

Editors:

Radim Cerkal Natálie Březinová Belcredi Lenka Prokešová

Proceedings of 28th International PhD Students Conference

10 November 2021, Brno, Czech Republic

Mendel University in Brno Faculty of AgriSciences



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Proceedings of 28th International PhD Students Conference 10 November 2021, Brno, Czech Republic

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PREFACE

Each year, the editors of the volume you are about to read are tasked with the responsibility of putting a coherent form to the proceedings from MendelNet, the international PhD Students Conference of the Faculty of AgriSciences of Mendel University in Brno.

The event which reached, this year, on November 10, 2021, its 28th edition, is traditionally aimed at both under and postgraduate students from the Czech Republic, Europe and beyond, and proudly welcomes the participants of various professional and cultural backgrounds. And while this time the people could not gather on-site due to globally-imposed covid-19 restrictions, the conference swiftly transformed itself into a virtual and fascinating beehive of results, opinions and brand new research paths and ideas.

Here in Brno, under the spell of great genetician G. J. Mendel and the guidance of skilled senior researchers and supervisors, students can introduce, defend and discuss their scientific results while those who do not feel confident enough to present and pen their paper in English are invited to join as spectators and follow-up discussion participants.

The best submissions are, after rigorous peer-review process, collected here and range from plant and animal production to fisheries and hydrobiology to wildlife research while agroecology and rural development, food technology, plant and animal biology, techniques and technology and applied chemistry and biochemistry also belong to the core areas being investigated.

The collection as varied and huge as this can succeed only as a team effort, both on authors' and editors' side, so we would like to express our thanks and gratitude to all committees and reviewers both for their outstanding work and invaluable comments and advice.

The Editors



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Alternative mechanical pre-treatment methods of hot-dip galvanising surface to increase of the organic coatings adhesion

Jaroslav Lozrt, Jiri Votava, Radim Smak Department of Technology and Automobile Transport Mendel University in Brno Zemedelska 1, 613 00 Brno CZECH REPUBLIC

x lozrt@node.mendelu.cz

Abstract: The content of this contribution is an evaluation of research on various mechanical pre-treatments of inorganic coating, which is part of the so-called duplex system applied to a steel sheet. In order to coating adhesion increase, the hot-dip galvanising surface was first pre-treatment using the standard light blast technology (synthetic brown corundum F40). Furthermore, pre-treatment was also carried out using alternative methods that can be used in conditions without blasting equipment – sandpaper regrinding (P40, P60, P80 and P100) and a corrosion-resistant steel brush (wire diameter 0.30 mm). Tools are designed e.g. for cleaning metal surfaces. Samples without mechanical pre-treatment and samples with blasted surface were used as a standard. The surface texture was evaluated based on the roughness height parameters Ra and Rz (according to ČSN EN ISO 4287 standard). The mechanical resistance of applied anti-corrosion protection was determined by means of a pull-of adhesion test (according to ČSN EN ISO 4624 standard). The experiment suggest results, that among the alternative methods, the use of P80 and P100 sandpapers and corrosion-resistant steel brushes seems to be the most suitable, as these tools are not as aggressive to the galvanised surface as P40 and P60 sandpapers.

Key Words: carbon steel, zinc coating, surface texture, abrasive, sweeping

INTRODUCTION

It is generally accepted that mechanical adhesion is improved by increasing surface roughness, since more active centres (anchorage points) are available. However, highly pronounced roughness can be detrimental as it can lead to local differences in the organic coating adhesion that could promote metals corrosion under the coating layer (Cabanelas et al. 2007, Votava et al. 2018). Thus, there is a critical point with an optimum roughness profile, which is particularly important for galvanized surfaces (Malone 1992). Increasing the surface roughness of galvanized steel can be achieved either naturally (by weathering) or by mechanical and chemical pre-treatment (Cabanelas et al. 2007, Votava et al. 2020). A suitable mechanical surface pre-treatment method for the application of an organic coating is the coarse removal of irregularities and lumps on the zinc coating with a coarse file, followed by a so-called sweeping with a sharp-edged abrasive (can be advantageously used especially for shape complex components - e.g. weldments) or sandpaper regrinding. The sweeping purpose is not only to achieve the desired degree of surface cleanliness, but also the necessary profile for anchoring the organic coating on the smooth zinc coating (Kuklík and Kudláček 2014, Poláková et al. 2018). Both of these factors contribute to increasing the coating adhesion to the substrate. Furthermore, blast pre-treatment also leads to an increase in the fatigue strength of metal surfaces (Hansel 1999). However, too much mechanical pre-treatment destroys and reduces zinc coating thickness or creates too much internal stress, which can later cause coating delamination. The surface pre-treatment should reduce maximum 10 µm of zinc coating (thus suitable for zinc coatings over 30 µm thickness). For this reason, the blasting air pressure should be 0.2–0.3 MPa and the distance of the nozzle end from the material surface should be 250-350 mm. However, the worker experience is also very important. The particle size for blasting galvanised steel should be between 200-500 µm (Hylák and Kudláček 2017). The authors Hylák et al. (2017) then specifically recommend brown corundum (alumina) and corrosionresistant chromium grit for light blasting of zinc coating.





MATERIAL AND METHODS

The experiment consists of two parts. The first part is focused on the surface roughness analysis (Ra and Rz parameters) and the material removal evaluation (h parameter). The second part deals with the coating adhesion to the galvanized surface. The blasting was carried out with a BlastRazor Z-25. All alternative mechanical pre-treatments were carried out on a vertical cantilever milling machine FA 3 AV. The tools used were brand new. A total of 48 samples in 7 sets were researched. One waterborne paint and one synthetic paint were tested. The basic paint was not applied, as both of these paints are so-called self-etching ("2 in 1"). A commercially available hot-dip galvanized sheet (continuously by the so-called Sendzimir method) based on ferritic-pearlitic steel S235JRG1 was chosen. The zinc coating thickness was $35 \pm 3 \mu$ m. The zinc coating thickness measurement was carried out using the Elcometer 456 non-destructive electromagnetic method in accordance with the procedure specified in ČSN EN ISO 1461 standard. The samples dimensions were $190 \times 65 \times 1$ mm. For the samples intended for alternative mechanical pre-treatment methods, a bend was made at one end (for clamping in a vice). After surface pre-treatment, the samples length was cut to 160 mm. Table 1 then shows a breakdown of the samples number in each set.

Set	Pre-treatment	Pressing force	No. of samples			
No.	(grain size [µm])	[N]	Total	Waterborne paint	Synthetic paint	
1	-	-	4	2	2	
2	F40 (355–500)	-	4	2	2	
3	P40	10 ± 2	4	2	2	
	(400-500)	40 ± 2	4	2	2	
4	P60	10 ± 2	4	2	2	
	(250-315)	40 ± 2	4	2	2	
5	P80	10 ± 2	4	2	2	
	(125-250)	100 ± 2	4	2	2	
6	P100	10 ± 2	4	2	2	
	(125-100)	100 ± 2	4	2	2	
7	Ctorel horsely	10 ± 2	4	2	2	
	Steel brush	100 ± 2	4	2	2	

Table 1 Samples No. in each set based on the various criteria

The blasting was performed with synthetic brown corundum, the air pressure was 0.25–0.30 MPa and the nozzle distance from the surface was 300-350 mm. The sanding papers grain was always synthetic corundum with open structure, synthetic resin binder, paper substrate and 75 mm die diameter. The sandpapers clamping was done by Velcro. The steel cup brush was made of corrosion-resistant corrugated wire with a 0.30 mm diameter. The brush diameter was 75 mm. The tools clamping to the fixture was realized by means of an M14 nut with a 2.0 mm thread pitch. The jig clamping was carried out using a milling head and a clamp with a 20 mm diameter. The tool vertical movement in the jig and torque transmission was ensured by a 5×20 mm groove and a M5 screw. The milling table transverse travel was always 450 mm/min and the tool revolutions were always 45 1/min. The tool pressure on the workpiece was exerted by a steel coiled compression spring. The spring wire diameter was 1.6 mm, outer diameter 12 mm, free length 45 mm, number of coils 11, material EN 10270-1 SH. The spring characteristics were measured on a ZDM 5/51 universal testing machine. The pressing force setting and change was realized by vertical sliding of the milling machine table. The two different pressing forces were chosen for verification in practice (using hand tools). For the P40 and P60 sandpapers, a pressing force of only 40 ± 2 N (instead of 100 ± 2 N) had to be chosen because of the high aggressiveness of these tools (a higher pressing force would have caused the zinc layer to failure to the steel substrate) – forces were determined experimentally, as there is no standard for determining the pressing force to ensure roughness. The tools themselves exerted a force on the sample of approximately 1-2 N. This force is included in the ± 2 N tolerance. A workplace view during sample machining is shown in the Figure 1.

Figure 1 Workplace layouts during sample machining



Legend: a - sandpaper; b - steel brush





The surface texture was evaluated based on the roughness height parameters Ra (average arithmetic deviation of the roughness profile under consideration) and Rz (roughness profile maximum height). The measurements were performed using a Mitutoyo SURFTEST SJ-201 mobile profilometer. The roughness profile was filtered according to ČSN EN ISO 4287 standard. Sampling length λc 2.5 mm, number of sampling lengths 5, Gauss filter, evaluation length ln 12.5 mm. The Ra and Rz parameters were measurements always parallel to the direction of workpiece movement during surface pre-treatment (approximately in the longitudinal axis – thus perpendicular to the tool grooves direction) and were carried out 100 times per set. Material removal was also evaluated. The zinc coating thickness was also measured 100 times before and 100 times after pre-treatment in one set. For low pressing forces (around 10 N), the calculation considered the zinc coating plastic deformation above the base material level, which results in the higher probe distance of the measuring instrument from the steel substrate. The material plastic deformation above the base material level (in belt grinding) is described - e.g.by Pandiyan and Tjahjowidodo (2019). Thus, the zinc coating thickness increases artificially and the actual material removal has to be determined by calculation - equation (1). It has then been verified by practical measurements that at higher pressing forces values (above 40 N) there is already a measurable decrease in the zinc coating thickness and the actual material removal needs to be determined by calculation according to equation (2):

$$h = Rz - \Delta t = Rz - (t2 - t1) [\mu m]$$
(1)

 $h = Rz + \Delta t = Rz + (t1 - t2) [\mu m]$ (2)

Legend: h – material removal [μ m]; Rz – roughness profile maximum height [μ m]; Δt – zinc coating thicknesses difference before and after mechanical pre-treatment [μ m]; t2 – zinc coating thickness after mechanical pre-treatment [μ m]; t1 – zinc coating thickness before mechanical pre-treatment [μ m]

Paints that are commonly available on the Czech market were tested. Instead of the trade name, the individual paints were designated "waterborne paint" and "synthetic paint". The main parameters of paints provided by the manufacturers are given in Table 2.

Type of paint	Non-volatile solids [weight %]	Specific density [g/cm ³]	Emissions of volatile organic compounds [kg/kg]	Zn ₃ (PO ₄) ₂ [weight %]
Waterborne	50.0	1.20-1.30	0.03	-
Synthetic	43.0-48.0	1.24–1.35	0.35-0.37	≤ 5.0

Table 2 Paints main parameters

The coating was applied by air spraying technology at an ambient temperature of 22 ± 1 °C. The relative humidity was 50–60%. The coating application was always carried out immediately after pre-treatment. The application interval of the individual each layers was at least 24 hours. Three coats were always applied with a tolerance of $20 \pm 5 \mu m$, so that the resulting coating thickness was always $60 \pm 5 \mu m$ in total (tolerance guaranteed by air spraying application by a very experienced worker). The coating thickness was measured after it had completely dried, again using an Elcometer 456 (according to ČSN EN ISO 1461 standard). The measurements were performed 10 times on each sample. Furthermore, the samples were subjected to a pull-off adhesion test (according to ČSN EN ISO 4624 standard) immediately after the paint had completely dried. The standard test cylinder diameter was 20 mm and the two-component glue "Araldite" was used. Tensile stress analysis was carried out using an Elcometer (measuring range 0–7 MPa) – 24 hours after test cylinder gluing. Due to the limited contribution scope was realized only basic testing using by the pull-off adhesion test.

RESULTS AND DISCUSSION

Surface texture

The following graphical representation (Figure 2) shows the Ra, Rz, t1, t2 and h parameters depending on the mechanical pre-treatment and the pressing force. From Figure 2, it can be seen that the lowest average values of Ra and Rz parameters were measured on the surface without mechanical pre-treatment. Therefore, if these measured values are lower than the values optimal for maximum paint adhesion, an increase in paint adhesion to the zinc substrate can be expected (Malone 1992). It is further evident from Figure 2 that the highest average values of the Ra and Rz parameters were measured on the blasted surface. Compared to alternative mechanical pre-treatments, the blasted surface also achieves very high differences for the minimum and maximum values of the Ra and Rz parameters. This can be attributed to the variable blasting conditions. The author Hansel (1999) states that any variation in nozzle





air pressure, nozzle to surface distance or abrasive impact angle will affect the resulting profile. Authors Guzanová et al. (2014) confirm that after brown corundum blasting, the resulting surface is rated as the most dissected. This is confirmed by the values shown in Figure 2. These authors further state that the surface cleanliness is average due to the increased dustiness. From this point of view, alternative mechanical pre-treatments appear to be preferable as they produce a more homogeneous surface, which is desirable for a relatively thin zinc layer ($35 \pm 3 \mu m$). Authors Cabanelas et al. (2007) reported an increase in the paint adhesion to the galvanised substrate when the Ra parameter value was increased from $0.95 \pm 0.19 \mu m$ (freshly galvanised surface) to $1.87 \pm 0.34 \mu m$ (low surface weathering). If Ra was further increased to $5.28 \pm 1.28 \mu m$ (high surface weathering), no further increase in paint adhesion was observed. Therefore, based on the results of this publication, it can also be predicted that any mechanical pre-treatment will lead to an increase in paint adhesion (compared to a surface without pre-treatment – average Ra = 1.29 µm). At the same time, however, there will no longer be measurable differences between the blasted (average Ra = 4.92 µm) and the alternatively pre-treated surface (average Ra = 1.56–2.45 µm). Correlation of parameters Ra and Rz were not monitored.





Pull-off adhesion test

The pull-off adhesion test result (Figure 3 and 4) is tensile stress necessary for the failure of the weakest interface (adhesion failure) or the weakest component (cohesion failure) of produced anti-corrosion system. Five measurements were always performed on each sample.



Figure 3 Average values of pull-off adhesion test and comparison with Ra parameter

In the case of waterborne paint, only minimal increases in average tensile stress values were observed for the pre-treated surfaces (compared to the surface without pre-treatment). The surface roughness without pre-treatment is therefore sufficient. In the case of this paint, it is also important





to take into account the fact that in the vast majority of cases there is adhesion failure between the coating layers (65-80%). This finding indicates lower strength bonding layers characteristics of the paint (Hylák et al. 2017). For this reason, the implementation of any pre-treatment is also of no benefit (for this particular paint). The adhesion measurement of waterborne paint to hot-dip galvanized substrate (without pre-treatment) was also investigated by Lozrt et al. (2021) and this experiment yielded similar results (average values of 1.66–3.38 MPa). The value of 1.66 MPa was achieved by paint with the same dry matter content and specific gravity. Values of 3.38 MPa can then be justified by the higher dry matter content and specific paint gravity. In the case of synthetic paint, the mechanical pre-treatment benefit has already been statistically demonstrated. Without pre-treatment, average values of 0.52 MPa were achieved, with 90% adhesion failure between the substrate and the first layer. This finding indicates a higher level of adhesion between the paint layers, compared to the paint adhesion to the zinc substrate. The very low measured values clearly indicate incompatibility between the substrate and the paint, as the surface cleaning before paint application was always very thorough. Average values in the range of 0.79–1.10 MPa were then found during surface pre-treatment. In the case of the maximum values (1.10 MPa), adhesive failure between the paint layers (75-80%) was already detected, indicating a higher adhesion degree of paint to the substrate, compared to adhesion between paint layers and also low strength bonding layers characteristics of the paint. This finding therefore clearly confirms the positive benefit of surface pre-treatment. Similar values were also measured by Hylák and Kudláček (2017), who concluded that brown corundum blast pre-treatment increases the paint adhesion to the substrate up to twice as much (1.0 MPa without pre-treatment and 1.5-2.0 MPa with pretreatment). However, it must be stressed that there are very large differences in the adhesion of different synthetic paints. In fact, the same authors found values in the range of 5.5–9.0 MPa with different paint, and blasting even had a negative effect on the paint adhesion here. These high values can be justified by the very good chemical paint adhesion with the zinc substrate, which leads to the formation of a very high adhesion bridge (Hylák et al. 2017). As expected the higher blasted surface roughness did not result in higher paint adhesion (compared to the alternative pre-treatments).

Figure 4 Examples of the coating failure character (test cylinder diameter 20 mm)



Legend: a – waterborne paint – without pre-treatment; b – waterborne paint – steel brush (10 N); c – synthetic paint – without pre-treatment; d – synthetic paint – steel brush (10 N)

CONCLUSION

In the case of waterborne paint, the mechanical pre-treatment benefit was minimal, as the zinc surface roughness without pre-treatment was near optimal. The pre-treatments use here would also be uneconomical due to the lower strength bonding layers characteristics of the individual paint layers. In this particular case, therefore, only a thorough surface degreasing before the paint application can be clearly recommended. For synthetic paint, mechanical pre-treatment can already be recommended. However, the applied synthetic paint shows low chemical adhesion to the zinc substrate. This may be due to the application of a self-etching paint ("2 in 1"). Among the alternative methods, the use of P80 and P100 sandpapers and corrosion-resistant steel brushes seems to be the most appropriate, as these tools are not as aggressive to the galvanised surface as P40 and P60 sandpapers. This fact is clearly confirmed by the measured material removal rates, e.g., P40 sandpapers achieve an average removal rate of $10.86 \ \mum (10 \ N)$ to $21.08 \ \mum (40 \ N)$ – thus, a fourfold increase in the pressing force results in an approximate doubling of the material removal rate. In contrast, P100 sandpapers, e.g., achieve an average removal rate of $9.06 \ \mum (10 \ N)$ to $12.86 \ \mum (100 \ N)$ – a tenfold increase in pressing force results in only a minimal average increase in material removal. This finding is crucial as it confirms the suitability of this technology for application in engineering practice.





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