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Research Article

A STUDY ON THE EFFECT OF PHARMACOLOGICALLY ACTIVE *LAWSONIA INERMIS* LINN. LEAF EXTRACT ON ZINC ELECTRODEPOSITION ON MILD STEEL

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ABSTRACT

Lawsonia inermis L. is a much branched shrub or small tree commonly known as henna which invites attention of the investigators worldwide for its pharmacological profile ranging from anti inflammatory to anti cancer activities. The present study is carried out to find the effect of Lawsonia inermis L. (henna) leaf extract (primary colouring agent is 2-hydroxy-1,4-naphthoquinone) on electrodeposition of zinc on mild steel. The effect of bath constituents, pH, current density and temperature on nature of deposits are studied through Hull cell experiments. The bath constituents and operating conditions are optimized. The increase in the current efficiency and throwing power reveals the action of henna on electrodeposition. Polarisation, SEM, EDAX and XRD photographs obtained from optimum bath reveal fine grained deposit in the presence of extract and hence modify the morphology of zinc deposit. IR spectrum of the scrapped deposit shows the inclusion of the addition agent.

Keywords: Carbon steel; Additive; Lawsonia inermis Linn; 2-Hydroxy-1,4-naphthoquinone; Hull cell test; SEM.

INTRODUCTION

Acid zinc-based baths have grown in popularity in the past few years. This phenomenal growth has resulted from the need to avoid the toxicity of cyanide-based baths and their costly effluent following stringent regulation against water pollution. For some years, the commercially-available proprietary additives have been used [1–4]. However, the present interest in further research necessitates the need to develop other environment-friendly non-commercial proprietary additives for the acid sulphate baths. The importance of electrodeposition (electroplating) in engineering products/facilities and in our daily lives is further made significant by 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. In recent works [5–7], surface characterization 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 were performed. The use of Lawsonia inermis leaf extract under different experimental working parameters/conditions in this study is an attempt to further extend these previous Investigations. Two types of non-cyanide zinc plating solutions are in use—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]. Zinc baths are used where it is desirable to have a high

plating rate and low cost. 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. The use of local plant, henna leaf extract, as addition agent in zinc electrodeposition from acid based solution, in this work, makes this study significant.

Lawsonia inermis Linn. most commonly known as 'Henna" invites attention of the investigators worldwide for its pharmacological profile ranging from anti inflammatory to anti cancer activities[14]. The principal colouring substance of henna is a red orange coloured molecule (lawsone, 2-hydroxy1,4-napthoquinoine) having molecular formula, C10H6O3 and M.P. 190o, present in dried leaves in a concentration of 1–1.4%w/w [15, 16, 17]. Upadhyay et al in 2010 confirmed that the quantitative estimation of leaves of L. inermis collected in different seasons showed variations in the active ingredient (lawsone) [18]. Aqueous extract of Henna is also used in corrosion and it is best inhibitor for iron in HCl acid and in aqueous solution containing 60 ppm of Cl– ion [19]. Thus it is expected to exhibit electrochemical activity of enhanced quality zinc electrodeposition on mild steel. It is also very environment friendly and its successful use as an additive, in the improved deposition of zinc on mild steel, will be technologically and economically beneficial.

EXPERIMENT

The chemicals used were of AR grade and easily soluble in water. For the preparation of solutions, distilled water was used. The standard Hull cell of 267 mL capacity was used to optimize the bath constituents. The Hull cell experiments with bath solution (Table 1) were carried out without agitation. The pH of the bath solution was adjusted with 10% hydrochloric acid or sodium carbonate solution. Zinc plate of 99.99% purity was added as anode. The anode was activated each time by immersing in 10% HCl followed by water wash. Mild steel plates (AISI-1079) of standard Hull cell size were mechanically polished to obtain a smooth surface. The scales and dust on the steel plates were removed by dipping in 10% HCl and were subjected to electrocleaning process. These steel plates were washed with water and used for the experiments as such. After plating experiment, the plates were subjected to bright dip in 1% nitric acid for 2 s followed by water washes. The nature and appearance of zinc plating was carefully studied and recorded through Hull cell codes as shown in Fig. 1.

The deposits were obtained at a constant current density from the optimized solution taken in a rectangular methacrylate cell of 2.5 l capacity. Polished, degreased and electro cleaned cathodes of 3 X 4 cm2 were used for plating. Experiments were done in triplicate. Standard experimental procedures (Parthasarathy 1989) were adopted for measurement of properties of the deposit such as ductility, adherence etc. In all the above studies the average thickness of the deposit was 20 µm.

Polarization studies were carried out by using a three-compartment cell. The area of zinc anode was 2 cm2.Mild steel was used as cathode with an exposed area of 2 cm2. The cathode potential was recorded galvanostatically with respect to standard calomel electrode at different current densities. Haring and Blum cell was used to measure throwing power and the current distribution ratio between anode and cathode was 1:5. IR spectra of the scrapped deposit were taken to know the inclusion of addition agent. SEM photomicrographs were taken to know the nature of deposit in the presence of addition agents.

RESULTS AND DISCUSSION

Hull cell studies

Effect of ethanolic henna extract

Basic bath solution gave coarse dull deposit between the current density range of 1 and 2 A/dm2 at 1A cell current. To improve the nature of deposit, Henna Extract was added to the bath solution. With increase in the concentration, the nature of deposition improved and at a concentration of 80ml of Henna Extract, the Hull cell panels were bright between the current density range of 0.4 and 4 A/dm2.With further increase in the concentration of henna extract, the nature of the deposit became burnt at higher current density region. Therefore, on the basis of the above observations, the concentration of Henna Extract was kept at 80 ml/l as optimum. The Hull cell patterns are shown in Fig. 1.

Effect of zinc sulphate

To find out the effect of zinc ion, the zinc sulphate concentration was varied from 15–250 g/l keeping Henna Extract at 80 ml/l. At low current density region, dull deposits and at high current density range, burnt deposits were obtained (Fig. 2).With increase in the concentration of zinc sulphate, the brightness range was extended to higher and lower current density regions. At a concentration of 80 g/l, a satisfactory bright deposit was obtained. Above this concentration of zinc sulphate, no improvement in the nature of deposit was observed. The concentration of zinc sulphate was fixed at 80 g/l as optimum.

Effect of sodium acetate

Sodium acetate was added to increase the conductance of the bath solution. The concentration of sodium acetate was varied from 2-50g/l. At lower concentrations, the Hull cell panels showed semi bright deposit at low current density region and burnt at high current density region. The semi bright and burnt regions were found to be reduced with increase in the concentration of sodium acetate and at 35g/l, the deposit was bright over a current density range of 0.4–4 A/dm2.Further increase in the concentration (>35g/l) did not introduced any effect on the nature of deposit and on the conductance also. So, the concentration of sodium acetate was fixed at 36 g/l in the bath solution. The Hull cell patterns showing the effect of sodium acetate are given in Fig. 3.

Effect of aluminium sulphate

Aluminium Sulphate was added to increase the conductance of the bath solution. The concentration of sodium acetate was varied from 2–50 g/l. At lower concentrations, the Hull cell panels showed semi bright deposit at low current density region and burnt at high current density region. The semi bright and burnt regions were found to be reduced with increase in the concentration of sodium acetate and at 35g/l, the deposit was bright over a current density range of 0–4A/dm2. Further increase in the concentration (>35 g/l) did not introduced any effect on the nature of deposit and on the conductance also. So, the concentration of sodium acetate was fixed at 36 g/l in the bath solution. The Hull cell patterns showing the effect of sodium acetate are given in Fig. 4.

Effect of pH

To know the effect of pH, the pH of the bath solution was varied from 2-5.At higher pH, the Hull cell panels showed burnt deposit at high current density region. At pH 3.5, satisfactory deposit was obtained. At lower pH (<3.5), the specimens had dull deposit at low current density region. From the above observations, the pH of the bath solution was kept at 3.5 as optimum. The Hull cell patterns are as shown in Fig. 5.

Effect of temperature

To study the effect of temperature on Hull cell experiments, the plating experiments were carried out in a thermostat. The temperature of the thermostat was varied from 293-323K. At room temperatures (<303 K), the deposition was bright in the current density range 1-4Adm2 at 1A cell current. Above 303K, the deposit was dull in the low current density region. Therefore, the optimum operating temperature range was 303K.The Hull cell panels showing the effect of temperature are shown in Fig. 6.

Effect of current

The Hull cell experiments were carried out at different cell currents (1–3A) for 10 min using optimum bath solution. It was found that at a cell current of 1A the deposit was bright in the current density range 0.8–4 A/dm2. At a cell current of 2 A, the deposit was bright in the current density range of 2–4 A/dm2. At a cell current of 3 A the deposit was bright over the current density range between 3 and 5 A/dm2. The Hull cell patterns are as shown in Fig. 7.

Current efficiency

At lower current density (1 A/dm2), the current efficiency of zinc electrodeposited carbon steel obtained from basic bath and optimized bath were found to be 50 %, and 80% respectively. At a current density range of 1–4 A/dm2, the efficiency was found to be increased and reached high at 4 A/dm2. The Efficiency obtained were 70%, and 98% respectively. Further increase in the current density was found to decrease the efficiency (Table 3). This showed the absence of hydrogen evolution at a current density range of 1–4 A/dm2, after this hydrogen evolution was started.

Table 1. Basic bath composition and operating conditions

Bath composition	Conc	Operating conditions
$ZnSO_4$	240 g/l	Temperature: Room temperature
CH_3COONa	30 g/l	Anode: Zinc metal (99.99% pure)
$Al_2(SO4)_3$	30 g/l	Cathode: mild steel
pH	3.5–4.5	

All the experiments were conducted at room temperature. A known amount of Henna Extract was added to the bath solution. The bath solution was stirred for 30 min and then used for the Hull cell experiments.

Table 2. Optimum bath composition and operating conditions

Bath composition	Conc.	Operating conditions
$ZnSO_4$	80 g/l	Anode: Zinc metal(99.99%pure)
$Al_2(SO_4)_3$	36 g/l	Cathode: mild steel
CH_3COONa	36 g/l	Temperature:303K
Ehanolic	80ml/l	pH: 3.5
Henna extract		Plating time:10min
		Bright current density range:0.8–4 A/dm^2
		Cell constant in Ampere:1A

Table 3. Current efficiency at different current densities

Current density(A/dm^2)	Current efficiency(%)	
	Basic bath	Optimised bath
1	50	80
2	56	85
3	65	91
4	70	98
5	62	85

Table 4. Throwing power at different current densities

Current density (A/dm^2)	**Throwing power (%)**	
	Basic bath	**Optimized bath**
1	18.4	27.5
2	21.1	29
3	23.2	33.6
4	25.5	37.2
5	26.8	30.8

Effect of Henna extract

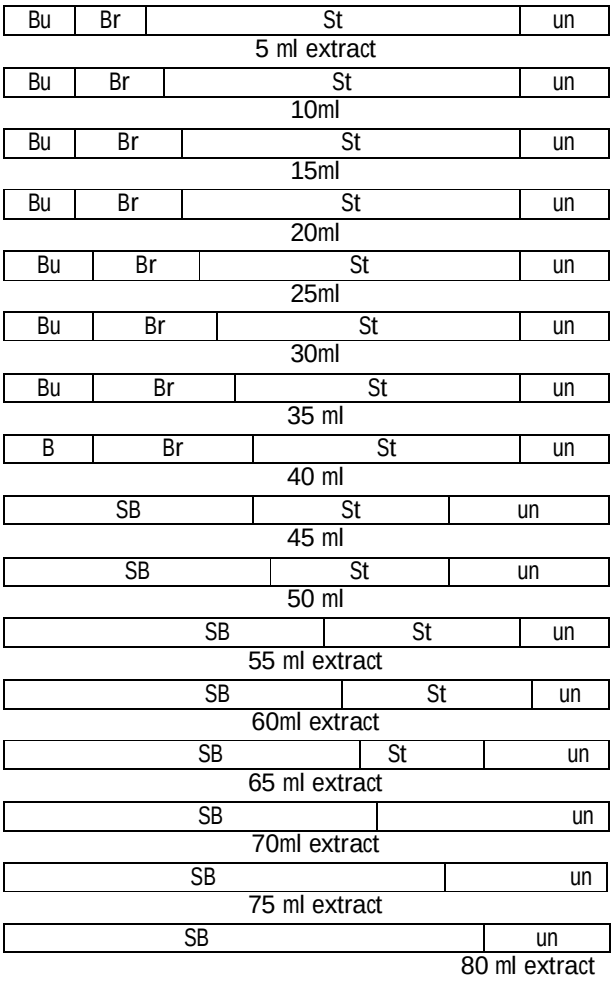

Fig. 1 Hull cell diagram: **Effect of henna extract**

Effect of Zinc sulphate 7.H_2O

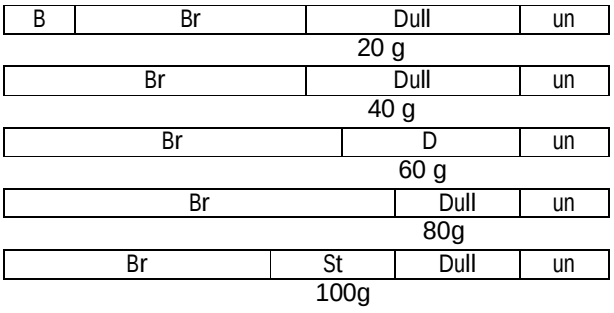

Fig. 2 Hull cell diagram: **Effect of $ZnSO_4$ $7H_2O$**

Effect of Sodium Acetate

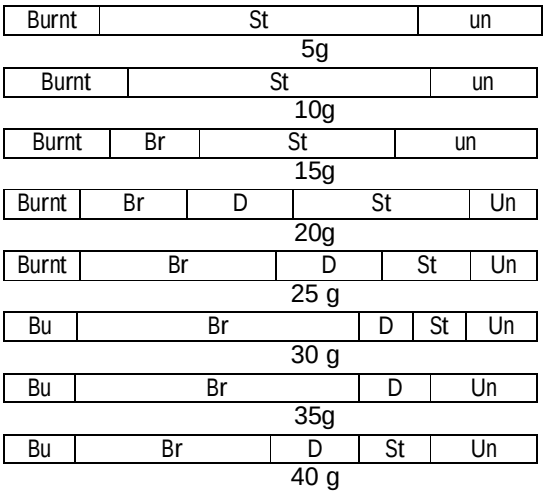

Fig. 3 Hull cell diagram: **Effect of sodium acetate**

Effect of Aluminium sulphate

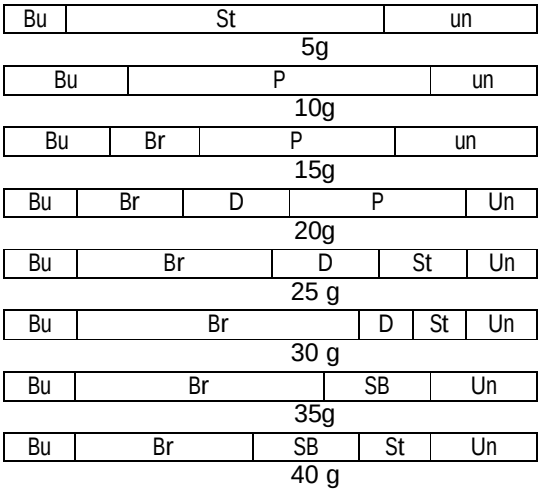

Fig. 4 Hull cell diagram: **Effect of aluminium sulphate**

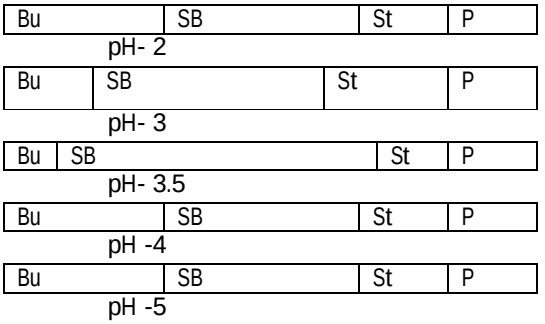

Fig. 5 Hull cell diagram: **Effect of pH**

Bu	SB	St	P

293K

Bu	SB	St	P

303K

Bu	SB	St	P

313K

Bu	SB	St	P

323K

Fig. 6 Hull cell diagram: **Effect of temperature**

Bu	Br

1A

Bu	SB	P

2A

Bu	SB	P

3A

Fig. 7 Hull cell diagram: **Effect of cell current**

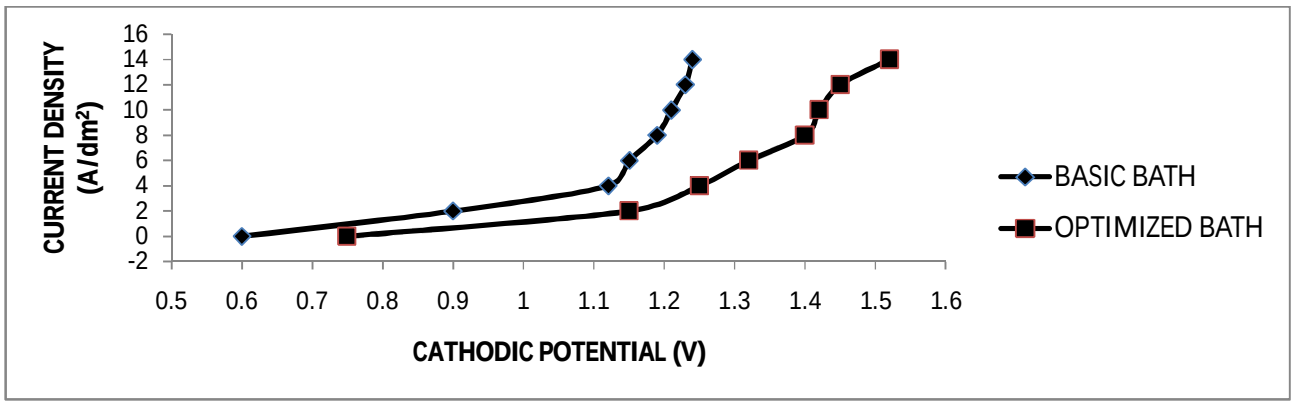

Fig .8 Effect of additive on cathodic polarization

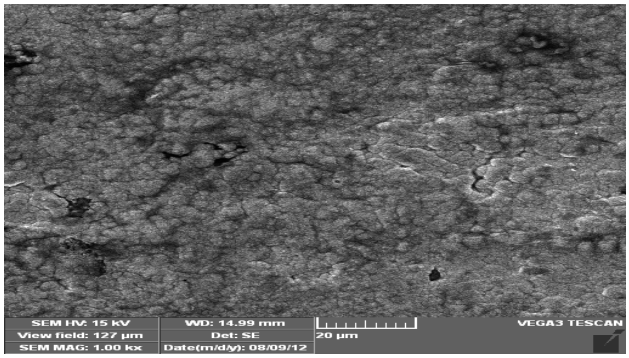

Fig. 9a SEM photo micrographs obtained in the absence of henna extract

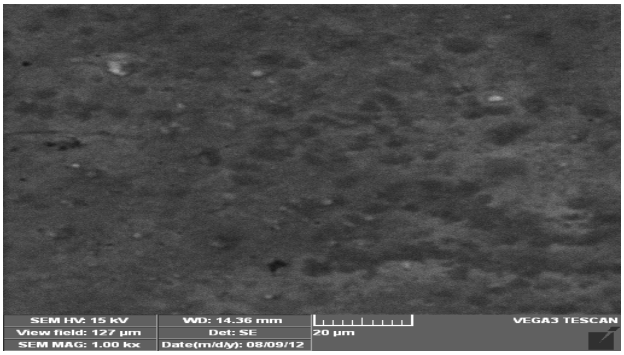

Fig. 9b SEM photo micrographs obtained in the presence of henna extract

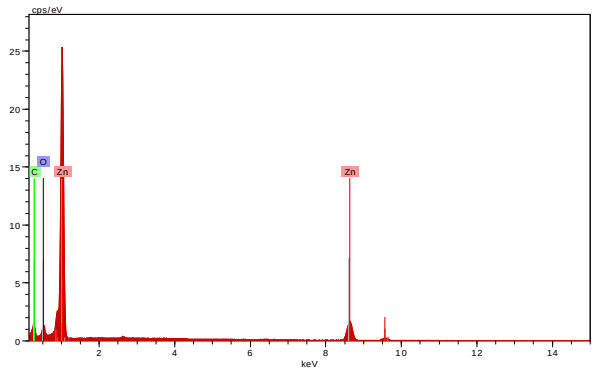

Fig. 10a EDAX photograph obtained for basic bath

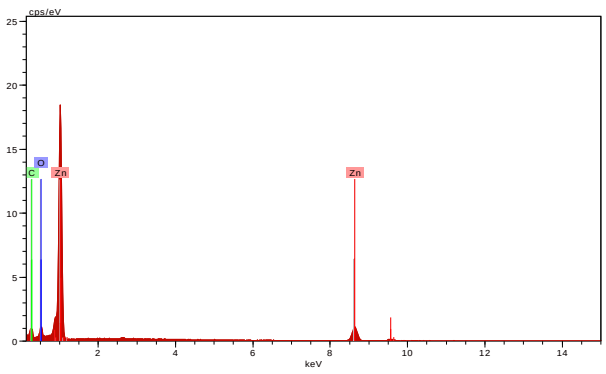

Fig. 10b EDAX photograph obtained for optimized bath

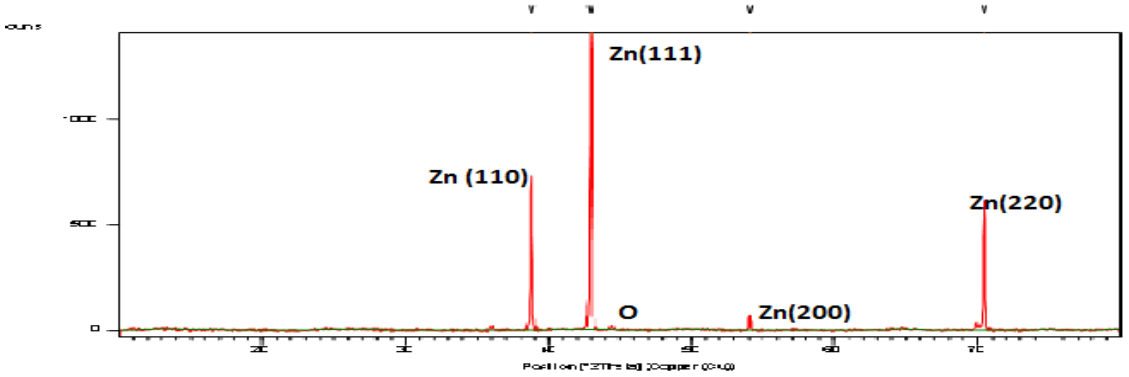

Fig. 11a XRD photograph obtained for basic bath

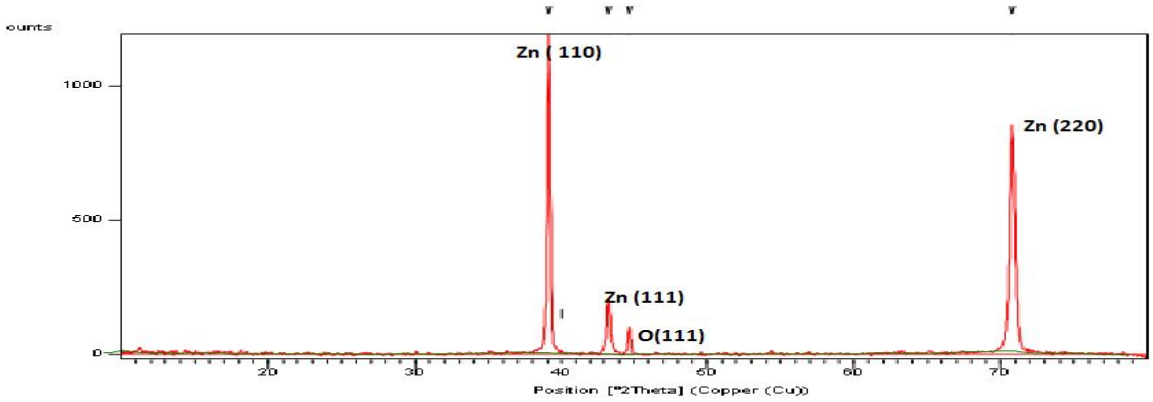

Fig. 11b XRD photograph obtained for optimized bath

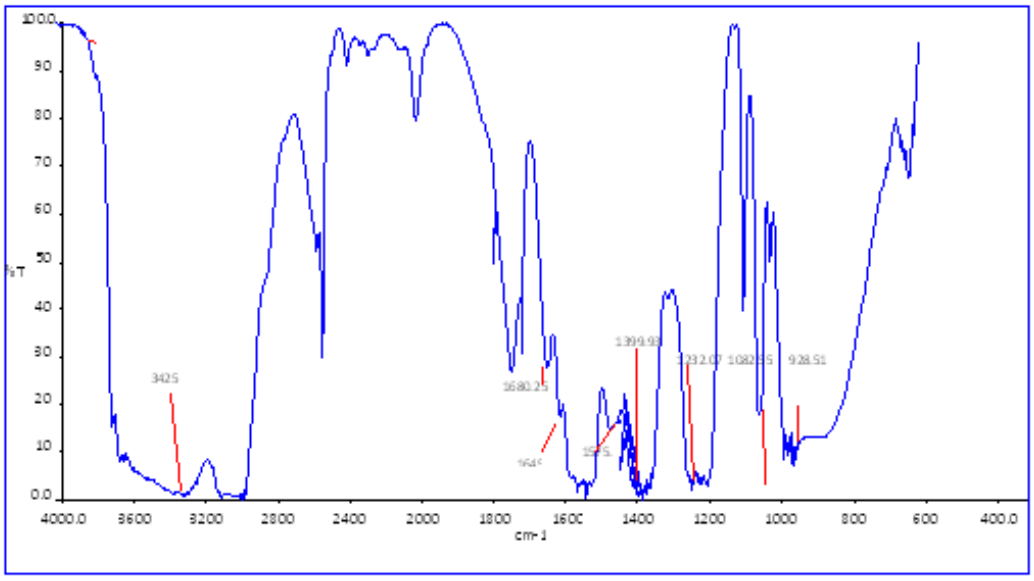

Fig. 12 IR spectrum of scrapped zinc deposit of optimized bath

Throwing power

Throwing power for basic bath and optimized bath was measured by using Haring Blum Cell at different current densities. It was found that the optimized bath had shown greater throwing power than Basic bath. At lower current density, throwing power for basic bath, and optimized bath were found to be 18.4 % and 27.5 % respectively. Further increase in current density, increased the Throwing Power and attained the maximum of 25.5% and 37.2% respectively at 4 A/dm2 (Table 4). Thorough literature survey revealed the presence of additive increased the throwing power on zinc electro deposition.

Polarization studies

The potential of steel cathode was measured galvanostatically with respect to saturated calomel electrode at different current densities. The variation of potential in the presence of different bath constituents is shown in Fig. 8. The shift in cathode potential towards negative direction was observed in the presence of henna extract.

Surface morphology study

The natural growth in the presence and the absence of henna extract was explained with the help of SEM photomicrographs (Fig. 9). In the Fig. 9a, the crystal growth is not uniform, the basic bath produced deposit having different and slightly larger crystal size. But in the presence of Henna Extract in the optimized bath showed uniform arrangement of crystals, refinement in the crystal size and hence gave a bright deposit (Fig. 9b).

EDAX

EDAX spectra of zinc electrodeposited carbon steel from Basic bath and optimized bath showed the presence of zinc, carbon and oxygen on the metal surface. The weight percentages of zinc, carbon and oxygen in zinc electrodeposited carbon steel from Basic bath were found to be 93%, 24.63 % and 8.7% respectively. But in the Zinc electrodeposited carbon steel from optimized bath had only 84.07% of Zinc. This showed that the some of the active sites of Zinc were occupied by Lawsonia inermis Linn extract. The weight percentage of Oxygen in the Zinc electrodeposited carbon steel from optimized bath was found to be increased to 10.27%. This confirmed the inclusion of the addition agent during zinc electrodeposition in the presence of extract.

X-ray diffraction studies

X-ray diffraction analysis carried out on the thin film of zinc electroplated carbon steel obtained from basic bath and optimized bath were shown in Figs. 11a, b. Intensity of peaks of zinc electrodeposited carbon steel from optimized bath was lower and the peak width was broader than that of zinc electrodeposited carbon steel obtained from basic bath. The average crystal size was found to be 0.76nm against 1.56nm of Zinc electrodeposited carbon steel obtained from basic bath. The incorporation of henna extract influenced the growth of zinc crystal such that it brought about a reduction in the crystal size. The henna extract included in the deposit acted as protrusions in a metal electrolyte interface resulting in a higher current density which increased the rate of nucleation and inhibited the growth of zinc crystal and finally gave fine grained deposits.

FTIR study

The IR Spectrum of the scrapped deposit obtained from optimized bath Fig. 6 was used to determine the inclusion of henna extract in the deposit. The IR Spectrum Contains the peak around 3400–3300 cm-1 corresponds to OH str, peak at 1680 cm-1 and at 1645 cm-1 corresponds to C=O, peak at 1575 cm-1 corresponds to C=C and peak around 1280–1232 cm-1 corresponds to C-O stretching. The absorption peaks in IR revealed the inclusion of chief component lawsone (2-hydroxy-1,4-naphthoquinone)in the deposit during the electrodeposition.

CONCLUSION

Using the henna (Lawsonia inermis) leaf extract as the addition agent, the experiments produced good zinc electrodeposition on mild steel surface in the acid zinc sulphate solution.

- Addition of henna extract which contains major amount of 2-hydroxy-1,4-naphthoquinone. This improved the hardness of the deposits, throwing power and current efficiency of the bath. IR spectra revealed the inclusion of additive in the deposit.
- At this optimum concentration the deposit was found to be crystalline and fine grained as evidenced from XRD and SEM studies.
- The particle size is very much reduced in the presence of additive.

- Hence the coating can be used for engineering applications where high corrosion resistance and hardness are required.
- The additive was a natural product that is environment friendly.

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