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Electrophoretic Deposition of Barium Titanate Nanopowder Coatings for Corrosive Protection of Carbon Steel

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ABSTRACT

Nanomaterials coatings were developed for corrosion protection of carbon steel. Barium titanate ($BaTiO_3$) possesses a distinctive layered structure that can be effectively utilized to enhance the resistance to corrosion in industrial coating systems. Homogeneous Barium titanate films were deposited successfully by electrophoretic deposition (EPD) using an aqueous suspension under altering deposition time. An investigation was conducted using electrochemical measurements to determine the impact of a Barium titanate $BaTiO_3$ coating on the corrosion resistance of carbon steel. Deposited layers characterizations such as adhesion and wettability have been investigated. X-ray Diffraction (XRD), a field emission scanning electron microscope (FE-SEM), and Energy Dispersive Spectroscopy (EDX) confirmed the successful incorporation of the composites in the coating. The corrosion protection properties of the prepared coatings were evaluated using OCP, Tafel, and Cyclic Voltammetry (CV) measurements. The surface morphology of the coatings after corrosion was studied using Field Emission Scanning Electron Microscopy (FESEM). The scanning electron microscope (SEM) analysis of the deposited thin film shows that the film is adhered, dense, and free of voids. This characteristic allows the coated carbon steel to have a high corrosion resistance.

1. Introduction

Carbon steel has been widely used in various industries and technologies for decades, particularly in applications requiring large amounts of steel, such as construction. This is mainly due to its abundant availability and low cost. Furthermore, it can be welded, exhibits toughness and durability, and is easily annealed. Nevertheless, it is considered to be one of the construction materials that are highly susceptible to rapid corrosion [1]. Carbon steel is susceptible to different types of corrosion caused by multiple factors, which lead to the degradation and breakdown of metal components in structures [2, 3]. Corrosion of metals is widely recognized as a significant issue that poses numerous challenges for societies, impacting the economy, safety, as well as health [4, 5] Applying external coatings, materials with a corrosion allowance, or corrosion resistance materials can all help control and prevent corrosion. To control or reduce the corrosion of carbon steel, a range of protective methods have been implemented, one of which involves the application of protective coatings. Coating is essential for enhancing the surface quality and protecting a substrate by isolating it from the surrounding environment [4].

Electrophoretic Deposition (EPD) is a highly notable method for applying coatings that relies

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on the process of electrophoretic deposition. Currently, there is a growing interest in ceramic coating processing technology from both academics and industrialists, owing to its wide range of potential applications [6]. Electrophoretic deposition (EPD) is a recently developed and highly adaptable method used in electronic applications. Electrophoretic deposition (EPD) is a versatile technique that can be used in a wide range of applications, including magnetic materials, bioceramics, and high-temperature ceramics. This method alters the form, morphology, structure, and porosity, along the surface area [7]. The primary benefits of this technique include its exceptional adaptability when applied to various materials and their combinations, as well as its cost-effectiveness due to the utilization of uncomplicated and inexpensive equipment. Furthermore, it can be utilized on a vast magnitude, including the application of a protective layer on objects with intricate forms, as well as the creation of composite micro- and nanostructures, and the production of near-net-shape objects with precise dimensions (micro- and nano-manufacturing) [6].

Barium titanate is an electro-active ceramic known for its piezoelectric properties, making it suitable for use. Barium titanate ($BaTiO_3$) is a ferroelectric substance that possesses exceptional characteristics, including a high dielectric constant, strong ferroelectric properties, and significant electro-optic as well as non-linear coefficients. $BaTiO_3$ thin films are highly regarded for their potential use in various device applications, including ferroelectric random-access memories, optical modulators, switches, waveguides, micro-electromechanical systems, and structural health monitoring [8].

Several researchers have conducted studies on the EPD process for $BaTiO_3$, however, there is currently no available report on evaluating the EPD process for carbon steel. Despite barium titanate's widespread use, its behavior in coating industrial applications is still unclear. No published research on the electrochemical properties of carbon steel electrophoretically coated with barium titanate has been located as far as we are aware. Investigating the microstructure and corrosion behavior of barium titanate is thus the unique aspect of this study. The $BaTiO_3$ nanopowder coating is deposited on the surface of carbon steel using electrophoretic deposition technique. The coated samples underwent various electrochemical tests to assess their corrosion resistance.

2. Experiment

2.1 Materials

Carbon steel specimens of dimensions 20 mm ×20 mm ×3 mm having composition C-0.098, Si-0.188, Mn-0.702, P-0.0104, S-0.0056, Cr-0.0147, Mo<0.002, Ni-0.0257, Cu-0.0399, Al-0.0430, and balance iron (all in weight percent) were used as the substrate materials for the deposition of nanopowders coatings. The specimens were abraded with a series of emery papers up to 500 grits degreased with acetone to remove the impurities rinsed thoroughly with double distilled water and finally dried in air.

Barium titanate nanopowder is procured commercially with a particle size of 50-70 nm and a purity of 99.9% (provided by SkySpring Nanomaterials, Inc.). Figure 1 shows the morphology of $BaTiO_3$ Nanopowder. It has been used as the nanopowder for coatings on carbon steel substrate.

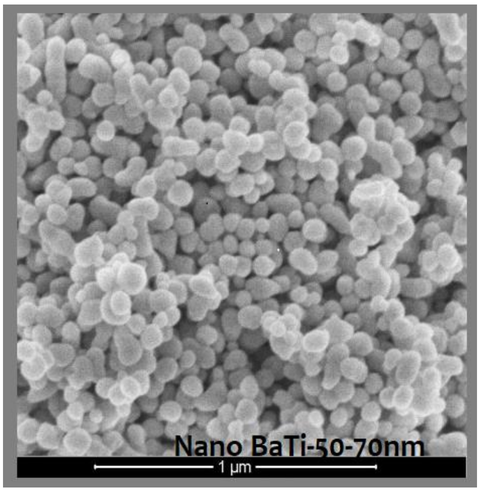

Figure 1. Morphology of $BaTiO_3$ Nanopowder

2.2 Electrophoresis Deposition

The $BaTiO_3$ Nanoparticles were dispersed in ethanol at room temperature using a magnetic stirrer for 4 hours to achieve a stable suspension and directly used in the electrophoretic deposition process as shown in Figure 2. The carbon steel samples were polished mechanically, degreased, and rinsed with water. Carbon steel sample was used as cathode and Graphite as anode with a distance of 1.5 cm for optimal process control. The electrodes were connected to a DC power supply of 3 V, current 0.01 A, and the deposition time (2, 4, and 6 min).

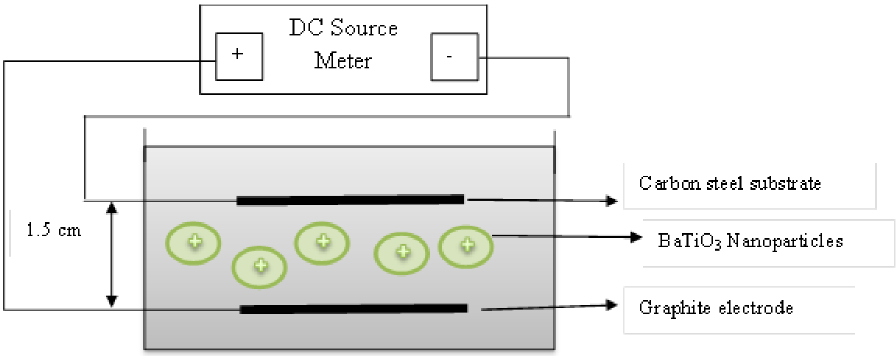

Figure 2. Schematic diagram of electrophoretic deposition cell.

2.3 surface characterization

The X-ray diffraction (XRD) technique was used to analyze the surface morphology of the deposited thin film, a field emission scanning electron microscope (FE-SEM), and energy dispersive X-ray (EDX). The X-ray diffraction XRD technique was applied, to determine the crystalline phase of the $BaTiO_3$ film. Furthermore, the XRD analysis facilitated the assessment of potential alterations in the microstructure resulting from the combining of the ceramic powder with the carbon steel. XRD was also used to assess the presence of any newly formed phases following the coating process. The XRD data were obtained using an X'Pert Pro Scanning Diffractmeter. The $BaTiO_3$ film was scanned from 10° to 90° in 2θ, with steps of 0.01° taken every 0.5 seconds. The surface morphology of the coatings and the elemental analysis of the deposited layer before and after corrosion were studied using an InspectTM F50 model of a field emission scanning electron microscope (FE-SEM)

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equipped with energy dispersive X-ray (EDX) technology.

2.4. Adhesion test

The interfacial adhesion between the coating and the carbon steel was evaluated under the ASTM-D3359 standard utilizing an adhesion tap kit (PA-2000, Paul N. Gardner Company Inc., Florida, USA) equipped with a standard blade (11-teeth with teeth spacing of 1 mm). Before subjecting the coating to the corrosive solution, the interface adhesion was assessed to investigate the coatings' long-term durability. The results of post-adhesion tests were obtained using a scanning electron microscope (SEM) model Zeiss LEO 1550, located in New York, USA. The SEM samples were affixed to the SEM holder using carbon tape and then coated with gold through sputtering for 120 seconds.

2.4 Electrochemical testing

The electrochemical measurements were carried out on a three-electrode cell using a CH Instruments Model 604E and controlled by computer software which was used for data acquisition and analysis under static conditions. The coated samples were utilized as the working electrode, while an Ag/AgCl (3.5M KCl) electrode served as the reference electrode, and a platinum plate functioned as the counter electrode. A surface area of 1 cm^2 is revealed. A potentiodynamic polarization method, open-circuit potential (EOCP)-time curves, and cyclic voltammetry curves (CV) were used to measure electrochemical corrosion. The tests were conducted in a solution containing 3.5 weight percent of NaCl at room temperature. Before conducting the tests, the working electrode was submerged in the solution for 1200 seconds to achieve a stable condition, as evidenced by the consistent open-circuit potential (EOCP). Subsequently, electrochemical measurements were carried out within a potential range of ±1.2V, using a scan rate of 0.1 V/s. The electrochemical corrosion parameters, such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), cathodic Tafel slope (βc), as well as anodic Tafel slope (βa), were automatically derived from the polarization curves. The corrosion rate is determined using the following equation [9]:

$$Corr.Rate = 0.00327\, I_{corr}\left(\frac{E_w}{DA}\right) \quad (1)$$

Where Corr. Rate is corrosion rate (mm/year), I_{corr} represents the corrosion current density measured in µA, while E_w is the equivalent weight, D represents the density measured in g/cm^3 and A is the electrode area (cm^2)

3. Results and discussion

3.1 Surface coating with nanopowders

The coatings were analyzed using X-ray diffraction (XRD) and the resulting diffraction spectra can be seen in Figure 3. The patterns show various diffraction peaks as a function of coating time, as indicated by the broad peaks that the material is $BaTiO_3$. Furthermore, the X-ray diffraction (XRD) patterns observed suggest that the nanoparticles of $BaTiO_3$ maintain their level of crystallinity. From Figure 3 the Iron structure is alpha iron is body-centered cubic according to 2θ values of 44.585, 64.980, and 82.297 deg. corresponding to (110), (200), and (211). $BaTiO_3$ exhibits a stereotypical behavior where it is tetragonal at room temperature, transitioning to a cubic structure above 120 ◦C, and an orthorhombic structure at lower temperatures, down to -90 ◦C. below -90 ◦C, it adopts a rhombohedral structure.

The cubic perovskite structure (a cubic Pm3m structure), was matched by the nano- $BaTiO_3$ samples, according to the XRD phase diffractgram (Figure 3). The panel shows the diffractogram so that the tetragonal line splits can be seen [10]. To enhance the visibility of the small-intensity peaks at higher angles, the intensity scales are reduced on the high-angle panel. Assuming a cubic Pm3m structure, the ideal cubic perovskite is a highly accurate fit for the XRD patterns obtained through the Pawley refinement method in the Bruker-TOPAS software [11]. This method is specifically designed for fitting line profiles and provides accurate measurements of peak positions and widths. It utilizes a model that takes into account the dependence of line width on line positions as well as the space group as its foundation. Despite the well-matched diffractograms, there were noticeable disparities between the calculated and experimental line profiles.

The commercially available nano-sized $BaTiO_3$ material claimed to be in the micrometer range, possesses a tetragonal crystal structure, thereby exhibiting a ferroelectric state. The coherence length of ferroelectrics is determined by the sizes of the ferroelectric domains. The local lattice distortion, quantified as "microstrain", was observed to be significant for the tetragonal phase of this powder. This relationship is likely to be anticipated, as the single tetragonal structure also closely resembles the gradient lattice strain proposed by the surface relaxation or the core-shell model [12].

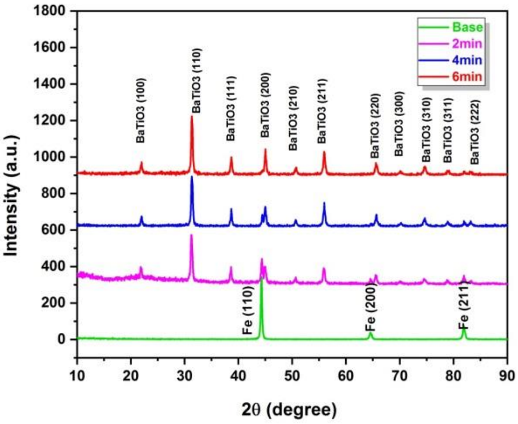

Figure 3. XRD patterns of barium titanate nanopowders deposited on the carbon steel samples

Typical top-view FeSem micrographs of $BaTiO_3$ nanoparticle coatings with various coatings times are displayed in Figure 4. The $BaTiO_3$ coating applied for 2 minutes exhibited a uniform and smooth appearance, devoid of any cracks (refer to Figure 2a). Nevertheless, the surface structure of the coatings underwent significant alterations as the coating time was extended to 4 minutes and 6 minutes. Aggregates were visible in the $BaTiO_3$ coating for samples that were coated for 4 minutes. The graph indicated that the number of aggregates increased as the coating time was extended to 6 minutes. This is because the materials have a high surface free energy, which is caused by the effects of a high surface area to volume ratio at nanometer length scales [13].

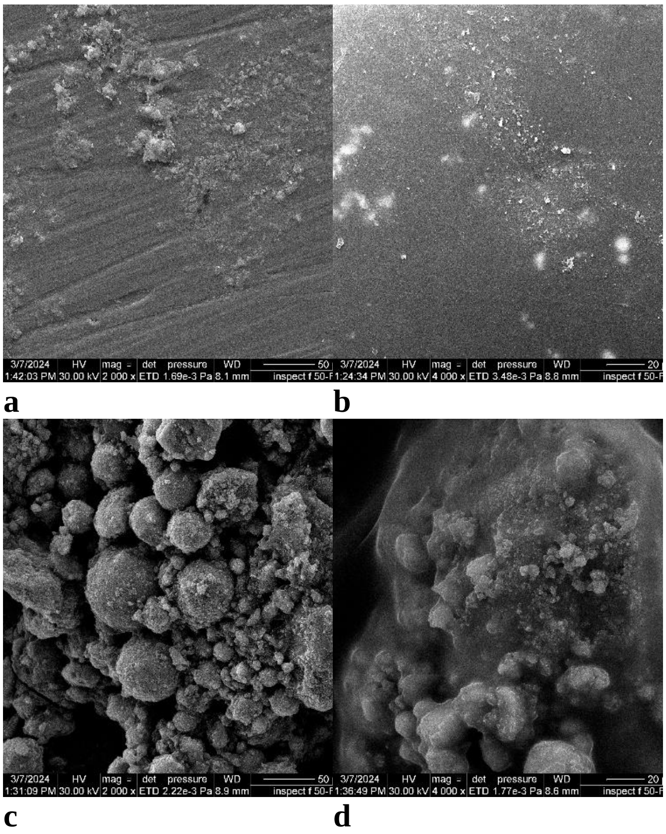

Figure 4. FeSem micrographs of carbon steel coatings (a) base (b) coating $BaTiO_3$ for (2 min) (c) coating $BaTiO_3$ for (4 min) (d) coating $BaTiO_3$ for (6 min)

3.2 Adhesion test

Prior to considering the coating as protective, it is crucial to evaluate the interface adhesion between the metal substrate and the coating. When the interface isn't properly adhered, it can cause the metal substrate and coating to become separated, creating spaces where corrosive substances can collect and accelerate the corrosion process. Thus, a high degree of interface adhesion is ideal in all cases. The carbon steel substrate's adhesion to the $BaTiO_3$ coatings was examined and evaluated using the standard test for adhesion tape, ASTM D3359. Before exposure to the corrosive solution, the test was conducted on $BaTiO_3$ coating samples. The adhesion test results are displayed in Figure 5 and Table 1, indicating that the coatings received a 2B and 3B rating for peelings, under the ASTM standard.

The state of the surface pretreatment before coating greatly affects how well the $BaTiO_3$ coating adheres to the carbon steel base. The $BaTiO_3$ coating layer was discovered to exhibit a high level of adhesion strength. The coated layer was discovered to possess a significant level of adhesion strength and was categorized based on ISO classification.

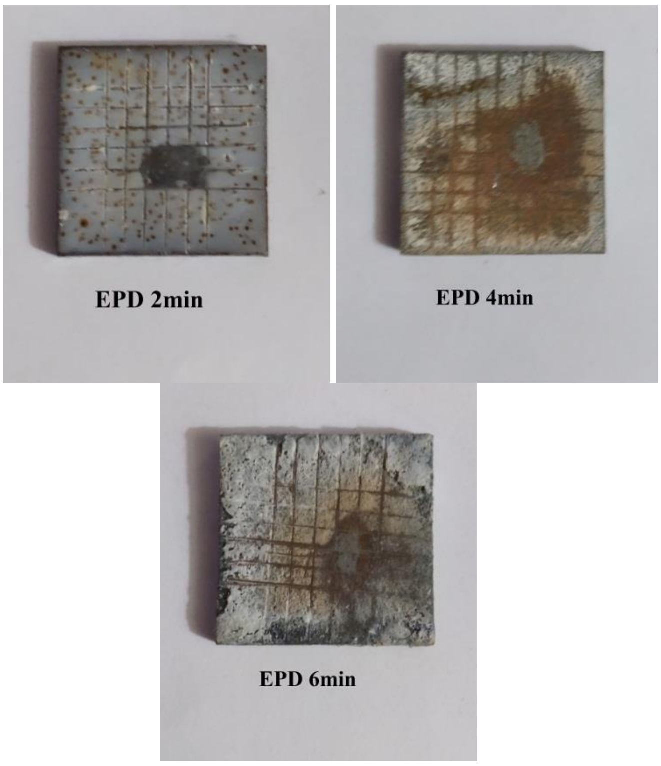

Figure 5. Images of post-adhesion test of (a) 2 min $BaTiO_3$ coating (b) 4 min $BaTiO_3$ coating (c) 6 min $BaTiO_3$ coating

Table 1: post-adhesion test of coated carbon steel samples

Sample	Classification	% Area Removed
EPD2min	2B	24%
EPD4min	3B	12%
EPD6min	3B	8%

3.3 Wettability

A barrier that effectively prevents oxygen and moisture from reaching the metal substrate is the principal mechanism by which $BaTiO_3$ nanopowder coatings on carbon steel protect against corrosion. Water contact angle measurements were used to evaluate the coatings' hydrophobic properties. As shown from the results all samples coated with nano-$BaTiO_3$ exhibited hydrophobic characteristics. It has the potential to significantly reduce the ability of the aggressive electrolyte to wet the surface, thereby restricting the absorption of water on the coating. Adding nanoparticles to coatings can make them less porous and better at blocking the movement of oxygen and water molecules, which slows the entry of harmful substances. Consequently, the obtained properties were deemed favorable, as indicated by the low corrosion rates and high impedance observed in the electrochemical measurements [13].

Table 2: Wettability test (contact angel)

Sample	Contact angle (degree)
EPD2min	0.0
EPD4min	0.0
EPD6min	38.195

3.4 OCP measurements

The corrosion resistance of the samples with a coating was assessed using the open-circuit potential (E_{OCP}) in 3.5wt% NaCl solutions. Figure 6 shows the plot of the E_{OCP} recorded for the coated and base samples as a function of immersion time. From initial values of approximately -0.60 V (Ag/AgCl) to values greater than -0.50 V (Ag/AgCl), the E_{OCP} values associated with the coated samples shifted substantially in the anodic direction; this tendency to shift to nobler potential was also noted as the coating time increased. Moreover, (E_{OCP}) values of electrodes coated with $BaTiO_3$ nanopowders for 2 minutes and 6 minutes exhibited a slower decrease compared to electrodes coated with $BaTiO_3$ nanopowders for 4 minutes when exposed to a corrosive environment for 20 minutes. Coatings with high E_{OCP} values generally have a high level of corrosion resistance [14]. Increasing the coating time greatly improved the barrier properties, as the increased E_{OCP} indicated that the $BaTiO_3$ nanopowders coatings were less porous and had a lower permeability against diffusing corrosive species [13, 15, 16]

Figure 6 displays how the E_{OCP} of carbon steel changes over time in a 3.5% NaCl solution after being coated with nano $BaTiO_3$ at different times. It is generally observed that as the coating time increases, the E_{OCP} becomes less negative. Specifically, in the original sample, (E_{OCP}) stabilizes at approximately -0.60 V (Ag/AgCl) after being immersed for 1200 seconds. By increasing the coating time, the (E_{OCP}) rapidly shifts towards a less negative value. This indicates that the coating has the ability to effectively control corrosion when immersed in the solution [17].

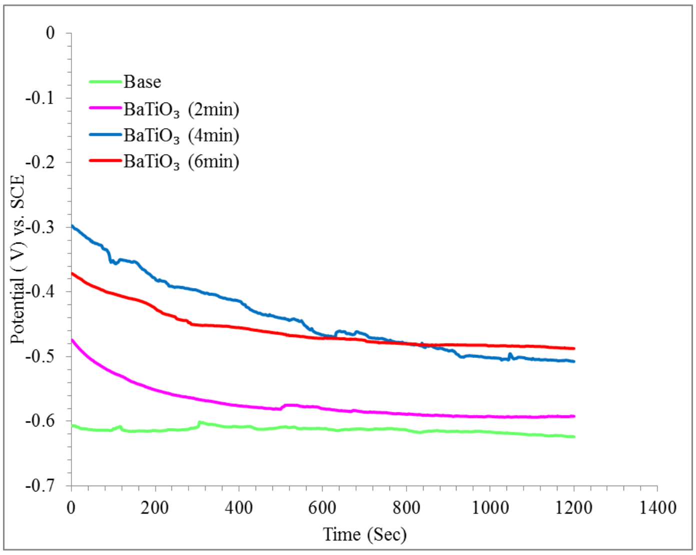

Figure 6. Open-circuit potential of carbon steel coating with nano $BaTiO_3$ in various time in 3.5 NaCl solution

3.5 Polarization curve measurements

Figure 7 shows the potentiodynamic polarization curves of carbon steel coating with nano $BaTiO_3$ for various deposition times in 3.5% NaCl solution. It has been observed that prolonging the deposition time raises the corrosion potential. Furthermore, the anodic curves exhibit a downward shift in current density. The impact of the duration of the coating process on the cathodic branches is not evident. Furthermore, when the coating time is 4 minutes, the electrode undergoes passivation, which is evident from the presence of passive curves in the anodic polarization branches. Clearly, prolonging the duration of the coating process effectively prevents the corrosion of carbon steel when exposed to a NaCl solution[17].

Figure 7 displayed the Tafel plots of uncoated carbon steel and coated samples with $BaTiO_3$ nanopowders for varying coating durations. It was observed that the application of the coating resulted in a significant decrease in both the anodic and cathodic current densities. The observed reductions in current indicate that the coatings effectively suppressed both the cathodic and anodic reactions. Furthermore, the anodic current of the sample with 2-minute duration decreased more significantly than the cathodic current when compared to the base sample. The disparity between the decrease in anodic and cathodic current can be easily observed, particularly when the coating time is 6 minutes. The results showed that longer coating time affected the anodic kinetics on the metal surface, and the $BaTiO_3$ coating acted as a protective layer, inhibiting anodic reactions and maintaining the metal in a more stable condition[13].

Table 3 displays the calculated values of different electrochemical corrosion parameters, such as I_{corr}, E_{corr}, CR, βa, and βc. Observations indicate that the corrosion current density and corrosion potentials decline as the coating time increases. Therefore, the application of a nano-$BaTiO_3$ coating has proven to be successful in preventing corrosion of carbon steel. Furthermore, the existence of anodic passive curves indicates that a protective film can be created on the surface of carbon steel to prevent corrosion.

Table 3 displays the values of Ecorr, Icorr, and corrosion rates (CR) for the samples. The Tafel extrapolation method can be used to determine the corrosion potential, corrosion current density, and corrosion rate of various samples based on the data provided in Figure 7. The Ecorr of the samples exhibited a positive increase as the coating time increased, ranging from approximately -0.574 V to -0.592 V (Ag/AgCl). The increase in Ecorr also indicated that the nanopowders of $BaTiO_3$ coatings reduced the anodic reactions on the carbon steel surface, thereby preventing the entire corrosion process in the system [18]. An initial decrease in the (CR) value was observed for the sample with a 2-minute coating, suggesting that the longer the coating time, the better the corrosion resistance of the sample. By extending the duration of the coating process to 4 minutes and 6 minutes, a significantly enhanced corrosion protection capability was achieved.

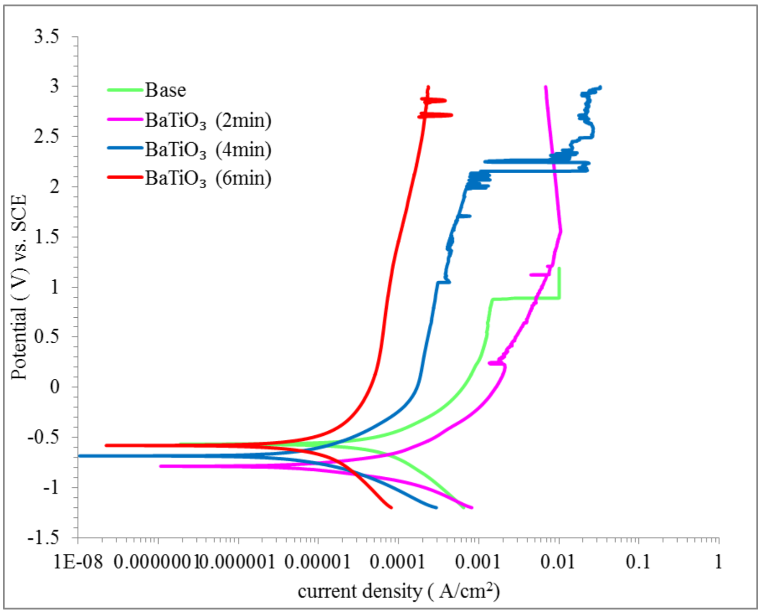

Figure 7. potentiodynamic polarization curves measured on carbon steel in 3.5% NaCl solution for various time of nano $BaTiO_3$ coating

Table 3: Electrochemical corrosion parameters for carbon steel and coated specimens immersed in a 0.5 M NaCl solution.

Sample	$E_{corr.}$ Volt	$I_{corr.}$ μA	CR mmpy	βa mV/dec	βc mV/dec
Base	-0.574	33.11	0.387	0.200	0.216
EPD2min	-0.775	20.49	0.239	0.116	0.133
EPD4min	-0.669	7.42	0.0868	0.188	0.172
EPD6min	-0.592	5.24	0.0613	0.227	0.210

The electrochemical process or corrosion reaction involves two half reactions, which are as follows:

$$Fe \longrightarrow Fe^{+2} + 2e^{-} \qquad \text{(anodic)} \qquad (2)$$

$$\frac{1}{2}O_2 + H_2O + 2e^{-} \longrightarrow 2OH^{-} \text{ cathodic)} \qquad (3)$$

The observed reactions are attributed to the ion diffusion process occurring through the pores of the $BaTiO_3$ coating on the metal surface. Based on the calculation mentioned above [Abdel Aal 2007], the $BaTiO_3$ film has a low porosity of 0.02. Consequently, the film exhibits lower conductivity or higher film resistance, measured at 1800 Ω [19]. Therefore, the creation of a titanate thin film with minimal porosity greatly enhances the carbon steel's ability to resist corrosion.

3.6. Potentiodynamic measurements

One typical approach to studying the electrochemical properties of metal coatings and substrates is cyclic voltammetry. The research used cyclic voltammetry tests on coated and uncoated carbon steel substrates using a three-electrode configuration.

Electrochemical response of the nanocoating on the carbon steel surface was determined using Cyclic Voltammetry analysis. Based on the observed data, it is evident that both the anodic and cathodic reactions are enhanced in the carbon steel curve. This suggests that $BaTiO_3$ nanopowders are sufficiently stable and can protect carbon steel under these conditions, preventing corrosion caused by current flow. The application of a nanocoating on $BaTiO_3$ demonstrates enhanced resistance to corrosion. The results demonstrate that the nanocoating exhibits superior corrosion resistance due to its excellent coverage[20, 21].

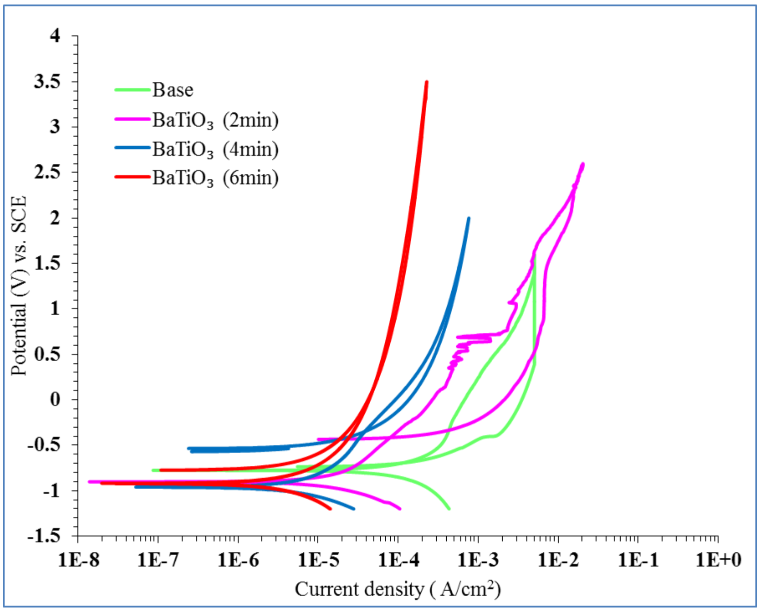

Figure 8. Cyclic Voltammetry curves measured on carbon steel in 3.5% NaCl solution for various deposition time of nano BaTiO$_3$ coating

3.7 FeSem characterization after corrosion

The FeSem surface morphology of carbon steel in the absence and presence of nano-BaTiO$_3$ coating is depicted in Figure 9 before and after immersion in a 3.5 weight percent NaCl solution. Without the application of a nano-BaTiO$_3$ coating, carbon steel experiences noticeable corrosion, resulting in a rough and uneven surface, as depicted in Figure 9(a). Applying the nano-BaTiO$_3$ coating results in a smoother surface on the specimen compared to the base specimen. However, there are still small spikes present along the mechanical scratches that were created during specimen preparation, as shown in Figure 3(b-d). The homogeneous film has the ability to shield the carbon steel from corrosion damage.

4. Conclusions

Electrophoretic deposition technique is successfully applied to coated carbon steel by nanopowder BaTiO$_3$. The electrophoretic coating process utilizes untreated BaTiO$_3$ nanoparticles powder. The deposition of a BaTiO$_3$ porous film layer onto a carbon steel substrate is achieved by utilizing the powder that is suspended in ethanol. A coating process with a duration of 6 minutes yields encouraging outcomes. The corrosion protection of BaTiO$_3$ coatings with varying coating times was assessed using electrochemical measurements. The study revealed that the application of BaTiO$_3$ coating significantly enhanced the carbon steel's ability to resist corrosion in 3.5 wt% NaCl solutions. The effectiveness of incorporating the respective components was confirmed through XRD and elemental analysis. The thin films of BaTiO$_3$ demonstrated strong adhesion to the substrate, minimal porosity, and therefore, exceptional resistance to corrosion.

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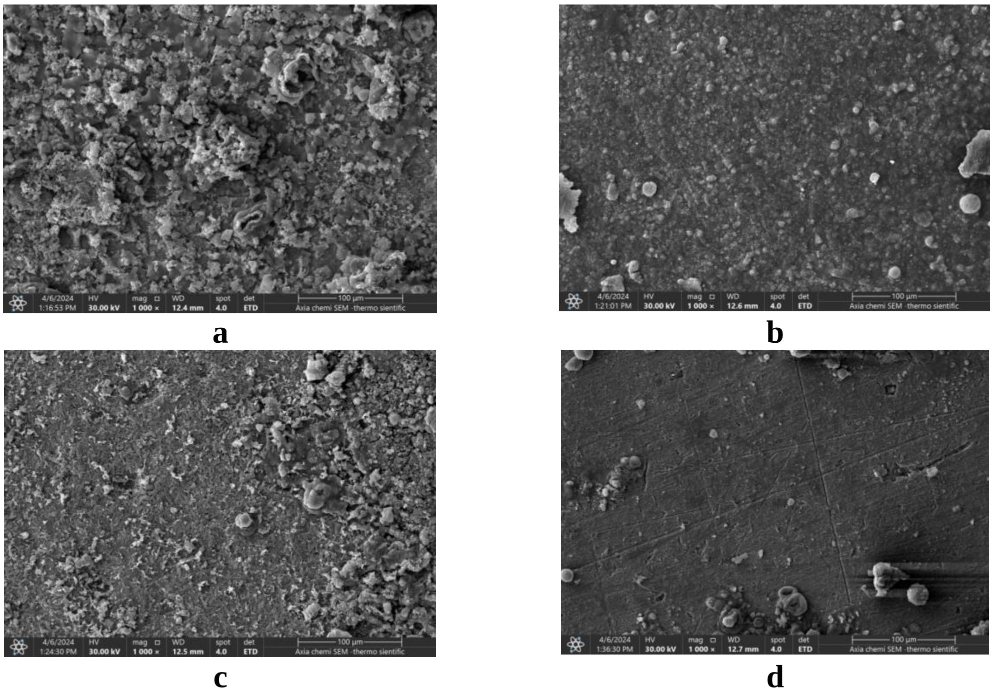

a **b** **c** **d**

Figure 9. FeSem images of carbon steel after immersion in 3.5% NaCl solution (a) base (b) coating $BaTiO_3$ for (2 min) (c) coating $BaTiO_3$ for (4 min) (d) coating $BaTiO_3$ for (6 min)

Conflict of interest Disclosure There are no conflicts of interest regarding the publication of this manuscript.

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