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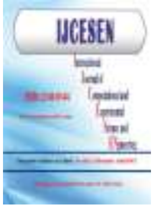

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Research Article

A Study to Examine the Effect of the Phosphate Coating Process on Adhesion and Corrosion Resistance in Structural Steels

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Abstract:

Industrial washing and drying machines are systems that operate in high-temperature and humid environments, which can therefore be considered aggressive in terms of corrosion. Stainless steel materials to be used in the body structures of these systems are the most important solution in this regard due to their superior corrosion resistance properties. However, due to the low initial investment cost, structural steels, especially from the non-alloy steel group with low carbon content, are widely used in the sector as an alternative to stainless steel in machine and system bodies by increasing the corrosion resistance by applying paint. However, it is an important necessity to apply some additional surface treatments to low carbon steel to supply protection against aggressive corrosive environments. Phosphate coating by spraying is a widely applied industrial method to increase the adhesion of metal surfaces to paint and their resistance to corrosion. Within the scope of this study, the corrosion behaviors of two different stainless-steel materials (AISI 304 and AISI 316) and a St-37 (S235JR) structural steel material, which was painted directly, painted after phosphate coated and painted+phosphate coated were examined. The aim of the study is to reveal the substitutability of structural steel material to stainless steel materials in terms of corrosion properties. Contact angle measurements, cross-cut test, microhardness and surface roughness value of the samples were analyzed. The lowest corrosion resistance was obtained with phosphate coated, while the highest corrosion resistance was observed in the phosphate coated+painted sample.

1. Introduction

304 and 316 austenitic stainless steels have high corrosion resistance due to the oxide layer formed on their surfaces[1][2]. St37, which is a low carbon steel, is frequently used in many areas of industry due to its easy machining, easy welding, physical and chemical properties. However, this steel is susceptible to corrosion. There are many methods to improve the corrosion resistance of materials. Phosphate coating process, which is one of the conversion coating methods frequently used in the sector, is also a preferred method to increase corrosion resistance[3][4][5][6].

Conversion coatings covered by organic coatings are typically used to shield steel from corrosive environments of outdoor applications. Among conversion coatings, phosphate coatings are commonly used to protect the surfaces of carbon steel, low-alloy steel and cast iron products such as cars, bikes, refrigerators, washing machines, office furniture etc.[7] Phosphate coatings are most commonly used as a paint base, owing to their good adhesion performance to the metallic surface[8][9].

Phosphate conversion coating is preferred due to its low corrosion rate, favorable production cost and environmental friendliness[3]

Phosphate coatings arises from the chemical reaction between the metallic surface and a solution including phosphoric acid (H_3PO_4), ions of bivalent metals (Zn^{2+}, Fe^{2+}, Mn^{2+}), and an oxidizing reagent (nitrate, nitrite, peroxide)[9][10] The properties of phosphate coatings depend on the type of phosphating baths. Phosphate coatings can be applied in three different ways: iron phosphate, manganese phosphate and zinc phosphate[11]. Manganese phosphate improves the wear resistance of steel surfaces, whereas zinc phosphate is beneficial for corrosion resistance[12][13][14].Zinc phosphate coatings are perfect for painting as well as for enhancing corrosion resistance. By creating a transfer film on the opposite surface during the coating process and functioning as a solid lubricant, manganese phosphate coatings increase wear resistance and reduce the coefficient of friction. Additionally, manganese phosphate coatings are favored due to their ability to retain oil [15][16]

Three different kinds of salts are produced by phosphoric acid (H_3PO_4): phosphates, hydrophosphates, and dihydrophosphates. The protective effect of the coating that forms on the metal surface is determined by the characteristics of the phosphate salts that are produced. Me is a divalent metal, and the dihydrophosphates $Me(H_2PO_4)_2$ are monosubstituted salts. They are created as soon as the metal and phosphoric acid come into contact. Further acid-metal interaction results in the formation of two-substituted ($MeHPO_4$) and three-substituted ($Me_3(PO_4)_2$) salts, at which point the acid concentration drops. The precipitation of phosphates and the dissolution of the base metal are the two primary processes that are seen during phosphating on the metal surface. The metal's surface layer is etched during the phosphating process as a result of an interaction with the phosphating fluid. Insoluble secondary and tertiary phosphates that stick to the base metal and are an essential component of it form a crystalline layer on the steel's surface[9].

Steel is phosphated primarily for two reasons: to increase paint adherence by creating a surface with numerous anchor points and to act as a barrier to prevent corrosion from spreading beneath the paint layer. Iron-phosphate coatings are employed infrequently compared to the phosphate coating types listed above due to their thinness and, as a result, lower corrosion stability. Iron-phosphate coatings do, however, have certain benefits, such as the ease of the technological process of coating deposition and the use of basic equipment. Furthermore, the neutralization procedure is essentially the only step in the straightforward waste water treatment process. The porosity of the iron-phosphate coatings may be reduced in the presence of accelerators in the plating bath, such as formic acid, chlorates, hydroxylamines, and $NaNO_2$. Because of all of these factors, iron-phosphate coating on steel is a promising steel pretreatment before the deposition of an organic coating[7]

Phosphating is the technique of treating metal surfaces by creating phosphate conversion coatings, which is intended to improve organic coatings' resistance to corrosion and strength of adhesion. Phosphate conversion coating has been demonstrated to offer corrosion resistance and a barrier against the propagation of corrosion processes beneath the paint layer. Iron-phosphate coatings have also been utilized occasionally, although their corrosion resilience was lower than that of zinc-phosphate coatings. Compared to the manufacture of zinc-phosphate coatings, the technological procedure for iron-phosphate coatings is easier and requires a lot less equipment. Waste water treatment is similarly straightforward. Good corrosion protection for steel can be achieved when iron-phosphate coatings are combined with organic coatings. When fine crystals of vivanit, $Fe_3(PO4)_2$, and strengite, $FePO_4$, were produced, it was demonstrated that iron-phosphate coatings on steel could be chemically created by immersing the steel in phosphate solutions with a pH between 3.5 and 5.5. Low steel surface coverage is the main issue with iron-phosphate coatings. Iron-phosphate coatings' limited surface coverage is typically linked to the metal substrate's high rate of corrosion. It was demonstrated that the phosphate layers' characteristics might be enhanced by additions used as accelerators. The iron-phosphate coating's surface coverage is increased when formic acid or nitrite is added to the phosphating bath. In another study, it was shown that extending the phosphating time resulted in improved surface finish of the base metal[14].

A variety of micro primary cells, such as microanodes and microcathodes, are created when a piece of steel is submerged in an acid conversion bath. The pH at the steel-bath interface can be altered by the dissolving of iron at microanodes and the evolution of hydrogen at microcathodes. This will then encourage the multistage hydrolysis of soluble primary phosphates in the bath to produce the phosphate ion (PO_4^{3-}). The cation (Zn^{2+}, Fe^{2+}, Ca^{2+}) from the bath and the insoluble phosphate from PO43- are then applied to the steel's surface, where they progressively develop into a PCC layer[17]. Thin crystalline layers of phosphate compounds that stick to the metal substrate's surface make up phosphate coatings. Zinc, manganese, or iron phosphate solutions can create porous phosphate crystals. The phosphate coatings offered by the three varieties range slightly in terms of coating thickness and crystal size. This makes it possible to choose a more tailored coating for the specific use needed for a portion of the structure. The substrate is sprayed or submerged in a solution to create the coating[10].

In this study, it is aimed to increase the corrosion properties and mechanical strength of St37 steel as an alternative to stainless steels by applying surface treatments on it. Research on the iron phosphate coating method applied within the scope of the study is limited in the literature, and the study conducted in this context aims to contribute to future studies by filling the information gap in the field.

2. Material and Methods

In this study, 304, 316 austenitic stainless steels and St37 steel have been used. The chemical compositions of the materials are shown in Table 1. Sanding, polishing and etching processes were applied to the samples for metallographic examination. Optical microscope examinations were carried out on the Nicon Eclipse LV150 device. Microstructure images of the samples are given in the Figure 1.

In the study, phosphate coating, painting and phosphate coating + painting processes were applied to St37 steel in 3 different ways to improve corrosion resistance. Iron phosphate coating, one of the phosphate coating methods, was applied to the samples by spraying method. Process parameters of phosphating and rinsing baths are shown in Table 2 and Table 3. Before the coating process, degreasing was applied, and the metal was activated in the activation bath to obtain a fine crystalline structure that will increase the corrosion resistance and adhesion properties during coating. Afterwards, phosphate coating was applied, and the samples were rinsed. At the last stage, paint was applied to the samples. A white colored paint with a density of 1.64 ± 0.05 gr/cm³ coded 08288.EWR57 was used. The average paint thickness was measured as 200 microns.The electrochemical corrosion behavior of samples was investigated with Metrohm Dropsens Potentiostat/Galvanostat equipment in 3.5 wt.% NaCl solution. The corrosion cell setup includes a 75 ml NaCl solution, an Ag/AgCl reference electrode, a graphite electrode, and the sample as a working electrode. The exposure area of the sample was set as 25 mm^2.

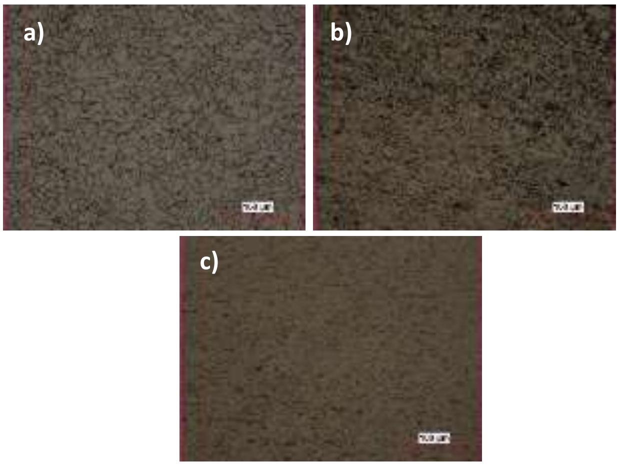

Figure 1. *Microstructure images of materials*

a) St37 b) 304 c)316

Table 1. *Chemical composition of materials*

Samples	**Chemical composition (%)**							
	C	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**	**Mo**
St37	0.11	0.03	0.56	0.007	0.005	0.07	0.03	-
304	0.08 max	0.75	2	0.045	0.03	18-20	8-10	-
316	0.08 max	1	2	0.045	0.030	16-18	10-14	2-3

Table 2. *Chemical Bath Content and Process Parameters of Degreasing and Phosphating Bath*

Bath Volume	5.4 m^3
Temperature	Ambient Temperature : 50 °C
Nozzle Pressure	1.0 -1.5 bar
Ring Number	10-10
pH	3.5 -5.5
Solution	Decorrdal 40-45 (Phosphoric Acid 10 - <25%, Laurylamine Ethoxylated 3 - <5%, Sodium 3-Nitrobenzenesulfonate 0.5 - <1%, Hydrofluoric Acid 0.1 - <0.5%, Sodium Hydrogendifluoride 0.1 - <0.5%)

The scan rate and the scanning range of the corrosion test were 0.05 V/s and -1V to 1V, respectively. The corrosion rate was calculated according to the Tafel extrapolation method using the Dropview software. Contact angle measurements using the sessile drop method were performed with a contact angle tensiometer (Attension Theta). Water slowly dropped onto the flat sample surface with a microsyringe at room temperature. Images of the droplets were photographed using a video camera connected to a light microscope after they remained on the surface for 10 seconds. The contact angle was then measured with imaging software. Also in this study, the Cross-cut (adhesion) test was performed to determine the paint adhesion resistance. The test was conducted according to the ISO 2409 standard. Microhardness examinations were carried out on the Future-Tech FM-7 microhardness tester. Vickers hardness measurements were carried out using a Vickers hardness tester with 100 gf load application. Surface roughness test was done Mitutoyo sj301 profilometer device according to EN ISO 4287 standard. The results of all tests performed were given at the results and discussion section.

3. Results and Discussions

3.1 Corrosion Test

Corrosion test results can be explained in two sections. The first section includes the comparison of corrosion behavior of low carbon steel, AISI 304, and AISI 316 stainless steels. In the second section, the influence of iron-phosphate coating and painting processes on the corrosion resistance of low carbon steel surfaces was discussed.

Low carbon steel without a surface modification exhibited the highest corrosion rate considering the corrosion rates of stainless steels. It was revealed that the corrosion rate of the low carbon steel is 6 and 7 folds of the corrosion rates of AISI 304 and AISI 316, respectively. It is well-known that the higher corrosion resistance of stainless steels arises from the existence of a protective oxide (Cr_2O_3) layer[18][19] Tafel curves of both AISI 304 and AISI 316 have a passive region in which the current density is almost constant against increasing voltage (Figure 2(a)). The surface is protected from the corrosion in the passive region. A further increase in potential increases the current density and the protective layer is damaged. Hence, the active corrosion region starts. Instead of a passive region, a pseudo-passive region was observed in low carbon steel. However, the pseudo-passive layer is not stable and protective below pH 5.6[20]

AISI 316 has the least corrosion rate among all steel samples which are investigated in the present study. The insignificant difference in corrosion rates of AISI 304 and AISI 316 (Figure 2(b)) mainly ensued from their chemical content. AISI 304 and AISI 316 include almost equal amounts of Cr and Ni, whereas AISI 316 has approximately 2 wt.% Mo, additionally. As reported by molybdenum improves the corrosion resistance of stainless steel by increasing the stability of the protective passive oxide layer restraining the invasion of chloride ions[21].

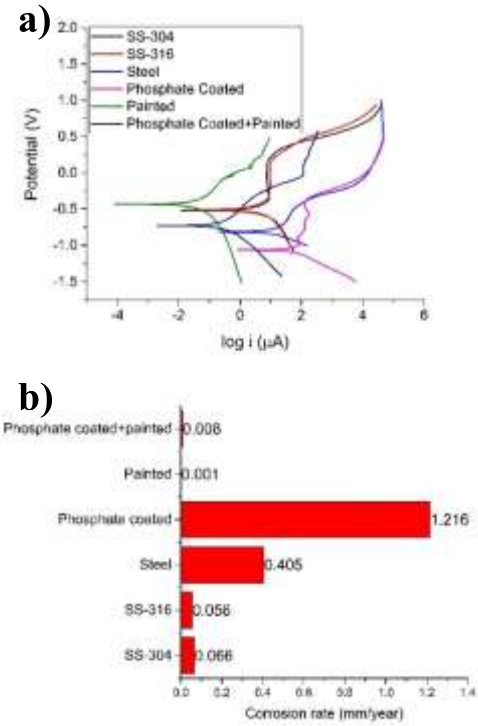

Figure 2. *a) Tafel curves of cold rolled steel before and after surface treatments and Tafel curve of stainless steels (AISI 304 and AISI 316), b) corrosion rates of samples*

According to corrosion test results it can be emphasized that the most effective way to protect the low carbon steel is by applying organic coatings (painting) on the surface without a prior treatment. Minimum corrosion rate was observed in the painted sample. Even the stainless steels showed higher corrosion rates than the painted steel surface. Iron phosphate coating can enhance the adhesion of organic coatings on steel surface, but it has no beneficial effect on corrosion protection of the low carbon steel. Phosphate coating + painted sample exhibited worse corrosion resistance than only painted sample. The only handicap of painting relies on its low tribological performance. Paintings as organic coatings can be easily removed by mechanical stimulations. Even a micro-scaled defect on an organic coating can cause an unexpected increase in corrosion rate. Aqueous corrosive environment can reach the substrate material surface through the defects on the organic coating by capillary action. The passive zone of the phosphate coated sample exhibits three distinct peaks whereas phosphate coating + painted, painted, and steel samples have S-shaped curves. Gattu et al. reported that the pseudo-passive region may consist of two or more separate regions[22]. It was observed that after the first passivation, the corrosion current density tended to increase which can be an indicator of increased dissolution rate. Yang et al. obtained a similar result in the corrosion of Ti-5Al-5Mo-5V-1Fe-1Cr in marine environment media[23]. They observed three separate passive regions and observed an increase in the current density in particular in the tertiary passive zone which can be explained by the dissolution of passive film and evolution of oxygen.

3.2 Contact Angle Measurements

Within the scope of contact angle measurement tests, three samples were analyzed and three random points per sample were measured. The contact angles measured on the uncoated sample and the iron phosphate coated sample are 77.4°±0.7° and 52.8° ±1.1°, respectively. The shapes of the water droplets on the surface of the uncoated and coated samples are shown in Figure 3.

Compared with the uncoated substrates, the coated samples have low contact values (θ), indicating that the iron phosphate coating can effectively improve the wettability of metal surfaces.

The wettability of the coating mainly is determined by the surface chemical composition and microstructure[24].

On the other hand, after applying paint to the coated and uncoated surfaces, no significant difference was observed in the contact angle.

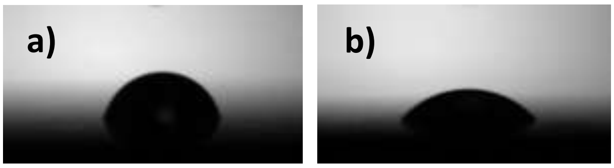

Figure 3: *Shape of water droplets on a) the uncoated sample and b)coated sample*

3.3 Cross-Cut Test

The purpose of the coatings' adhesion tests is to ascertain the adhesion parameter between the coating and the surface or between the system's separate layers. Surface treatment of materials is an important condition for good adhesion. The cross-cut test determines the resistance of the coating on the specimens to separation from the substrate[25]. It is used to evaluate the adhesion strength of paints, coatings and thin film layers in particular. The adhesion strength of the coating is evaluated according to the amount of peeling and the results are as shown in Table 3.

Table 3. *Cross-cut test results*

Painted Sample	Class	Class
Phosphate coated and painted sample	GT 0 (5B)	GT 0 (5B)

A number between 0 and 5 is assigned to the test's final result, whichever best represents the damage found[25].The test result resulted in a GT 0 (5B) grade (Table 4). GT0 or 5B is an adhesion grade used to evaluate cross-cut test results and represents the highest adhesion quality. A GT0 or 5B grade indicates that there is no peeling or separation between cuts and that the coating has excellent adhesion to the surface. This result indicates that the paint or coating is particularly strong and durable on the substrate.

3.4 Microhardness Test

The average of the measurements taken from each sample is given Figure 4. When the results are examined, it is seen that is has 304 185.5; 316 178.55 HV value. The hardness value increased from 171.78 HV to 179.65 with phosphate coated on St37 steel.

3.5 Surface Roughness

Surface roughness has a significant impact on coating performance and adherence.

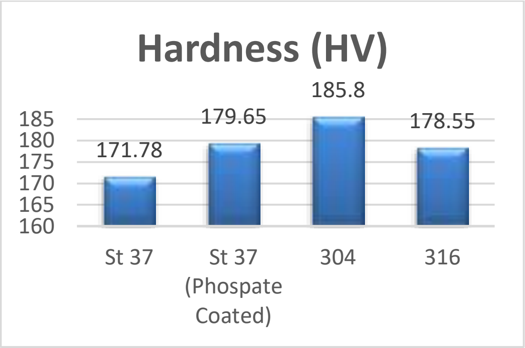

Figure 4. *Microhardness results of the samples*

Furthermore, the substrate's surface roughness might affect the coating's adherence since a rough surface offers more mechanical interlocking sites for the coating to stick to[26]. The Table 4 shows the average Ra values of the measurements taken from the samples. When the results are examined, it is seen that while the surface roughness value of St37 steel is 1.26 µm, it increases to 1.36 µm when phosphate coating is applied. It is seen that the phosphate coated painted sample has the highest surface roughness value. Phosphate coating is known to provide better adhesion of paints by creating a porous and rough surface on the surface and the increase in the results support this statement.

Table 4. *Surface roughness results*

	Surface Roughness Value (Ra- µm)					
	Phosphate	**St37**	**304**	**316**	**Painted**	**Phosphate+ painted**
1	1.30	1.27	0.79	0.73	1.77	1.96
2	1.29	1.24	0.78	0.72	1.59	1.78
3	1.51	1.26	0.82	0.72	1.6	1.86
4	1.23	1.26	0.76	0.73	1.65	1.82
5	1.34	1.3	0.78	0.7	1.65	1.85
6	1.31	1.25	0.78	0.7	1.73	2.01
7	1.44	1.3	0.75	0.72	1.85	1.65
8	1.44	1.23	0.77	0.71	1.65	1.94
Average	1.36	1.26	0.78	0.72	1.69	1.86

4. Conclusions

In this study, the substitution of St37 steel to 304 and 316 steels in terms of corrosion resistance through iron phosphate coating and painting processes was investigated. With this regard, mentioned materials were compared in terms of their mechanical properties and corrosion resistance. As a result of the study, it was observed that the corrosion resistance of the structural steel material after painting, exceeded that of stainless steels. When the painting process is not taken into account, 316 stainless steel was found to be the steel with the highest corrosion resistance. This result is supported by the fact that stainless steels have a protective oxide layer and the 2% Mo in the structure of 316 stainless steel increases the stability of the protective oxide layer.

When phosphate coating, painted and phosphate coating+ painted processes are applied to St37 steel in three separate categories, it is seen that phosphate coating significantly reduces corrosion resistance. However, the benefit that the painting process adds to corrosion resistance compensates for this situation. Iron phosphate coating can increase the adhesion of organic coatings to the steel surface, but no effect on the protection of steel against corrosion was observed. The increased corrosion resistance with the painted sample showed a slight decrease when applied together with the phosphate coating.

When the contact angle measurements of the samples are evaluated, FeP coating has low contact values compared to uncoated samples, indicating that phosphate coating can effectively increase wettability on surfaces. In other words, it can be said that an increase in paint adhesion is achieved by increasing wettability.

The highest adhesion quality was found when the cross-cut test used to measure paint adhesive resistance was examined. Thus, it can be said that the paint and coating are durable on the substrate. According to the results of surface roughness, an increase was observed in St37 steel with phosphate coating. This result is supported by the fact that the surfaces on which phosphate coating were applied have a rougher structure and thus provide paint adhesion. In future studies, it is recommended to work on different phosphate coating methods in order to improve both paint adhesion resistance and corrosion resistance properties.

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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