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Comparative investigation of corrosion rate on A-36 steel with different coatings include ZnO and TiO_2 nanoparticles using linear polarization resistance technique

Maaz Akhtar[a,*], Muhammad Imran Lashari[b], Muhammad Muzamil[a], Mohsin Sattar[c], Muhammad Imran Shabir[b], Sumiya Mohsin[a], Muhammad Samiuddin[d]

[a] Mechanical Engineering Department, NED University of Engineering and Technology, Karachi, 75270, Pakistan

[b] Mechanical Engineering Department, Swedish College of Engineering and Technology, Rahim Yar Khan, Pakistan

[c] Department of Mechanical Engineering, Universiti Teknologi PETRONAS, Perak, Malaysia

[d] Department of Metallurgical Engineering, NED University of Engineering and Technology, Karachi, 75270, Pakistan

(*Corresponding author: maaz@neduet.edu.pk, maazakhtar@hotmail.com)

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ABSTRACT: The current study is conducted to develop an optimized corrosion resistant method. Low carbon steel (A-36) is used with five different coatings along with an uncoated sample, to characterize the behavior against corrosion. Specimens are coated with red oxide primer, oil paint, and oil paint-primer. Coatings are also made by mixing nanoparticles of titanium oxide (TiO_2) and zinc oxide (ZnO) with oil paint. One molar nitric acid (HNO_3) solution is used to produce acidic medium, one molar sodium hydro-oxide (NaOH) solution is used to make basic medium and distilled water is used as a neutral medium. The linear polarization resistance (LPR) technique is used to determine the corrosion rate of different coatings in all conditions. In the acidic environment, the bare sample gives maximum corrosion of 191.5 mpy. The corrosion rate is decreased when coated with primer and paint respectively. But the minimum value of 0.302 mpy is observed in zinc oxide nanoparticles based coatings. In basic medium corrosion rate is observed to be low in bare and all types of coatings compared to the acidic environment. It shows that mild steel produces less metal oxides in a basic environment. The corrosion rate trend in the basic medium is the same having maximum in the bare sample (i.e. 0.1044 mpy) while minimum in zinc oxide-based coating (i.e. 0.000261). In distilled water, the bare sample gives maximum corrosion rate of 12.98 mpy as expected. Comparing three environments, acidic medium gives the highest corrosion rate in bare samples and in all coatings. Hence proper attention should be given when mild steel is being used in an acidic environment. The maximum corrosion rate is observed in bare samples while minimum in specimen coated with zinc oxide-based coatings. Hence it can be concluded that for better corrosion resistance, a coating made by mixing paint with zinc oxide nanoparticles should be used that works in all environments. Current study can be considered as easy to use solution for corrosion prevention in different corrosive environments.

KEYWORDS: Acidic medium; Basic medium; Corrosion rate; Linear polarization resistance (LPR); Low carbon steel; TiO_2 nanoparticles; ZnO nanoparticles.

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RESUMEN: *Comparación de la velocidad de corrosión del acero A-36 recubierto con nanopartículas de ZnO y TiO_2 utilizando la resistencia de polarización lineal.* El estudio se llevó a cabo para desarrollar un método optimizado de resistencia a la corrosión. El acero A-36, con bajo contenido de carbono, se utilizó con cinco recubrimientos diferentes y una probeta sin recubrir. Las probetas se recubrieron utilizando una imprimación de óxido rojo, pintura al óleo e imprimación de pintura al óleo. Dichos recubrimientos se fabricaron mezclando nanopartículas de óxido de titanio (TiO_2) y óxido de zinc (ZnO) con pintura al óleo. Una solución molar de ácido nítrico (HNO_3) se utilizó para obtener un medio ácido, una solución molar de hidróxido de sodio (NaOH) para conseguir un medio básico, y agua destilada para obtener un medio neutro. La técnica de resistencia de polarización lineal (LPR) se utilizó para determinar la velocidad de corrosión. En medio ácido, la probeta sin recubrimiento produjo una velocidad de corrosión máxima de 191,5 mm por año. La velocidad de corrosión disminuyó al aplicar el recubrimiento de imprimación y acabado con pintura. El valor mínimo de velocidad de corrosión (0,302 mm por año) se observó en recubrimientos a base de nanopartículas de óxido de zinc. En medio básico, se observó que la velocidad de corrosión era pequeña con todo tipo de recubrimientos y sin protección adicional, en comparación con el medio ácido. Lo que indica que el acero A-36 produce menos óxidos metálicos en medio básico. La tendencia de la velocidad de corrosión en medio básico es la misma, teniendo el máximo de velocidad de corrosión en la probeta si protección adicional (0,1044 mm por año), mientras que el mínimo se produjo con el recubrimiento a base de óxido de zinc (0,000261 mm por año). En agua destilada, la probeta sin protección adiconal produjo, como se esperaba, una velocidad de corrosión máxima de 12,98 mm por año. Al comparar los tres medios, el ambiente ácido proporciona la velocidad de corrosión más alta en probetas sin protección adicional y con todos los recubrimientos. Por lo tanto, se debe prestar atención al utilizar el acero A-36 en medio ácido. La máxima velocidad de corrosión se observó en probetas sin protección adicional, mientras que la mínima se obtuvo en probetas recubiertas con recubrimientos a base de óxido de zinc. Por tanto, se puede concluir que, para una mejor resistencia a la corrosión, se debe utilizar un recubrimiento elaborado mezclando la pintura con nanopartículas de óxido de zinc que funcione en todos los medios.

PALABRAS CLAVE: Acero bajo en carbono; Nanopartículas de óxido de titanio; Nanopartículas de de óxido de zinc; Medio ácido; Medio básico; Resistencia de polarización lineal (LPR); Velocidad de corrosión

ORCID ID: Maaz Akhtar (https://orcid.org/0000-0002-5061-8090); Muhammad Imran Lashari (https://orcid.org/0000-0002-9371-949X); Muhammad Muzamil (https://orcid.org/0000-0001-8380-0502); Mohsin Sattar (https://orcid.org/0000-0002-5531-0765); Muhammad Imran Shabir (https://orcid.org/0000-0002-5082-0504); Sumiya Mohsin (https://orcid.org/0000-0002-1395-8344); Muhammad Samiuddin (https://orcid.org/0000-0002-2350-6114)

1. INTRODUCTION

Metals are being used in diverse structural or machine applications due to good strength (under fatigue and different load conditions), better manufacturing capabilities, etc (Akhtar *et al.*, 2018). Steel and aluminum are being the top on the list. Steel is an alloy of iron that is the fourth most abundant material present in the earth's crust. Kumar *et al.* (2019) discussed the applications related to composite having titanium dioxide (TiO_2) as a reinforcing particle and aluminum (Al 3003) as a matrix. A process known as friction has been applied successfully as a stir procedure for synthesizing the formation of aluminum metal composites. Efforts were done to check out the effect in different combinations of reinforcing particles on the weight percent ratios within the matrix. Typically, steel has 95% or more iron with relatively small alloying elements such as C, Mn, Si, Cr, Ni, Mo, Al, Ti, V, Nb, and Co. Steel has a wide variety of applications from simple households to complex engineering solutions like cars, bridges, mega-structures, turbines, etc. Applications made of steel have a measure issue of corrosion. Samaniego-Gámez *et al.* (2020) investigated the effect of corrosion of AA2099 aluminium alloys under different environments. Authors concluded that 6 wt.% $Na_2Cr_2O_7$ sealing treatment resulted in improving the corrosion resistance.

Many authors investigated steels in different conditions and provide a solution for corrosion prevention (Finšgar and Jackson, 2014; Lu *et al.*,2016; Deng *et al.*, 2019). Finšgar and Jackson (2014) study the number of corrosion-inhibitors for steel in the solutions of acid. Steel is kept in HCl solutions for experiments, and emphasizes is made on high temperature. A mixture of surfactants, enhancers, inhibitors of corrosion and solvents are used for the study. Mixture improves the effectiveness at high temperatures for individual compounds and making it very useful information for a variety of industrial areas, such as acidification procedures and the applications where steel corrosion-inhibitors are required. Dastgerdi *et al.* (2019) investigated the degradation of surfaces on the performance of stainless steel materials. Authors used the design of experiments to check out the effects of pH, chloride-concentration in both crevice and pitting corrosion of stainless-steel at high temperatures. The design of experiments reduces the set of experiments required to investigate the effect of different parameters on passive breakdown potential and found that temperature's effect was more important in pitting corrosion. Hossain *et al.* (2019) explore the cinnamaldehyde effect as an environmentally protecting inhibitor that reduces the corrosion's damage of aerated NaCl (3% w/w) mild steel in which potentio-dynamic polarization and weight loss methods are used. Cinnamaldehyde moderate prevention efficiency reached about 70% by making an adsorption layer on the metal surface at the optimal level of 0.5 $g\cdot L^{-1}$. The corrosion rate becomes slow when the cinnamaldehyde dose reached the optimum level, but slowly increasing at moderate temperatures.

Melia *et al.*, (2019) investigated corrosion in stainless steel by the effect of processing and microstructure-defects in directed energy deposition (DED). The Materials is formed by DED having heat input of 0.45 and 0.03 $kJ\cdot mm^{-1}$ in comparison of forged 304L. Because there is no fusion pore in DED-material, the breakdown- potential (Eb) is controlled by 1st order and reduced it up to 400 mV compared with 0.6M NaCl base-material. Pradhan *et al.* (2019) discussed the effect of grain size, grain distribution, and residuals in corrosion by pitting in stainless steel using two types of polarization tests of potentio-static and potentio-dynamic. Authors found that the higher the dislocation density of the deformed specimen, the higher will be the pit generation rate. Haldhar *et al.* (2019) researched Acacia-concinna resistive properties on mild steel when it is extracted in a medium of 0.5 M sulfuric acid. The composition and structure of the surface film were examined with SEM and an atomic-force microscope. Perform electrochemical impedance and potentiometric spectroscopy. The electrochemical analysis showed that the 250 $m\cdot L^{-1}$ inhibitor in concentration showed the best results.

Zhang *et al.* (2018) review some benefits and limitations of the self-healing procedure for preventive organic-coatings that can be used for corrosion resistivity purposes. Autonomous healing methods are mostly possible by implanting polymeric healing materials or deterioration preventers used in the coating matrix. In the case of a non-autonomous procedure, the effects of healing are activated by an outside source that becomes the cause of compound reactions that are useful for bonding. For the current study A-36 low carbon steel is used that has a density of 7800 $kg\cdot m^{-3}$, Young's modulus value of 200 GPa, Poisson's ratio of 0.26, and shear modulus of 75 GPa (Dai *et al.*, 2017; Li *et al.*, 2017). Many authors investigated the corrosion rate of material using experimental methods like linear polarization resistance (LPR), weight-loss method, etc. Erfani *et al.* (2015) discussed the use of the LPR method to investigate corrosion in the operation of CO_2 removal units. The study shows the effect of temperature, inhibitor, and CO_2 content on mild-steel and stainless steel in both laboratory and long duration tests in an operating plant. The result shows 1 to 11 mpy corrosion rate for temperature ranges from 60 °C to 80 °C. The use of salicylic acid decreases the corrosion rate by 43%. Zaferani and Shishesaz (2014) used a dye named AYGG (Alizarin yellow GG) as an inhibitor. This method is used to study the corrosion prevention in carbon steel in an acidic environment. The results show better resistance to corrosion by newly made inhibitors even in the highly corrosive environment. Barranco *et al.* (2004) investigated the behavior of different paints along with zinc-based coatings on steel in3% solution of sodium chloride. The results of this study give better corrosion prevention for painted specimens as compared to bare samples. Zinc-based coatings also give very good prevention against corrosion. Parhizkar *et al.* (2018) studied the effect of epoxy-based cerium nano-coatings on steel samples and produce coatings like Ce, Ce-AGO, and Ce-GO, and changes in morphology were investigated using field emission SEM. The outcome of the experimental study showed that cerium nano-coatings can enhance adhesion, prevention against corrosion, and delaminating of the films. Osarolube *et al.* (2008) studied the effect of corrosion on high-to-low carbon steels in different acidic environments. Samples are exposed for a week and investigated the corrosion rate using a weight loss technique. It was concluded that the fastest corrosion rate occurs in nitric acid, medium in per chloride, and slowest in hydrochloric acid. Techniques used by different authors provide the solution to many problems against corrosion but they usually need complex, and expensive methods or sometimes required more time and effort. Sambathkumar *et al.* (2021) studied corrosion resistance of Al 7075 matrix alone and Al 7075-redmud based composite. Corrosion rate is found out to be decreased in case of redmud-matrix composite as compared to matrix alone.

The current study is based on providing a simpler, quick, and relatively cheaper method for corrosion prevention. Low carbon steel (A-36) is being used for experimental investigation of corrosion rate under different coatings. Five different coatings of red oxide primer, oil paint, primer plus oil paint, paint mixed with TiO_2, and ZnO based nanoparticles are applied. To explore the effect of environmental conditions all tests were performed in acidic, basic, and neutral environments. The following sections provide details of experiments performed along with the results obtained.

2. EXPERIMENTAL METHOD AND SETUP

2.1. Linear Polarization Resistance (LPR)

LPR method is an electrochemical process that is widely used to determine the polarization resistance of a material that helps in determining the corrosion level (ElBatanouny *et al.*, 2013). Figure 1 gives the experimental setup of LPR used for the current study.

The electrical conductivity of the fluid is related to the ability to produce corrosion. This technique establishes the relationship between the current of charged electrons and electrochemical potential. It generally includes two or more electrodes which on erosion generates polarization resistance that ultimately gives the degree of corrosion. Material is first polarized in an open circuit to ±10 to ±20 mV. Current usually flows

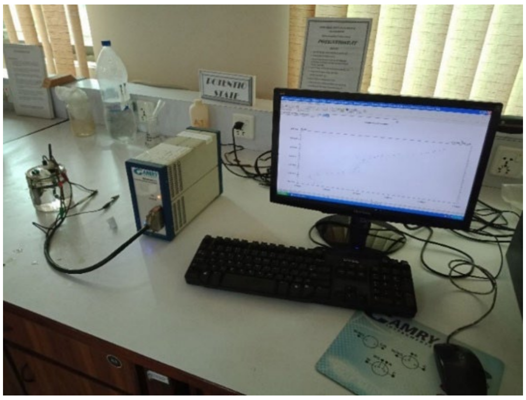

Figure 1. Linear Polarization Resistance (LPR) Experimental Setup.

between electrode made of material under consideration and counter electrode (Amin *et al.*, 2010; Boikanyo *et al.*, 2018; Lu *et al.*, 2017). Polarization resistance (PR) is calculated by determining the slope of electrochemical potential and current. Using the Stern Geary equation (Angst and Büchler, 2015) this PR can be converted into a corrosion rate.

$$R_p = \frac{B}{i_{corr}} \quad (1)$$

where, B is the proportionality constant for the particular corrosion system and ιcorr is its corrosion current density (in units of $\mu A \cdot cm^{-2}$). Tafel plot is used to determine proportionality constant (B) by measuring the slopes of anode (β_α) and the cathode (β_b) empirically.

$$B = \frac{\beta_\alpha \beta_b}{2.3\,(\beta_\alpha + \beta_b)} \quad (2)$$

$$R_p = \frac{\beta_\alpha \beta_b}{2.3\,(\beta_\alpha + \beta_b)\, i_{corr}} \quad (3)$$

Tafel constants are dependent on electrode used and the environment. Many authors determined these constants for many electrodes under different acidic or basic solutions (Bates and Pinching, 1949).

2.2 Experimental setup

A-36 structural steel is used in the present study for the determination of the corrosion rate (CR). CR is typically stated as a degree of penetration deep inside the material. This phenomenon takes time hence it is normally given in terms of the degree of penetration occurred over a period like inches per year or melts per year (MPY) where melts equals to 0.0254 mm (1/1000 inch) (Mendonça, *et al.*, 2017). Test specimens of dimensions 1 cm x 1 cm are cut from the plate of A-36 steel material. The specimens are grinded and polished by using different grades of sandpapers from coarse to finer grit sizes. The final finishing step is done using the cloth-wheel machine with Alumina powder. Specimen than soldered with copper wire (10 inches long) to make connections and later molded in epoxy material as shown in Fig. 2. Corrosion testing has been done using LPR equipment.

For the experimental investigation, different coatings were made and resistance to corrosion in each condition is evaluated. Coatings are applied using

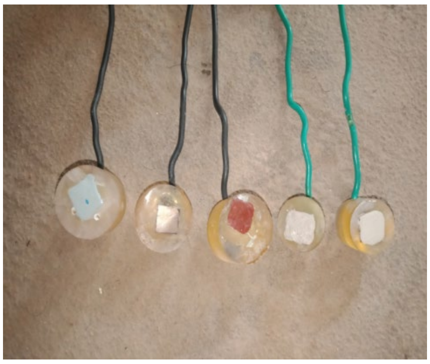

Figure 2. Linear Polarization Resistance (LPR) Specimen.

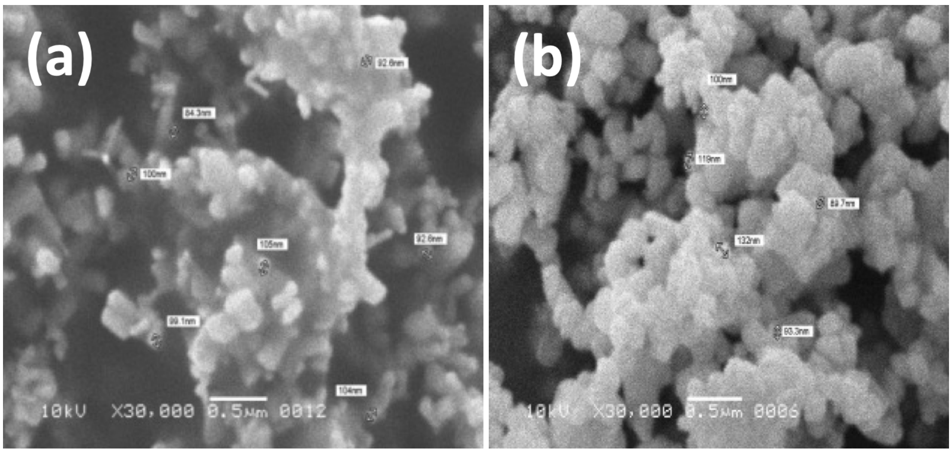

Figure 3. SEM images of a) Zinc-oxide and b) Titanium-oxide nanoparticles.

Primer (red-oxide), Paint, Primer and Paint, Paint with zinc oxide nanoparticles, and Paint with titanium oxide nanoparticles. Figure 3 gives the Scanning electron microscope (SEM) images of zinc oxide (ZnO) and titanium oxide (TiO_2) nanoparticles that were used in coatings. SEM image gives the average size of ZnO nanoparticles of 97.5 nm and for TiO_2 average size comes out to be 106.8 nm. To investigate the correct response for zinc-oxide and titanium oxide-based coatings, nano-particles for about 5-10% by weight are added in the paint as described by Schaefer and Miszczyk (2013). Three environmental conditions are provided to the specimen to investigate the effect in different situations like acidic, basic, and neutral. Standard 1M solution of NaOH is used to produce a base environment, 1M solution of HNO_3 is used to provide the acidic environment and the neutral condition is maintained by distilled water. For each environment, six conditions were tested (five with coatings and one bare sample).

Coatings are applied manually to each specimen. The thickness of coatings is measured using an electrometer and found to be 50 to 60 microns in each case. The next section discusses different results obtained by experimental investigation and analysis of those results.

3. RESULTS AND DISCUSSION

In this section results obtained by the LPR, the experiment is described. One sample without coating (bare) and five with different coatings are tested in acidic, basic, and neutral environments.

3.1. Acidic environment

To provide an acidic environment, specimens are kept under 1 molar HNO_3 solution in LPR equipment. Sample without coating is tested first. Equipment through controller sends the data to computer software that gives a graph between the voltage in millivolts vs reference current in milli-ampere. For illustration purposes, the linear polarization graph for the bare specimen is shown in Fig. 4 as a sample. The graph shows a linear increasing trend.

The above curve is used to determine polarization resistance using the Stern-Geary equation as described above. For the bare sample, the value of the corrosion rate is found to be 191.5 mpy. This value shows a relatively higher amount of corrosion. For the remaining samples, a similar process is used to determine the melts per year. Following Fig. 5 shows the corrosion rate in melts per year for all the specimens in the acidic environment.

It can be seen that the bare sample showed the fastest corrosion rate as expected. While the sample in the paint gives the corrosion rate of 44.31 mpy, primer alone gives 56.96 mpy, paint plus primer gives 61.3 mpy in acidic environment respectively.

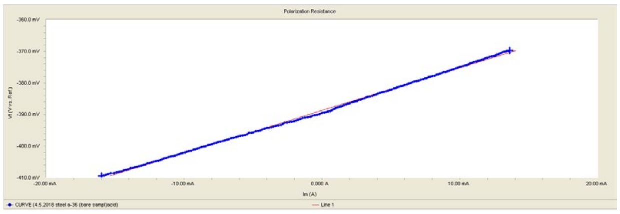

Figure 4. LPR experimental graph between voltage and current for bare sample in Acidic Environment.

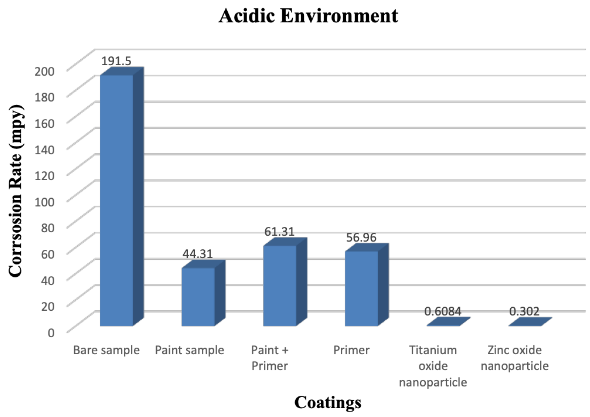

Figure 5. LPR experimental results for different coatings in Acidic Environment.

The addition of nano-particles in paints gives a significant improvement in the corrosion rate. For titanium-based nano-particles paint, coatings resulted in a huge decrease in corrosion rate i.e. 0.6084 mpy that further dropped to 0.302 mpy in case of zinc oxide-based nano-particles paint coatings. The addition of zinc oxide nanoparticles in paints improves the cathodic protections by creating additional conductive paths between the nano-particles. These nano-particles start to be oxidized confirming better corrosion prevention to the steel specimen. Titanium also gives good prevention against corrosion due to its chemical affinity to form to oxides that help in cathodic protection.

3.2. Basic environment

Figure 6 gives the results obtained by LPR testing for different coatings to determine the corrosion rate in the basic environment. Samples were tested under 1 molar sodium hydro-oxide solution that serves as the base environment. Corrosion rates obtained are 0.1044 mpy, 0.0281 mpy, 0.0129 mpy, and 0.0307 mpy in the bare sample, painted, primer based and paint plus primer samples respectively. Paint coating with titanium oxide-based nano-particles gives 0.07083 mpy that is slightly higher than the paint/primer samples. Zinc oxide nano-particles based paint gives the minimum corrosion rate of 0.000261. As expected, in base condition overall better resistance to corrosion is achieved. That's the reason that in highly

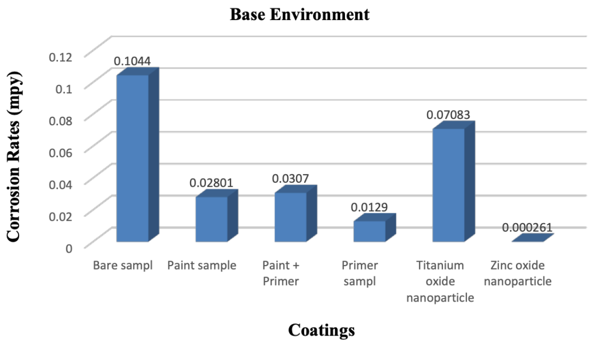

FIGURE 6. LPR experimental results for different coatings in Base Environment.

corrosive environment inhibitors were used to provide a basic environment.

3.3. Neutral environment

Figure 7 gives the LPR results obtained for different coatings in a neutral environment. Neutral condition is provided by distilled water. It can be seen that the bare sample gives the corrosion rate of 12.98 mpy, paint gives 0.022 mpy, primer gives 0.0255 mpy, and primer plus paint gives 0.0204 mpy. Zinc-oxide nano-particles and titanium oxide nano-particles based paint coatings give the corrosion rate of 0.00265 mpy and 0.435 mpy, respectively. Again it can be observed that nano-particles based coatings give the best resistance against corrosion.

The bare sample gives the highest corrosion rate of 191.5 mpy in the acidic, the medium of 12.98 mpy in neutral and lowest of 0.1044 mpy in the base environment. This similar trend follows nearly in all samples coated with paint, primer, and primer plus paint. Titanium oxide nanoparticles based paint coating gives highest to lowest corrosion rate in acid, neutral, and base environment respectively. Zinc oxide nano-particles based paint coating follows the similar trend giving maximum resistance to corrosion in the base environment and minimum resistance in an acidic environment. In order to have better understanding and study the effect at a glance a three-dimensional plot is added (Fig. 8), that relates corrosión rate, coatings and environments in a single graph. On the comparison between the acidic, base, and neutral environments, it can be observed that the highest corrosion rate occurred in an acidic environment. The neutral environment also shows more resistance to corrosion as compared to the acidic environment. Best cathodic protection is found in the base environment. Amongst different coatings, bare samples gave the worst corrosion resistance while zinc oxide-based nano-particle paint coatings give minimum corrosion rate (or maximum prevention against corrosion) because of results already explained above. Zinc oxide nano-particles based paint provide coatings that tremendously decreases the corrosion rate from 191.5 mpy to 0.302 mpy in the worst environmental condition (acidic). The current study provides a simple, easy, and less expensive method of synthesis of protective coatings that provide excellent prevention against corrosion.

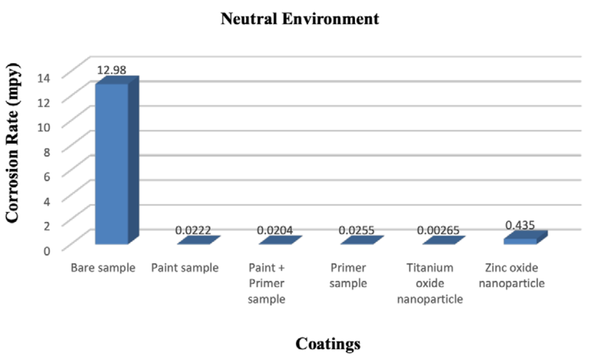

FIGURE 7. LPR experimental results for different coatings in Neutral Environment.

4. CONCLUSIONS

- The current study investigated the corrosion prevention capabilities of different coatings. Low carbon steel is selected for evaluating the corrosion rate using paint, primer, primer plus paint, special nano-particles based (zinc-oxide and titanium oxide) paint coatings. Three environmental conditions are provided (acidic, base, and neutral) to all coated specimens. All experiments were conducted using LPR technique for determining the corrosion rate. The bare sample was also tested to observe the worst scenario of corrosion prevention. In the acidic environment, the bare sample gives 191.5 mpy, neutral condition gives 12.98 while the base environment gives 0.1044 mpy corrosion rate.
- The corrosion rate showed a decreasing trend in paint, primer, and primer plus paint in an acidic environment. Titanium oxide-based coating

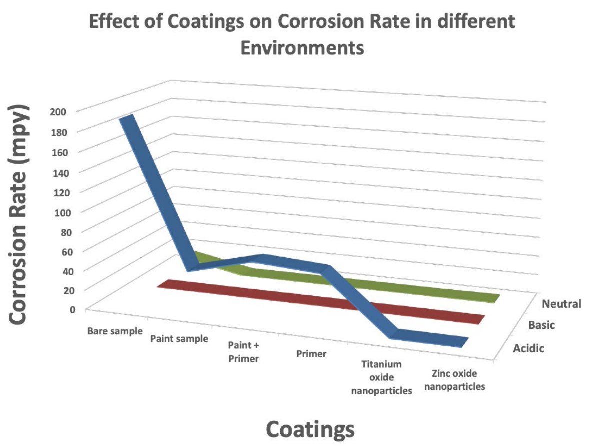

FIGURE 8. 3D Plot of Effect of Coatings on Corrosion Rate in different Environments.

gives a 0.6084 mpy corrosion rate. The lowest rate is seen in zinc oxide nanoparticles based coatings (0.302 mpy). As compared to the acidic environment, samples showed a lesser corrosion rate in the basic medium for all bare and coated samples. In distilled water, again bare sample showed the largest corrosion rate as estimated.

- Experimental observations anticipated the analogous drop in corrosion rate in primer, paint, primer plus paint, titanium oxide, and zinc oxide-based coatings respectively. Hence it can be resolved that the base environment gives the best prevention against corrosion. On comparison amongst different environmental conditions provided, acidic medium gives the largest corrosion rate in bare samples as well as in all coatings. Zinc oxide based coating gives the maximum cathodic protection amongst all the coatings while the bare sample gives the worst.
- It can be concluded from current research that zinc oxide nanoparticles coating made by mixing with paint shows the maximum resistance against corrosion in all acidic, base, and neutral environments. Hence in different applications having the worst environmental conditions, this coating provides easier, reliable, and cheaper solutions against corrosion.

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