Canadian Journal of Pure and Applied Sciences
Vol. 9, No. 1, pp. 3267-3277, February 2015
Online ISSN: 1920-3853; Print ISSN: 1715-9997
Available online at www.cjpas.net

EFFECT OF *ALLIUM CEPA* (ONION) EXTRACT ADDITIVE ON THE MORPHOLOGY OF ZINC ELECTROPLATED MILD STEEL IN ACID CHLORIDE SOLUTION

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ABSTRACT

An investigation of the plating quality effect of *Allium cepa* extract as addition agent on the electroplating of zinc on mild steel in acid chloride solution was done in the laboratory. Different extract concentrations, different plating time and fixed pH conditions were used to perform the experiments. A DC – supplier at defined operating parameters was used to perform the electroplating of zinc on mild steel. Scanning electron microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS) was used to examine the surface of the plated steel for surface morphology and surface elemental composition analysis. Variable surface characteristics were obtained depending upon the concentration of the additive and the plating time. Potential measurement, corrosion current and gravimetric methods were used to monitor and determine the corrosion resistance of the plated surface. The coating efficiencies were also determined. Entirely new and different metal substrate surface morphology were produced due to variations in the plating parameters used. The microstructural features of the plated surfaces showed that the quality of the electro-deposition of zinc was good.

Keywords: Electroplating, onion juice extract, steel surface, acid chloride solution, corrosion.

INTRODUCTION

Electroplating is primarily used for beautification of the object being plated and corrosion resistance. Hence, zinc electrode position on mild steel has been of very good importance in surface engineering research and in metals' industry. Addition agents are especially required in this process to enhance the surface finishing quality. In spite of the fairly long period of time that the available proprietary additives have been used (Schneider, 1987; ASM, 1982; Pushpavanam, 1986; D'Angelo, 1986), it is still considered necessary to develop other environment – friendly and non-commercial proprietary additives for the acid chloride bath. This has recently been generating increased research interest. This necessitates the present investigation. This work therefore aims at a further contribution to the recent investigations (Loto and Olefjord, 1990; Loto and Olefjord, 1992; Venkatesha *et al*., 1987; Kanagalasara *et al*., 2011; Loto *et al*., 1992) which characterized the surface effects of additives on the electrodeposition of zinc on mild steel in acid-chloride solution under different conditions. Many other authors (Schlesinger and Paunovic, 2000; Darken, 1979; Zemanova, 2009; Nayana *et al*., 2011) have also reported in different areas of zinc and zinc alloys electrodeposition and on synergistic effect of electrodeposited alloys. The effect of addition agents and corrosion resistance characteristics using different bath solutions were also documented. Chloride zinc solution gives improved bath efficiency and exceptional brightness, in addition to eliminating cyanide in plating.

Use of plant extracts as inhibitors for the corrosion of metals/alloys in recent time have been reported by various authors (Loto, 2000, 2001, 2003, 2005; Okafor *et al*., 2007; Davis and Fraunhofer, 2003; Fraunhofer 1995; Fraunhofer *et al*., 2001; Fraunhofer, 2000). However, the application of these plant extracts to zinc electroplating on metallic alloy such as mild steel is still relatively new (Loto *et al*., 2014).

Onion (*Allium cepa*) is known to contain on analysis,

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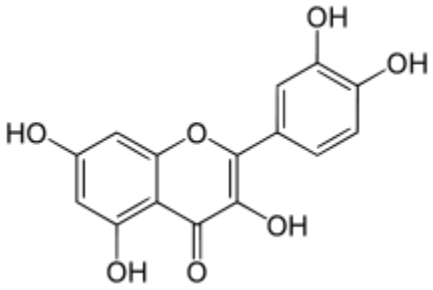
vitamin C, vitamin B_6, folic acid and other nutrients though in small quantities (National Onion Association, 2014). Onions have also been found (Slimestad *et al*., 2007) to contain other chemical compounds such as phenolics and flavonoids. These have been described (Williamson *et al*., 1996) to include quercetin and its glycosides quercetin 3, 4'-diglucoside and quercetin-4'-glucoside.



Quercetin, a typical flavonoid, is a polyphenol

The complex chemical compositions contained in *Allium cepa* may exhibit electrochemical activity such as zinc electroplating on mild steel. It is expected the result obtained in this work will be of economic benefit.

Materials and Methods

Fresh onions weighing 2 kg and collected from a local farm were cut into small chunks and slices and fed into a blender with about 10ml water to assist the process. The onion blend was then poured unto a sieve and then filtered to remove chaff and particles from the onion juice. The juice extract obtained, were prepared and used as the addition agents in the electroplating process as further described below.

Experimental set-up

Flat mild steel, with 0.1 cm in thickness was used in this investigation. The experimental set up was just as previously described in recent studies (Loto, 2012; Loto *et al*., 2013; Loto and Loto, 2013). The nominal composition of the steel is: 0.038% C, 0.195 Mn and the remainder Fe. It was cut into several specimens and each of 10.0 cm long and 1.0cm wide. At one end of each specimen, a portion of 1.0 cm in length was marked for the zinc electrodeposition. The test specimens were degreased ultrasonically for 5 minutes with an alkaline degreasing chemical. They were subsequently rinsed in distilled water, immersed in methanol, and air dried. The specimens were, in turns, etched for 50 seconds in 3M HCl, and further rinsed in distilled water. They were then immersed in methanol; then removed and air dried and stored in a desiccator for further experimental process.

Electroplating bath of acid chloride solution was made of ZnCl (71g/l), KCl (207g/l) and H_3BO_4 (35g/l). Varying concentrations of extracts of *allium cepa* of: 5, 10 and 15 ml / 50ml of acid chloride solution were used in turns as the addition agents. Electroplating of zinc on steel was performed by the partial immersion of the steel specimen and the zinc electrodes in the plating solution contained in the beaker used as the plating bath. A wire connection of the steel specimen was made to the negative side of a DC supplier while the zinc two electrodes were also connected with a wire to the positive side (Fig. 1). The pH of the plating solutions was adjusted with potassium hydroxide. Variable plating time of 15 and 18 minutes were separately used for the plating process. Weight of the steel specimen was taken before and after the electroplating process to determine the weight of zinc deposited. This was done by finding the difference between both weight readings. The results obtained are presented in figure 2.

The plating solution was stirred gently while the plating was being carried out to ensure even plating. The other operating conditions were: pH of the solution, 5; temperature, 27- 30^{o}C; current 0.08A; Voltage, 13V DC; plating time, 15 and 18 min. After each plating experiment, the specimen was taken out, rinsed in distilled water, immersed in methanol, and quickly air-dried. The specimens were stored in a desiccator for further analysis. The respective samples were labelled as presented in table 1.

Table 1. Plated samples identification.

H	5ml Onion juice (15mins)
I	5ml Onion juice (18mins)
J	10ml Onion juice (15mins)
K	10ml Onion juice (18mins)
L	15ml Onion juice (15mins)
M	15ml Onion juice (18mins)

SEM/EDS characterisation

A scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) was used to examine the surface morphology and/or microstructure of each of the plated test specimens. The cut and stub mounted specimens were separately examined in turns in the SEM. Electron micrographs were made of the representative areas of the surface at different magnifications. The energy dispersive spectroscopic (EDS) analysis was also done on the selected surface portions. This was performed to determine the elemental composition of the steel plated surface.

Adhesion test

Cellotape fastened to the surface of the zinc plated steel and subsequently pulled off was used to test for the adhesion of zinc to the plated surface. The cellotape was visually observed for any zinc stripping from the plated steel's surface. A further test was done by using a scalpel to scratch the plated steel surface to test for the zinc adhesion.

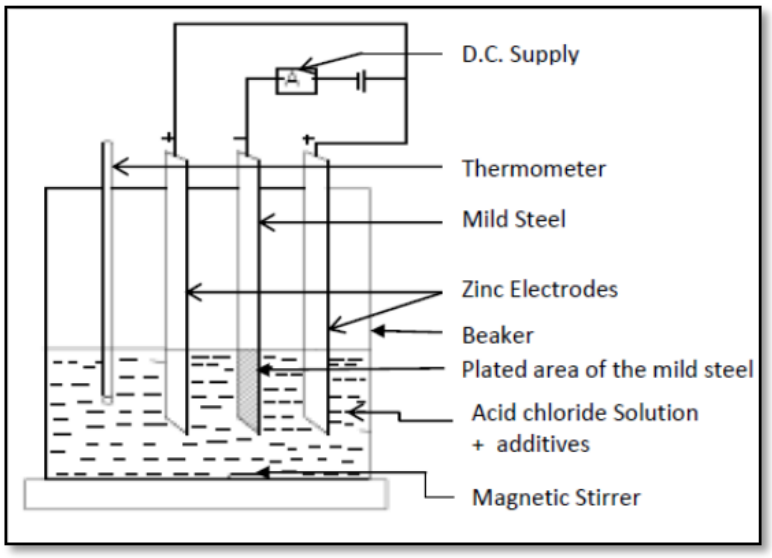


Fig. 1. Schematic diagram of the experimental set-up.

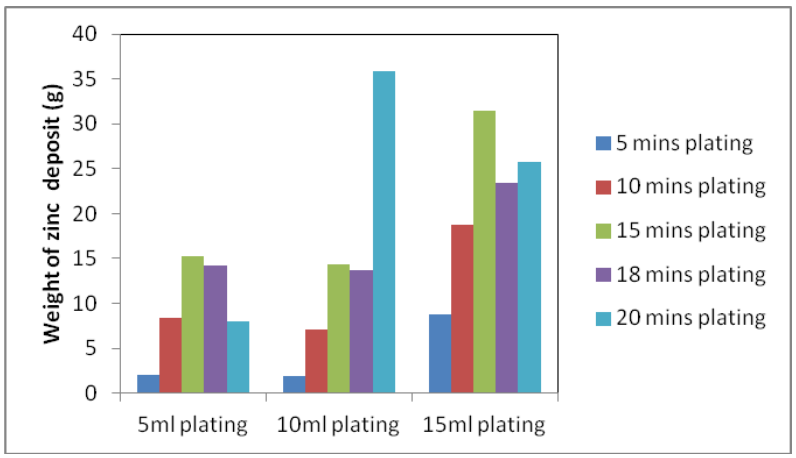


Fig. 2. Weight of zinc deposited on mild steel samples versus additives concentrations and the different plating time.

Corrosion resistance

Weight loss method

The electroplated mild steel was tested for corrosion resistance. This was performed by the weight loss method and by the use of potential and current measurements. Plated mild steel test specimens were separately immersed in 0.5M HCl. Readings of their weight loss recorded every two days for a period of 24 days. In addition, the corrosion rate (C.R.) values were calculated from these weight loss values by the use of the formula presented below:

$$\text{C.R.} = \left[\frac{87.6W}{DAT}\right] \quad \ldots\ldots 1$$

Where:

W = the weight loss in milligrams;
D = the density in g/cm^2;
A = the area in cm^2; and
T = the time of exposure in hours.

Potential and Current measurements

Mild steel samples were cut into small coupons and welded/soldered with copper wires to each of them. They were then mounted in resins (araldite); to expose only the plated sample surface area to the acid test medium. The measurements were performed with multi meter (for voltage) and zero resistance ammeter (for current) measurements. All the experiments were performed using 75 ml each in a beaker of 0.5M HCl. In all the corrosion tests, only few selected specimens were used.

Efficiency of Coating

The coating efficiency was determined with the use of this formula:

$$EC\% = \frac{Rcn - Rca}{Rca} \quad \ldots\ldots\ldots 2$$

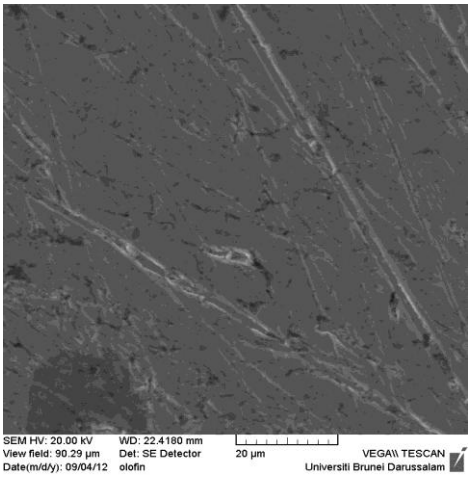
Where, Rca is corrosion rate of sample plated with additive, and Rcn is the corrosion rate of samples plated without additives.

RESULTS AND DISCUSSION

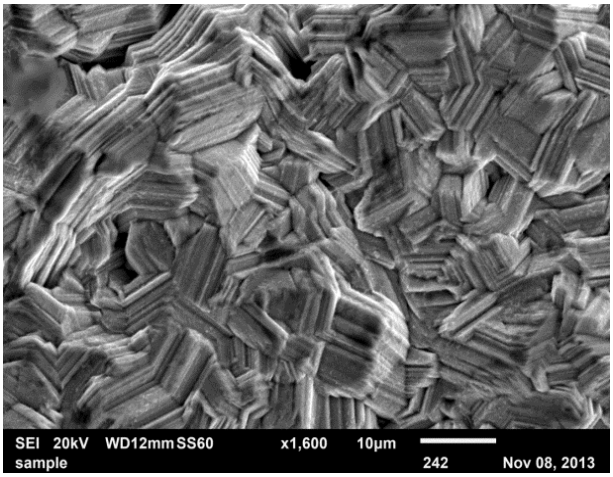
Electrodeposition of zinc

Plating with no additive

Scanning electron microscopy (SEM) micrograph of the surface of the mild test sample before zinc electroplating is presented in figure 3 (i), while figure 3 (ii) shows the electron micrograph of the zinc electroplated mild steel test samples from acid–chloride solution without any additive. The slipped-stack-like crystals were distinctly and uniformly distributed throughout the plated surface, closely packed and with very low microscopic porosity in just two places. The surface crystals feature was not smooth. The structure was coarse and this could be due to the absence of levelling agents in the acid solution and the poor throwing power of the acid solution. The surface morphological structure was however, uniquely patterned in shape.

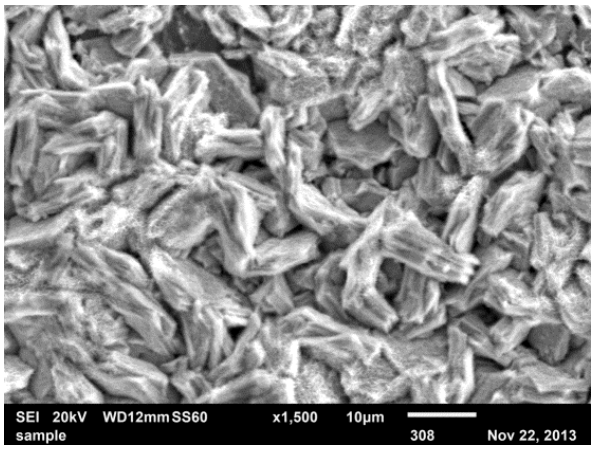


(1) Unplated

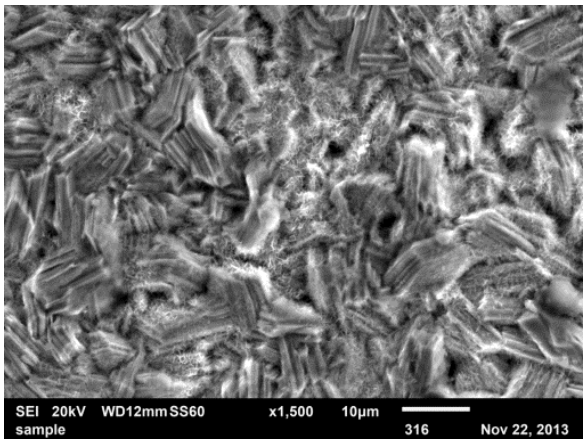


(ii) Plated without additive

Fig. 3. Micrographs of mild steel test sample: (i) Unplated sample (ii) Plated without onion juice extract addition.



(i)



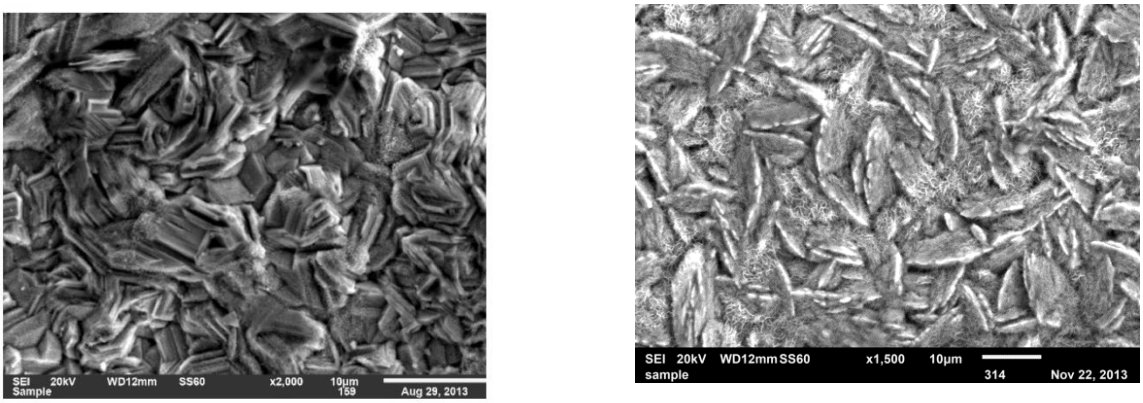
(ii)

Fig. 4. Micrographs of steel surface after zinc plating with 5ml /50ml of acid- chloride solution at (i) 15 and (ii) 18 minutes, respectively.

Plating with same additive concentration and different plating time

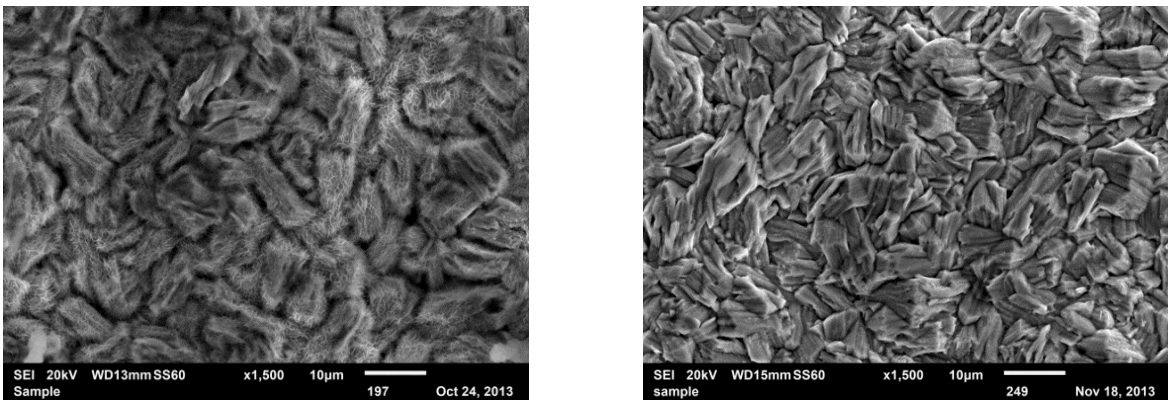
5 ml / 50 ml additive at 15 and18 minutes plating time

The micrographs made with plating at 15 and 18 minutes respectively with the onion juice extract concentration of 5ml / 50ml of acid chloride solution are presented in figure 4. The microstructure in figure 4 (i) appears more uniquely shaped and defined than in figure 4 (ii) though they both have fine small grains with rod-like appearance. The surface microstructure is levelled and looks bright. The crystal particles were very close-packed and no porosity observed. Obviously, the observed very fine grains and levelling difference in surface morphology as evidenced in figure 4 (i-ii), when compared with those in figure 3 (i-ii), emanated from the addition of the onion juice which has complex chemistry such as phenolics and polyphenols, particularly, flavonoids. These include, as mentioned above, quercetin and its glycosides quercetin



(i) (15 minutes) (ii) (18 minutes)

Fig. 5. Micrographs of steel surface after zinc plating with 10ml /50ml of acid chloride solution at (i) 15 minutes and (ii) 18 minutes, respectively.



(ii) 15minutes (ii) 18 minutes

Fig. 6. Micrographs of steel surface after zinc plating with 15 ml /50ml of acid chloride solution at (i) 15 min. and (ii) 18 minutes, respectively.

3,4'-diglucoside and quercetin-4'-glucoside (Williamson *et al*., 1996); they could exhibit electrochemical activity of electro-deposition. The difference in surface morphology in figures 4 (i) and (ii) appears not significant but the grains of the latter (the 18 minutes plating time) are smoother, more compact and more levelled. The non-porous unique microstructure observed in figure 4 (i-ii), confirmed good zinc electroplating. The mass of zinc deposited was weighed to be 15 and 14 g, respectively (Fig. 2).

10ml/50ml of additive at 15 and 18 minutes plating time

When the concentrations of the extract additive were changed from 5ml to 10ml onion juice extract/50 ml of acid chloride solution at both the 15 and 18 minutes plating time, another different surface morphological structures as shown in figure 5 (i - ii) were obtained. The very little increase in the volume/concentration of the extract brought about very significant surface morphological changes. The surface crystals are clcosely packed and thus presenting very compact and non-porous surface features. The surface structures are smooth and levelled as could be observed thus presenting a good surface morphology. However, the surface feature for the 18 minutes plating time was very much different from that of 15 minutes. The flat fish-like platelets feature of the surface morphology of the 18 minutes plating time, figure 5 (i), presents an uncommon unique surface microstructure. This observed feature, apparently, could be plausibly associated with concentration and plating time effects. A very levelled, smooth, well defined and compact, non-defective surface feature such as observed here, has the positive inclination of enhancing better surface plating and hence corrosion protection. The plated zinc was expected to corrode sacrificially to protect the mild steel substrate. The mass of zinc deposited was 14 g each for the 15 and 18 minutes plating time.

15ml /50ml additive at 15 and 18 minutes plating time

Figure 6 (i - ii) show the scanning electron microscopy micrographs of steel surface after zinc electroplating with

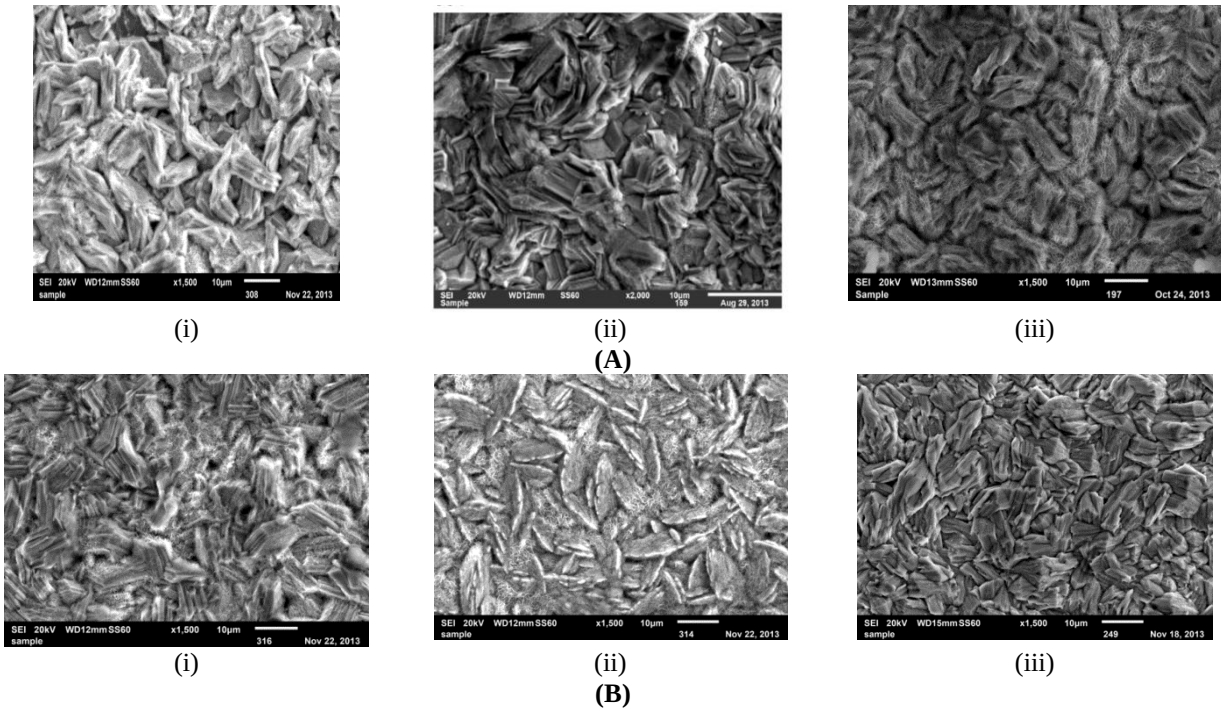


Fig. 7. SEM micrographs for the samples plated at different concentrations and at the same plating time: (**A) –** (i) 5 ml, (ii) 10ml and (iii) 15ml/50ml for 15 minutes; (**B**) (i) 5 ml, (ii)10 ml, and (iii) 15ml at 18 minutes, respectively.

15 ml /50ml of acid chloride solution at 15 and 18 minutes, respectively. The crystal grains were compact and well levelled but dissimilar in shape. The grains were well close-packed and thus presenting a smooth surface characteristics. The plated surface morphological microstructure was therefore expected to give very good surface protection to the mild steel substrate. The mass of zinc deposited was 31g for 15 minutes plating time and 23g for 18 minutes plating time. The increase in the additive concentration seemed to have made significant contribution to the observed compactness, smooth and levelled surface morphology. It is important to mention here again that the chemical composition of onion juice extract is indeed very complex. This would, undoubtedly, have appreciable beneficial impact on the recorded positive results obtained in this work.

The smoothness and the compactness of the surface morphology in the plated steel substrate were therefore expected to give a better surface corrosion resistance. It is also important to mention that the 18 minutes plating time enhanced its levelling behaviour.

Different additive concentrations and same plating time

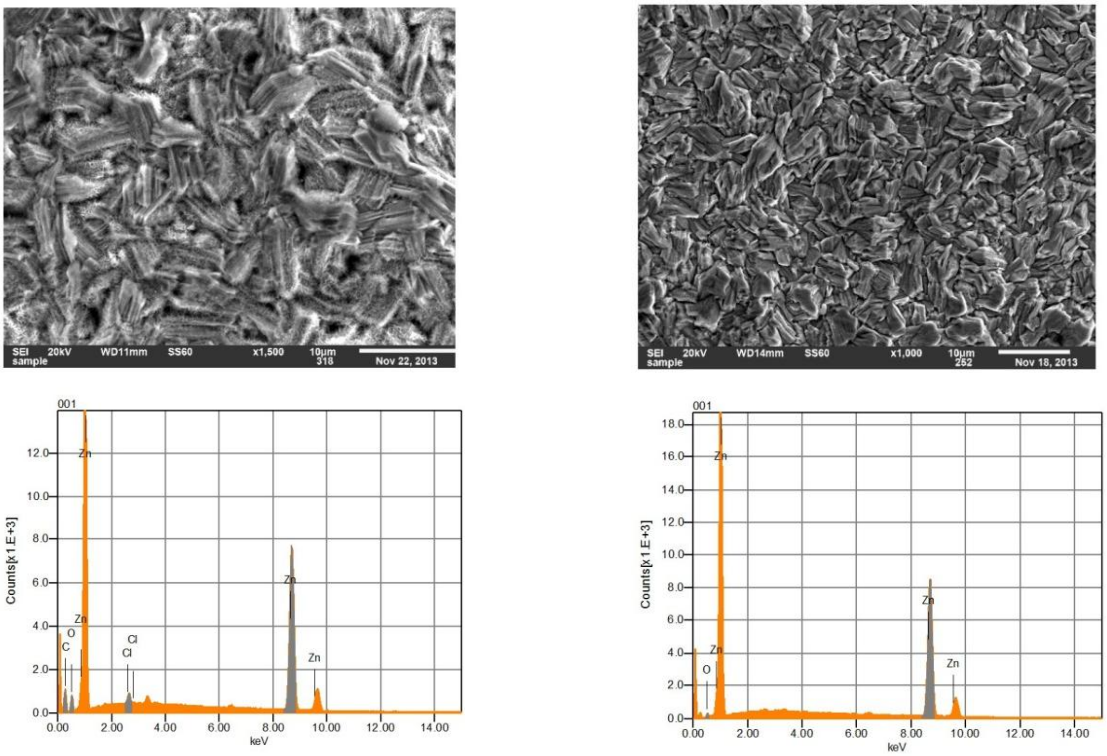
Presented in figure 7 are, the various SEM micrographs for the samples plated at different concentrations of 5, 10, and 15 ml / 50ml and at the same plating time of: (**A)** 15 minutes and (**B**) 18 minutes respectively. The surface morphology of each of these micrographs had been described above. For surface morphological structural comparison, they are represented in figure 7. Though not very much significant, there were apparent differences, in general, in the surface structural morphology for the plating time of 15 and 18 minutes at each of the concentrations used. However, the 18 minutes plating time gave the most compact, smoothest and the most levelled surface microstructure at all the concentrations. The surface morphology for each of the plating time for the three different concentrations shows different features with good levelling and dense packing.

Energy dispersive spectroscopy (EDS) analysis

The results of energy dispersive spectroscopy analysis (EDS) of figure 4 (ii) and figure 6 (ii)) are presented in figure 8. The surface microstructure was predominantly zinc and a little amount of iron in the plating without additive which apparently was in trace form. The EDS analysis confirmed the effective coverage of the zinc deposition and the dense packing that left no room for porosity.

Corrosion resistance of the zinc plated mild steel

Plated samples of some representative concentrations and plating time values were selected for test because of their observed better surface plating structure. All the plated samples with additives with 15 ml / 50ml concentration of acid chloride solution at 15 and 18 minutes plating time showed higher corrosion rate values than those of 10 and 5 mil / 50ml of acid chloride solution at the same plating



(i) 5ml Onion juice (18mins) x1500 (ii) 15ml Onion juice (18mins) x1500

Fig. 8. EDS analysis of the plated surface of samples in (i) Fig. 4, and (ii) Fig. 6(ii).

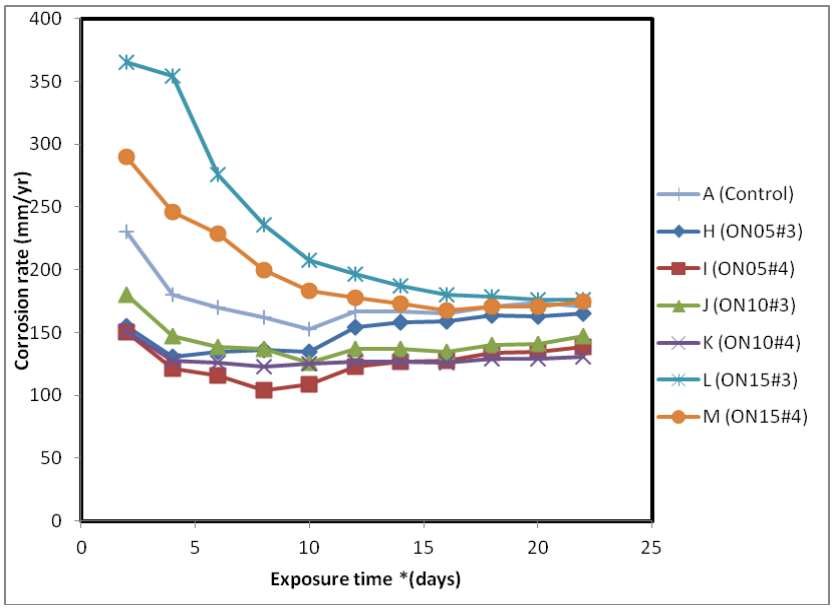


Fig. 9. Plot of corrosion rate with exposure time for the zinc plated mild steel samples in 0.5M HCL (onion juice additive at concentration of 15 ml / 50ml acid chloride solution); and 5, 10, 15 and 18 minutes plating time).

time. All the tested samples of the latter concentration of acid chloride had lower corrosion rate than the plated samples without onion juice additive with corrosion rate values ranging from 129 to 165mm/yr on the 20th and the 22nd day of the experiment. They all had lower corrosion rates. The sample plated with the concentration of 10ml / 50ml acid chloride solution has the lowest corrosion rate of all the samples at all the plating time and at all the concentrations used as presented in figure 9. It should be noted, however, that additives function more as levelling

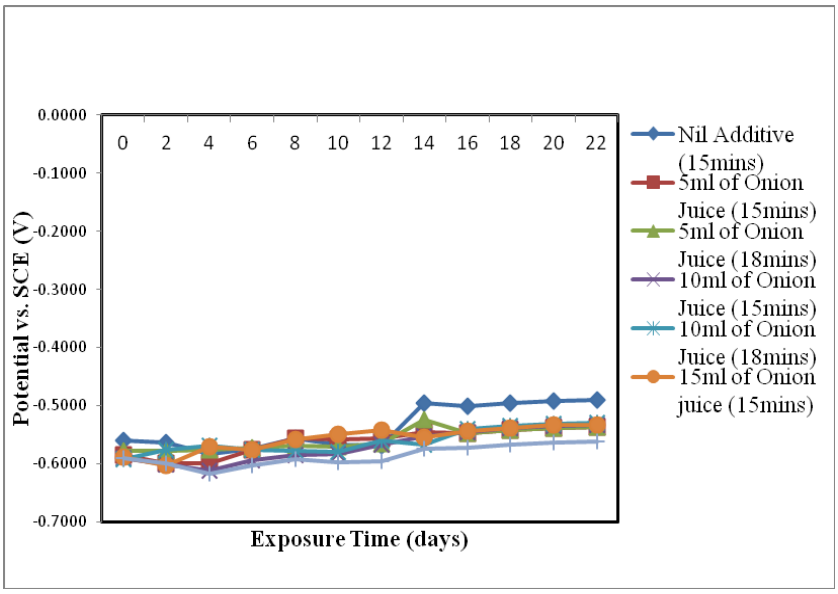


Fig. 10. Curves of potential versus the exposure time for the zinc plated mild steel in 0.5 M HCl with variable plating time and onion juice extract additive concentrations.

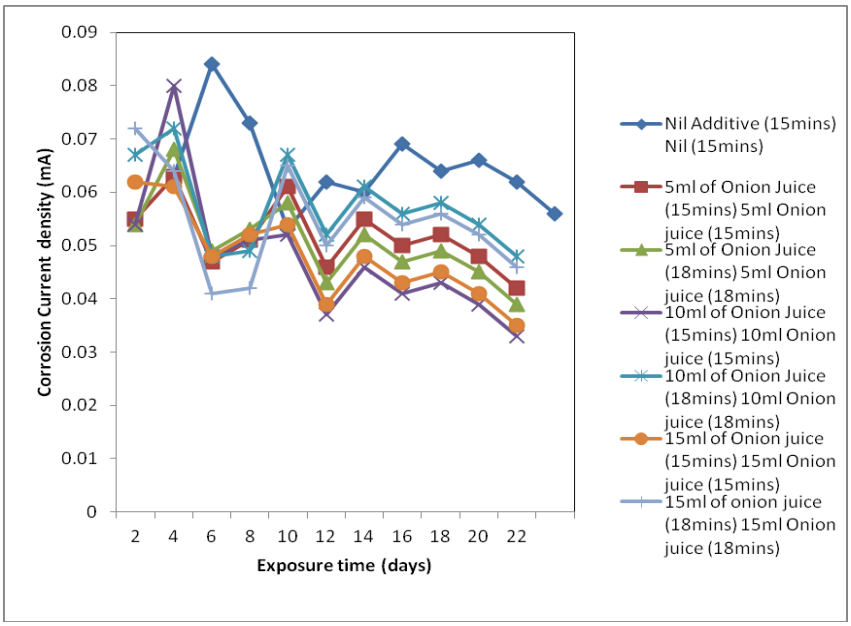


Fig. 11. Curves of corrosion current density with the exposure time for the zinc plated mild steel in 0.5 M HCl.

and brightening agents presented in figure 10 are the results obtained for variation of potential with the exposure time for the zinc plated mild steel in 0.5 M HCl with variable plating time of 15 and 18 minutes and 5, 10 and 15ml / 50 ml acid chloride solution concentrations of onion juice extract additive. All the results appeared to be in the active corrosion reactions potentials ranging between -0.4570 V (-457 mV) and -0.6170V (-617mV) while maintaining an almost steady state corrosion reactions phenomena but with minimal potential fluctuations. Plausibly, the close results could be associated with the anodic dissolution of the plated zinc in the acidic test medium.

The zinc corroded to protect the mild steel substrate by cathodic reactions process. There was no particular or significant effect of the plating time variation and/or the additive concentration effect, exhibited from the potential measurement values. The additives function mainly to enhance surface microstructural levelling and as

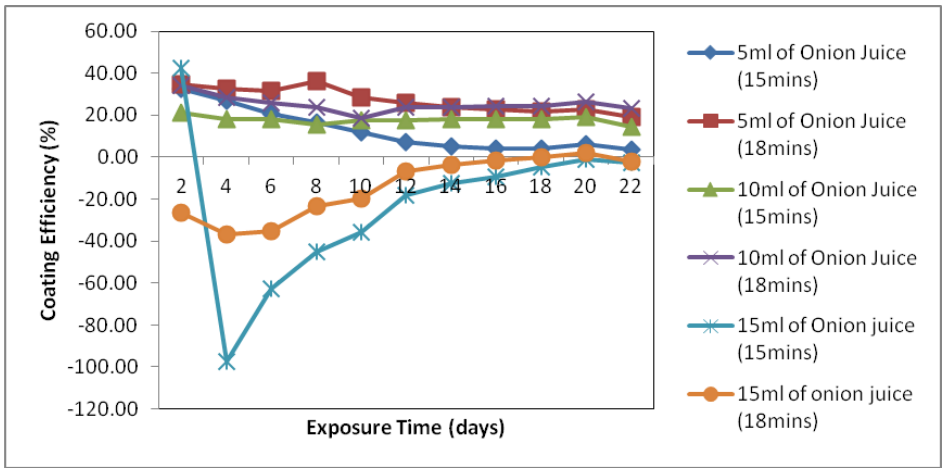


Fig. 12. Plot of corrosion current density with the exposure time for the zinc plated mild steel in 0.5M HCl using different concentrations of onion juice.

brightener for the zinc electrodeposit on the metal substrate thus improving the surface electroplating quality.

Results obtained for the plot of corrosion current density versus the exposure time for the zinc plated mild steel in 0.5 M HCl with variable plating time of 15 and 18 minutes and 5, 10 and 15ml/50 ml acid chloride solution concentrations of onion juice extract additive are presented in figure 11. Just like in a previous report (Loto *et al*., 2014), the general trend of the corrosion reactions was that of reducing corrosion current density from the beginning with time of exposure throughout the experimental period and with the fluctuations of the corrosion current density values. This trend of results was an indication of reducing corrosion rates reactions with the samples exposure time. The results obtained here showed clearly that the plating without additive had the highest corrosion current density and hence the highest corrosion rate. In addition, the lowest corrosion current density and hence the lowest corrosion rate were achieved with the 15ml / 50 ml acid chloride solution of onion juice extract concentration at both the 15 and18 minutes plating time respectively. These trends of results are in agreement with the results obtained for corrosion rate by weight-loss method.

Figure 12 presents the relationship of coating efficiency with the exposure time for the plated test samples immersed in 0.5M HCl. At an efficiency that ranges between 42.48 and 23.52%, the juice extract additive concentration of 5 and 10ml / 50ml of acid chloride solution and the plating time of 18 minutes respectively, could be described as the most effective and hence providing the best electroplating quality in terms of levelling and surface integrity that is devoid of porosity. These result seemed to be out of trend for the results obtained for the corrosion current density and the corrosion rate from the gravimetric method. Relatively the lower and the average extract concentrations gave better coating efficiency.

It is important to mention that the results obtained for corrosion resistance performance of the samples bear some close correlation with the surface microstructure in the micrographs and also to the mass of zinc deposited on the plated portions. The more compact, levelled and smooth, the surface crystal particles of the plated samples are, the more the corrosion resistance and hence the less anodic dissolution of the plated zinc observed.

Visual inspection could not reveal any visible particle removed from the plated steel surface. The cellotape test confirmed the strong adhesion of the zinc to the steel surface.

CONCLUSION

Onion juice *(allium cepa)* extract as an additive, gave good quality zinc electroplating with compact, levelled, smooth and non- porous close-packed crystal grains on mild steel surface in the acid zinc chloride solution. Scanning electron microscopy examination of the mild steel zinc plated surface, showed different but good surface morphology, depending on the plating parameters. Good corrosion resistance of the tested samples achieved in the strong acid (HCl), when compared with the unplated samples confirmed the protective capabiliy of the zinc plated metal and by extension the positive effect of the onion juice extract.

An enirely new and different surface crystal strucure is produced from the variable plating parameters. The electrodeposition process was sensitive to changes in

additive concentration and plating time. The effectiveness of the onion juice extract additive was demonstrated by the bright zinc deposition obtained. *Allium cepa* extract additive used was environment friendly.

ACKNOWLEDGEMENT

The authors acknowledge the laboratory investigation contribution of Mr. Joel Egile Oniso, and also, the Department of Mechanical Engineering, Covenant University, Ota for the provision of research facilities for this work.

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Received: July 28, 2014 ; Revised: Nov 13, 2014;
Accepted: Dec 24, 2014