



ORIGINAL ARTICLE

Effect of ginger, pomegranate and celery extracts on zinc electrodeposition, surface morphology and corrosion inhibition of mild steel



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Received 29 January 2020; revised 17 February 2020; accepted 11 March 2020
 Available online 31 March 2020

KEYWORDS

Electrodeposition;
 Zinc;
 Mild steel;
 Corrosion

Abstract Electrodeposition of Zn on mild steel (MS) within an acid bath consisting of admixed $ZnCl_2$, H_3BO_3 and KCl solution at specific concentrations of ginger (*Zingiber officinale*), celery (*Apium graveolens*) and pomegranate (*Punica granatum*) extracts, and plating time was studied. The surface of electrodeposited MS was analyzed with scanning electron microscope (SEM). Energy dispersive spectroscopy (EDS) was employed for elemental analysis. Results show the presence and increase in extract concentration is associated with the amount of Zn electrodeposited on MS. Ginger extracts induced the highest weight gain with values between 0.0867 g and 0.1219 g. Morphology studies showed good electrodeposition of Zn on MS, with closely packed Zn particles on the surface with respect to additive concentrations and plating time. EDS results showed pomegranate induced the highest %wt. content of Zn on MS. Corrosion resistance of the unplated and Zn electrodeposited MS with and without extract additives were evaluated with potentiodynamic polarization method. Results showed the presence of ginger, pomegranate and celery extracts improved the corrosion protection of Zn electrodeposited MS with optimal corrosion rates of 2.48 mm/y, 2.78 mm/y and 1.95 mm/y compared to 7.96 mm/y for unplated MS and 6.58 mm/y for Zn electrodeposited MS without additives.

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1. Introduction

Corrosion is the chemical and electrochemical deterioration of the metallurgical, mechanical and physical properties of metallic and non-metallic materials due to molecular interaction with ionic species within their environment of operation

[1,2]. The adverse effect of corrosion is prevalent on steels used for structural applications due to their gradual degradation in service such as in electric towers, bridges and highways, petrochemical, chemical processing, automobile and manufacturing plants etc. endangering the safety of personnel and operators [3]. Steels are produced from iron ores with the application of huge amount of energy to extract them from their ores. The higher energy state of the extracted metals results in gradual release of energy and return of the metal alloy to their natural state at lower energy [4]. The consequences of corrosion such as total shutdown of power plants, refineries, chemical

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 Peer review under responsibility of Faculty of Engineering, Alexandria University.

processing plants etc. can be detrimental leading to serious production and economic problems [5]. Corrosion products can also endanger safety due to rapid wear of metallic parts, leading to contamination of food products and chemicals, fire and explosion hazard from leaking containers and fuel tanks etc. [3,6]. Mild steel has exceptional mechanical properties; it is relatively cheap, recyclable and easily fabricated which gives it versatile application in nearly all industries [7]. However, mild steel has very weak corrosion resistance even in industrial environments where the presence of corrosive anions can be damaging. Electrodeposition is used to reduce corrosion on metallic materials in situ by the passage of an electric current onto a conductive material placed within a solution consisting of a salt of the protective metal to be deposited on the steel [8]. The basic concept of electrodeposition is to separate the steel from the corrosive environment by affixing a layer of a highly-resistive metal – usually zinc [9,10].

Coatings are a layer of electrically padding materials over metallic surfaces to be protected with extensive application in underground pipelines [11]. Coatings technology is used in industries such as in shipping, automotive, building, petrochemical pipelines etc. due to the continual failure of materials in astringent conditions [12]. Deposition through electroplating is performed to obtain a finer material surface. Electrodeposition of zinc on mild steel is a widely accepted method of corrosion protection [13]. Zinc coating exhibits strong corrosion resistance properties by forming a protective layer over the steel when in contact with air [14]. Zinc coatings can also have their functional and protective properties enhanced which is an improvement on the galvanizing problems of other methods [15]. The usage of electroplated zinc coatings as a protective coating for many products has led to the development of zinc alloys. Zinc can be coated from cyanide, non-cyanide alkaline or acid solutions. Factors which affect good deposition include: the nature of the bath constituents, surface of the substrate being coated and plating time [16]. The electrodeposition of zinc from ionic liquids has been the subject of interest over the years because use of ionic liquids in zinc electrodeposition is environmentally friendly compared to the traditional aqueous solutions which are corrosive and very toxic [17].

A lot of developments have been achieved in the use of ionic liquids in electrodeposition. However, a major limitation is the application of brighteners. Additives can be used to improve the deposit morphology and physical characteristics by affecting the mechanism in which zinc is electrodeposited [18]. Application of organic additives in zinc electrodeposition improves the operating performance by decreasing power consumption as well as increasing current efficiency [19]. They also affect the zinc deposition and the process of crystal building. Another major role of organic additives is in control of acid mist over the electrolytic cell. However, organic additives are readily decomposed thus the need for sustainable and environmentally sustainable additives [20]. Organic additives significantly influence the growth of deposits, deposit structure and deposit glossiness. Application of additives also influence deposit brightening, diminishing stress, enhances current density range and decreases pitting [21]. Use of organic compounds extends beyond electrodeposition to application as corrosion inhibitors though their toxic nature limits their versatility [22,23]. Compounds of natural origin are non-toxic, readily available, environmentally friendly, cheap and

biodegradable [24]. Research has documented the use of plant extracts in the electroplating of zinc on metals. However, application of plant extracts in the electroplating of zinc on metals and their alloys is still evolving [25]. Electrodeposition and disposal of liquid wastes from cyanide-based baths are considered to be very toxic and costly. The aesthetic value and corrosion resistance of the electroplated mild steel can be further improved upon with considerable economic and technological benefit [26]. This research aims to study the effect of natural extracts of ginger (*zingiber officinale*), celery (*apium graveolens*) and pomegranate (*punica granatum*) on the zinc electrodeposition on mild steel at specific concentrations and plating times.

2. Experimental methods

2.1. Materials preparation

Mild steel (MS) plate used in this research was purchased from a vendor in Lagos, Nigeria. The steel was cut with hacksaw to final dimensions of 100 mm × 10 mm × 1.5 mm (length, breadth and thickness). The nominal (%) weight composition of MS is shown in Table 1. Morphological characterization and analysis was carried out with scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). Ginger, celery and pomegranate fruits containing the required plant extract solution for the research were purchased from the open market in Lagos, Nigeria. The extracts were obtained by separately cutting the fruits into small pieces, mashing them and subsequently sieving out the solid particles. The extracts solution was stored without the use of preservatives and subsequently used in the experiment before the onset of fermentation.

2.2. Electrodeposition metal properties and electrodeposition bath

Zn was obtained in powdery form with density of 7.14 g/cm³, melting point value of 419 °C and boiling point of 907 °C. Zn is characterized by its excellent resistance to many corrosive environments. Zn powder is mainly used as a major material in high performance coating. The Zn plate was cut to dimensions of 100 mm × 10 mm × 0.1 mm. The electrodeposition bath consists of ZnCl₂, H₃BO₃ and KCl. H₃BO₃ (obtained from Sigma-Aldrich, USA) which functioned as the buffer in the electroplating solution maintaining the pH value of the solution. KCl supplies the chloride content which is a primary contributor to the conductivity of the solution. ZnCl₂ was used to maintain the Zn content of the bath by the dissolution of Zn anodes which substitute the ZnCl₂ in the solution. The acid chloride solution was obtained by dissolving 71 g of anhydrous ZnCl₂, 207 g of crystalline KCl and 35 g of H₃BO₃ in 1 L of deionized H₂O. The solution was passed through a filter paper to remove impurities. Plant extract additives were then added to the acid chloride in volumetric concentrations shown in Table 2 to form the electroplating solution.

2.3. Electroplating Set-Up

The variable DC supply was set to 13 V and 0.08 A. 50 ml of the plating solution was poured into the beaker. The electrode-

Table 1 Elemental content (wt. %) of MS.

Element	C	Si	Mn	P	S	Cu	Ni	Al	Fe
Composition	0.40%	0.17%	0.44%	0.01%	0.01%	0.08%	0.01%	0.03%	Balance

Table 2 Concentration of plant extracts in bath solution.

Plant Extract Additives	Volumetric Concentration (L/L)
Ginger	0.08
	0.12
	0.16
	0.08
Celery	0.12
	0.16
	0.08
	0.12
Pomegranate	0.16

position process was carried out in the baths with different additives while varying the plating time. During the plating process, the bathing solution was placed in a 250 ml beaker and the beaker was covered using a perspex cover. MS was dipped in the acid chloride solution and made the cathode by connecting it to the negative terminal of the DC supply. The anode was also placed in the bath solution and connected to the positive terminal of the DC supply. The anode is a 99.99% pure Zn rod. The plating time was varied between 15 min and 18 min. Thereafter the samples were cleansed in deionized H₂O to rinse the acid solution off the samples plated immediately after the plating process. The samples were rinsed for 5 s and eventually air dried.

2.4. Characterization of electroplated samples

Comparative analysis of the electroplated and non-electroplated MS was done through SEM analysis. SEM was used to study morphology of the surface microstructure, electroplated coatings, grain size, surface defects and inclusions, and nature of grain deposits.

2.5. Electrochemical analysis

Electrochemical analysis of electroplated MS was performed at 30 °C ambient temperature. A triple electrode cell consisting of Pt rod counter electrode, Ag/AgCl reference electrode with 3 M KCl electrolyte at pH of 6.5 and MS working electrodes were placed inside a cell containing 200 ml of 0.5 M H₂SO₄ solution. The electrode configuration was interfaced with Digi-Ivy 2311 potentiostat and computer. MS working electrode was encased in hardened resin metallographic mounts. Polarization curves were plotted at scan rate of 0.0015 V/s between potentials of -1.5 V to +1.5 V. Corrosion current density J_{cr} (A/cm²) and corrosion potential E_{cr} (V) values were obtained Tafel extrapolation. Corrosion current I_{cr} (A) was obtained from the intercept between the anodic-cathodic polarization plots. Corrosion rate, C_R (mm/y) was calculated as follows;

$$C_R = \frac{0.00327 \times J_{cr} \times E_v}{D} \quad (1)$$

E_v represents the equivalent weight (g) of MS, 0.00327 is a constant, and D represents the density (g/cm³). Inhibition efficiency η (%) was calculated from the equation below;

$$\eta_2 = \left[1 - \left(\frac{C_{R2}}{C_{R1}} \right) \right] \times 100 \quad (2)$$

C_{R2}/C_{R1} is the ration of corrosion rate of MS with and without the plant extracts in the acid solution.

3. Results and discussion

3.1. Electrodeposition results

Tables 3–6 shows the electroplating parameters and weight gained for Zn electrodeposition on MS with respect to plating time (15 min and 18 min) and extract concentration (0.08 L, 0.12 L and 0.16 L). Electroplating parameters for Zn electrode-

Table 3 Electrodeposition parameters without plant extracts.

Sample Identification	Extract Concentration (l/l)	Time (min)	Weight Gained (g)
NA-15	–	15	0.0558
NA-18	–	18	0.0621

Table 4 Electrodeposition parameters with celery extracts.

Sample Identification	Extract Concentration (l/l)	Time (min)	Weight Gained (g)
C-4-15	0.08	15	0.0763
C-4-18	0.08	18	0.0883
C-6-15	0.12	15	0.0995
C-6-18	0.12	18	0.1011
C-8-15	0.16	15	0.1041
C-8-18	0.16	18	0.1152

Table 5 Electrodeposition parameters with ginger extracts.

Sample Identification	Extract Concentration (l/l)	Time (min)	Mass Gained (g)
G-4-15	0.08	15	0.0867
G-4-18	0.08	18	0.1115
G-6-15	0.12	15	0.1096
G-6-18	0.12	18	0.1109
G-8-15	0.16	15	0.1178
G-8-18	0.16	18	0.1219

Table 6 Electrodeposition parameters with pomegranate extracts.

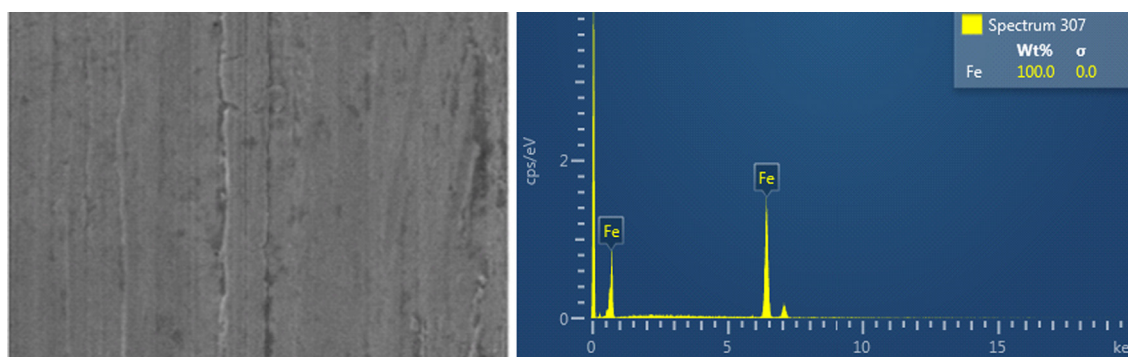
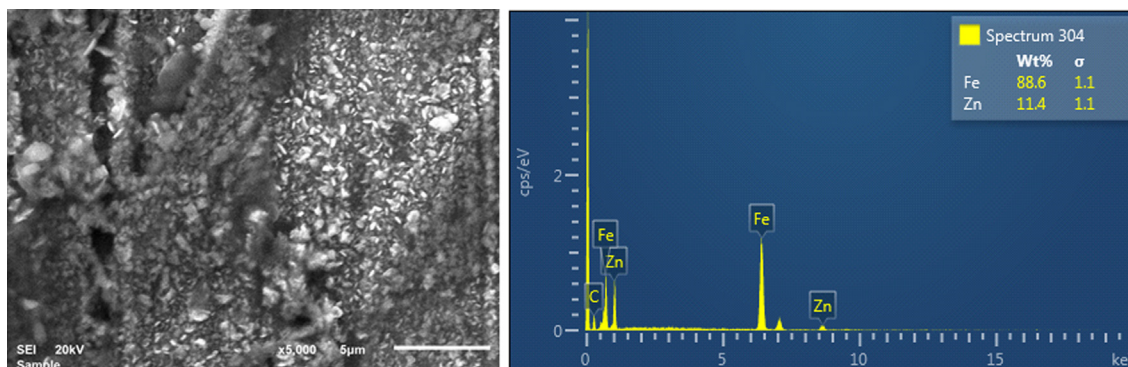
Sample Identification	Extract Concentration (l/l)	Time (min)	Mass Gained (g)
P-4-15	0.08	15	0.0582
P-4-18	0.08	18	0.0602
P-6-15	0.12	15	0.0664
P-6-18	0.12	18	0.0620
P-8-15	0.16	15	0.0723
P-8-18	0.16	18	0.1614

position on MS without the addition of plant extracts is shown in Table 3. Tables 4–6 show the electroplating parameters for Zn electrodeposition on MS at specific concentrations of celery, ginger and pomegranate extracts. The presence and increase in extract concentration is strongly associated with the amount of Zn electrodeposited on MS. The weight addition of Zn on MS at 0.0558 g and 0.0621 g (Table 2) is relatively lower than the values obtained for Zn electrodeposition in the presence of celery, ginger and pomegranate extracts at 15 min and 18 min. Observation of Tables 4–6 shows the performance of gingers extracts on the amount of Zn electrodeposited on MS were generally better than the values obtained for celery and pomegranate between concentration of 0.08 L – 0.16 L and optimal plating time of 15 min. Results obtained for weight gained in the presence of pomegranate were generally the lowest. However, the highest weight gained can be seen in sample P-8-18 having a value of 0.1614 g.

3.2. SEM/EDS analysis of unplated and Zn electrodeposited MS

The surface morphology and %wt. content of unplated and Zn electrodeposited MS samples with and without plant extract additives was analysed through SEM and EDS. Micrographs were produced at specific magnifications. The microscopy and spectroscopy results were obtained for MS sample with optical performance of Zn electrodeposition. Fig. 1 shows the SEM morphology (mag. 1000 \times) and EDS result of MS before Zn electrodeposition. Fig. 2 shows the morphology (mag. 5000 \times) and EDS result of Zn electrodeposited MS without any plant extract additive in the acid chloride bath after 15 min of exposure. It is observed that the microstructural features are not so bright especially coupled with the presence of surface defects. The surface crystals are not well defined, though closely packed they are very coarse with significant porosity. The absence of additives (levelling agents) does not provide sufficient protection against corrosion. EDS results of the Zn electrodeposited MS surface shows %wt. of Fe is significant at 88.6% while the presence of Zn gave a wt.% of 11.4% on MS surface.

MS surfaces (mag. 2500 \times) electrodeposited with Zn and 6 ml concentration of ginger extract as additive at plating time of 15 min and 18 min are shown in Figs. 3 and 4. Fig. 3 exhibits a closely packed petal-like morphology with limited porosity at 15 min of exposure. The morphology appears brighter than the in Fig. 2 due to the brightening effect of the ginger extract. The morphology in Fig. 4 is significantly brighter than Fig. 3 due to

**Fig. 1** SEM/EDS result of unplated MS specimen.**Fig. 2** SEM/EDS result of Zn electrodeposited MS no extract additive at x5000 Magnification.

increase in plating time to 18 min with the microstructure being more compact, hence and less susceptible to corrosion. EDS analysis in Fig. 3 shows the wt.% content of Zn electrodeposited on MS has increased to 55.5% compared to 11.4% in Fig. 2. Further increase in plating time to 18 min (Fig. 4) results in electrodeposition of more Zn on MS surface (at 67.5 wt.% content). At this plating time the microstructure in Fig. 4 is even more closely packed with the petal-like structure being more rose-like. The aesthetic value of the Zn electrodeposited surface has increased due to the brightening effect resulting from prolonged time.

Figs. 5 and 6 (mag. 5000 \times) shows the SEM morphology and EDS results for Zn electrodeposited MS surface with pomegranate as additive at 4 ml and 6 ml for 15 min. The

microstructure is observed to be closely packed with uniformly distributed Zn crystal particles over MS surface coupled with limited porosity. However, the degree of brightness is limited though greater than Zn electrodeposited MS surface without extracts. EDS result showed increase in %wt. of Zn electrodeposited on MS increased to 78.2%. This signifies pomegranate extract enhances the electrodeposition of Zn on MS greater than the effect of ginger though ginger produces more brightening effect. Increase in pomegranate concentration to 6 ml produces had minor effect of the electrodeposition of Zn on MS though decrease in %wt. Zn content was observed from EDS result at 75.8%.

Morphology of Zn electrodeposited on MS with celery additive (4 ml concentration) for 15 and 18 min is shown in

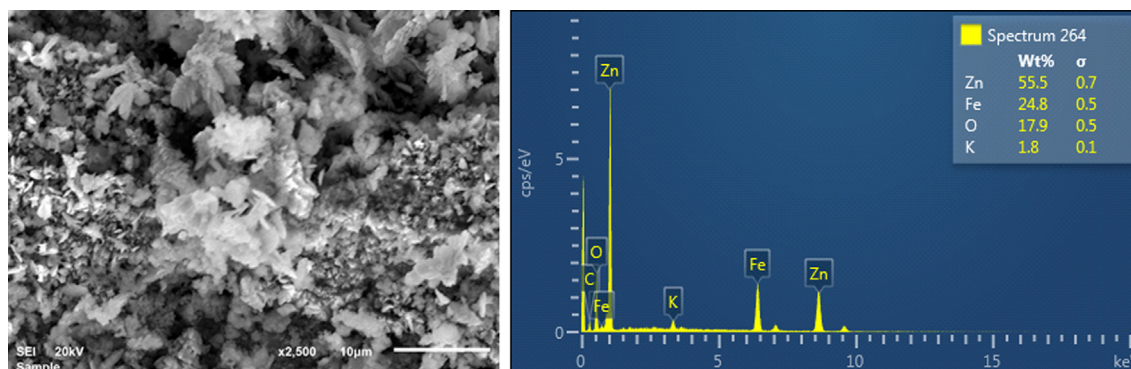


Fig. 3 SEM/EDS result of Zn electrodeposited MS with 6 ml ginger extract addition for 15 min (mag. 2500 \times).

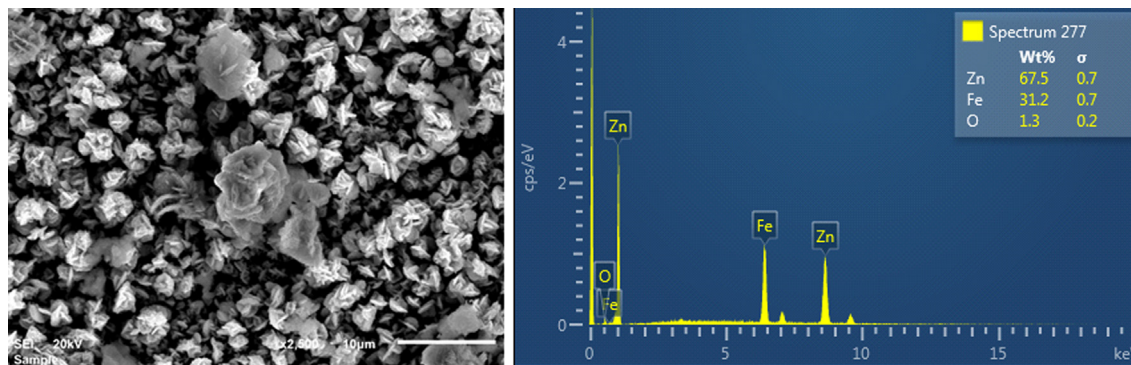


Fig. 4 SEM/EDS result of Zn electrodeposited MS with 6 ml ginger extract addition for 18 min (mag. 2500 \times).

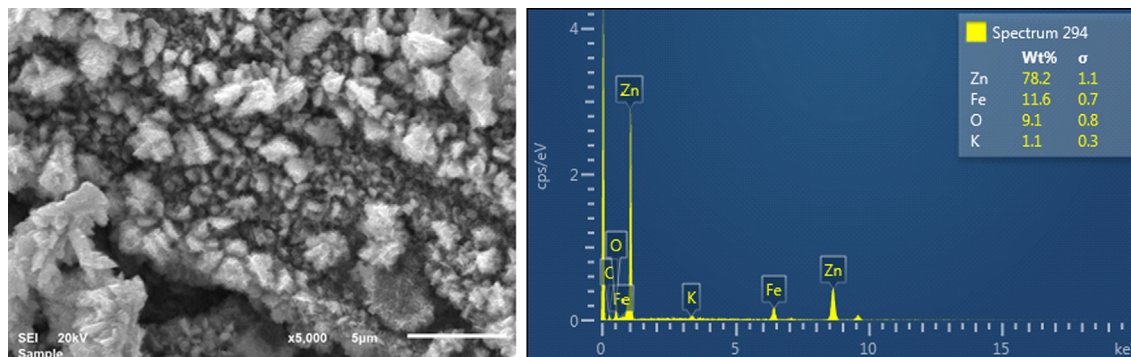


Fig. 5 SEM/EDS result of Zn electrodeposited MS with 4 ml pomegranate extract addition for 15 min (mag. 5000 \times).

Figs. 7 and 8 (mag. 5000 \times). The morphology exhibited good zinc electrodeposition which significantly contrasts the image for electroplated MS without additive (Fig. 2). The effect of the additive at 15 min is visible on MS morphology, considering the brightness and compactness of the flake like crystal particles. However, these attributes do not translate to enhanced electrodeposition of Zn on MS as shown in the EDS result whereby only 40.2 wt.% content of Zn was deposited on MS. Further increase in electroplating time to 18 mins marginally influenced the outcome of the electrodeposition process on MS. The Zn crystals in Fig. 8 appears to be more concentrated and closely packed compared to Fig. 7 signifying brighter morphology. However, EDS results shows slight

improvement in Zn electrodeposition from 40.2 wt.% to 47.4 wt.% This ensures higher corrosion resistance of MS after electroplating for 18 min.

3.3. Corrosion polarization studies

Potentiodynamic polarization plots for unplated and Zn electrodeposited MS at specific extract concentration and plating time is shown from Figs. 9–11. Fig. 9 shows the plots for unplated MS, Zn electrodeposited MS and Zn electrodeposited MS with ginger extract additive at 4 ml and 6 ml after 15 mins of plating time. Fig. 10 shows the plots for unplated MS, Zn electrodeposited MS and Zn electrodeposited MS with pome-

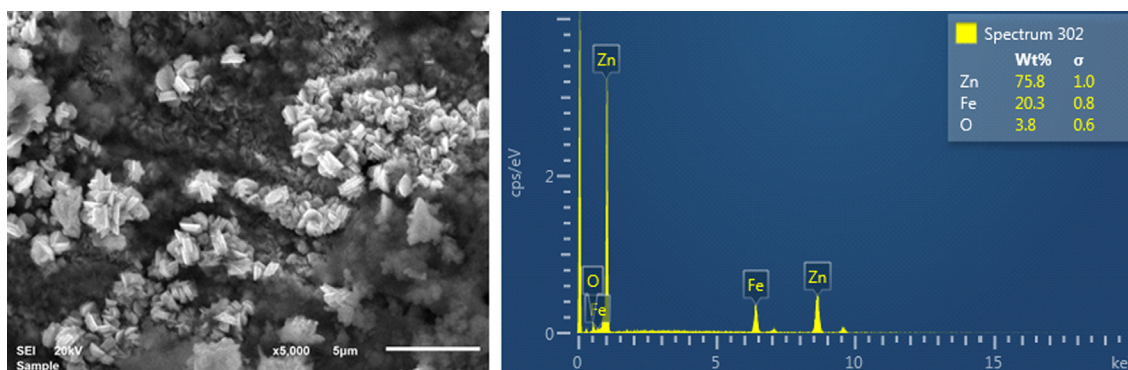


Fig. 6 SEM/EDS result of Zn electrodeposited MS with 6 ml pomegranate extract addition for 15 min (mag. 5000 \times).

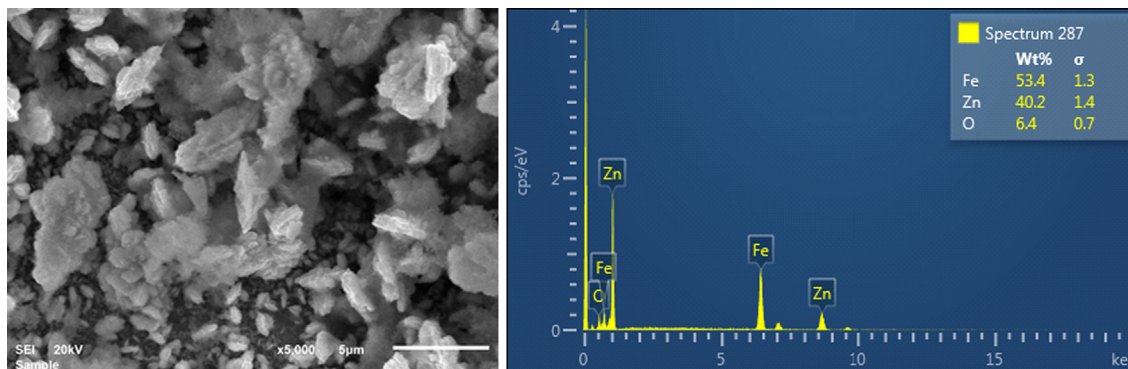


Fig. 7 SEM/EDS result of Zn electrodeposited MS with 4 ml celery extract addition for 15 min (mag. 5000 \times).

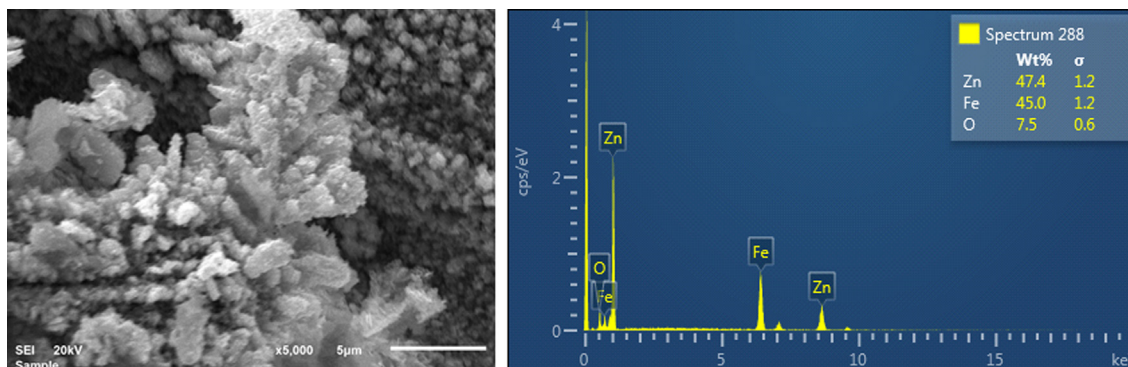


Fig. 8 SEM/EDS result of Zn electrodeposited MS with 4 ml celery extract addition for 18 min (mag. 5000 \times).

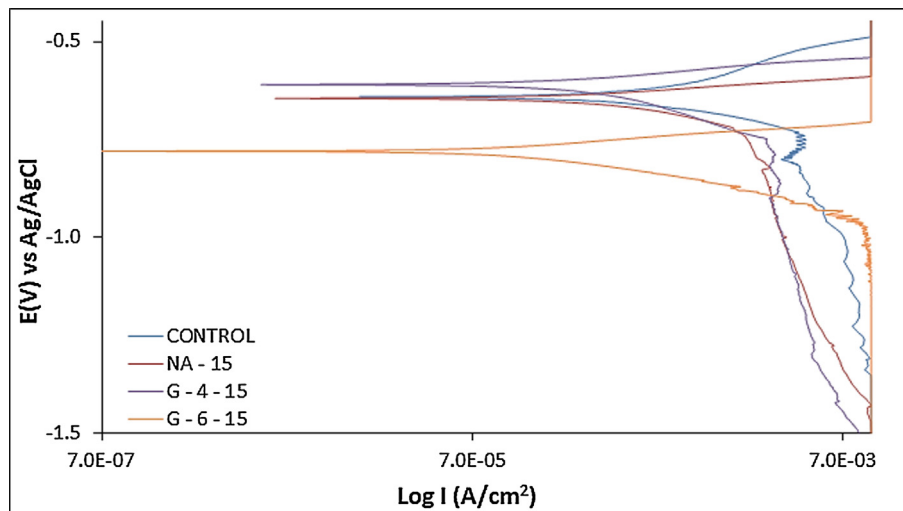


Fig. 9 Potentiodynamic polarization plot for un plated MS, Zn electrodeposited MS and Zn electrodeposited MS with ginger extract additive at 4 ml after 15 min and 18 min of plating time.

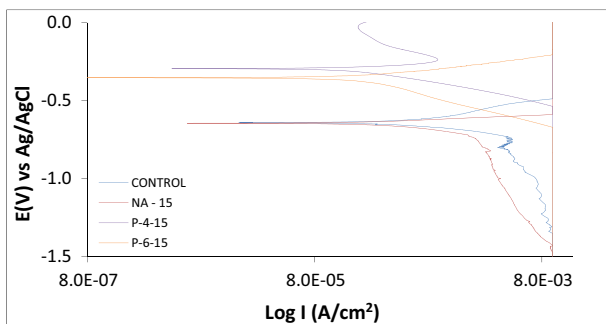


Fig. 10 Potentiodynamic polarization plot for un plated MS, Zn electrodeposited MS and Zn electrodeposited MS with pomegranate extract additive at 4 ml and 6 ml after 15 mins of plating time.

granate extract additive at 4 ml and 6 ml after 15 mins of plating time, while Fig. 11 shows the plots for un plated MS, Zn electrodeposited MS and Zn electrodeposited MS with celery extract additive at 4 ml after 15 and 18 mins of plating time. Data obtained from the polarization plots are shown in Table 7. The corrosion rate of control MS (7.96 mm/y) is significantly higher than the values obtained for Zn electrodeposited MS with different extract additives. The value corresponds to corrosion current density of $6.76 \cdot 10^{-4}$ A/cm² and polarization resistance of 32 Ω. This observation is due to oxidation of Fe²⁺ ions into the acid chloride solution and corresponding H₂ evolution reactions. As a result, the redox electrochemical process accelerates due to the weakness of MS to electrochemical degradation. Electrodeposition of Zn to MS surface slightly decreased the corrosion rate value to 6.58 mm/y as shown in Table 7. The polarization resistance value increased to 39.3 Ω. However, the change in electrochem-

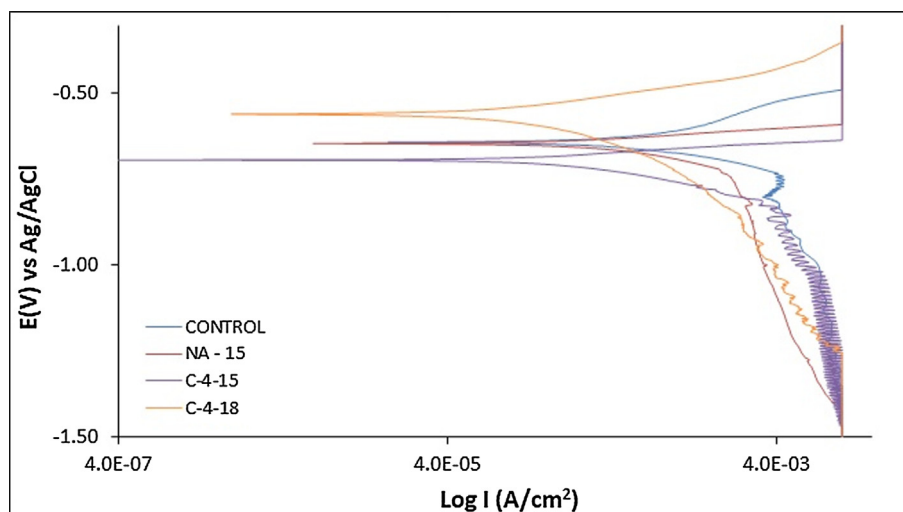


Fig. 11 Potentiodynamic polarization plot for un plated MS, Zn electrodeposited MS and Zn electrodeposited MS with celery extract additive at 4 ml and 6 ml after 15 mins of plating time.

Table 7 Potentiodynamic polarization results for unplated and Zn electrodeposited MS at specific extract concentration and plating time.

Control and No Additive						
Sample	Extract Conc. (l/l)	Corrosion Rate (mm/y)	Corrosion Current (A)	Corrosion Current Density (A/cm ²)	Corrosion Potential (V)	Polarization Resistance (Ω)
Control	–	7.96	7.91E–04	6.76E–04	–0.641	32.5
NA-15	–	6.58	6.55E–04	5.59E–04	–0.645	39.3
Ginger Additive						
Sample	Extract Conc. (l/l)	Corrosion Rate (mm/y)	Corrosion Current (A)	Corrosion Current Density (A/cm ²)	Corrosion Potential (V)	Polarization Resistance (Ω)
G-4-15	0.08	4.40	4.38E–04	3.74E–04	–0.610	58.7
G-6-15	0.12	2.48	2.47E–04	2.11E–04	–0.780	104
Pomegranate Additive						
Sample	Extract Conc. (l/l)	Corrosion Rate (mm/y)	Corrosion Current (A)	Corrosion Current Density (A/cm ²)	Corrosion Potential (V)	Polarization Resistance (Ω)
P-4-15	0.08	3.73	3.71E–04	3.17E–04	–0.295	69.3
P-6-15	0.12	2.78	2.76E–04	2.36E–04	–0.353	93
Celery Additive						
Sample	Extract Conc. (l/l)	Corrosion Rate (mm/y)	Corrosion Current (A)	Corrosion Current Density (A/cm ²)	Corrosion Potential (V)	Polarization Resistance (Ω)
C-4-15	0.08	3.75	3.72E–04	3.18E–04	–0.693	69
C-4-18	0.08	1.95	1.94E–04	1.66E–04	–0.689	132

Key: X-Y-Z. X: Additive Name (No Additive, Celery, Ginger & Pomegranate); Y: Additive Amount (4 ml, 6 ml & 8 ml); Z: Plating Time (15 min, 18 min).

ical parameters with Zn electrodeposition, while signifying improvement is insufficient to prevent corrosion considering the weak corrosion resistance of MS. Addition of ginger extract at 4 ml and plating time of 15 min decreased the corrosion rate of Zn electrodeposited MS to 4.40 mm/y. Further increase in volume of ginger extract to 6 ml improved the corrosion resistance of MS further to 2.48 mm/y which corresponds to polarization resistance of 104 Ω . The corrosion rate of Zn electrodeposited MS in the presence of pomegranate additive at volumetric content of 4 ml and 6 ml is 3.73 mm/y and 2.78 mm/y which is generally similar to the values obtained with ginger as the additive. The corrosion rate value obtained in the presence of celery extract (3.75 mm/y) at 4 ml also falls within the range of values obtained in the presence of ginger and pomegranate extract. However, at volumetric content of 4 ml and plating time of 18 min the corrosion rate value significantly decreased to 1.95 mm/y. The corrosion rate value corresponds to corrosion current density of 1.66×10^{-4} mm/y and polarization resistance of 132 Ω , signifying effective protection against corrosion.

4. Conclusion

Natural extracts of ginger, pomegranate and celery proves usable with excellent results as additives in the zinc electroplating of mild steel within solutions of combined admixture of ZnCl₂, H₃BO₃ and KCl. The crystal structure of the electroplated steel varied significantly with respect to additive concentration and plating time. Micrographs of electroplated surface with additives were significantly brighter than the images of the control specimen. Increase in extract concentration and

plating time positively influenced the brightness of the electroplated metal samples with the surface structure being finer and more closely packed. The electroplated steel with ginger additive had the brightest and finest micrographic image. Results from potentiodynamic polarization in 1 M H₂SO₄ showed the electroplated steel samples were more corrosion resistant than the control steel. The corrosion resistance of the mild steel samples reduced with increase in concentration of the natural extracts and plating time with the electroplated steel with ginger extract having the lowest corrosion rate. The unplated mild steel exhibited very low resistance to corrosion.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

The authors are grateful to Covenant University for their support of the research and funding for publication.

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