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RESEARCH ARTICLE

Chronopotentiometric Deposition of Polypyrrole on Mild Steel in Tartrate Medium for Corrosion Prevention

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Abstract

This work discusses the polypyrrole (PPy) deposition on mild steel (MS) from 0.4 M pyrrole in 0.1 M sodium potassium tartrate (Na-K tartrate) by chronopotentiometrically and the corrosion-resistant behavior in acid media. The current densities of 1, 2, and 3 $mAcm^{-2}$ were used for the electrodeposition of PPy on MS. When current density 2 mA cm^{-2} was applied, no induction period associated with metal dissolution was observed. This behavior showed that very little dissolution of the MS occurs as the current density applied is ≥2 mA cm^{-2}. Thus, the obtained polypyrrole (PPy) coated MS surface was examined by optical and scanning electron microscopy (SEM) which showed uniform, compact and adherent PPy coatings. The elemental composition of the PPy coating film was established by energy-dispersive x-ray (EDX) analysis. The corrosion performance was investigated by using potentiodynamic polarization (PDP) in 0.1 N H_2SO_4. It revealed that PPy coating considerably reduced corrosion current density, with an inhibition efficiency of 90.44%. The coating also behaved as a mixed inhibitor.

Keywords: Electropolymerization; Chronopotentiometry; Mild Steel; Polypyrrole; Tartarate.

Introduction

The use of metals is one of the indicators of the development of the nation. Metals are essential for modern society because of their usage in infrastructure, machinery, buildings, and automobiles [1,2]. Predominantly, iron, mild steel, copper, aluminium called "major metals," have been used by humans for a very long time, fulfilling fundamental human requirements. Nevertheless, a number of environmental issues, including biodiversity loss, climate change, the health effects of particulate matter, and corrosion are being raised [1,3]. In order to minimize future effects on our society and to comply with climate and other environmental targets it is necessary to investigate the complex and uncertain future developments of metal supply and the environmental impacts associated with them. Mild steel is one of the extremely demanded metals [1,4] due to its outstanding mechanical characteristics, which are influenced by the microstructure and composition of the material. However, it undergoes severe corrosion leading to huge material loss and economy. Galvanization, use of inhibitors, polymer coating, etc are used to prevent corrosion of MS. The conducting polymer coating is one of the surface coating techniques which is the most competent corrosion inhibition method for steel.

Polyaniline and polypyrrole are the most investigated conducting polymers for the prevention of corrosion. In comparison to many other conducting polymers, polypyrrole (PPy) is one of the most studied polymers owing to its outstanding water solubility and low oxidation potential. It is also easy to prepare, has good environmental stability, and has a higher conductivity [5].

Polypyrrole (PPy) is synthesized using aqueous polymerization without using harmful chemicals, and could also be a viable choice [6, 7]. Electrochemical deposition of PPy is the most effective method for coatings due to its limited processability. Coating thickness and shape are also easily controlled by varying potential or current density. It is also a cost-effective process, and it can be accomplished by various electrochemical techniques like potentiostatic, galvanostatic, and cyclic voltammetry [8]. Despite these benefits, because of the high oxidation potential of the monomer, one of the most problematic elements of electrochemical polymerization of pyrrole is the risk of metal dissolution prior to coating development. So Polypyrrole coatings are only fashioned on MS when electrochemical conditions allow the metals to passivate without blocking pyrrole electropolymerization. Only a few electrochemical systems have been known to be capable of fulfilling this requirement [9]. Troch-Nagels *et al.*, used Na_2SO_4 solution as a supporting electrolyte. The conductive PPy coatings ensured the substrate's corrosion resistance but were brittle and had poor adherence to the substrate [10,11]. Schirmeisen and Beck confirmed the fact that polypyrrole films can be deposited on Pt, Au, Ti, and V2A but not on iron in an aqueous medium containing several anions such as BF_4^-, ClO_4^-, HSO_4^-, Tos^-, HCO_3^-, H_2PO_4-, HPO_4^{2-}, and $H_2BO_3^-$, but discovered that well-adhering polypyrrole layers are obtained when NO_3^- ions are added to the aqueous medium [12]. Beck *et al.,* electropolymerized pyrrole on iron in an oxalic acid [12], having very poor adhesion. A manganese oxide layer was employed on the previously formed working surface. to improve PPy adhesion. Successful outcomes were obtained by Bruyne et al. by lowering the pH to 1.4 [13,14]. The work of Ferreira's *et al.,* showed that a 10% aqueous nitric acid treatment inhibits iron dissolution while enabling pyrrole oxidation to proceed. In various aqueous mediums including Na_2SO_4, $K_2C_2O_4$, or KNO_3 extremely adherent and thick PPy films were formed at constant current condition [15,16]. Su and Iroh [17] found that the adherence of PPy to the substrate increases when the pH of oxalic acid decreases. The polymer films made from extremely acidic solutions are coarse and turbid, alkaline solutions can be used to make thin films [18]. Both aqueous and non-aqueous solutions containing the monomer and a suitable electrolyte serving as dopant or counter-ion (including organic and inorganic ions) such as, oxalate , perchlorate, naphthalenesulfonate, p-toluene sulfonate (pTS), chloride, tetrabutylammonium tetra-floroborate (TBAFB), sulfate, styrenesulfonate (SS) and polystyrenesulfonate (PSS) are found to be able to prepare Polypyrrole [6,19].

Conducting polymer coatings has generated significant interest due to their efficiency in safeguarding metal surfaces. The corrosion resistance of these coatings was tested by synthesizing them on aluminum, iron, and alloy surfaces [6]. Several studies have been conducted on the ability of these polymer coatings to protect oxidizable metals from corrosion [20, 21]. Herrasti *et al.*, demonstrated that Polypyrrole coatings can protect stainless steel from corrosion by shifting the corrosion potential to more positive values and reducing the corrosion current [22]. The type of dopants used in the PPy coating had a significant impact on its capacity to protect the metal surface against chloride ion attack [8]. Long-term immersion studies show that phosphoric acid-doped PPy coatings provide stronger and more persistent corrosion resistance than PPy coatings doped with sulphuric and oxalic acids [23, 24].

Polypyrrole (PPy) coatings doped with oxalic acid were electrodeposited by cyclic voltammetry technique to improve the corrosion protection of a copper current collector [24]. Using varying amounts of monomer, oxalic acid (OXA), and dodecylbenzene sulfonic acid (DBSA) solutions, polypyrrole (PPy) coatings were produced on the surface of copper electrodes using an electrochemical polymerization process. While all of the coatings offered corrosion protection for the copper electrode, the coating with 0.1 M monomer, 0.1 M OXA, and 0.1 M DBSA concentration provided the best protection [25].

In this regard, polypyrrole coating was electrodeposited on mild steel using Na- K tartrate solvent by galvanostatic polarization technique. The corrosion protection behaviours of the obtained coating has been tested in 0.1 N H_2SO_4.

Materials and Methods

Materials

Commercial-grade mild steel MS, which is sold in Nepal's local market, was selected and cut into 3 cm x 3 cm x 0.15 cm pieces. A succession of emery papers ranging in grade from #100 to #2000 was then used to abrade the samples until the working surface became smooth. The abraded samples were washed with hexane and degreased in ethanol. Then samples were subjected to air drying and stored in a desiccator prior to each measurement. Pyrrole (C_4H_4NH), ethanol (C_2H_5OH), Sodium potassium Tartrate ($KNaC_4H_4KO_6.4H_2O$) Acetone (C_3H_6O), Hexane (C_6H_{14}) were acquired from Merck, India. Double-distilled water was used to prepare the solutions of the needed concentration and all experimental procedures. 0.1 M H_2SO_4 solution was also prepared in double-distilled water.

Electrochemical Deposition of Polypyrrole (PPy)

A Hokuto Denko HA-151 potentiostat consisting of three-electrode system was used for the electrodeposition of polypyrrole (PPy) and potentiodynamic polarization. The potentiostat was operated by self-made lab VIEW software and connected to an IBM computer [27]. Among the three, a saturated calomel electrode (SCE) was employed as the reference electrode, a graphite rod as the counter electrode, and an MS sample as the working electrode. The PPy was chronopotentiometrically deposited with the application of an anodic current to the MS sample immersed in a solution of 0.4 Pyrrole containing 0.1 M sodium potassium Tartrate (Na-K tartrate). The current density ranged from 1 mA cm^{-2} to 3 mA cm^{-2} and was applied for the deposition of PPY on the MS. The PPy-coated MS was placed in a desiccator for further study after being rinsed with distilled water and air-dried.

Surface Characterization

The polypyrrole (PPy) coated MS surface was examined by both optical microscopy and scanning electron microscopy (SEM). The optical image was taken using a light polarizing microscope (Radical Scientific, India) in reflection mode with a USB Procam attached. Surface morphology was investigated by SEM (JEM-1200EX electron microscope, Tokyo, Japan). The elemental composition of the PPy coating film was established by energy-dispersive x-ray (EDX) analysis.

Corrosion Study by Potentiodynamic Polarization (PDP)

The effectiveness of the PPy coating in preventing corrosion in a 0.1 M H_2SO_4 solution was investigated using potentiodynamic polarization (PDP). The potential was scanned at a rate of 1 mv/s from the cathodic to the anodic direction, with the polarization potential limit set at ± 300 mV vs open circuit potential (OCP). Before carrying out polarization, the OCP was recorded for thirty minutes.

The corrosion inhibition efficiency (IE) was obtained by the following equation (1).

$$\text{Inhibition Efficiency (IE)} = \frac{icorr - i'corr}{icorr} \times 100\ \%..\ (1)$$

Where i_{corr} is the corrosion current density of MS and i'_{corr} is the corrosion current density of PPy-coated MS. Using Tafel extrapolation, corrosion current density, and corrosion potential were obtained.

Results and Discussion

The galvanostatic deposition of PPy onto MS was achieved at various current densities. **Fig. 1** represents the chronopotentiometric performance of MS immersed in the solution of 0.4 pyrrole containing 0.1 Na-K tartrate at a current density of 1, 2, and 3 mA cm^{-2} respectively. When the applied current density was 1 mA cm^{-2} during the polarization process, the potential rapidly increased from -0.744 V to -0.255 V and was almost steady for 332 seconds. This is the time for incubation similar to the deposition of PPy and polyaniline (PANI) in oxalic acid [27–29]. Then, from 332 sec to 359 sec, it rose from -0.000183 V to 1.82 V. The potential then stayed almost constant at 1.81 V for 428 seconds. However, when the current density was 2 mA cm^{-2} or more, no induction period associated with metal dissociation was observed. This behavior showed that very little dissolution of the MS occurred as current was applied in this current density range.

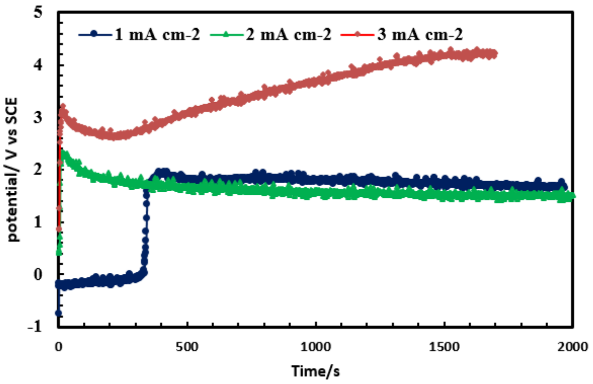


Fig. 1: Chronopotentiometric deposition of PPy on MS in 0.4 M pyrrole + 0.1 Na –K tartrate

Depending on the applied current density, **Fig. 1** illustrates that the MS dissolves anodically with time while the potential remains at roughly -0.226 V during the initial phases of polarization. Before the potential increases, the charge (Q) flowing through the electrode is determined by the relation (2)

Q= It ....(2)

I represents the current density in mA cm^{-2} and t represents the time in seconds. The chronopotentiogram shows the oxidation of pyrrole needs a 502 mC charge. But in one second, the potential was 0.407 V at a current density of 2 mA cm^{-2}. After four seconds, the potential reached 2.13 V. This occurred as a result of the MS surface passivating due to an increase in current density [29]. Following a slight decrease in potential brought on by passivation, the continuous formation of PPy and surface coverage result in a constant potential. The voltage then dropped a little to 1.62 and then stayed steady. In the case of a current density of 3 mA cm^{-2}, the potential was 0.8566 v on 1 Sec. When a nearly constant voltage is reached, polymerization on the outside surface is invisible. The potential marginally increases as the polarization advances, revealing evidence of exogenous polymerization. Ultimately, the visible growth of PPy on the outer surface and the resulting thickness cause the potential to stable for a longer amount of time. These results are similar to those that have been documented for the electrodeposition of pyrrole onto porous silicon. Firstly, iron dissolves as indicated by the initial potential value.

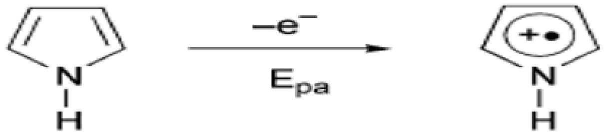
$Fe \rightarrow Fe^{2+} + 2e$ ……………………… (3)

The dissolved Fe^{2+} produces an insoluble Fe-tartrate and precipitates at the MS surface in the presence of tartarate ion in the solution, producing a passivation layer.

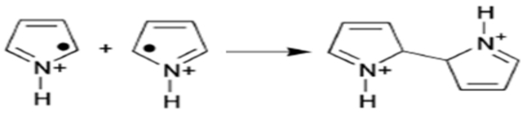
$Fe^{2+} + C_4H_4O_6^{--} \rightarrow FeC_4H_4O_6 \downarrow$ …… (4)

When the active dissolution of the metal is halted, the available charge is used for the oxidation of pyrrole, which results in polymerization and the creation of radical cations. The dissolution, passivation, oxidation, and polymerization mechanisms are described as:

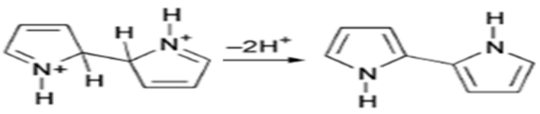
Oxidation of monomer: The first stage of the reaction is the electrode oxidation of monomer molecules yielding radical cations with the radical state delocalized over the pyrrole ring.



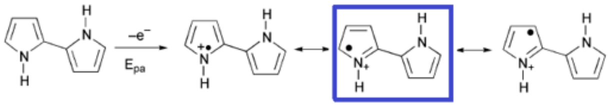
Coupling of radicals: The radical cations dimerize



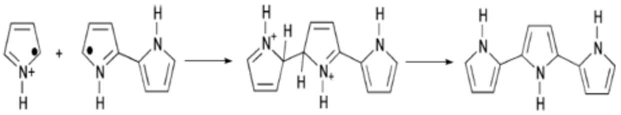
Stabilization: Loss of proton to form a dimer



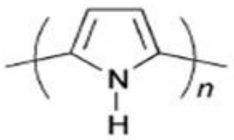
Oxidation of Dimer: The dimeric molecule can experience subsequent oxidation,



Formation of trimer: The chain growth proceeds by the addition of a newly formed radical cation to an oligomeric one.



Final product: Polypyrrole polymer



Morphology of PPy coating

The shape of PPy coating is substantially influenced by current density. **Fig. 2** displays the optical picture of the MS surface coated with PPy at different current densities. The optical image shows a homogenous, spherical, and compact PPy coating at a current density of 2 mA cm^{-2}. It is revealed that the ppy coating is composed of larger particles as the current density rises.

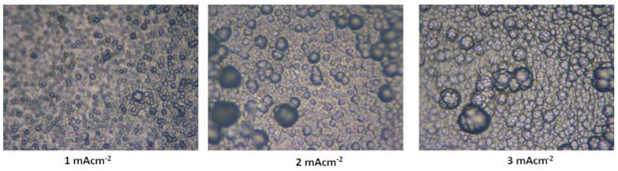
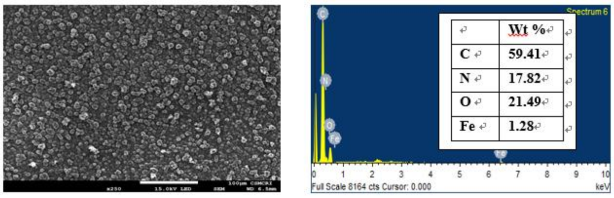

1 $mAcm^{-2}$ 2 $mAcm^{-2}$ 3 $mAcm^{-2}$

Fig. 2: Optical images of PPy deposited on MS chronopotentiometrically at various current densities.

Fig. 3 shows the SEM image of PPy coated MS on chronopotentiometric deposition at 2 mA cm^{-2} current density. It shows uniform globular type spheroidal morphology and compact PPy coating. The PPy coating looks like the structure of cauliflower [5,19,30]. The EDX confirms the formation of PPy with identifying the presence of C, N and O elements.



	Wt %
C	59.41
N	17.82
O	21.49
Fe	1.28

Fig. 3: SEM and EDX Spectrum of PPy coating at 2 mA cm^{-2} current density.

Corrosion Performance of the PPy coating

Fig. 4 shows the corrosion performance of PPy coated MS in 0.1 M H_2SO_4. The corrosion behavior of PPy was examined by potentiodynamic polarization (PDP). The open circuit potential (OCP) was recorded for 30 minutes and it was found to be -0.540 V for PPy coated MS which was slightly negative than MS.

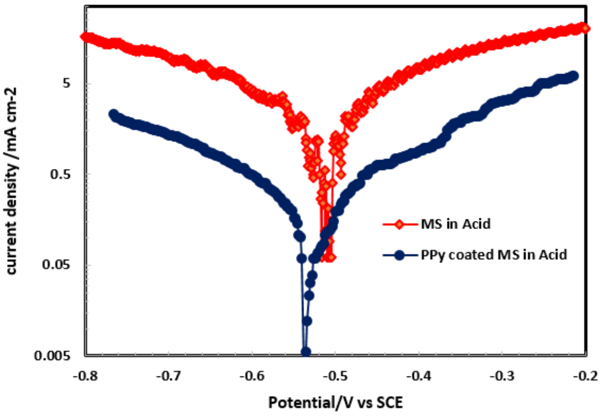


Fig. 4: Potentiodynamic behavior of MS and PPy coated in 0.1 M H_2SO_4.

After applying a current density of 2 mA cm^{-2}, the corrosion inhibition efficiency (IE), corrosion potential (E_{corr}), and corrosion current density (i_{corr}) of PPy-covered MS electrodeposited by galvanostatic polarization are displayed in Table 1. It demonstrates that PPy coating considerably reduced corrosion current density, with an inhibition efficiency percentage of 90.44% **(Table 1)**. It reflected the diminishing of the hydrogen reduction, as indicated by the suppression of the cathodic current without altering the reaction process [31,32].The corrosion potential (Ecorr) of PPy coating moved somewhat in the negative direction than bare MS in acid solution. The fact is that the PPy coating shifts by less than 85 mv revealing a mixed-type inhibitor [28, 33].

Table 1: Corrosion Behavior of PPy coated MS

Sample	**β_a (V/decade)**	**β_c (V/decade)**	**i_{corr} (mA cm^{-2})**	**E_{corr} (V)**	**Inhibition Efficiency (%)**
MS	0.217	-0.283	2.24	-0.522	-
PPy coated MS	0.199	-0.183	0.214	-0.516	90.44

Conclusions

Polppyrrole (PPy) was chrono-potentiometrically deposited onto the MS using a 0.4 M pyrrole containing 0.1 M tartrate solution. When current density 2 mA cm^{-2} was applied, the PPy coating was adherent, uniform and compact. Similarly, there was very little dissolution of the MS occurrs on the application of 2 mA cm^{-2} current density. The corrosion performance of PPy was studied by using potentiodynamic polarization (PDP) in 0.1 N H_2SO_4 which revealed that PPy coating serves as excellent corrosion prevention in acidic medium. The inhibition efficiency was found to be approximately 90.0 %. The open circuit potential (OCP) and corrosion potential (E_{corr}) reveal that PPy coating behaved as mixed inhibitor.

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Author's Contribution Statement

Sanjay Singh: Writing – original draft, Investigation, Formal analysis, Data curation. **Nabin Karki:** Writing – review & editing, Project administration. **Dipak Kumar Gupta:** Writing – review & editing, Supervision, Formal analysis. **Amar Prasad Yadav:** Writing – review & editing, Supervision, Conceptualization.

Conflict of Interest

The authors do not have any conflict of interest throughout this research work.

Data Availability Statement

The data supporting this study's findings are available from the corresponding authors upon reasonable request.

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