Review on Systematic Approach to Phosphating and Passivation
For Improvement in Surface Properties

Vishal Takle[1], Ravindra Karwande[2], Swapnil Dhole[3], Mohmmad Irfan[4]
[1,]PG Student, Department of Mechanical Engineering,
[2]'Asso. Prof. Mech. Engineering Dept., MSSCOET, Jalna.
[3,4] Research Scholar, Dr. BAMU, Aurangabad

Abstract

We outline the ability of zinc phosphate coatings, obtained by chemical conversion, to protect steel against localized corrosion, generated by chloride ions in alkaline media. Phosphating is the most widely used metal pretreatment process for the surface treatment and finishing of ferrous and non-ferrous metals. Due to its economy, speed of operation and ability to afford excellent corrosion resistance, wear resistance, adhesion and lubricative properties, it plays a significant role in the automobile, process and appliance industries. Though the process was initially developed as a simple method of preventing corrosion, the changing end uses of phosphated articles have forced the modification of the existing processes and development of innovative methods to substitute the conventional ones.

Keywords: *Phosphating and Pasivation. alkaline medium, Process parameters, Performance parameters, Optimization.*

I. INTRODUCTION

The use of phosphate coatings for protecting steel surfaces has been known since the turn of the century and during this period the greater part of the World's production of cars; refrigerators and furniture were treated this way. The first reliable record of phosphate coatings applied to prevent rusting of iron and steel is a British patent of 1869 granted to Ross. In the method used by him, red hot iron articles were plunged into the phosphoric acid to prevent them from rusting The phosphating of steel, galvanized steel, zinc, and sometimes aluminum represents an essential stage in many areas of surface finishing. It is employed for the purpose of pretreatment prior to painting, increasing corrosion protection, assisting cold forming, and improving the sliding friction properties of components in sliding contact. Each of these areas will be covered with application examples. Zinc phosphate is a crystalline conversion coating that is formed on a metal substrate utilizing the chemical reaction between metal ions that have been dissolved in mineral acids and then diluted with water to form the process solution. Contrary to traditional plating operations where electrical current forces the coating formation, zinc phosphating processes rely on the basic pickling reaction that occurs on the metal substrate when the process solution comes in contact with the metal. The mineral acids that are normally used to dissolve the metal ions are nitric acid and phosphoric acid. Metals, such as zinc, nickel, and manganese, are dissolved depending on the process necessary. Several other metals can be dissolved to create specific characteristics. Nickel plays a major role in achieving an acceptable corrosion resistance of the coating as well as accelerating the process chemistry. More recent developments have created nickel-free processes that can compete with the nickel containing processes. Accelerators are added to phosphating processes for specific reasons such as reaction speed, hydrogen elimination, and sludge formation control. Accelerators can be used as single materials or they can be mixed to achieve the most effective combination. Several materials can be used including nitrite/nitrate, chlorate, nitro guanidine, hydroxylamine, peroxide, and organic compounds such as sodium nitrobenzene sulfonate.

II. CORROSION, PHOSPHATING & PASSIVATION

Corrosion

Corrosion is an electrochemical reaction that deteriorates a metal or an alloy. Reclamation's structures are particularly susceptible to this deterioration based on their proximity to bodies of water and atmospheric exposure. Structures like gates, valves, penstocks, piping, and other exposed features are among some of the most prone to corrosion from condensation, spray, rain, and other environmental conditions. Corrosion of steel requires four elements to complete the electrochemical cell: an anode (the metal that readily gives up electrons, a cathode (the

metal that readily accepts electrons), and an electrolyte (the liquid that helps the electrons move), and a metallic pathway for electron movement. The interaction of these elements is also known as a corrosion cell. Corrosion inhibitors interrupt the corrosion cell by removing one of these elements. In general corrosion of steel, the steel acts as the anode, with adjacent areas acting as cathodes. In order to complete the corrosion cell, the steel is also a metallic pathway, and water or soil can be the electrolyte. Elemental iron is naturally unstable and releases electrons at the anode in the presence of oxygen to become a more stable ferrous iron, $Fe2+$. Those electrons move to the nearby catholic sites on the surface, forming hydroxyl ions ($OH-$) as they combine with oxygen and water.

Phosphating

Phosphating process can be defined as the treatment of a metal surface so as to give a reasonably hard, electrically non-conducting surface coating of insoluble phosphate which is contiguous and highly adherent to the underlying metal and is considerably more absorptive than the metal. The coating is formed as a result of a top chemical reaction, which causes the surface of the base metal to integrate itself as a part of corrosion film.

Phosphate coating is used on steel parts for corrosion resistance, lubrication, or as a foundation for subsequent coatings or painting. It serves as a conversion coating in which a dilute solution of phosphoric acid and phosphate salts is applied via spraying or immersion and chemically reacts with the surface of the part being coated to form a layer of insoluble, crystalline phosphates. Phosphate conversion coatings can also be used on aluminium, zinc, cadmium, silver and tin.

The main types of phosphate coatings are manganese, iron and zinc. Manganese phosphate are used both for corrosion resistance and lubricity and are applied only by immersion. Iron phosphates are typically used as a base for further coatings or painting and are applied by immersion or by spraying. Zinc phosphates are used for corrosion resistance (phosphate and oil), a lubricant base layer, and as a paint/coating base and can also be applied by immersion or spraying.

Passivation

The corrosion resistance of mild carbon steel is due to chromium rich complex. A thin oxide film that forms on the surface of the steel. This is the normal condition for steel surface and is known as a "Passivation". The passive condition on the surface of the mild carbon steel is obtained by formation of a chromium oxide film on the metal's surface. This is accomplished by passivating the metal. Chemical passivation procedures using alkalines, wetting agents, chelants, and organic or inorganic acids are used to clean the metal surface thoroughly and in conjunction with an oxygen rich atmosphere, a uniform chromium oxide film forms. The key is to have a uniform chromium oxide film with an acceptable chrome to iron ratio (typically>1) and thickness (20-30microns). It is only a few atomic layers in thickness where the ratio of chromium to iron (Cr/Fe) is at least 1.5-to-1. The chromium binds with oxygen to create a chemically inert, "passive" surface.

Although passivation occurs naturally in corrosion resistant and chromium-rich alloys, given the proper conditions, a new stainless steel vessel or part needs to be passivated prior to being placed into service. Fabrication, machining and welding leave behind contaminants such as metal oxides, inclusions, fabrication debris, and tramp iron, thereby compromising the metal's natural ability to resist corrosion.

III. APPLICATIONS, ADVANTAGES AND LIMITATIONS

Application

Phosphating:
- Corrosion protection in conjunction with organic coatings, such as paints and polymer films
- Facilitation of cold-forming processes, such as wire drawing and tube drawing, or deep drawing
- Corrosion protection in conjunction with oils and waxes
- Corrosion protection with no subsequent treatment
- Improving anti-friction properties, such as break-in, wear resistance, anti-galling and coefficient of friction
- Providing strong adhesion bonding for subsequent painting or other organic coating

Passivation:
- Passivation removes surface contamination
- Passivation increases corrosion resistance
- Passivation reduces the risk of product contamination
- Passivation allows you to extend system maintenance intervals

Advantages

Phosphating:

> ➢ Increasing corrosion protection
> ➢ Improving friction properties of sliding components
> ➢ It provides is strong adhesion and corrosion protection
> ➢ Improves the friction properties of sliding components

Passivation:

> ➢ Passivation Removes Surface Contamination
> ➢ Passivation Increases Corrosion Resistance
> ➢ Passivation Reduces the Risk of Product Contamination
> ➢ Passivation Allows Extended System Maintenance Intervals.
> ➢ Passivation Saves Money and Reduces Downtime.

Limitations

As with most processes, passivation has disadvantages. Most notably different from electro polishing, it does not smooth out the surface of the metal; it only protects it from corrosion. It also requires a pre-cleaning operation, which will extend the time required to complete the process.

It is seldom impossible to phosphate a part because of its shape. However, shapes can restrict production or limit the choice of process. Parts with complex passages must be immersion coated, because spray phosphating cannot reach all areas of the passages. Cup-shape parts, phosphate coated by either method, present problems of handling to achieve complete drainage. Blind holes or cavities may entrap air, preventing phosphating solution in the immersion process from contacting all areas to be coated.

IV. LITERATURE REVIEW

The specific analysis is performed in the different areas as discussed by this review paper

A. M. El-Shamyet et.al. (2020): In this work, the aqueous Lawsonia inermis extract (LI) is investigated as an economic and green deterioration inhibitive formula for steel alloy 4130 in 3.5 wt.% NaCl solutions.)e water-based extraction process is considered as one of the cheapest techniques for preparation of active ingredients of natural products. These ingredients play an important role in corrosion mitigation of steel alloy 4130 in saline media.is extract was subjected to three different parameters: inhibitor concentration, rotation speed, and temperature in 3.5 wt.% NaCl solutions.e electrochemical techniques are used to perceive the corrosion behavior, and the obtained results were dedicated to theoretical explorations to assess the features of corrosion inhibition and the adsorption over the steel substrate in 3.5 wt.% NaCl solutions. Affording to the electrochemical techniques of LI showed very promising results against corrosion depending on the inhibitor concentrations. The inhibition efficiency of LI was additionally appraised at three

diverse temperatures, and the results disclosed that the inhibition efficiency is decreased. Additionally, the theoretical aspects illuminated that the main active ingredients of LI have a proclivity to coagulate on the steel substrates allowing these areas to paradigm a protecting layer on the steel surfaces. Is behavior is in provision of investigational results. Statistical studies were

used to examine the consequence of chief constraints (i.e., inhibitor concentration, temperature, and rotation speed) on the inhibition efficiency and the rate of corrosion of steel alloy 4130. The inhibitive effect of LI in contradiction of the corrosion of steel alloy 4130 surfaces is considered by resources of DFT/6-31G(d) calculations. quantum chemical parameters interrelated to the

inhibition efficiency are considered.)e characteristics of corrosion inhibition of green inhibitor

based on Lawsonia inermis extract (LI) in 3.5 wt.% NaCl solutions on steel alloy 4130 surfaces were estimated by collective investigational and computational approaches. Examinations by EIS designated that, by cumulating the load of LI from 1 to 10%, the protection efficacy pointedly amplified and touched 89.19%

E.Gurunathan et.al. (2020): This paper reports an investigated to evaluate the corrosive resisted characteristics of Zinc Phosphating coated mild steel. Phosphating is the process of dipping or immersing the oil seal made of cold rolled mild steel in the stock solution to resist the effects made by the engine such as corrosion resistance, rubber to metal bonding, abrasion etc. Now by making time study in the Hi Tech Arai Company, according to that the statistical tool used to quality and the process can be optimized. The influence of the phosphate processing parameters on the corrosion resistance of zinc phosphate conversion coatings and the optimization of the process are investigated using the Taguchi method. The pieces are tested by salt spray method, and the results are analysis the quality of the oil seals was improved. That's the tested material was investigated by SEM Analysis for optimization, which is used for purpose of achieving best designed components. The electrochemical corrosion method to

corrosive rate of the Phosphate conversion coated material is analyzed. The aim of the present work is to control the iron concentration in the Phosphating bath in order to improve the quality and adhesive properties of zinc Phosphating coating on mild steel. Salt spray test to corrosion resistance and weight loss of the material is calculated. Weight difference to SN value and Range of each parameter are calculated using the S/N equation. Averaging the S/N ratio for each parameter to Range and rank of the parameter are obtained and to that Better Phosphating parameter is determined. Electrochemical corrosion test to corrosive rate of the coated and non-coated materials are analyzed.

Jakub Duszczyk et.al. (2018): Manganese phosphate coating could be used to protect the surface of steel products. However, it is essential to determine the effects which process parameters, as well as the types of additives used, have on the efficiency of coating deposition. Thus, we present here a process of phosphatization of low-alloy steel (for 15 min at 95 C) in manganese/nickel baths followed by a passivation process with the use of a silicon and zircon compounds. The microstructure and morphology of the surface were analyzed by SEM EDX and XRD methods. The obtained results showed that the manganese phosphate could be effectively formed at 95 _C in the solution containing nickel and guanidine derivatives. Anodic polarization of manganese coating was investigated in 0.5 M KCl by the analysis of polarization resistance. The effects of the activation process on corrosion properties of the coating have been examined. It was observed that an increased concentration of activating substances in the activation bath results in the enhancement of corrosion resistance.

Hafez Alizadeh et.al. (2018): The aim of this study is to investigate the effect of nitrate and nitrite on the weight, morphology and electrochemical properties of phosphate Zn-%12Ni electrodeposited coatings. In order to investigate the phase structure and surface morphology of samples, X-ray diffraction and scanning electron microscopy were employed. Also, to measure the corrosion resistance behavior of the coats, Potentiostat/Galvanostat test was used. The results showed that nitrite accelerator reduces coating weight and surface porosity simultaneously obtaining by phosphating solution. Furthermore, coatings being obtained by the nitrite accelerator had a higher corrosion resistance than that of the nitrate accelerator For phosphate coating, cylindrical steel samples with 10mm diameter and 10mm thickness were used. For surface preparation, the samples were ground with sandpaper No 240-1000 first and then mechanical preparation was done to achieve uniform and suitable surface. In order to remove pollutions, oil, and corrosion products, degreasing by 60g/l NaOH at 60-70oC temperature for 20 minutes, and acid cleaning in 30% HCl solution for 1 minute at ambient temperature were carried out. Finally, to form the Zn-12Ni coating on samples, a bath with chemical composition and working condition Hence, an aqueous solution with 0.015% chromic acid was used. After phosphating, samples were immerged in the mentioned solution for 15 seconds at room temperature. Figure 1 shows schematic of phosphating process steps. For phase structure study, X-ray diffraction XRD) was performed with D8 advance diffractometer (Brokers) using Cu radiation (Cukα=1.54oA). The step-scan mode was 2–theta from 10° to 60° with a step size of 0.033°. The surface morphology of samples was investigated with CAMSCAN2300 Scanning electron microscopy (SEM). Coating weight as a main factor in determining the phosphate coating quality was measured for bath parameters and operating conditions standardization. The weight of the phosphate coating was determined by weighing the phosphated samples before and after stripping in 25g/l chromic acid solution for 2 minutes at 50oC temperature.

Filip Pastoreket.al. (2016) High strength low alloy (HSLA) steels exhibit many outstanding properties for industrial applications but suffer from unsatisfactory corrosion resistance in the presence of aggressive chlorides. Phosphate coatings are widely used on the surface of steels to improve their corrosion properties. This paper evaluates the effect of a manganese phosphate coating prepared after various mechanical surface treatments on the electrochemical corrosion characteristics of S355J2 steel in 0.1 M NaCl electrolyte simulating aggressive sea atmosphere. The manganese phosphate coating was created in a solution containing H3PO4, MnO2, dissolved low carbon steel wool, and demineralised H2O. Scanning electron microscopy (SEM) was used for surface morphology observation supported by energy-dispersive X-ray analysis (EDX). The electrochemical corrosion characteristics were assessed by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PD) measurements in the solution of 0.1 M NaCl. Method of equivalent circuits and Tafel-extrapolation were used for the analysis of the obtained results. Performed experiments and analysis showed that the morphological and corrosion properties of the surface with manganese phosphate are negatively influenced by sandblasting surface pretreatment.

VI. CONCLUSIONS

This paper investigates the implementation of Taguchi design in the estimation of optimum minimum corrosion rate of steel in cooling tower that uses saline solution of different concentration and different working parameters. It had been concluded that Taguchi design prepare a useful methodology for the setup and optimization of corrosion rate with minimum numbers of trials in comparison to other experimental design.

The Taguchi method provides a systematic and efficient methodology for the design and optimization of corrosion protection parameters with far less effort than would be required for most optimization techniques.

Sludge: This thick, moist substance has been a necessary evil of the phosphating process, costing significant resources and downtime for the cleaning of equipment and tanks. Additionally, the cost to dispose of this sludge can be prohibitive, particularly in the quantities it is produced. Experts say that the ideal new technology would reduce sludge output by 40 to 50 percent or more, and would facilitate the continuous removal of the sludge, virtually eliminating the downtime factor from the equation.

Water Consumption: The current phosphating process requires multiple rinse steps that requires high volumes of water, and also produces a significant amount of wastewater that needs internal pre-treatment before it can be discharged to sewer systems. Scientists say that a new, more environmentally friendly process could be developed which would eliminate the need for rinses – and result in a zero-discharge system.

High Temperatures: The high temperatures generated by the phosphating process create incredible inefficiencies – requiring costly energy to heat process tanks up to 200°F. The mists and vapors generated by heated tanks are hazardous for employees, requiring removal from the environment using ventilation. An ideal solution could potentially be developed, say engineers, that would allow for the operation of the phosphating process at room temperature, reducing energy consumption and eliminating ventilating equipment.

Space: Zinc phosphating is a "production-line" process, requiring a large footprint to complete its cleaning, rinsing, activating, phosphating, a second round of rinsing, and drying steps – plus the addition of ancillary equipment to treat the waste; ductwork for ventilation; hoists to move baskets; and so on. Condensing this process to less than half the floor space of the current scheme, while producing equivalent volumes of output, is the "Holy Grail" of phosphating production – particularly when considering rising real estate prices and the attendant energy costs and other carrying costs related to physical plant.

Color: Zinc phosphating typically has delivered a finished product that has a gray matte finish – requiring interim steps, depending upon the application. For fasteners, this often means dipping the completed part in a colored wax or other topcoats to facilitate the distinction of one part from another; for larger parts, it requires heavy coats of primer or finish preparation prior to painting. Engineers say a process can be developed which would allow for multiple primary base colors, including blue, green, red and yellow. This would eliminate the interim colorization step, in many cases, and also cut down on paint coats for larger applications.

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