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Technical Report

Effect of *Saccharum officinarum* Juice Extract Additive on the Electrodeposition of Zinc on Mild Steel in Acid Chloride Solution

C.A. Loto [1, 2,*], *A. Olofinjana* [3], *A.P.I. Popoola* [2]

[1]Department of Mechanical Engineering, Covenant University, Ota, Nigeria
[2]Dept. of Chemical and Metallurgical Engineering, Tshwane University of Technology, Pretoria,
[3]Department of Mechanical Engineering, Universiti Brunei, Brunei Darussalam
[*]E-mail: akinloto@gmail.com

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Experimental investigations have been performed to examine the electrodeposition of zinc on mild steel in acid chloride solution using as additive different concentrations of *saccharum officinarum* (sugarcane) juice extracts. The experiments were performed under different plating time, different additive concentrations and fixed pH conditions. Zinc electrodeposition on mild steel was performed using a DC – supply at defined operating parameters. The surface of the plated steel was examined using scanning electron microscopy (SEM); and Energy Dispersive Spectroscopy (EDS) for surface elemental composition analysis. Different surface characteristics were obtained depending upon the concentration of the additive and the plating time. The corrosion resistance of the plated surface was also determined by gravimetric method. The quality of electrodeposition of zinc was good as indicated by the plated surface microstructural morphology.

Keywords: Electrodeposition, sugarcane, steel, acid chloride solution, corrosion, surface characteristics

1. INTRODUCTION

The growth in popularity of acid zinc-based baths in the past few years has resulted from the need to avoid the toxicity of cyanide –based baths and their costly effluent following stringent regulation against water pollution. Though commercially-available proprietary additives [1-4] have now been used for some years, the need to develop other environment –friendly non-commercial proprietary additives for the acid chloride bath has generated the present interest in further research. There is also the need to prevent corrosion and toxicity, and to enhance the aesthetic value of steel

components in the automotive, construction, electronics, electrical appliances, recreational and materials handling industries and in our daily lives. This has, in addition, led to an enlarged interest in the field of electrodeposition.

Surface characterisation of the effects of organic additives on the electrodeposition of zinc on mild steel and the influence of organic additives on the surface characteristics of zinc electrodeposition on mild steel in acid-chloride solution under different conditions have been demonstrated in recent works [5-7]. This article extends these previous investigations by further looking into the effect of plant juice extracts starting with the *saccharum officinarum* (sugarcane) under different experimental working parameters/conditions.

Non-cyanide zinc plating solutions can be divided into two types – mildly acid solution using chloride or sulphate anions, and alkaline-zincate solutions [8]. The mild baths generally consist of zinc chloride dissolved in solution of excess ammonium chloride. More recently, potassium chloride processes, which are far less corrosive, have been marketed and ammonia free-formulation is now the most popular in production [4]. Zincate baths consist solely of a small concentration of zinc metal dissolved in approximately 100g/l of sodium hydroxide solution [9]. Chloride zinc solution does not only eliminate cyanide in plating, it also gives improved bath efficiency and exceptional brightness. And zinc baths are used where it is desirable to have a high plating rate and low cost. Chloride zinc plating offers considerable advantages over cyanide-based systems, although it is not without its share of routine operating problems [3]. Use of the acid sulphate process is increasing due to its relatively low cost, safety features and pollution control characteristics, but throwing power and insufficient brightness from an acid sulphate bath are disadvantages [9]. Many other authors [10-13] have also reported in different areas of zinc and zinc alloys electrodeposition and on synergistic effect of electrodeposited alloys/ effect of addition agents and also on their corrosion resistance characteristics using different bath solutions.

In this work, the use of local plant, sugarcane (*saccharum officinarum*) juice as addition agent in zinc electrodeposition from acid based solution, makes this study unique. Sugarcane juice is obtained from the plant. The juice expressed from cane is an opaque liquid covered with froth due to air bubbles entangled in it. Its color varies from light grey to dark green, depending on the coloring matter in the rind of the cane crushed. It contains in solution all the soluble substances like sucrose, fine particles of bagasse, wax, clay (adhering to the cane), coloring matter and albumen. The proportion of albumen increase when unripe cane or green cane tops are crushed with the ripe cane. Chemically, it contains glucose and fructose, vitamin B2, potassium, etc. Sugarcane contains about 70% of water, in which sucrose & other substances are held in solution, forming about 88% by weight of juice in the stem [14, 15]. The remaining 12% represents the insoluble cane fiber component. Further characterisation and compositions are contained in Tables 1 and 2. Cane wax, cane fiber and starch, being insoluble in the juice, are removed along with those substances precipitated by lime. Cane wax, $C_{24}H_{50}O$, occurs as a whitish deposit on the exterior surface of the cane stem, close to each node. The cane juice has an acidic pH ranging from 4.9 to 5.5. The juice acidity corresponds to about 0.2%. About an equal quantity occurs in combination with the mineral bases as salts. Mineral salts are all derived from the soil, and constitute the incombustible ash of the plant. The total quantity of these salts does not exceed 1% by weight of the entire cane plant, proving that the sugarcane

removes less mineral matter from the soil than many other crops. Another major component of the juice is the colloids. The colloids are particles existing in a permanent state of fine dispersion and they impart turbidity to the juice. These colloids do not settle ordinarily unless conditions are altered. The juice is viscous owing to the presence of colloids as waxes, proteins, pentosans, gums, starch and silica. The gums belong to the polysaccharide group of carbohydrates. Sugarcane juice, considering its chemical constituents, is expected to exhibit electrochemical activity of enhanced quality zinc electrodeposition on mild steel. As an edible natural compound it is also very environment friendly.

Table 1. Composition of sugarcane juice [14]

PARAMETER	VALUE (%)
Water	70 to 75%
Sucrose	11 to 16% (avg. = 13.0%)
Reducing sugars	0.4 to 2%
Organic non-sugars	0.5 to 1%
Mineral matters	0.5 to 1%
Fiber	10 to 16%

Table 2. Composition of non-sugars in sugarcane juice [14]

Acids	Nitrogen Compounds	Coloring Matters	Other Organic Non-sugars	Mineral Matters
Organic Acids Glycolic Malic Oxalic Succinic Tannic	*Organic Compounds*- Albumin Albumoses Amines amino-acids nuleins Peptones Xanthene Compounds	*Soluble* anthocyanin, saccharetin	*Soluble* pectin gum (xylan)	*Mostly soluble* alumina, lime, magnesia, potash, soda, sulphur, chlorine
Inorganic acids- Phosphoric Sulphuric	Inorganic compounds of ammonia and nitrogen	Insoluble chlorophyll	Insoluble cane fiber, cane wax	Insoluble silicates

2. MATERIALS AND METHODS

Flat mild steel, SIS 14147, 0.1 cm thick, with a nominal composition of 0.038% C, 0.195 Mn and the remainder Fe, was cut into several test specimens of 10.0 cm long and 1.0cm wide. A portion

of 1.0 cm in length was marked off at one end for the electrodeposition of zinc. The test specimens were degreased ultrasonically for 5 minutes with an alkaline degreasing chemical – Henkel VR 6362-1, and then removed from the solution, rinsed in distilled water, immersed in methanol, and air dried. The specimens were, in turns, etched for 50 seconds in 3M HCl, rinsed in distilled water, immersed in methanol, air dried and stored in a desiccator for further experimental process.

The acid chloride solution for the electrodeposition consisted of ZnCl (71g/l), KCl (207g/l) and H_3BO_4 (35g/l). Extracted *Saccharum officinarum* (sugarcane) juice of varying concentration -4, 5, 6 ml/50ml of acid chloride solution (80;100;120ml/l) were used in turns as the addition agents (Table 3).

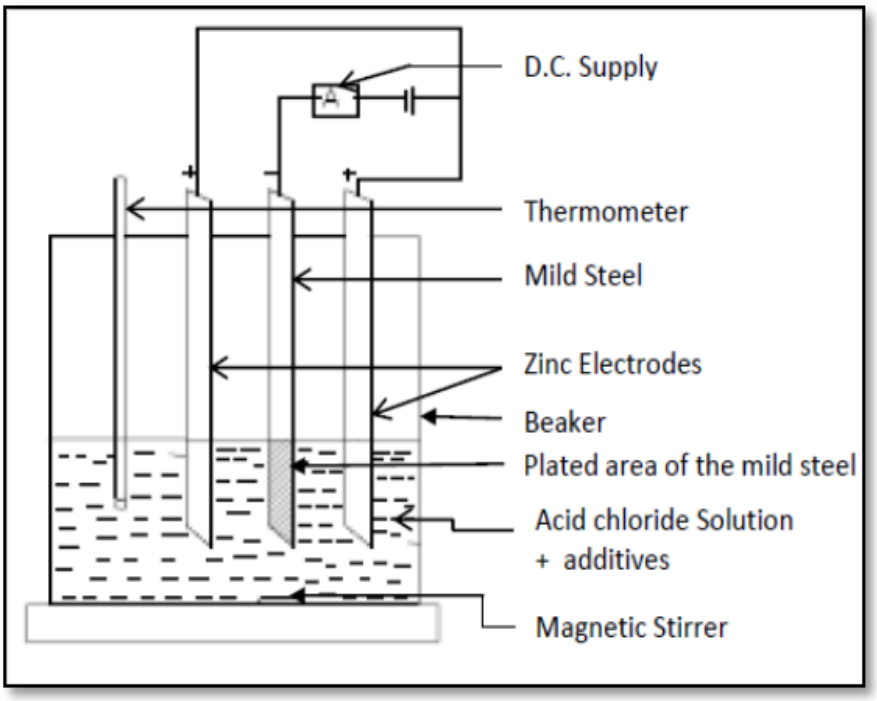


Figure 1. Schematic diagram of experimental set-up

Electrodeposition of zinc on steel was performed by partially immersing the steel specimen and the zinc electrodes in the plating solution (20mm deep) through the rectangular hole made on prepared Perspex cover for the 250ml beaker used as the plating bath. The steel specimen was connected to the negative side of a DC supplier while the zinc electrodes were also connected with a wire to the positive side, Fig. 1. The plating solutions were put in turns into the beaker and their respective pH was obtained by adjusting the original solution with potassium hydroxide. The plating times used for each bath were 15 and 18minutes. The weight of the steel specimen was taken before and after the electroplating process in order to determine the weight of zinc deposit by finding the difference between both weight readings, (Table 4). 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.

2.1. SEM/EDS characterization

A scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) was used to examine the surface topology and morphology of each of the plated test specimens. A small portion of each of the specimens was cut and mounted on a stub. The specimens were examined in turn in the SEM, and electron micrographs were made of the representative areas of the surface at different magnifications. The EDS analysis was also done to determine the composition of the surface of the plated metal.

2.2. Adhesion test

The adhesion of the zinc coating to the steel substrate was tested by using cellotape fastened to the surface and later pulled off and visually observed for any zinc stripping from the plated steel's surface. The plated surface was scratched with a scalpel to test for the zinc adhesion. The specimens were too small for a bending test.

2.3. Corrosion resistance testing of electroplated specimen

Corrosion resistance of the electroplated mild steel was tested in sea water gravimetrically. The water was obtained at the Alpha Beach, Lekki, Lagos, Nigeria from the Atlantic Ocean. Each of the test plated mild steel test specimens was partially immersed in the seawater test environment. The seawater was topped up to replace the amount lost due to evaporation. Weight Loss measurements were taken every two days for a period of 24 days. Corresponding corrosion rates values were determined from these weight loss values by calculation using this formula:

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

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

Table 3. The bath addition agent and concentration used

Additive	Quantity/50ml of acid chloride	% Concentration
Sugarcane	a. 4.0ml	8
	b. 5.0ml	10
	c. 6.0ml	12

Table 4. Mass of zinc deposited on steel substrate during plating

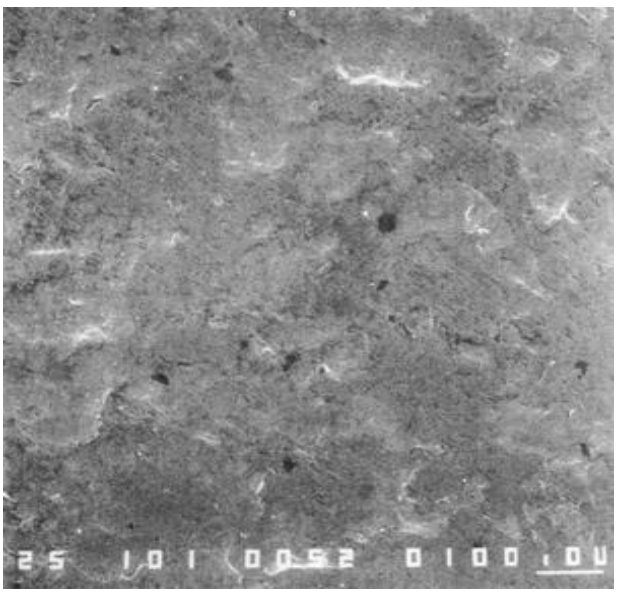
Sample	Mass Deposited (g)
G2	0.025
H2	0.0298
I2	0.0265
K1	0.0409
L2	0.0334

3. RESULTS AND DISCUSSION

It is important to state here that the major variables that will describe and explain the results obtained in this work will be the electrodeposition time effect and the amount of the sugarcane extract additive used in the acid chloride solution.

3.1. Electrodeposition of zinc in acid chloride solution without additive

The SEM micrograph of the surface of the mild test samples before zinc electrodeposition is presented in Fig. 2(i). Electrodeposition of zinc on mild steel test samples from acid – chloride solution without any additive showed, curiously, very little apparent porosity. The crystals were not clearly defined in shape with some coarse but closely packed. The surface microstructural feature was not so smooth morphologically. The implication of this for the plated metal's surface integrity in corrosive environment seems difficult to predict.



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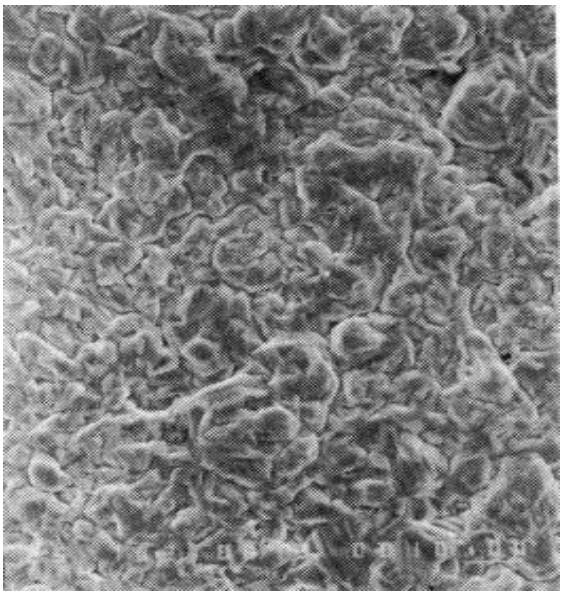

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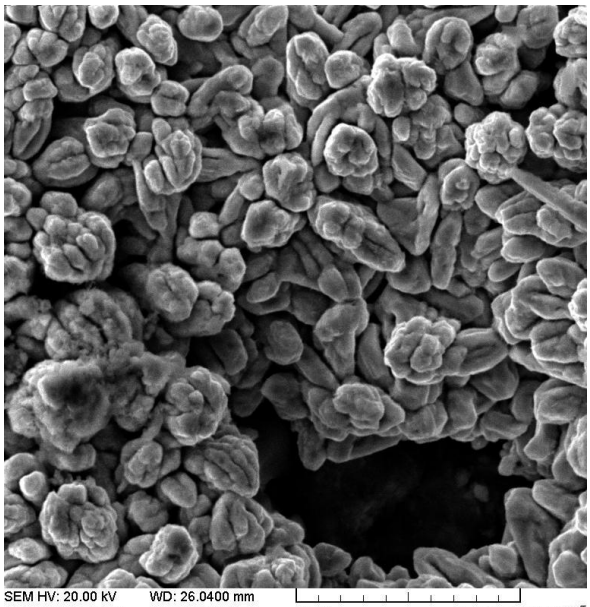

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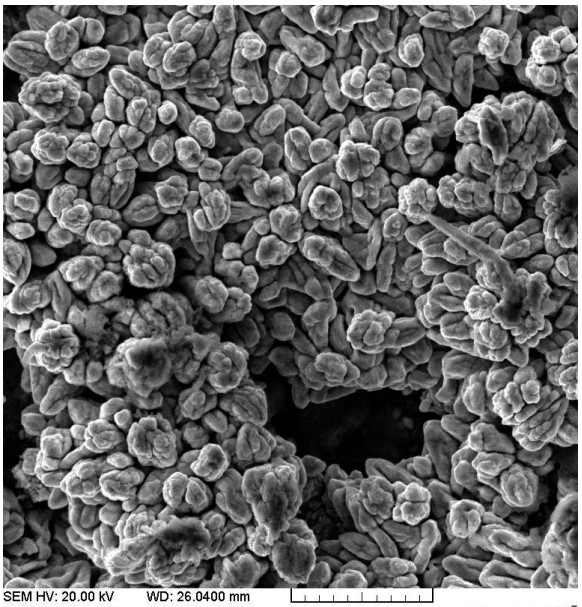

(iv)

Figure 2. SEM micrographs of mild steel test sample: (i) Before zinc electrodeposition (ii) Plating without additive; (iii) and (iv), plating with 4ml /50ml of acid chloride solution at 15 min. and at the Magnification of: x5000 and x3000 respectively.

However, this observation seems to indicate that zinc – electroplating of ferrous metals in acid chloride solution without any additives may not be wholly protective to corrosion attack. The slightly coarse structure could be due to the absence of levelling agents in the acid solution. The viability of this type of zinc plating will certainly depend on the conditions under which the experiments are performed. The observed amorphous crystal structure of the plated sample surface could be due to the poor throwing power of the acid solution.

3.2. Addition of Sugarcane (saccharum officinarum) juice extracts

3.2.1. 4ml sugarcane juice/50ml of acid chloride solution at 15 minutes plating time

With 4ml /50ml of acid chloride solution at 15 minutes plating time, Fig. 2 (iii) and (iv) a very well defined round crystal structure could be observed. While the plating time remained the same with the plating without added additive, the difference here is the addition of the juice extracts and the drastic change in the surface structure is very significant. This unique structure, which is evidence of good zinc electrodeposition, arises mainly from the addition of the sugarcane (*saccharum officinarum)* juice. In spite of the very high magnification of the SEM used, a closed crystal structure could be seen. However, at the same time a porous defect could be seen that indicated the poor throwing power characteristic of the acid solution bath. Within the plating period, a mass of 25mg zinc was deposited on the steel substrate, Table 4.

3.2.2. 4ml sugarcane juice /50ml of acid chloride solution at 18 minutes plating time

The variation in plating time yet produced another apparent and significant surface structure which is totally different from the previously discussed, Fig.3 (i-iv).

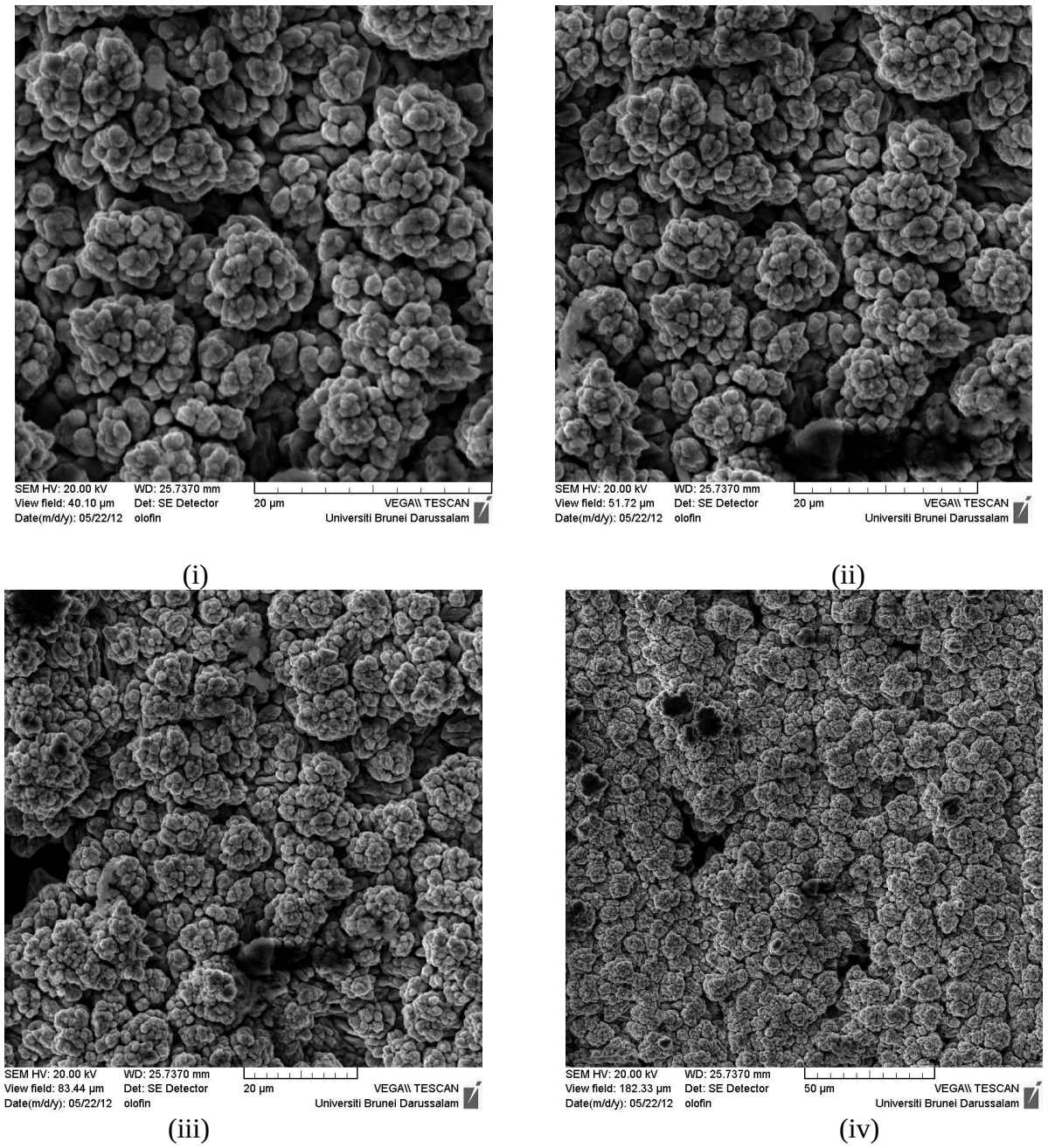

Figure 3. SEM micrographs of steel surface after zinc electrodeposition with 4ml /50ml of acid chloride solution at 18 min. (i) x7000; (ii), (iii) and (iv) are x5000, x3000, and x1000 magnification respectively.

Different magnifications of SEM micrographs that ranged from x1000 to x7000 are presented here. A not well –defined surface structure at the magnification of x1000 gave rise to a mushroom-/

broccoli-like structure at the very high magnifications of x3000, x5000, and x7000. A shift in the plating time from 15 to 18 minutes gave rise to this significant zinc electrodeposition feature. The implication then, is that zinc electrodeposition on mild steel in acid solution bath is very time sensitive/dependent. It could also be said that the addition agent worked differently with electrodeposited zinc on mild steel substrate as the plating time varies. Just like in Fig. 2, some porosity defects were observed but not significant. A densely packed surface structure, as just seen in Fig.3, is expected to give appreciable corrosion resistance performance. The brightening effect of the added juice extract was good. The mass of zinc deposited was weighed to be 29.8mg, Table 4. That sugarcane juice is effective as an addition agent in zinc electrodeposition is not unexpected when its complex chemical compositions are considered. As presented in Tables 1 and 2, chemically, sugarcane juice consists of sucrose\e and fructose, Vitamin B2, potassium and many others. The non-sugars in the juice composition are complex combinations which in no doubt will, together with sucrose, fructose and others, enhance the electroplating effectiveness and activity by synergism.

3.2.3. 5ml sugarcane juice /50ml of acid chloride solution at 15 minutes plating time

A change in the juice additive concentration from 4ml to 5ml sugarcane juice extract/50 ml of acid chloride solution at 15 minutes provided another different surface feature/characteristic as presented in Fig. 4 (i – iii). A close look at the surface microstructure shows it to be very fine granulated crystals clustered together and very well tightly packed. At the magnification of x1000, there was no apparent porosity at all, but at higher magnifications of x3000 x5000, some very little porosity could be seen. The surface morphology shows it to be a very good electrodeposition of zinc on the mild steel steel substrate. This apparent result confirms the improvement in the quality of the electrodeposition of zinc with increased amount of the juice extract additive used. It means that the increased concentration of sucrose, fructose and all the other chemical constituents in the juice extracts synergistically combine to give a good plating with improved brightness. The very little porous defects indicates an improvement in the acid solution throwing power that was necessitated by the increased addition agent concentration. The chemical reactions sensitvity of the plating process was also obvious in this regard. The mass of zinc deposited was 26.5mg. Though slightly less than the former, the coverage here was near perfect. In addition, the plating time was less – 15 minutes instead of 18 minutes of the former. It is expected that an improved corrosion resistance would be obtained in this plating.

3.2.4. 6ml sugarcane juice /50ml of acid chloride solution at 15 minutes plating time

SEM micrographs of steel surface after zinc electrodeposition with 6ml of sugarcane juice extract/50ml of acid chloride solution at 15 min are presented in Fig. 5 (i – iii) at different magnifications of x8000, x5000, and x3000 respectively. The surface feature once gain is totally different from each and every one of the above discussed Here the surface crystals are predominantly round in shape and interspersed with rod-like shaped crystals and all well - defined. The crystals are

closely packed even at the very high magnifications of x5000 and x8000. Porosity defect was minimal. The mass of zinc deposited was 40.90mg. The increased amount of the added additive and the increased concentration of the reacting additive chemicals would have contributed significantly to the present surface crystal structure which could be considered to be a very good electrodeposition of zinc on the mild steel substrate. The porosity at

the upper edge of Fig.5 (iii) could be associated with the characteristic throwing power of the acid solution bath that is normally relatively poor.

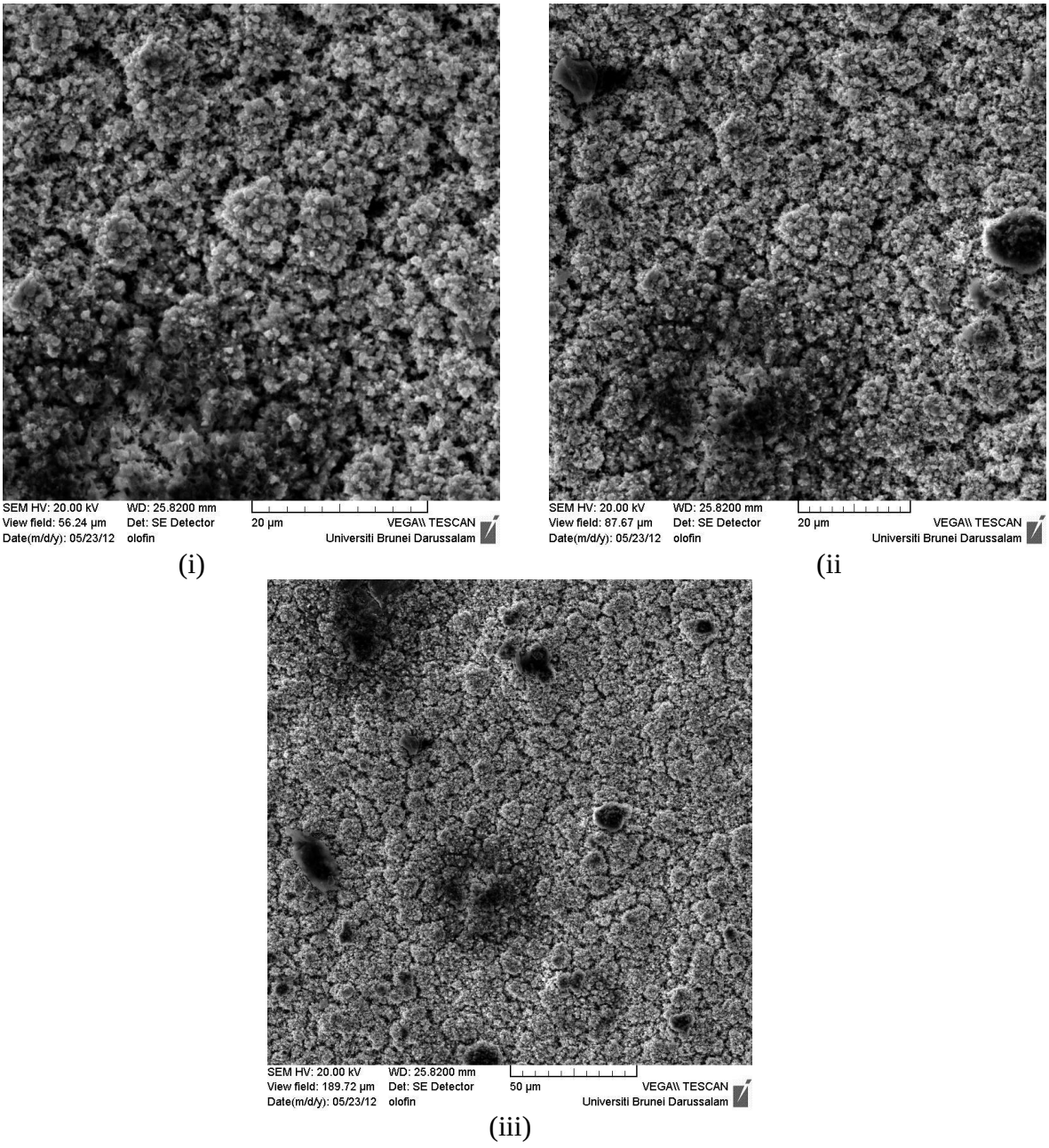

Figure 4. SEM micrographs of steel surface after zinc electrodeposition with 5ml /50ml of acid chloride solution at 15 min. (i) x5000; (ii) and (iii) are x3000, and x1000 magnification respectively

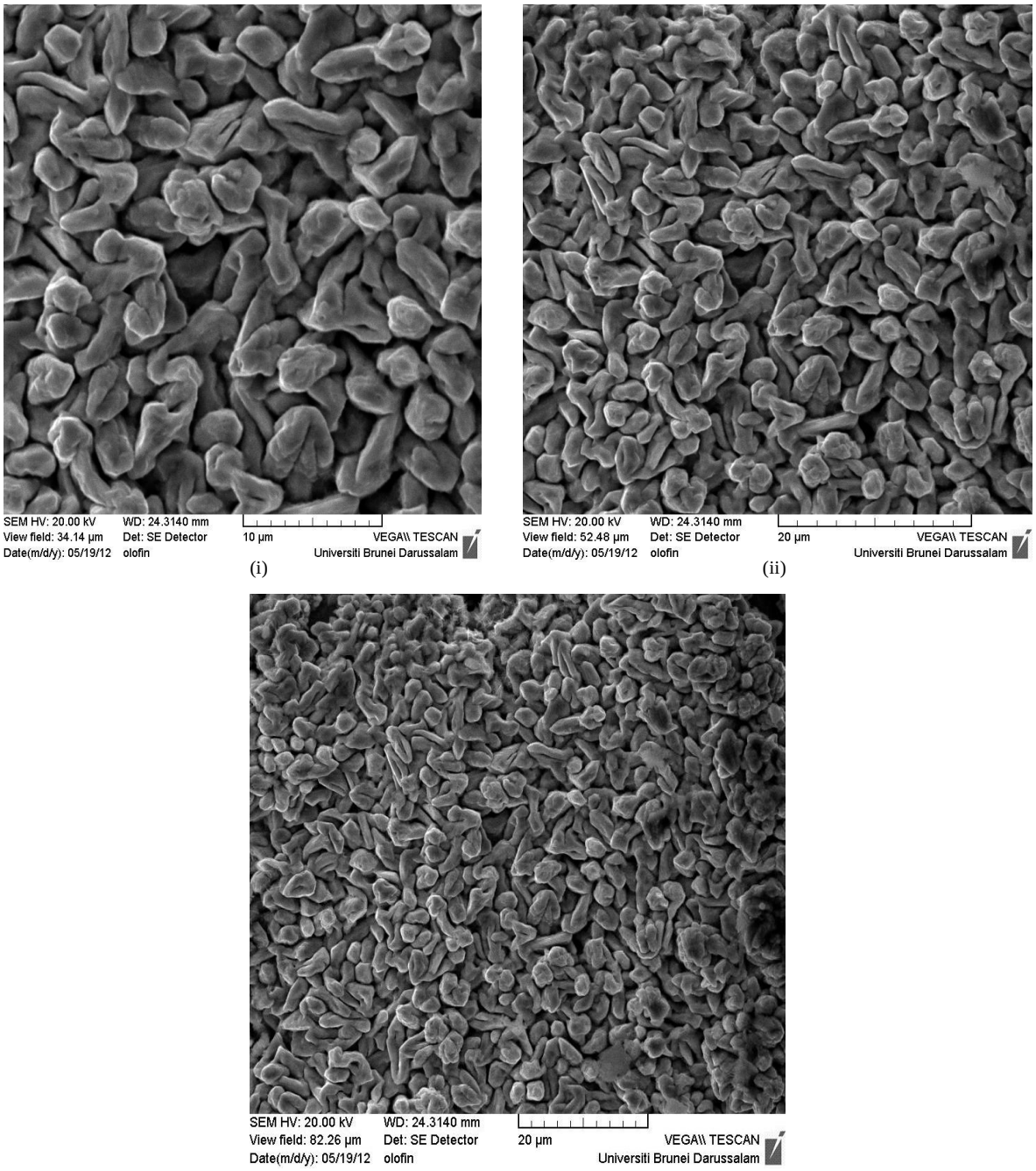

Figure 5. SEM micrographs of steel surface after zinc electrodeposition with 6ml /50ml of acid chloride solution at 15 min. (i) x8000; (ii) and (iii) are x5000, and x3000 magnification respectively

3.2.5. 6ml sugarcane juice extract /50ml of acid chloride solution at 18 minutes plating time

Presented in Fig. 6 (i and ii) are the SEM micrographs for the mild steel surface after zinc electrodeposition with 6ml of sugarcane juice extract /50ml of acid chloride solution at 18 minutes plating time. Though these micrographs were made at very high magnifications of x5000 and x7000 respectively, the fine microstructure and uniform distribution of zinc crystal particles all over the steel substrate surface with minimal porosity, is apparent. A fairly bright zinc deposition was obtained. The

mass of zinc deposited on the plated portion was 33.40mg. Both the increased volume/concentration of the additive and the increased plating time combined to give a good electrodepostion of zinc on the steel. The surface microstructure could be seen again to be different from all the rest above thus confirming both the additive concentration and plating time effects on the plated surface morphology. The complex chemical composition of the non-sugar content of the juice, in addition to its sucrose and fructose content, just as previously mentioned above, would have combined by synergism to produce this unique plating feature.

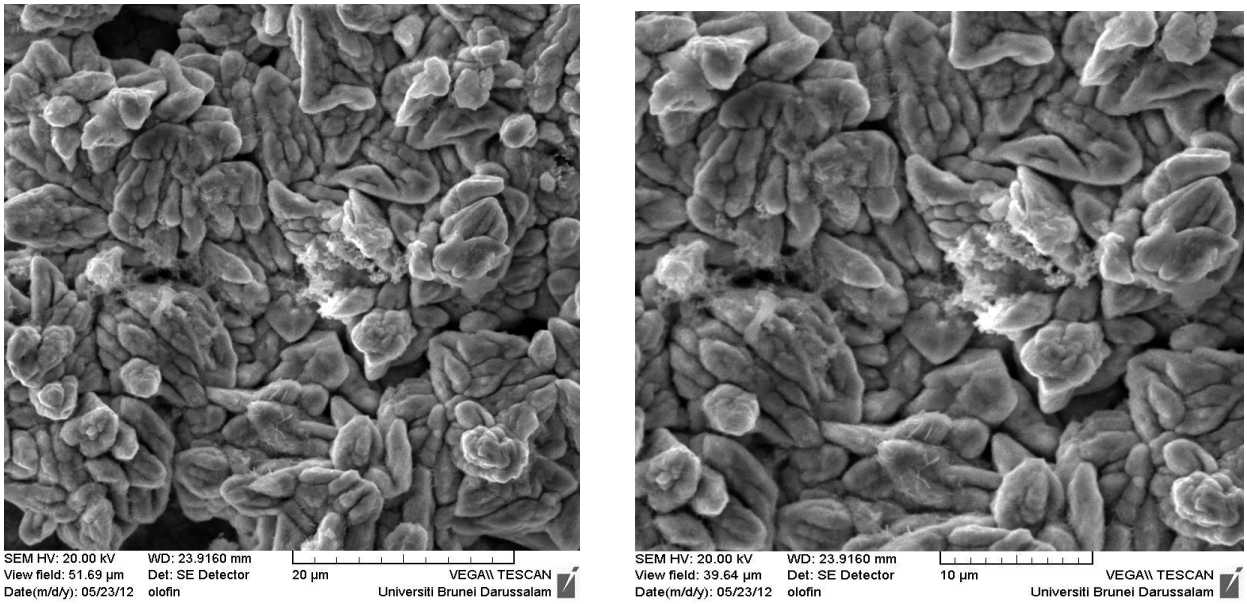

Figure 6. SEM micrographs of steel surface after zinc electrodeposition with 6ml /50ml of acid chloride solution at 18 min. (i) x5000 and (ii) x7000 (iii) magnification respectively

3.4. SEM/EDS Analysis

The result of energy dispersive analysis (EDS) of Fig. 3 (iv) is presented below as Fig. 11.; and a Table of analysis as Table 5. The surface microstructure showed it to be mainly zinc and a little amount of tin which could be in trace form in the zinc metal that was co-deposited.

Table 5. eds analysis of plated surface of sample in fig.3 (iv)

Element	App Conc	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
Zn K	12.33	1.0108	87.21	1.58	92.53
Sn L	1.51	0.8460	12.79	1.58	7.47
Totals			100.00		

3.5. Corrosion resistance of the zinc plated mild steel substrate

The results of the weight- loss method and the calculated corresponding corrosion rates of some of the zinc plated mild steel samples that were tested in the sea water environment are presented in Figs. 8 to 11. Fig. 8 shows the curves of the weight loss versus the exposure time at variable additive concentrations and at the plating time of 15 minutes for each of the test samples. All the plated samples showed better corrosion resistance than the unplated samples.For example,the sample plated with 6ml juice extract/50ml acid chloride solution recorded a weight loss value of 19.30mg on the 30^{th} day of the experiment while the unplated sample recorded a weight loss of 39.70mg at the same period of 30 days of the experiment. However, the trend of corrosion resistance performance did not, in general, followed the amount of the juice extracts used as additive; the sample plated with 4ml juice extract/50ml acid chloride solution showed less weight loss (19.30mg) than the one plated with 5ml extract /50ml acid chloride solution (31.30mg) on the 30^{th} day of the experiment. This could be an experimental abnormality; the other results followed the expected results of corrosion resistance performance. The weight loss recorded for the plated samples was due mainly to the anodic zinc dissolution in the test environment after a long period of 30 days.

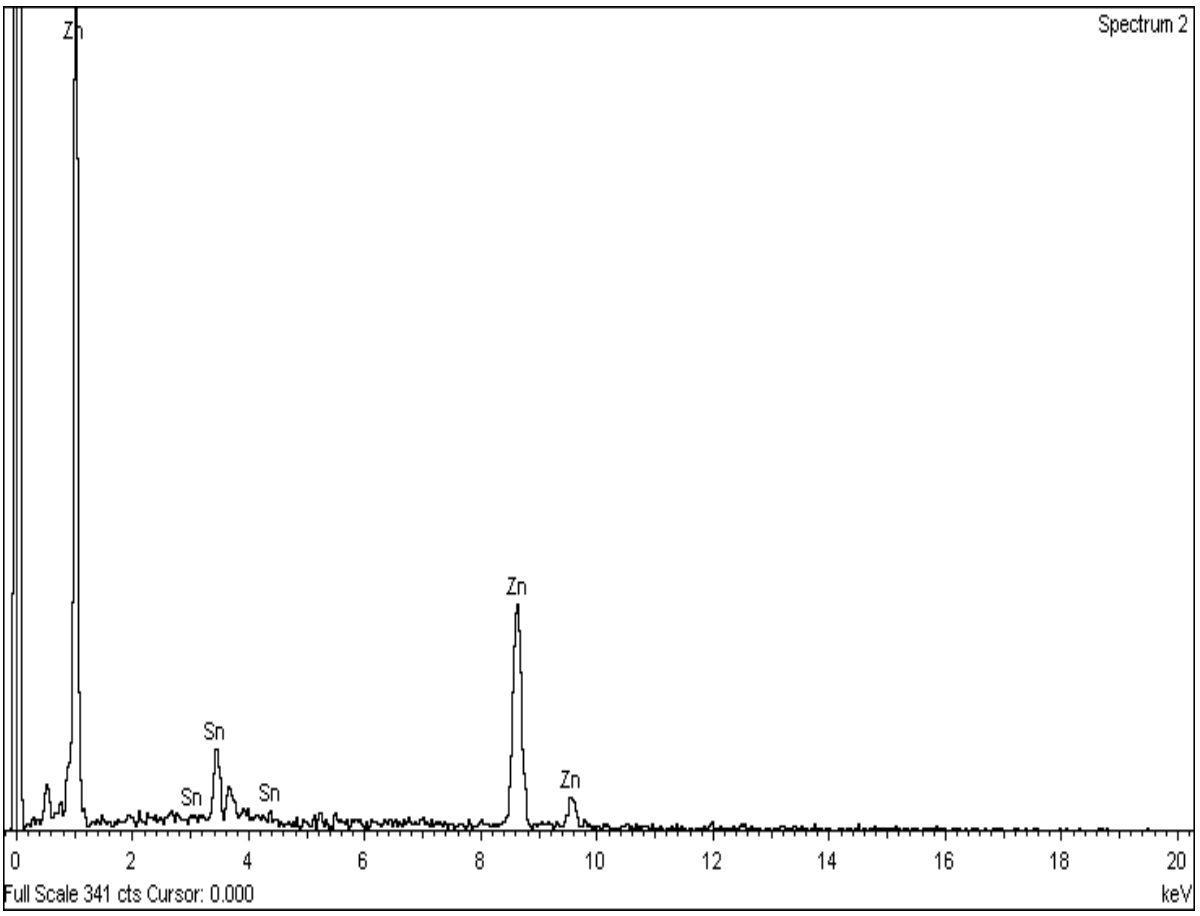

Figure 7. EDS analysis of the plated surface of sample in Fig.3 (iv)

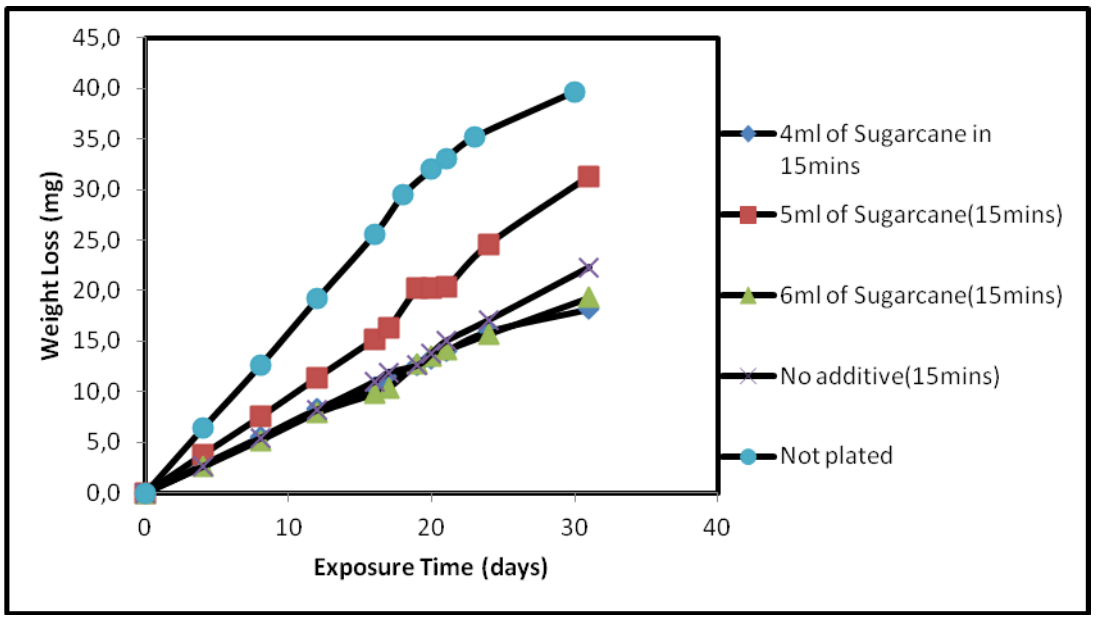

Figure 8. Variation of weight loss with exposure time for the zinc electrodeposited mild steel - sample in seawater. (Variable additive concentrations and 15 minutes plating time)

The corresponding corrosion rates curves, Fig.9 followed the same trend as above. The unplated test sample recorded the highest corrosion rate (0.2635 and 0.2127mm/yr) on the 18th and 30th day of the experiment respectively; while the sample plated with 4ml juice extract/50ml acid chloride solution recorded the corrosion rates of 0.1064 and 0.1066 mm/yr at the same peeriod of the experiment respectively. The difference in corrosion rate is significant.

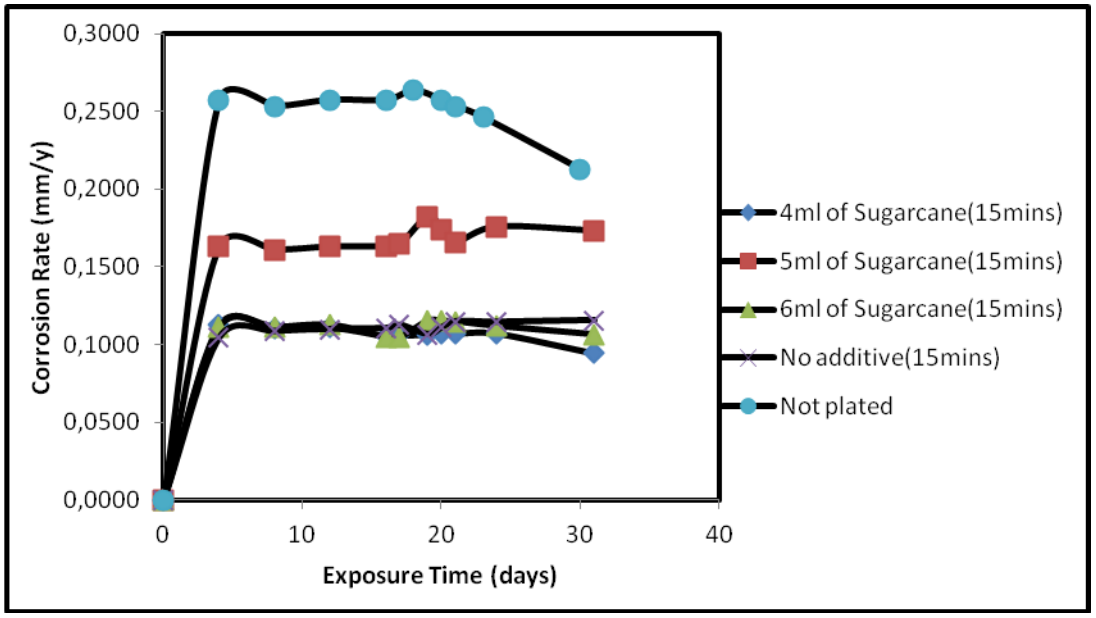

Figure 9. Variation of corrosion rate with exposure time for the zinc plated mild steel samples in seawater. (Variable additive concentrations and 15 minutes plating time)

While other plating parameters remain the same, the results presented in the curves in Figs. 10 (weight loss) and Fig.11 (corrosion rates) were obtained with the plating time of 18 minutes.

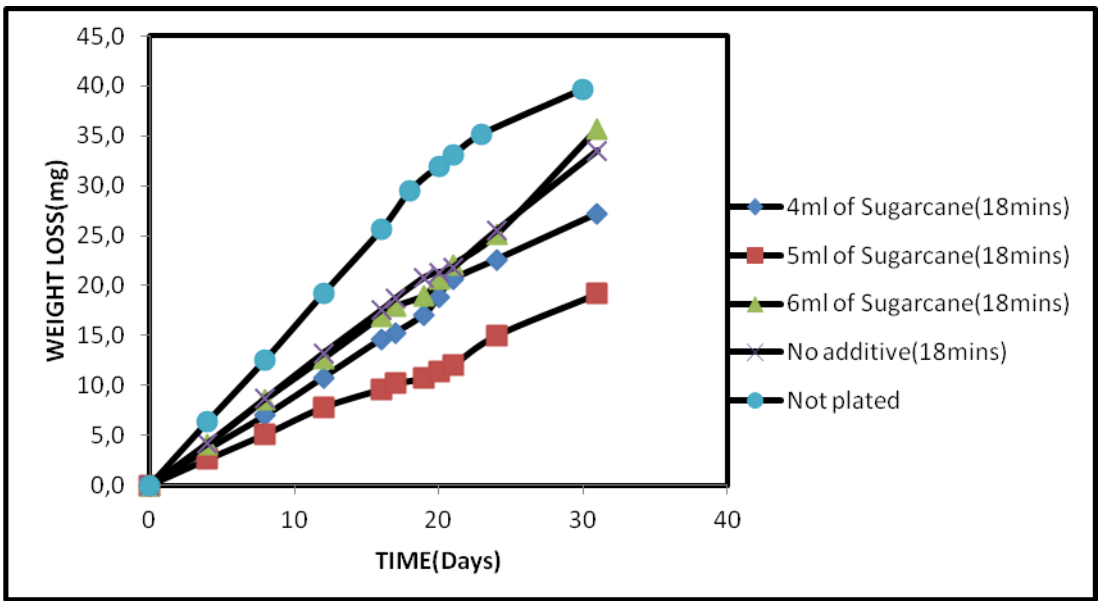

Figure 10. Variation of weight loss with exposure time for the zinc plated mild steel samples in seawater. (Variable additive concentrations and 18 minutes plating time)

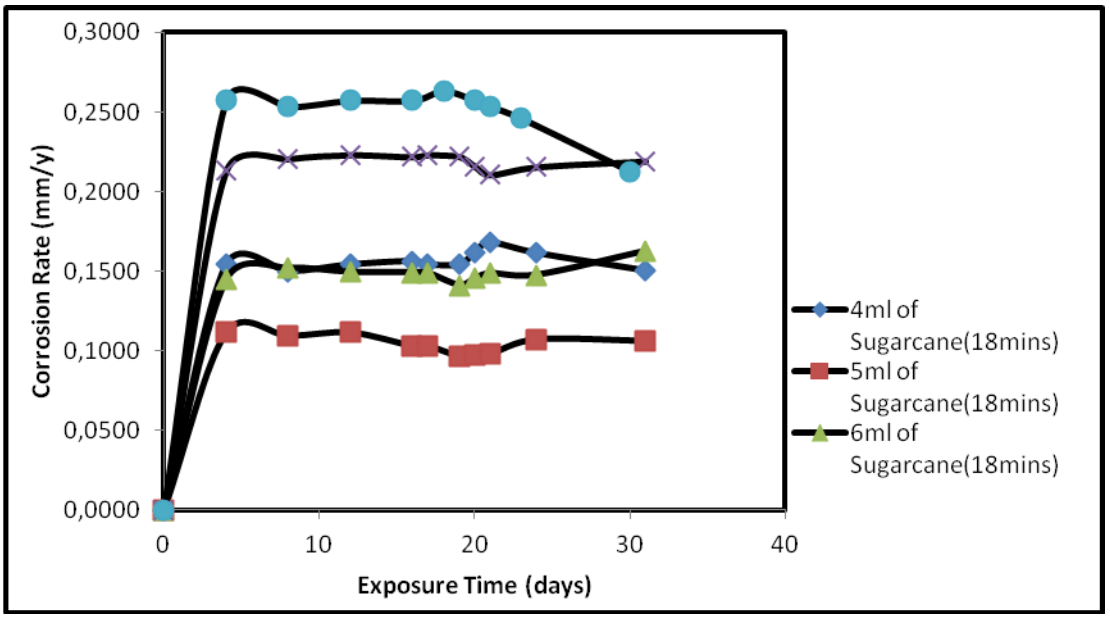

Figure 11. Variation Of Corrosion Rate With Exposure Time For The Zinc Plated Mild Steel Samples In Seawater. (Variable additive concentrations and 18 minutes plating time)

The trend here was as in Fig. 8 for the weight loss values; however, the samples plated with 5ml juice extracts (as additive) gave the lowest weight loss (19.20mg) on the 30th day of the

experiment. The unplated sample recorded a weight loss value of 39.70mg at the same period of the experiment while with the use of 4ml and 6 ml juice extract /50ml aci chloride solution the weight loss values were 27.20 and 33.50mg respectively. The corrosion rate for the sample with 5ml juice extract additive frecorded 0.1062mm/yr; with 4ml and 6 ml juice extracts, the recorded corrosion rate values were the sameat 0.1626 mm/yr. The unplated sample and the one without additive respectively recorded the same value of corrosion rate of 0.2127 at the 30^{th} day (end) of the experiment. In all, the plated samples were more corrosion resistant and hence more protective.

It is important to mention that the results obtained for corrosion resistance performance of the samples bear very close correlation with the surface microstructure in the micrographs and also to the mass of zinc depodsited on the plated portions. The more compact the surface crystal particles; the finer the crystal structure and the less amount of porosity in the plated samples, the more the corrosion resistance observed.

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

4. CONCLUSIONS

1. A good zinc electrodeposition on mild steel surface could be obtained in the acid zinc chloride solution using the sugarcane (*Saccharum officinarum*) juice as the addition agent.
2. Charactrerisation of the zinc plated surface of the mild steel substrate showed different surface features depending upon the plating conditions.
3. The electrodeposition process was very additive concentration and plating time sensitive. Any variation produced an enirely new and different surface crystal strucure.
4. The plated samples were found to exhibit good corrosion resistance in seawater test when compared with the unplated samples and thus confirming their protective capabiliy as expected.
5. The plating produced very fairly bright deposition. Though not as bright as the cyanide bath, the surface structures obtained indicate that the plating can serve several useful protective purposes that could be technologically and economically viable.
6. The additive used was an edible agricultural product that is environment friendly.

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