Gt1, as does the Volkswagen TL52451:2019-04 standard.

The purpose of this transformation is to create a response, a dependent variable, that meets most of the requirements for the adhesion of the cathaphoretic layer according to individual standards in the automotive segment. The basic representation of the individual adhesion types in the original scale (Gt0 to Gt5) and at the same time the standard for assigning individual repeated measurements ($n=5$), for each sample to individual adhesion groups is shown in Figure 2.

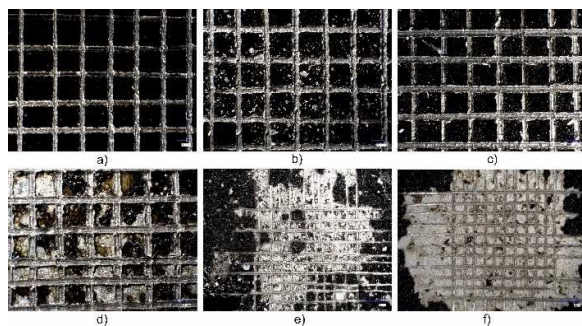


Fig. 2. Basic characteristics of the individual adhesion levels of the cathaphoretic layer (a) Gt0; b) Gt1; c) Gt2; d) Gt3; e) Gt4; f) Gt5).

Given the fact that the measured response within the experimental activity is ordinal in nature and takes on values from 0 to 5 and 5 repeated measurements were performed on each sample, however, transformed from the original to a binary character (0; 1) for the needs of a new response, the maximum value from 5 repeated measurements on each sample was always selected. The optimal method of processing the measured data with respect to the nature of the investigated response appears to be logistic regression analysis.

The logistic regression analysis model itself, where the dependent variable response is the adhesion of the formed layer, is created in accordance with the planned experiment. The independent variables were the temperature of the degreasing solution (temperature), the concentration of the degreasing solution (concentration), the time of deposition of the sample in the degreasing solution (time of deposition), the type of degreasing solution (degreasing) and the average thickness of the formed cathaphoretic layer (th_{aver}).

Basic results of the logistic model, omnibus test likelihood ratio test is a statistical test used to assess the overall significance of a model by comparing its fit to a simpler, nested model (often the null model). It determines whether a model with explanatory variables provides a significantly better fit to the data than a model without these variables.

Essentially, it tests whether the explained variance in the data is significantly greater than the unexplained variance, at the chosen significance level $\alpha=0.05$, as shown in Table 1.

Table 1

Omnibus Likelihood Ratio Tests			
Predictor	χ^2	df	p
concentration [$g \cdot l^{-1}$]	12.150	1	<0.001*
time of deposition [min]	5.280	1	0.022*
temperature [$^{\circ}C$]	3.012	1	0.048*
th_{aver} [μm]	6.370	1	0.012*
degreasing solution	6.210	2	0.045*

The overall suitability of the applied logistic model (basic parameters) for predicting the achievement of the adhesion value of the cathaphoretic layer for the condition $Gt \leq 1$ is presented in Table 2.

Table 2

Model Fit Measures					
Deviance	AIC	R ² _{McF}	Overall Model Test		
			χ ²	df	p
44.7	58.7	0.325	21.5	6	0.001*

Table 2 shows that the proposed logistic model of the probability of achieving the adhesion value of the cataphoretic coating $Gt \leq 1$ is significant ($p=0.001$) at the selected significance level $\alpha=0.05$. The value of the Akai information criterion (AIC) is at the level of 58.70 and the value of the Bayes information criterion (BIC) at the level of 71.8.

The estimates of the regression coefficients of the logistic model are presented in Table 3.

Table 3

Model Coefficients				
Predictor	Estimate	SE	Z	p
Intercept	-1.023	3.17	-0.319	0.750
concentration	0.252	0.234	1.077	0.021
time of deposition	0.132	0.152	0.868	0.016
temperature	0.118	0.023	0.803	0.022
$th_{aver.}$	0.096	0.114	0.137	0.041
degreasing solution:				
ODM_01 – ODM_02	-3.821	1.650	-1.438	0.049
ODM_03 – ODM_02	-4.992	1.390	-2.814	0.005

Table 3 shows that the basic input variables, namely the concentration, application time and temperature of the degreasing solution, but also the thickness of the formed cataphoretic layer, have a statistically significant effect on the probability value of achieving the adhesion criterion of the cataphoretic layer in the form $Gt \leq 1$ at the significance level $\alpha=0.05$. Based on Table 3, it is also possible to write a logistic equation in the form:

$$Gt1(1) = 0.252 \cdot x_1 + 0.132 \cdot x_2 + 0.118 \cdot x_3 + 0.096 \cdot x_4 \quad (1)$$

where x_1 - concentration of the degreasing solution, x_2 - deposition time in the degreasing solution (time of deposition), x_3 - temperature of the degreasing solution (temperature), x_4 - thickness of the formed cataphoretic layer. From the above equation (1) it is clear that by increasing the concentration of the degreasing solution by $1.0 \text{ g} \cdot \text{l}^{-1}$, the probability that the adhesion value of the cataphoretic layer will

reach the value of Gt_0 or Gt_1 will increase by 28.659%. Furthermore, it is clear from the equation (1) that by extending the deposition time by 1 min, the probability that the adhesion of the cataphoretic coating will reach the monitored criterion ($Gt \leq 1$) will increase by 14.122% and an increase in the temperature of the degreasing solution by $1 \text{ }^\circ\text{C}$ will increase the probability of achieving the specified adhesion criterion by 12.524%. Likewise, it is clear from Table 3 that the probability of achieving adhesion of the cataphoretic coating at the Gt_0 or Gt_1 level is 282.1% higher when using the degreasing solution ODM_01 and 399.2% higher when using the degreasing solution ODM_03 than when using the degreasing solution ODM_02 (water).

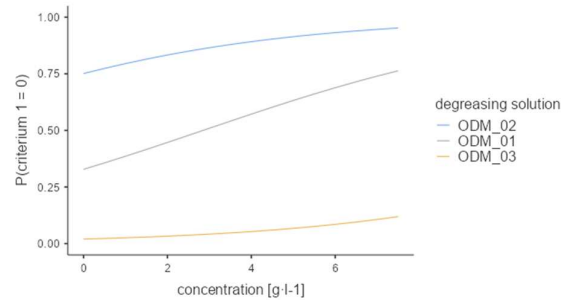


Fig. 3. The influence of the concentration of the degreasing solution on the probability of achieving the specified adhesion criterion of the cataphoretic coating ($Gt \leq 1$).

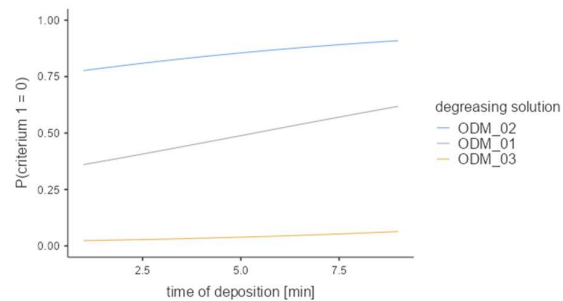


Fig. 4. The influence of deposition time in a degreasing solution on the probability of achieving the specified adhesion criterion of a cataphoretic coating ($Gt \leq 1$).

A graphical representation of relation (1) and table 3 for the influence of the concentration of the degreasing solution on the monitored response ($Gt \leq 1$) for individual degreasing solutions is shown in Figure 3.

Figure 4 shows the influence of the deposition time in the degreasing solution on the

monitored response ($Gt \leq 1$) for individual degreasing solutions. Figure 5 shows the influence of the temperature of the degreasing solution on the monitored response ($Gt \leq 1$) for individual degreasing solutions.

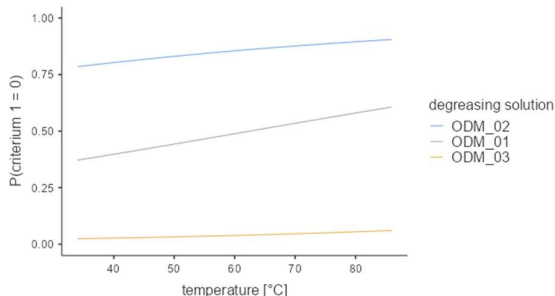


Fig. 5. The influence of the temperature of the degreasing solution on the probability of achieving the specified adhesion criterion of the cathoretic coating ($Gt \leq 1$).

The verification of the suitability of the logistic model (1) for predicting the probability of achieving the specified adhesion criterion of the cathoretic layer ($Gt \leq 1$) in the form of a classification table is presented in Table 4.

Table 4
Classification Table

Observed	Predicted		% Correct
	1	0	
1	21	5	80.8
0	5	17	77.3

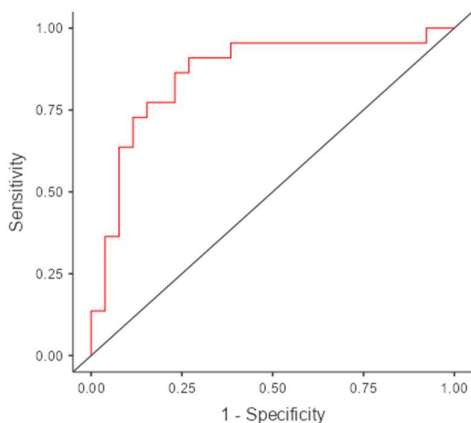


Fig. 6. ROC curve of the logistic model (1).

From Table 4 it is clear that the logistic model (1) correctly reported the values that met the established criterion ($Gt \leq 1$) in 80.8% of cases and the values that did not meet the established criterion in 77.3% of cases. The ROC curve of the logistic model is shown in Figure 6.

The total value of the area under the ROC curve (AUC) reaches a value of 0.860, which indicates a very good predictive ability of the logistic model (1).

4. CONCLUSION

Technological processes of surface treatments represent multifactorial, stochastic processes where individual factors, input variables act on the resulting properties of the layer either as main effects or in mutual interactions.

The research and analysis of the properties of the created layers is complicated in this case by the necessary technological operations (degreasing, pickling, phosphating, etc.) before the layer creation process itself. The adhesion of the cathoretic layer to the base material was chosen as the dependent variable for the study.

The basic research question was: How does the degreasing process affect the adhesion of the created layer?

The obtained results show that the basic input variables, namely the concentration of the degreasing solution (concentration), the deposition time in the degreasing solution (time of deposition), the temperature of the degreasing solution (temperature) but also the thickness of the formed cathoretic layer, have a statistically significant effect on the probability value of achieving the adhesion criterion of the cathoretic layer in the form of $Gt \leq 1$ at the significance level $\alpha=0.05$.

Based on the achieved results, we will continue with further important research.

5. ACKNOWLEDGEMENTS

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Influența pretratării suprafeței asupra aderenței acoperirii în vopsirea cataforetică a pieselor din aluminiu

Rezumat: *Articolul tratează influența pretratării suprafeței asupra aderenței stratului de acoperire în timpul vopsirii prin cataforeză a pieselor din aluminiu. Diverși agenți de degresare au fost aplicați pe probele de testare realizate din material de aluminiu. Agenții de degresare au fost aplicați în diferite condiții, specificate de experimentul planificat. Ulterior, probele au fost tratate la suprafață folosind tehnologia de vopsire prin cataforeză. Probele testate au fost supuse unui test cu grilă pentru evaluarea aderenței, conform ISO 2409:2020. Ulterior, a fost evaluată aderența la suprafața probelor testate. În urma analizei, s-a constatat că grosimea medie a stratului de acoperire are cea mai mare influență asupra aderenței stratului de acoperire prin cataforeză.*

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